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SUSTAINABLE DEVELOPMENT OF AN INTEGRATED SOLID WASTE AND WASTEWATER TREATMENT

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A thesis submitted in partial fulfilment of the requirements of The Robert Gordon University for the degree of Doctor of Philosophy

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Declaration

I hereby declare that the work or any portion of it, referred to in this thesis has not been submitted in support of an application for another degree or qualification of this, or any other university or institute of learning. This is an original piece of work undertaken by myself. All results and work other than my own are clearly citied and acknowledged.

Ling Say Wong

Abstract

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Degree of Doctor of Philosophy

SUSTAINABLE DEVELOPMENT OF AN INTEGRATED SOLID WASTE AND WASTEWATER TREATMENT

This PhD thesis investigated solid and liquid waste treatment systems for Sureclean, a waste management company based in the North of Scotland. Sureclean receives a diverse range of waste streams and the increasing need for sustainable development as well as stringent environmental legislation motivated this research to develop an integrated waste treatment system.

Waste characterisation was conducted using a range of analytical instrumentation to identify the TPH, COD, heavy metals content, TOC, and particle size of Sureclean waste streams. From there, four treatment systems were investigated utilising Sureclean waste streams: mechanical separation, chemical treatment, electro-coagulation and the advanced oxidation process. Laboratory and field trials were conducted using these different treatment techniques and the analysis was performed to verify the treatment results.

The result of these trials led to the development of four modular waste treatment units, that form the outcome of this research: the Sureclean Water Treatment System (SWTS), a filtration based mechanical separation system was shown to reduce the TSS, BOD and TOC content of an oily wastewater; the Sureclean Sludge Separation System (SSSTS), a chemical-enhanced filtration based system was demonstrated to reduce 52.6 % of the sewage sludge volume; the Sureclean Electro-coagulation Water Treatment System (SEWTS), a system that agglomerates colloid particles and demulsifies oil removed 99.9 % of TPH from Sureclean interceptor

effluent; and the Sureclean Advanced Water Treatment System (SAWTS), an advanced oxidation process which was demonstrated to reduce the TPH of a contaminated groundwater collected from an ex-gas work. The treated effluent could be discharged to Sureclean interceptor.

The four treatment units developed in this research expanded Sureclean waste treatment capabilities and an integrated system was developed to treat different waste streams and to improve the treatment efficiency thus increasing the revenue and future waste stream options for Sureclean.

Keywords: Sustainable development, oily wastewater, solid waste, decanter centrifuge, chemical coagulation, Electrocoagulation, photocatalysis, advanced oxidation process.

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List of Abbreviations

µg/g	Microgram per gram
µg/L	Microgram per litre
µL	Microlitre
µL/L	Microlitre per litre
µm	Micrometre
A	Amperage
AC	Activated Carbon
Acm^{-1}	Peak area
AFM	Activated Filter Media
Al	Aluminium
Al_3SO_4	Aluminium sulphate or Alum
$AlCl_3$	Aluminium chloride
A.M.U.	Atomic Mass Unit
AOP	Advanced Oxidation Processes
API	American Petroleum Institute
As	Arsenic
ATU	Allyl thiourea
BAT	Best Available Technique
BOD	Biological Oxygen Demand
BSP	British Standard Pipe
C=C	Carbon-carbon double bond
Cd	Cadmium
CF	Cartridge Filter
CH_4	Methane
Cl^-	Chloride ion
cm^{-1}	Wavenumber
cm	Centimetre
Co	Cobalt
CO_2	Carbon dioxide
COD	Chemical Oxygen Demand
CPS	Corrugated Plate Separators
Cr	Chromium

CSR	Corporate Social Responsibility
Cu	Copper
DAF	Dissolved Air Flotation
DE	Distillery Effluent
DECC	Department of Energy and Climate Change
DEFRA	The Department for Environment, Food and Rural Affairs
DF	Drilling Fluid
DO	Dissolved Oxygen
E^0	Oxidation potential
EA	Environmental Agency
EC	Electrocoagulation
ELV	Emission Limit Value
EMS	Environmental Management System
EPA	US Environmental Protection Agency
EU	European Union
EWC	European Waste Catalogue
Fe^{2+}	Ferrous salt
$FeCl_3$	Ferric chloride
FTIR	Fourier Transform Infrared
g	gram
g/L	gram per litre
GBF	Glass Bead Filter
GBRs	General Binding Rules
GC-FID	Gas Chromatography with Flame Ionisation Detection
GC-MS	Gas Chromatography/ Mass Spectrometry
GHG	Greenhouse Gases
gpm	Gallons per minute
GRP	Glass Reinforced Plastic
H_2	Hydrogen
H_2O_2	Hydrogen peroxide
H_2O_2/Fe^{2+}	Fenton's reagent
HCl	Hydrochloric acid
Hg	Mercury
HP	High Pressure

IBC	Intermediate Bulk Container
ICAPAES	Inductively Coupled Plasma Atomic Emission Spectrometry
ICPOES	Inductively Coupled Plasma-Optical Emission Spectrophotometer
IE	Interceptor effluent
IEP	Isoelectric Point
IPPC	Integrated Pollution Prevention and Control
ISO	International Organisation of Standardisation
kg	Kilogram
KHP	Potassium Hydrogen phthalate
kW	Kilowatt
L	Litre
L/min	Litres per minute
LOD	Limit Of Detection
m	Metre
M	Molar
m^3	Cubic metre
m^3/h	Cubic metre per hour
m/s	Metre per second
MB	Methylene Blue
MBR	Membrane Biological Reactor
MC	Moisture Content
mg/L	Milligram per litre
min	Minute
mm	Millimetre
Mo	Molybdenum
MSc	Masters of Science
NaCl	Sodium chloride
NaOH	sodium hydroxide
Ni	Nickel
NOM	Natural Organic Matter
O_2	Oxygen
O_2^-	Superoxide (hyperoxide)
O_3	Ozone

OBM	Used Oil Based Mud
OBMW	Used Oil Based Mud and Water
OECD	Organisation for Economic Co-Operation and Development
OFT	Offshore storage tank
OGP	The International Association of Oil & Gas Producers
OH^-	Hydroxide ion
$OH^.$	Hydroxyl radicals
O-O	Oxygen bond
OOH^-	Hydroperoxyion
OSPAR	The Convention for the Protection of the Marine Environment of the North-East Atlantic
PAC	Polyaluminum chloride
PAHs	Polyaromatic hydrocarbons
PAM	Polyacrylamides
PARCOM	Paris Convention
Pb	Lead
PDMS	Polydimethylsiloxane
PW	Produced water
PEI	Polyethylenimine
PPC	Pollution Prevention Control
ppm	Parts per million
PSA	Particle Size Analysis
psi	Pound per square inch
rpm	Revolutions per minute
RSD	Relative Standard Deviation
SAWTS	Sureclean Advanced Water Treatment System
SD	Standard deviation
SEPA	Scottish Environmental Protection Agency
SEWTS	Sureclean Electrocoagulation Water Treatment System
SIPC	Sureclean Inclined Plate Clarifier
Sn	Tin
SPME	Solid Phase Micro Extraction
SS	Suspended Solids
SSSTS	Sureclean Sludge Separation Treatment System

SSU	Sureclean Separation Unit
SWTS	Sureclean Water Treatment System
TDS	Total Dissolved solids
Ti	Titanium
TiO_2	Titanium dioxide
TOC	Total Organic Carbon
TPH	Total Petroleum Hydrocarbon
TS	Total Solids
TSS	Total Suspended Solids
TTCE	Tetrachloroethylene
UHP	Ultra High Pressure
UK	United Kingdom
UKAS	UK Accreditation Service
UN	United Nations
UNCED	United Nations Conference on Human Environment
UNEP	United Nations Environment Program
USA	United States of America
UV	Ultra violet
UV/TiO_2	Semiconductor photocatalysis
UV/Vis	Ultra Violet/Visible
V	Voltage
W	Watt
WAC	Waste Acceptance Criteria
WFD	Waste Framework Directive
WTS	Waste Transfer Station
WWTP	Wastewater Treatment Plant
λ	Wavelength
λ_{max}	Maximum wavelength
Zn	Zinc

Chapter 1   Introduction

1.1   Sustainable Development in Waste Treatment

Sustainable development is defined as the development that "meets the needs of the present without compromising the ability of future generations to meet their own needs" (World Commission on Environment and Development 1987 p.8). The concept of sustainable development was first introduced in 1987 in the Bruntland Report prepared by the United Nations (UN). In 1992, the UN developed an action plan called 'Agenda 21' and this has been implemented into government policies around the world to promote sustainability. The momentum of sustainability has been slow, nonetheless, increasing stress on natural resources as well as a more widely spread awareness of this issue have led to pressures from the public worldwide to re-energise the call for sustainable development (International Energy Agency-World Energy Outlook Team 2010, European Commission 2010). The European Union (EU) even goes as far as saying that "*Sustainable development is a fundamental objective of the European Union*" (Council of The European Union 2008 p.2).

Sustainable development promotes the integration of three key areas: environmental, economic and social matters. Businesses undeniably have impacts on all the key areas of sustainable development. This led to the growth of "corporate social responsibility" (CSR) which was defined by the European Commission in 2001 as "a concept whereby companies integrate social and environmental concerns in their business operations and in their interaction with their stakeholders on a voluntary basis" (European Commission 2001). There are clear benefits for companies to develop CSR policies into their business strategy as these actions could enhance relationships with internal and external stakeholders, entice potential trade collaborations, improve company image, minimise CSR-related risks and potential cost-saving (Du, Bhattacharya and Sen 2010; Manuela 2008; Burke and Logsdon 1996). In terms of reducing environmental impacts, companies could adopt voluntary environmental regulation in the form of

environmental management system (EMS); the most dominant form of EMS is ISO 14001 standard (Benn and Bolton 2011). The ISO 14001 standard was instigated by the International Organisation of Standardisation (ISO), which requires participating establishments to commit to continuous improvement in their actions to reduce environmental impacts.

1.1.1   Drivers to Waste Treatment

Solid waste and wastewater treatment plays a significant role in sustainable development. Effective treatment of waste can help protect natural resources, minimise pollution to the environment and safeguard public health. Water and land are important environmental media and are considered scarce resources. Anthropogenic activities such as deforestation, industrial processes, power generation and water irrigation have caused pollution and degradation to the environment. Major pollutants in water and land include heavy metals, aromatic hydrocarbons and halogens (Tchonobaglous, Burton and Stensel 2002). Industrialisation and modernisation of society have both led to the increase release of toxic material such as heavy metals from electronic waste, polyaromatic hydrocarbons (PAHs) from petroleum by-products and waste, as well as synthetic non-biodegradable chemicals used in the manufacturing sector.

1.1.1.a   Population increase

The UN projected that the global population will grow from 6.1 billion in 2000 to 8.9 billion in 2050 as a result of low mortalities and longer life expectancies (United Nations 2004). This puts more pressure on natural resources to sustain modern life style. According to the fourth Global Environment Outlook published by the United Nations Environment Program (UNEP), we would need 1.4 Earths to sustain our current rate of resource consumption (United Nations Environment Program 2007). The International Decade for Action: Water for Life (2005-2015), a program run by the United Nation's General Assembly stated that 1.1 billion people, approximately 18 % of global population lack safe drinking water (United

Nations Office to support the International Decade for Action 'Water for Life' (2005-2015) 2008). More severely, the UN also suggested that by 2025, approximately 5.5 billion of the global population will suffer severe fresh water stress (United Nations 1997). These key challenges arise from global population increase has an effect on both developed and developing countries.

1.1.1.b   Depletion of Natural Resources

Global demand for natural resources such as fossil fuel and minerals continues to increase due to population increase as well as the urban lifestyles we have chosen to adopt (Pamuk 2007). Water is used in daily domestic life, as well as for commercial and agricultural purposes which ultimately produces wastewater. Along with fresh water, this wastewater undergoes the hydro-geological cycle as demonstrated in Figure 1-1. As the fresh water supply travels from their sources and is turned into wastewater, the process decreases the fresh water supply as well as degrading the water quality. Therefore, it is critical to conserve water sources as well as the quality. This can be achieved by effective wastewater treatment.
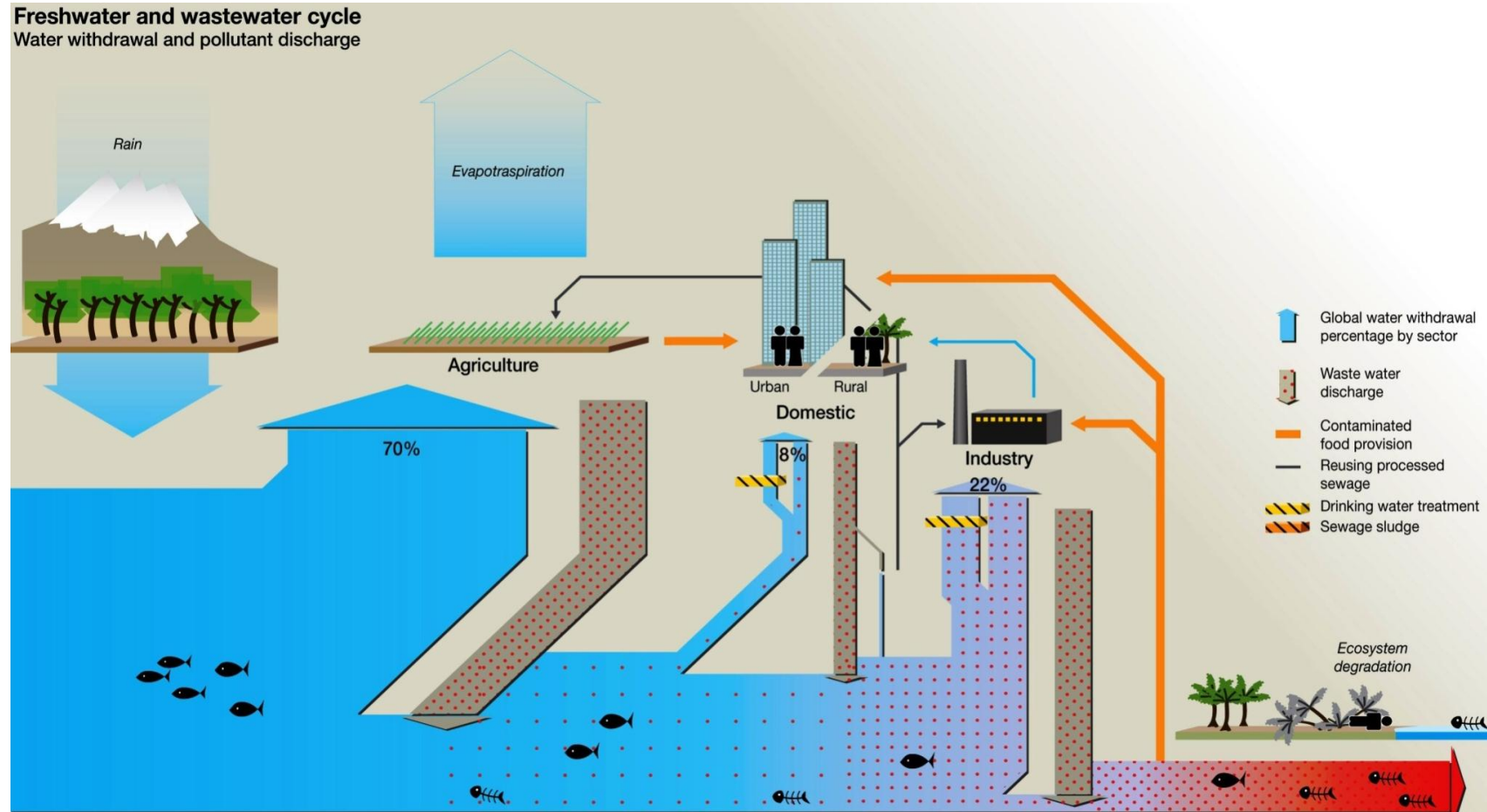
Figure 1-1 Fresh Water and Wastewater cycle (UNEP/GRID-Arendal 2010)

1.1.1.c   Industrialisation

The Environment Agency estimated that there may be some 300,000 hectares of land and groundwater in the UK affected to some extent by industrial or natural contamination (Ashworth et al. 2010). Oil pollution in the aquatic bodies especially in the marine environment is also one of the main environmental concerns; this was evident in the Deep Horizon oil spill disaster in the Gulf of Mexico in 2010. The Global Marine Oil Pollution Information Gateway stated that an estimated 470,000 to a possible 8.4 million tonnes per year of oil is entering the marine environment through natural sources, accidental spills from oil tankers, and operational discharges from oil and gas production (Global Marine Oil Pollution Information Gateway 2005). Oil that ends up in water bodies could be an environmental disaster; for example an oil film forming on a water surface could deprive aquatic life of oxygen and sun light.

1.1.1.d   Climate Change

Carbon dioxide (CO_2) and Methane (CH_4) are two of the major contributory greenhouse gases (GHG) in the atmosphere. In a report published by the International Panel on Climate Change, the world leading climate change research stated that "The global increases in carbon dioxide concentration are due primarily to fossil fuel use and land use change" (International Panel on Climate Change 2007). In the same report, the International Panel on Climate Change also stated that atmospheric CO_2 increased from the pre-industrialisation figure of 280 parts per million (ppm) to 379 (ppm) by 2005; this reflected the increase in global energy consumption since the pre-industrialisation era.

CO_2 and CH_4 are the main gases released in active landfill sites and are produced during anaerobic decomposition of organic matter in compacted waste. Therefore a reduction of waste to landfill will contribute to a decrease in the level of greenhouse gasses. In the United States of America (USA), the Natural Resources Defence Council (Natural Resources Defence

Council 2010) stated that climate change will have considerable effect on water supplies throughout the US in the coming decades, with over 1,100 counties facing larger risks of water scarcity due to climate change.

1.2 Scottish Industries and Their Waste

The Department for Environment, Food and Rural Affairs (DEFRA) stated that Britain produced over 280 million tonnes of waste every year (The Department for Environment, Food and Rural Affairs 2011). The Scottish Environmental Protection Agency (SEPA) estimated 17.11 million tonnes of waste was generated in Scotland in the 2009 (Scottish Environmental Protection Agency 2011). This figure incorporates household, commercial and industrial waste, construction and demolition waste, non-mineral waste from the mines and quarries as well as agricultural waste. In 2009, Scotland produced 1.80 million tonnes of industrial waste and 4.89 million tonnes of commercial waste (Scottish Environmental Protection Agency 2011). SEPA also estimated 79633.22 tonnes of oil and oil/water mixtures was produced in 2009. Figure 1-2 demonstrates total waste arising from Scotland.
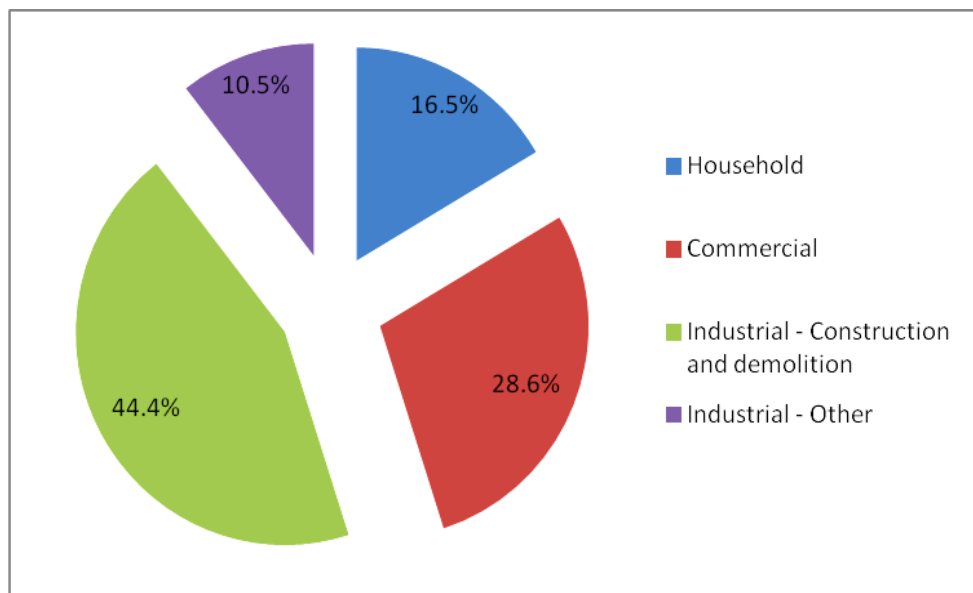
Figure 1-2 Controlled waste arising from Scotland in 2009 (adapted from Scottish Environmental Protection Agency 2011).

1.3 Potential Impacts of Enviromental Pollutants

Organometallic, halogen and aromatic compounds are highly toxic pollutants to living organisms and have the potential to bio accumulate in the ecosystem. Natural water bodies and soil have the ability to assimilate nutrients and restore its original ecological equilibrium. Overloading of nutrients such as nitrogen and phosphorus and other pollutants can upset this ability.

One of the main roles of solid waste and wastewater treatment is to reduce the level of pollutants going into the environment. In most cases, the agricultural and aquatic environments have their own natural processes that involve the breakdown of chemical substances by plants, animals or micro-organisms. However, human activities such as industrial processes have introduced a variety of pollutants that are not easily broken down by nature such as PAH, pesticides, chlorinated solvents, micro-organisms such as epidemic-causing bacteria, as well as bigger objects such as foreign sedimentation and even plastic bags. These pollutants significantly increase stress on the environment, and potentially pose danger to human health (Salvato, Nemerow and Agardy 2003). Elements with atomic density higher than 6 g/cm^3 such as chromium (Cr), nickel (Ni), copper (Cu) and lead (Pb) are heavy metals. Overloading of heavy metals in the environment could harm the normal biological cycle (Warey 2006).

1.3.1   Pollutants affecting the Aquatic Environment

Oil is one of the most well-known pollutants in the marine environment due to oil and gas production. The International Association of Oil & Gas Producers (OGP) estimated 1.5 tonnes of oil is spilled into the sea globally for every million tonnes of hydrocarbon produced in the oil and gas industry (International Association of Oil & Gas Producers 2010). Oil and grease that covers the surface of the water bodies decrease the air-water interaction and leads to a reduction of oxygen transfer. On top of that, spillage also contains harmful heavy metals which further downgrade the watercourse (Harrison 1999).

Sediments are usually brought to the aquatic environment, such as lakes or rivers, by runoff which carries loose soil and dissolved substances to the watercourse. This in turn increases the total dissolved solids (TDS) and the suspended solids (SS) content of the water. Sediments cause a decrease in dissolved oxygen and could lead to a decline in fish habitats and micro-invertebrates. The increase in sedimentation could also escalate the potential for flooding (University of Michigan 2005). Another major concern for the water bodies is inappropriate or minimal sewage treatment (Tebbutt 1998). The minimally or untreated sewage that is discharged to the watercourse causes an intensification of organic pollutants and thus increases the biological oxygen demand (BOD) of the water body (Tebbutt 1998, Harrison 2001).

1.3.2 Pollutants affecting land

Urban runoff is one of the major contributors in land contamination. Runoff may dissolve toxic heavy metals and bring them to their watershed (Tang, Ku and Yue 2007). These heavy metals tend to bio-accumulate in organisms and take a long time to be removed or purified. Heavy metals have a detrimental effect on both the human and any organisms that feed on the water. Another major contribution to contaminated land is industrial process. Chemical manufacturing, coal processing and landfill are amongst the few major industries that cause significant contamination to land (Hestor and Harrison 1997).

1.4 Sureclean Limited: Company background

The waste materials in this research were obtained from Sureclean Limited, a waste management company based in the North of Scotland. Sureclean was formed in 1985 in Alness. The company has evolved over the years and it is now a specialist company in waste management, industrial cleaning, asbestos management and high and ultra-high pressure water jetting. Sureclean environmental management system is accredited with ISO 14001:2004 and the company is committed to continuous improvement in

their environmental performance. Sureclean has business both onshore and offshore, nationally and globally. Sureclean aimed to expand its waste treatment facilities to a wider clientele nationally and globally. SEPA has granted the Pollution Prevention and Control (PPC) licenses to Sureclean two waste transfer stations that mainly receive hazardous waste; that include waste from both offshore and onshore. Hazardous waste is called 'special waste' in Scotland. In this research project, the term *solid waste* applies to special waste and does not apply to municipal non-hazardous waste. *Wastewater* applies to any form of wastewater that is required to be treated prior to discharge to sewer or water course. Sureclean Alness waste transfer station (WTS) has a six-stage interceptor that collects wastewater accumulated around the site via a series of drains (as shown in grey in Figure 1-3). The wastewater usually contains suspended solids, some heavy metals and hydrocarbon, and therefore SEPA has requested that the effluent is to be treated prior to discharging into the public sewer. Table 1-1 shows a summary of the most updated (2009) discharge consent. Prior to 2009, Tin (Sn) and Zinc (Zn) were included in the previous version of Sureclean discharge consent in the total heavy metals count, therefore some trials performed in this research included the two metals, when this is the case, it would be stated clearly in the trial methodology.

Parameters	Discharge Limits
pH	Between 5 - 11
Total Suspended Solids (TSS)	1000 mg/L
Chemical Oxygen Demand (COD)	3000 mg/L
BOD	1000 mg/L
Total Petroleum Hydrocarbon (TPH)	100 mg/L
Total Toxic Metals: As, Co, Cr, Cu, Mo, Ni, Pb	2 mg/L
Volume	Maximum 80 m^3 in 24 hours
Temperature	Less than 40°C

Table 1-1 Summary of Sureclean Alness Waste Transfer Station (PPC license: PPC/A1016691) trade effluent discharge consent
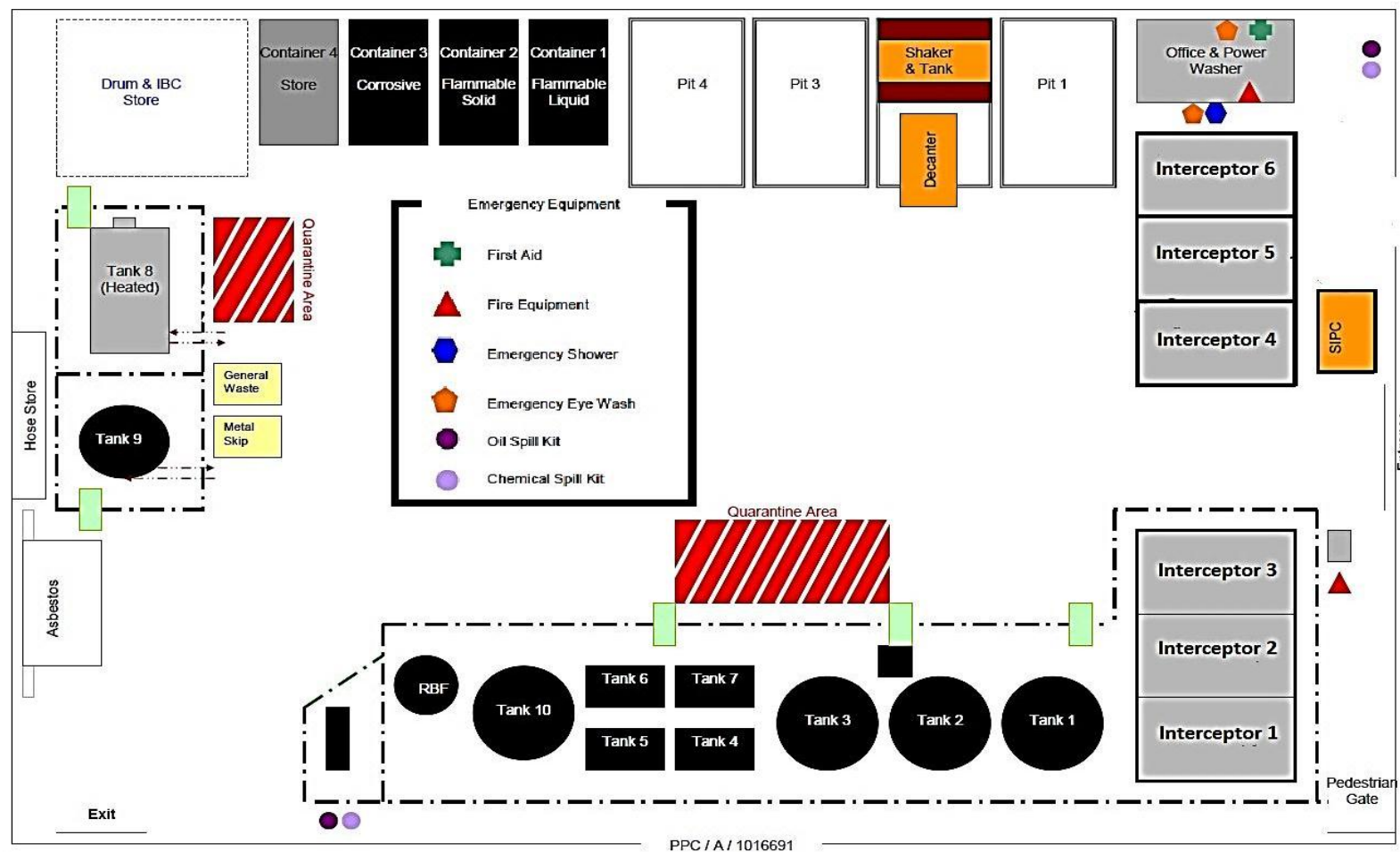
Figure 1-3 the Sureclean Alness Waste Transfer Station Site Plan.

1.4.1 Sureclean Waste Treatment Facilities: The Challenge

Sureclean was treating its waste streams from a diverse range of sources. In 2009, Sureclean received 1194.12 tonnes of commercial waste which included solid and liquid hazardous and non-hazardous waste (Sureclean 2010). Figure 1-4 shows a breakdown of the waste streams handled in both Aberdeen and Alness Waste WTS; the data was compiled and reported in Sureclean 2009 Environmental Report (Sureclean 2010). The highest waste input were wastes such as waste oil, oily water, laboratory wastes, waste paints, and chemical wastes.
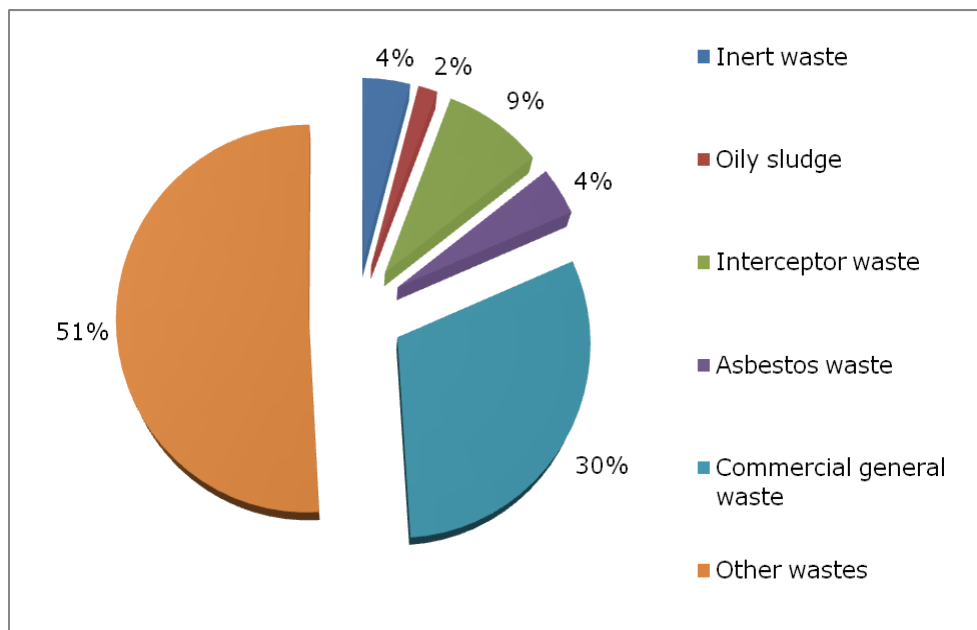
Figure 1-4 the Sureclean Waste Input from Client in 2009 (Sureclean 2010).

The stringent environmental, regulatory and legislative constraints are the main driving forces for the treatment of hazardous waste to a safe level prior to final disposal. Sureclean also recognised the importance of its CSR; therefore they made a substantial time and financial investments in the last few years in expanding their in-house treatment processes for both liquid and solid waste. However, these two waste streams had been treated separately, and this failed to maximise operation efficiencies. Moreover, the

cost and the difficulty of many of the methods prompted the need for providing efficiencies in both treatment and operation. By integrating its solid and liquid waste, Sureclean anticipated that the company would provide a comprehensive service from start to end for their clients.

1.5 Integrated Waste Treatment

A holistic view of treatment is essential for effective treatment of waste; this can be achieved by integrating solid and liquid waste treatment. Wastes that were investigated in this project were mainly solid materials such as soil, grits, sands, cuttings, metals and wastewater such as oily wastewater, bilge water, interceptor effluent and sewage. As stated by the UNEP (United Nations Environment Programme, Division of Technology Industry and Economics, International Environmental Technology Centre 2002), "One principle that logically emerges from adopting an integrated approach to waste management is that different types of waste should not be mixed." Each of these wastes can pose environmental damage if no treatment was carried out to reduce the pollutant loading. Therefore, the main strategy of this research is not about mixing solid and liquid waste, rather adopting a practical approach when treating each waste.

1.5.1 Fundamentals of Treatment Technologies Design

Woodard (2006) proposed that the first step to approach waste treatment design was to understand the waste source, the processes that give rise to the waste and undergo a waste characterisation. Figure 1-5 demonstrates the fundamental flow process for waste treatment design. Treatment objectives were based on the information gathered from review of literature, current market research and waste streams analysis. Environmental regulations play a significant role in determining the treatment objective. Laboratory or field based trials provide the essential data towards the treatment system selection or modification of an existing treatment technology.

Best Available Techniques (BAT) is defined as "the most effective and advanced stage in the development of activities and their methods of operation which indicate the practical suitability of particular techniques for providing, in principle, the basis for Emission Limit Values (ELVs) designed to prevent and, where that is not practicable, generally to reduce Emissions and the impact on the environment as a whole" (European Union 2008). Effective waste treatment techniques represent a balance and judgement of cost, benefits and affordability. A sound waste management plan forms the key framework for ensuring identification, implementation and employment to BAT in waste treatment technologies.
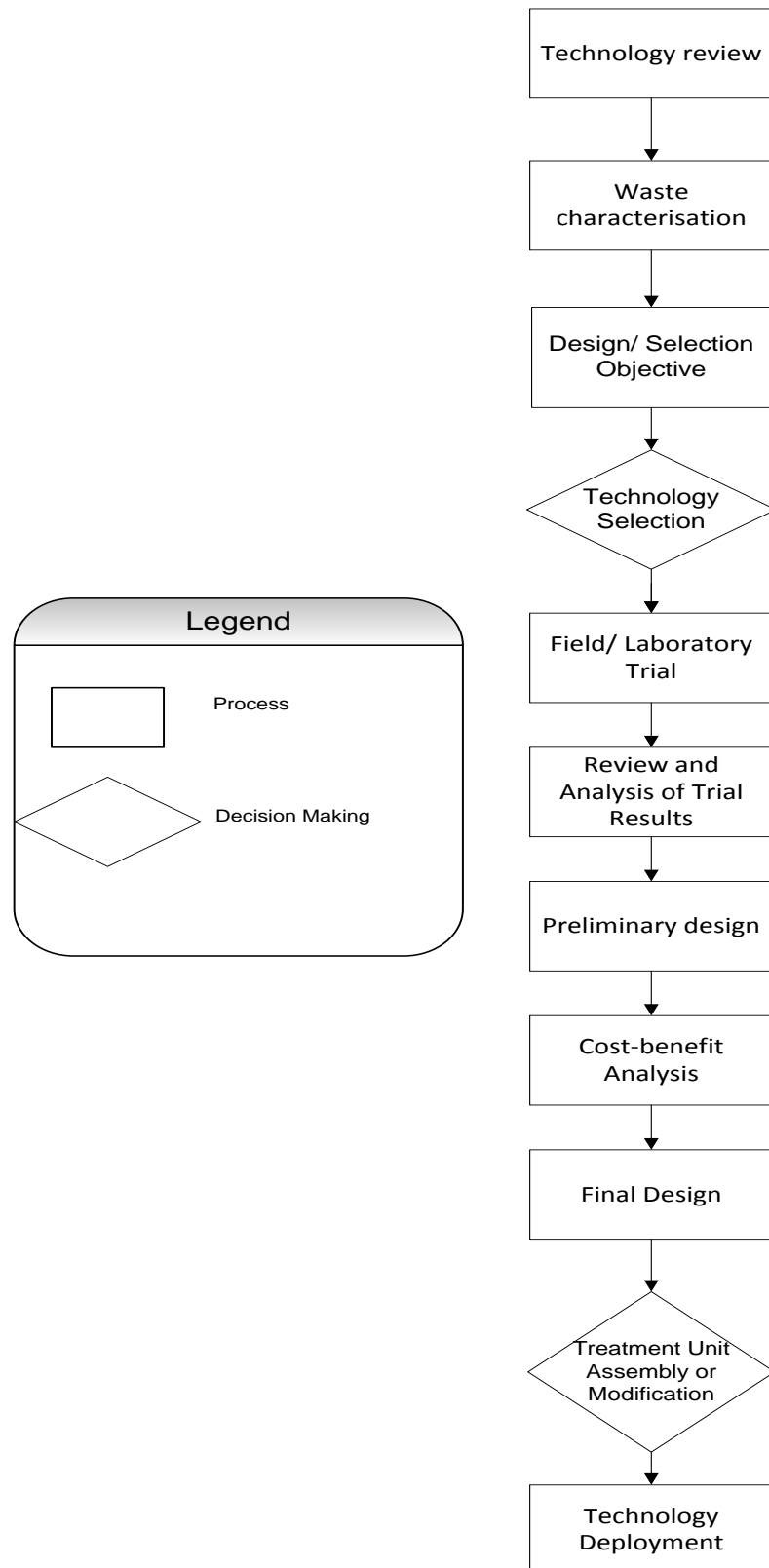
Figure 1-5 shows a flow diagram of an approach to design a waste treatment technology (Adapted from Woodard (2006)).

1.6 Current Waste Treatment Technologies

Solid waste and wastewater treatments can be classified as biological, physical or chemical treatment. Biological treatment involves the use of micro-organisms to reduce mainly organic contaminants. Physical treatment is the use of physical methods such as filtration, high speed centrifuge and sedimentation to aid or achieve treatment requirement. Chemical treatment in wastewater treatment aids treatment processes to remove colloidal particles that are difficult to remove just by physical means (i.e. filtration). The addition of coagulants and flocculants can improve sedimentation of solids for COD and TSS reduction (Kemmer 1998). Advanced treatment systems such as incineration and advanced oxidation processes (AOP) can provide enhanced treatment to remediate solid waste or wastewater to the required standard. These treatment processes have their pros and cons; therefore the focus of this research is to use a combination of treatments to achieve a final effluent that is fit and safe to be discharged to sewer and solid waste that is fit for landfill.

1.6.1   Biological Waste Treatment Technologies

Biological treatment has been applied to reduce the organic matter in conventional wastewater treatment processes. According to Gray (2005), the aim of wastewater treatment is to convert the waste materials into stable products so it can be safely disposed to inland waters. Furthermore, this is to ensure legal compliance to environmental regulations. There are many ways to classify wastewater treatment processes. In a conventional municipal wastewater treatment regime, this can be categorised according to the order of the treatment stages, i.e. preliminary or primary treatment is usually the first stage of the treatment processes, followed by secondary and tertiary or advanced treatment. Lin and Lee (2007) stated that conventional secondary treatment can remove 85 to 95 % of BOD and TSS and minor portions of nitrogen, phosphorus, and heavy metals. Unfortunately, it does not efficiently remove pathogens, heavy metals and non-biodegradable organics, which all may require advanced treatment (Qasim 1994).

1.6.2   Mechanical Separation

Mechanical separation is the use of physical methods to aid or achieve waste treatment without the requirement to alter the chemical composition of the waste prior to treatment (Woodard 2006). Mechanical separation which is more commonly known as physical treatment has been historically important in the wastewater pre-treatment process as well as primary treatment (Kiely 2007). These technologies include grit removal and solid screenings to remove large and rigid objects from the wastewater treatment plant to protect the down flow pipe works. In conventional municipal wastewater treatment plant, these unit processes are placed in the initial stage and they play an integral role in ensuring the efficiency of the downward treatment process.

In solid treatment, mechanical separation increases the speed of the natural processes such as solid settling in liquid phase, sludge drying and dewatering. Mechanical separations are ideal for the removal of large solids, some suspended solids and floating oil. However, chemicals are often added to aid solid separation, these are known as coagulants or flocculants, and will be discussed in Chapter 5. Physical treatment can be broadly divided into two categories: liquid-liquid separation units and liquid-solid separation units.

1.6.2.a   Liquid-Solid Separation

Solid separation from wastewater is a vital process in wastewater treatment to enhance the on-going treatment process. Chemical and electro-chemical treatment in solid and liquid waste can enhance the process but may be more costly. If time is not a factor, solid settling can be accomplished in a sedimentation tank or interceptor. However, in most industrial wastewater processes, as well as on offshore oil platforms and installations, waste has to be processed in a timely manner due to space requirement and back-loading. The decanter centrifuge is a form of accelerated sedimentation of solids from slurries by allowing heavier particles to be settled (Bouse 2005). Centrifuges employ centrifugal force to separate solid from liquid by

enhanced settling. The decanter centrifuge is able to provide continuous mechanical separation of liquids from solids in a more rapid and controllable manner. The liquid runs round the helical scroll and is discharged over weir plates fitted at the parallel end of the bowl. The solids are moved by the conveying action of the helical scroll up the gentle slope of the conical section, out of the liquid and finally out of the machine.

Oil separation from the liquid phase can be achieved by mechanical means such as a clarifier or interceptor. The interceptor is a gravity separation similar to a sedimentation tank. These treatment systems are a cost-effective way for separation of suspended particles based on their density or particle size. Another form of solid separation unit that is often used in the oil and gas offshore platform for drill cuttings treatment is the shale shaker. The shale shaker can be defined as a "cylindrical sieve or vibrating table that removes the drill cuttings from the circulating mud stream" (Grace 2007).

Filtration is a process where the solid in the liquid phase is retained on filter media. Removal of organic and inorganic colloidal and suspended solids is typically accomplished by filtration. Filtration is a mechanical separation of solids from a suspension in a liquid by means of a porous medium or screen (Puchas and Sutherland 2002). Particles are removed based on their size and this is the defining factor for the selection of a suitable filtration unit. There is a vast diversity of filter media present in the market place as stated by Purchas and Sutherland (2002); these include inorganic materials, carbon or charcoal, glass, metals, natural organic fibres, synthetic organic fibres and synthetic sheet material. These filter media have different pore sizes; the smaller the pore size, the finer and more solids it retains; however, this can lead to faster choking of the filter. Membrane filters may retain particle sizes down to less than 0.1µm.

1.6.3 Chemical Waste Treatment Technologies

Colloidal particles in wastewater which are not readily settable can be chemically enhanced to coagulate. In wastewater treatment, coagulation is the reaction where chemical destabilisation of particles occurs to form aggregation (Cosgrove 2010). Chemical coagulation/flocculation is an important pre-treatment process to water and wastewater treatment. The main purpose of chemical treatment is to remove suspended solids and heavy metals, thereby aiding in mechanical separation (by filtration or centrifuge decanter) (Spellman 2011, American Water Works Association 2011). Among the inorganic coagulants, the multivalent metal salts such as $AlCl_3$, $FeCl_3$ and Al_3SO_4 are more commonly used than electrolytes (Wakeman and Tarleton 1999). These metal salts are thought to be more effective and cheaper in comparison to electrolytes (Bratby 2006). Electrocoagulation is an electrolysis process where current is applied to the wastewater to destabilise colloid particles in effluent (Wang et al. 2010). The destabilisation mechanism is similar to chemical treatment using metal salts (Arvanitoyannis 2008).

1.6.4 Advanced Waste Treatment Technologies

Chemically stable pollutants such as some surfactants and PAHs (Surampalli 2004) are difficult to be treated by conventional treatment processes such as biological, chemical and physical processes. Effective treatment of these recalcitrant pollutants is imperative to prevent long-term biological effects if they are released to the environment (Ostroumov 2006). AOPs processes employing ozone, ultra-violet (UV) radiation or hydrogen peroxide (H_2O_2) are systems that produce chemical species that have high oxidation ability to degrade recalcitrant substances (Beltrán 2003; Tunay 2010). AOPs have been shown to be effective in remediation of wastewater from the textile, pharmaceutical and chemical industries (Tunay 2010).

1.7 Aims of This Research

This literature search has clearly shown that there are increasing concerns over the environmental impact of waste and wastewater, therefore the need for sustainable development of waste treatment is crucial for our future generations to meet their needs. The extensive literature search carried out shows that there is an opportunity for improvement in the whole range of waste treatments for the Sureclean solid waste and wastewater treatment.

This project aims to investigate conventional and new technologies for the combined treatment of solid and liquid waste streams; the emphasis is on industrial solid and liquid waste treatment. The treatment technologies are intended to have a small footprint (modular) so that transportation can be achieved either by a lorry or inside a shipping container (mobile). The treatment solutions aimed to have low start up time, simplicity in operation and little maintenance. The ideal design operation flow rate is 5 m^3 per hour. The resulting final discharge effluent must meet the Sureclean Alness WTS discharge consent (as shown in Table 1-1) whilst any solid waste is expected to be fit for inert landfill disposal in the United Kingdom (UK). To achieve this, the following objectives have been set.

1.7.1　MRes Objectives

1. Literature review on environmental legislation
2. Conduct market research on treatment technologies of solid waste and wastewater.
3. Review Advanced Oxidation Processes (AOP) in treatment of contaminated water and soil.
4. Conduct a comprehensive analysis of the waste streams of the Sureclean waste treatment company, thus identifying major pollutants.

1.7.2　PhD Objectives

5. Conduct laboratory and field based technology trials with identified waste streams.

6. Evaluation and interpretation of results from field trials.

7. Evaluation and interpretation of results from laboratory trials.

8. Implementation of processes based on field trial reports and cost-benefit analysis.

Chapter 2   Environmental Legislation

We are entering an era described as "Anthropocene", the term first proposed by a Nobel Prize winner Paul Crutzen in 2002 to describe an era where human activities has resulted in global scale impacts especially in geological terms (British Geological Survey 2011; Ehlers 2006). Human activities such as mining and the landfilling of waste have profound implications for the environment. Waste characteristics, for example have changed; modern society's reliance especially on hazardous industrial processes has created waste that is costly to the environment and the economy (Daven and Klein 2008). In the 19th century, the main forms of waste disposal management were open dumping into a large pits or open burning; it was not until the 1950s that land was specially engineered for waste landfill, also known as sanitary landfill (Tammemagi 1999). Therefore adequate regulation of waste management is now an important driver to protect the environment.

Environmental accidents in the past have had a very prominent influence on environmental legislation. In 1969, one of the largest oil spill incidents in United States history occurred when disaster struck on Platform Alpha 21 off the coast of Santa Barbara Channel that led to almost 100,000 barrels of oil spilled into the Gulf of Mexico (King 2010). The devastation has defined the US environmental legislation. With pressure from the public, the US Government established the US Environmental Protection Agency (EPA) as well as the passage of the US Clean Air and Clean Water Act (Juhasz 2011; offshore-technology.com 2011). Another example was in 1972 in the UK, when a series of illegal dumping of cyanide waste occurred, with the most serious involving 36 drums near Coventry. Public outrage forced the government to rush through parliament and passed The Deposit of Poisonous Waste Act 1972 (Levitt 1980; Williams 1998). The Act, one of the earliest pieces of environmental legislation passed in the UK, also introduced a pre-notification system of poisonous waste, where authorities

had to be notified, prior to the movement of waste, with appropriate documentation that included the details of the waste (Levitt 1980).

It can be seen that regulation of waste is crucial to protect public health and safety as well as to ensure environmental protection (Williams 1998). According to the United Nations Human Settlements Program (2010 p.20), "safe removal and subsequent management of solid waste sits alongside the management of human excreta (sanitation) in representing two of the most vital urban environmental services." Poor waste management in urban communities could lead to epidemic outbreaks as seen in the Black Death during the medieval Europe period, where the deadly plague spread rapidly due to inadequate public health services.

Environmental legislation can be reviewed in multiple angles. Wolf and Stanley (2003) described environmental law as a mixture of primary legislation (Acts of Parliament), secondary legislation (regulation or statutory Instruments), judicial decisions reported in law reports, common law principles, European Community legislation (usually in the form of Directives which are transposed into national law) and International law. Environmental pollution does not have boundaries and therefore the role of international law is crucial. As described by Birnie and Boyle (2002), international law aims to address transboundary issues as well as national and domestic environmental problems that affect humans as a whole such as the protection of heritage, biodiversity and sustainable development. One source of international law is in the form of treaties also labelled as protocols, conventions, covenants, acts or pacts (Birnie and Boyle 2002). There have also been a growing number of multilateral and bilateral treaties in the past thirty years sparked by the 1972 United Nations Conference on Human Environment (UNCED) or the 'Earth Summit' held in Stockholm (Wolfrum and Matz 2003). Sitaraman (2009) stated that treaties function as the stepping stone for the creation of international organisation to deal with common problems. However, it is up to the state to decide how the treaties are intended to take effect in domestic law (Kaczorowska 2010).

The UK is a Member State of the EU. The EU Directives and Regulations play a significant role in UK legislation or Acts of Parliament. According to the Environmental Data Services (Zukas 2007), the EU Regulations are binding in all Member States' legal systems and EU Directives set out final goals that all Member States have to meet. Environmental regulation enforcement are conducted by so called 'devolved administrators', i.e. Environmental Agency (EA) in England and Wales; SEPA in Scotland and the Environmental Protection Agency in Northern Ireland. On the whole, the DEFRA and all the devolved administrators are responsible for transposing EU Directives into UK national law (Organisation for Economic Co-Operation and Development 2009). In Scotland, the Scottish Executive is in charge of environmental policy and legislation development; while SEPA is the principal environmental regulatory authority (Organisation for Economic Co-Operation and Development 2009).

Legislation exerts a core influence on solid waste and wastewater treatments especially in specifying treatment outcome. The Sureclean target waste streams for this project focussed on oily sludge, drill cuttings from onshore and offshore oil and gas drilling, and sewage sludge. As stated in Chapter 1, the resulting final discharge effluent is expected to meet the Sureclean Alness Waste Transfer Station discharge consent, whilst any solid waste is expected to be fit for inert landfill disposal in Scotland and in a wider context in the UK. Therefore it is pertinent to understand the regulations governing these outcomes, as well as the correct management of hazardous waste.

2.1 Legal Aspects of Waste

According to Waste Online (2005), the UK national control on waste first started from the Control of Pollution Act 1974 and was then followed by the introduction of the Environmental Protection Act 1990. As stated by the Organisation for Economic Co-Operation and Development (2007), the development of waste legislation in the UK was predominantly driven by the need to transpose EU Directives into national legislation. In the UK, the

Environmental Act 1995 requires the Secretary of State to construct a national waste strategy for England and Wales; whilst in Scotland, this is constructed by SEPA (Thompson 2011; Organisation for Economic Co-Operation and Development 2002). Figure 2-1 shows a simplified demonstration of waste policy in the UK. The circles in the figure represent sub-groups of waste and the triangle represents methods of waste management; the shaded ovals in the figure symbolise particular waste types that are addressed in their individual policy.
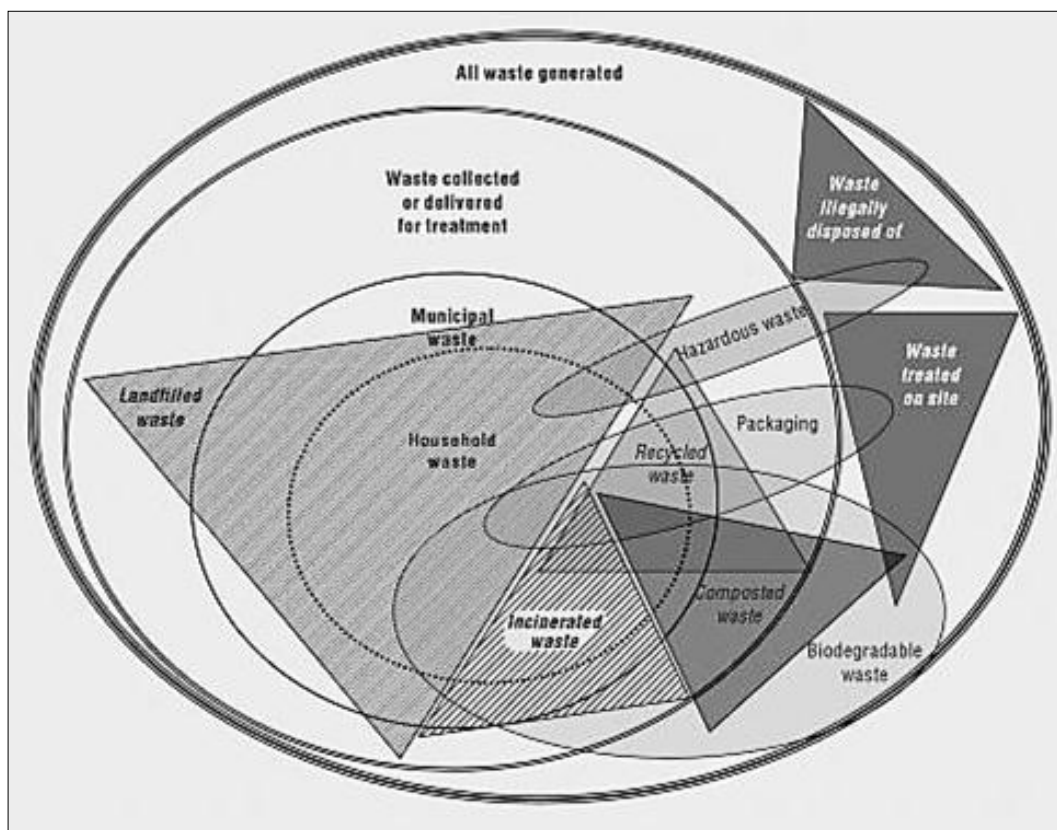
Figure 2-1 Waste Categories and Treatment Options (Organisation for Economic Co-Operation and Development 2007).

According to Zero Waste Scotland (2011), an organisation funded by the Scottish Government, the National Waste Strategy set out a framework to reduce the amount of waste production and encourage sustainable waste management, which include diversion from landfill. Within Scotland, the National Waste Plan and Area Waste Plan outline and implement targets set

in the National Waste Strategy. To reduce waste to landfill, the Scottish Government announced an ambitious plan for Zero Waste Scotland. The Zero Waste Plan published in June 2010 aimed to reduce environmental impacts to Scotland by increased resource recovery through waste, as well as significantly reducing waste to landfill (Scottish Government 2010). This provided a strong driver for Sureclean to develop waste treatment systems that are aligned with the strategy by diverting waste from landfill.

2.1.1   Waste Framework Directives (WFD)

The Waste Framework Directive (WFD) (2006/12/EC) on waste is considered one of the most important Directives that affect waste management legislation (Hawkins and Shaw, 2006). The Directive provides a clear definition of 'waste' and also illustrates the key concepts such as waste, recovery and disposal. There has been an on-going debate as to what constitutes waste. The Directive (Waste Framework Directive 2006/12/EC) stated that "Waste" is defined as any substance or object in the categories set out in Annex I of the Directive which the holder discards or intends or is required to discard. The definition of waste is significant as it will impact on legislation governing what is potentially to be waste. The treatment of waste and the philosophy of waste hierarchy have changed the outset of "waste", as what was constituted as "waste" no longer stands as it contains economic value.

However in 2008 a revised WFD (amended as the WFD 2008/98/EC) was introduced and in 2010 it repealed the existing Waste Framework Directive (2006/12/EC), the Waste Oils Directive (75/439/EEC) and the Hazardous Waste Directive (91/689/EEC). The revised WFD incorporates provisions dealing with the issues that were addressed by the repealed Directives into the single new Directive. The revised WFD retained the definition of 'Waste' and launched a fresh view to waste management that promote the prevention of waste by adopting a five-step hierarchy of waste management selections (waste prevention, re-use, recovery, recycling and safe disposal) which must be applied by Member States when developing their national

waste policies (Council of European Union 2008). The Directive set a new recycling and recovery targets for Member States to be achieved by 2020, where 50 % of household wastes would be set for re-use and recycling and 70 % of construction and demolition waste would be set for re-use, recycling and other recovery through waste management plan and waste prevention programme.

Although the new WFD revoked the Hazardous Waste Directive (91/689/EEC) but the provisions remain much the same. The Directive defined 'hazardous waste' as waste that display hazardous properties listed in Annex III: explosive, oxidising, highly flammable, flammable, irritant, harmful, toxic, carcinogenic, corrosive, infectious, toxic to reproduction, mutagenic, capable of release toxic gases, sensitising, ecotoxic and waste that yield another substance (such as leachate) after disposal. Articles 17 to 20 of Directive 2008/98/EC laid down the requirements for the handling of hazardous waste. Traceability by means of labelling, record keeping and monitoring of waste from production to final destination (disposal) and control of hazardous waste is obligatory according to the Directive. The Directive also bans mixing of hazardous substances with other waste material, which includes the dilution of hazardous waste. According to DEFRA, tight control of hazardous waste is necessary from point of production to its movement, management, recovery and disposal due to its hazardous properties which could potentially harm humans as well as the environment (Department of Environmental Food and Rural Affairs 2011).

The Waste Framework Directive has been implemented in Scotland through Waste Management Licensing (Scotland) Regulations 2011 and Waste (Scotland) Regulations 2011 (Tam 2011). In Scotland, special waste and hazardous waste means the same. Waste that is classified as hazardous in the European Waste Catalogue (EWC) is special waste. The EWC was established by the European Commission (2000/532/EC) based on the waste type and the industry or process that produces the specify waste. EWC code that is marked by an asterisk (*) shows that the waste is hazardous. Examples of special waste that Sureclean receives regularly

include oil containing drilling muds and wastes (01 05 05*), tank bottom sludges (05 01 03*), oily water from oil/water separators (13 05 07*) and insulation materials containing asbestos (17 06 01*). In Scotland, hazardous waste is regulated according to the Special Waste Regulation 1996 and Special Waste Amendment (Scotland) Regulations 2004 and in April 2011, the Hazardous Waste Regulations were amended to implement the revised Waste Framework Directive.

2.1.2 Landfill Directives

According to the DEFRA, the UK historically used landfill as its main means of disposing waste (Department for Environmental, Food and Rural Affairs 2010); however, this is now considered as a last option especially for biodegradable waste. In the EU, the Landfill Directives (99/31/EC) covers the technical requirements for landfills. The directive is transposed to Scottish law in The Landfill (Scotland) Regulations 2003. Productions of leachate and landfill gas are the two major pollutions associated with a landfill site (Hester and Harrison 2002). The directive requires separate sites for hazardous, non-hazardous and inert wastes. As a result, the co-disposal of hazardous waste and non-hazardous waste to landfill was completely stopped in 2004.

As stated by the Zero Waste Scotland (2011), the Landfill Directive is one of the most important drivers behind the National Waste Strategy in Scotland. Pre-treatment of waste before landfilling is a pre-requisite by the Directive, which aimed to both reduce waste volume and minimise the environmental impact of disposal. Pre-treatment of waste involves the physical, thermal, chemical or biological process, as well as reducing the volume and changing the characteristic of the waste (Zero Waste Delivery Team 2011). The Directive set the detailed conditions for safe disposal of waste called the Waste Acceptance Criteria (WAC). The WAC are based on assessments on basic waste characterisation through the waste's leaching behaviour, periodic spot testing to ensure compliance and on-site verification to ensure that the waste is the same as the waste characterisation documentation

(WRAP 2010a). The Directive has also set progressive targets to ban the landfill of biodegradable waste to 35% of 1995 levels by 2020 (Hester and Harrison 2002).

2.2 Legal Aspects of Water

The Water Framework Directive (2000/60/EC) is an integrated approach to the protection, improvement and sustainable use of all European water bodies including surface and ground water, fresh and saline water. According to the Environmental Agency (2008), the Directive aimed to replace the following Directives:

- Surface Water Abstraction Directive - 75/440/EEC
- Exchange of Information on Surface Water Decision - 77/795/EEC
- Surface Water Abstraction Measurement / Analysis Directive - 79/869/EEC
- Freshwater Fish Directive - 78/659/EEC
- Shellfish Waters Directive - 79/923/EEC
- Groundwater Directive - 80/68/EEC
- Dangerous Substances Directive - 76/464/EEC

Another EU Directive that is equally important is the Urban Wastewater Directive (91/271/EEC). The main aim of the Urban Wastewater Directive is to impose specific treatment standards depending on the size of the community and the location of discharge. A minimum of secondary treatment is required for urban wastewater. Advanced treatment applies depending on the discharging water bodies.

The Water Environment and Water Services (Scotland) Act 2003 implements Water Framework Directive in the protection of the water environment in Scotland. The regulation is also known as Controlled Activity Regulations (Scottish Environmental Protection Agency 2008). The act also aims to promote sustainable use of water and prevent pollution or emission to the water environment. River basin districts are designated by the

Scottish Minister. SEPA is responsible for characterising, monitoring and implement measures in regards to each district within Scotland.

2.2.1   Legal Aspect of Trade Effluent

When designing a wastewater treatment system, it is vital to set the objectives for the system. Legislation plays an integral part on setting the criteria for wastewater treatment design and inevitably the final effluent quality.   Wastewater legislation emphasised the effluent discharge point. Trade effluent is any wastewater that is discharged from industrial or business premises other than surface water and domestic sewage (Businesslink, 2011a). Trade effluent can be discharged to foul sewer or direct to local water bodies after treatment. According to Business Link (2011a), a UK government's online resource for business, most owners that discharge effluent to a sewer are most likely to require consent from the local water company. Local water companies may levy a charge for the services and this also requires businesses to conform certain requirements of the discharged effluent.   In Scotland, the Sewerage Authorities Act under the Sewerage (Scotland) Act 1968 requires licensing of discharges to the sewer (Organisation for Economic Co-Operation and Development 2009). The discharge consent is issued by SEPA and the interface between the water companies still remains. In order to preserve the efficiency and safety of the treatment work and to protect the surrounding watercourse, the water company must apply standards for the discharged effluent in terms of flow, pollutant strength and characteristic. Parameters of a typical consent include: maximum permitted daily flow (m^3/day), maximum BOD or COD value. Other parameters may be measured depending on the nature of the business: e.g. oil, metals and organic chemicals.

The main legislation that affects trade effluent discharges are as follows (Thompson 2011):

- Control of Pollution Act 1974 laid down the requirements for controlling trade effluent discharges into public sewers.

- Sewerage (Scotland) Act 1968 regulates discharges of trade effluent through consent or by agreement.
- Water Environment and Water Services (Scotland) Act 2003 laid down conditions to regulate polluting activities.
- Water Environment (Controlled Activities) (Scotland) Regulations is intended to control impacts on the water environment.

2.3 Integrated Pollution Prevention and Control (IPPC)

To regulate and control industrial activities that may cause pollution, previous EU legislation was designed to target specific activities or pollutants (Institute for European Environmental Policy, 2009). However, Council Directive 96/61/EC on Integrated Pollution Prevention and Control (IPPC) was adopted in 1996 to integrate prevention and control of air, water and land pollution. According to O'Malley (1999), the IPPC mainly covers six groups of industrial activities: energy, production and processing of metals, mineral industry, chemical industry, waste management and other areas such as paper and pulp, tanning, and certain agricultural activities. The implementation for IPPC in Scotland is transcribed as the Pollution Prevention and Control (Scotland) Regulations made under the Pollution Prevention and Control Act 1999. Operators of industrial and waste installations covered by Annex I require authorisation of a permit. In Scotland, the permit is granted by SEPA which covers a wide range of environmental impacts such as emission to air, water and land, waste production and disposal, energy use, accidents and site maintenance.

Under the Pollution Prevention and Control (Scotland) Regulations SI 2000/323, a stationary technical unit where one or more activities listed in Part 1 of Schedule 1 are carried out requires a PPC Part A. Examples of activities listed in Part 1 of Schedule 1 are "Disposal of Waste Other Than by Incineration or Landfill", "Activities Involving Asbestos" and "Treatment of Animal and Vegetable Matter and Food Industries". As Sureclean operates a hazardous waste transfer station, they obtained a PPC Part A permit that set

out conditions for their operations to prevent pollution. These conditions are also known as ELVs set by SEPA that acts as a standard for determining the responsibility of the permit holder in respect to pollution prevention and control. Using the BAT method ensure that any cost connected to applying the technology is not disproportionate to the level of environmental protection it provides (WRAP 2010b). Site specific "General Binding Rules" (GBRs) are fixed sets of conditions that apply to relevant unit in the PPC permit. Permit holder or operator of the regulated installations are required to carry out regular monitoring and recording of data such as environmental releases which the permit holder is mandated to supply to SEPA. The Regulations highlight the use of efficient and suitable management systems to ensure the protection of the environment.

2.4 OSPAR Convention

The Convention for the Protection of the Marine Environment of the North-East Atlantic (known as the "OSPAR Convention") forms the fundamental basis for national laws governing the discharge of offshore drilling wastes in the waters of the OSPAR signatory states, which includes Great Britain (Wills 2000). The Convention has been ratified by Belgium, Denmark, Finland, France, Germany, Iceland, Ireland, Luxembourg, Netherlands, Norway, Portugal, Sweden, Switzerland and the UK and approved by the European Community and Spain (Offshore Industry Committee expert assessment panel 2010). Understanding these regulations enables Sureclean to set design parameters for the solid and liquid waste treatment systems intended for offshore oil and gas installations.

According to OSPAR (2011), there are currently more than 1200 offshore installations in operation in the OSPAR maritime area. The Offshore Petroleum Activities (Oil Pollution Prevention and Control) Regulations 2005 and the Offshore Petroleum Activities (Oil Pollution Prevention and Control) (Amendment) Regulations 2011 controls all deliberate oil discharges such as produced water (Great Britain 2009). Other supporting legislation that regulates oil produced water discharges in offshore installations included the

Paris Convention (PARCOM) which set the discharge limit for water contaminated with oil at 40 mg/L (Offshore Industry Committee expert assessment panel, 2010). In 2011, the OSPAR recommendation, made in 2006, of 30 mg/L monthly average dispersed oil in water discharge came into effect.

2.5 Environmental Legislation Conclusions

Legislation plays a central role in business decision. As stated by Business Link (2011b), the UK government's online resource for businesses "there are legal requirements for managing your waste efficiently and safely. By meeting these requirements your business will avoid enforcement action and may cut costs". The overview of the main regulations and legislation that affect the waste and wastewater industries will shape the design of the treatment regime at Sureclean. Although not required by law, companies such as Sureclean are encouraged to use effective environmental management systems such as the internationally recognised ISO system or other recognisable system such as the British national standards for environmental management, BS8555 (Organisation For Economic Co-Operation And Development 2009). Engagement with relevant stakeholders at an early stage of any environmental project is important. Therefore early involvement of stakeholders such as local environmental agencies and councils are crucial in the success of this research project.

Chapter 3   Waste Stream Analysis

The purposes of waste treatment technologies are: to reduce pollutants that are discharged in to the environment, to enable reuse or recycle any valuable elements, therefore it is an important step to identify the most suitable treatment approach and it is part of waste profiling. Waste profiling can identify: the volume of waste to be treated, major pollutants present and their quantity and legislation that govern the waste. This information is fundamental to design the treatment strategies and targets.

Waste streams identification and characterisation are important steps for the effective design of a solid waste and wastewater treatment system. Woodard (2006) described waste characterisation as the process of determining chemical, biological, and physical characteristics of wastewater and solid waste streams. Waste streams are analysed using combinations of environmental analytical techniques and instrumentation. According to Hardy (2010), analytical chemistry consists of the separation, determination and identification of components in a given sample by a quantitative or qualitative method. These analytical methods include gravimetry, titrimetry, spectrometry, chromatography, thermal analysis, electrochemical analysis and radiochemical analysis (Kealey and Haines 2002).

Understanding the physical and chemical characteristics of the waste stream is an important determining factor in the selection of the correct treatment technology. Sureclean receives a diversity of waste streams from a range of sources including manufacturing sectors, power generations and food and drink industries but primarily the waste comes from the oil and gas sector hence the wastes are mostly oil contaminated. These wastes have different waste profiles; in fact the waste profile varies from client to client and from site to site within the same sector and therefore the analysis is vital to provide a fundamental understanding to Sureclean waste streams. This study aimed to conduct a comprehensive waste analysis, which could provide an overview of pollutants in each waste stream.

As discussed in Chapter 1, Sureclean has been subjected to a discharge consent issued by SEPA (as seen in Table 1-1). The main parameters that are governed by the discharge consent are: pH, heavy metals, TSS, BOD, COD and TPH. However, Sureclean was also interested in pollutants such as PAH and Total Organic Carbon (TOC) of each waste stream for future waste profiling and treatment.

3.1 Waste Materials

In this study, the waste materials were characterised by mixtures of solid and liquid waste that were mostly contaminated with oil. Six waste streams were investigated in this study. The wastes were obtained from Sureclean clients and collected from Sureclean Alness WTS: distillery effluent (DE), drilling fluid (DF), interceptor effluent (IE), produced water (PW), used oil based mud (OBM) and used oil based mud and water (OBMW).

3.1.1 Distillery Effluent (DE)

Scotland has a rich history in distilleries and has been famous for its whisky since the 15th century. The whisky industry has a gross income of £464 million and employs over 10,000 workers (Verso Economics, 2010). According to the Scottish Whiskies Association (2010), there are 101 working malt distilleries and 7 grain distilleries in Scotland. More than half of these distilleries are located in and around North Highlands and Speyside, which are within a few hours road travel from Sureclean Alness and Aberdeen WTS (as showed in Figure 3-1). Sureclean collected the DE or distillery spent wash from a routine wastewater tank cleaning project in a distillery located in Tain, Ross-shire Scotland.

Figure 3-1 Whisky Distilleries location around Sureclean (figure adapted from Athens Hash House Harries 2012).

3.1.2   Interceptor Effluent (IE)

As discussed in Chapter 1, Sureclean has a six-stage interceptor that collects wastewater accumulated around the site via a series of drains. Sureclean interceptors are a type of interceptor called the American Petroleum Institute (API) separator and consists of a rectangular tank where the effluent flows horizontally to enable free oil to rise to the surface due to difference in specific gravity (Foster 2003). Sureclean interceptor effluent may also contain other wastes from external sources such as residual effluent from a tank cleaning project, oil-in-water emulsions from garages, petrol-station forecourt and oil rigs that were based inshore for maintenance.

3.1.3   Produced water (PW)

According to the Oil and Gas UK (2009a), produced water (PW) is water trapped in an oil and gas reservoir or injected water that is brought to the surface admixed with oil and gas. PW have complex mixtures and the main pollutants in produced water are oil, heavy metals, radionuclei, treating chemicals, salt and dissolved oxygen (Ray and Engelhardt 1992). According to Stewart and Arnold (2009), the composition of produced water may vary between oil fields as well as different zones in the same field. Sureclean collected the PW from a tank cleaning project on an oil rig that was docked in Invergordon harbour for routine maintenance.

3.1.4   Drilling Fluids (DF)

Drilling fluids (DF), also known as drilling mud are a mixture of natural and synthetic compounds that is used to help in the drilling operations both onshore and offshore. According to Bucksch (1997), DF generally includes all fluids, gas, air, water, oil and mud; and drilling mud usually refers to a suspension of solids in water or oil. DF could either be water-based or oil-based depending on the drilling and formation requirements (Fink 2003). According to the American Society of Mechanical Engineers- Shale Shaker Committee (2005), drilling fluids are used in drilling operations where the fluid is circulated from the surface to the borehole, along the drill string and bits (as seen in Figure 3-2). DF is vital to providing a barrier for well control, suspend cuttings, control formation pressure and cooling and lubricating the drill bits (Rigzone 2012; American Society of Mechanical Engineers- Shale Shaker Committee 2005). Drilling fluids are important in upstream oil and gas, however according to the Oil and Gas UK (2009b), drilling fluids is also the main source of chemical discharge reaching the marine environment. Therefore it is pertinent to ensure safe management and disposal of DF. The DF sample was the liquid phase of the OBM from a tank cleaning project on an oil rig that was docked in Invergordon harbour for routine maintenance.
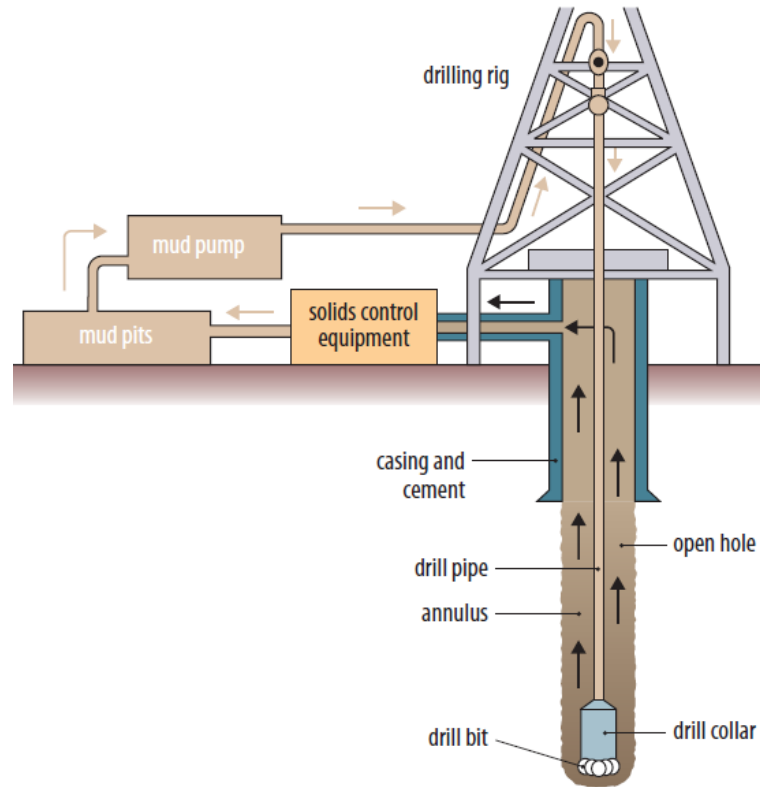
Figure 3-2 shows a DF system of a rig and well (International Petroleum Industry Environmental Conservation Association/ International Association of Oil & Gas Producers 2009).

3.1.5   Oil-based Mud (OBM) and Used oil-based mud and water (OBMW)

As mentioned in Section 3.1.4, drilling mud is a suspension of solid in water or oil and sometimes drilling mud is mentioned as DF (Mian 1992). Drilling mud is broadly classified as oil-based mud (OBM) or water-based mud (WBM) and Fink (2003) described drilling mud as a special class of DF for deep wells. The main components in drilling mud are: bentonite, clay and chemical additives. OBM can be composed of diesel, minerals, synthetic oil and brine (Hyne 2001). Weighting material such as barite or galena could be used to increase the weight of the mud to exert more pressure in the well (Hyne 2001). The un-used OBM as well as the OBMW was from an oil rig that was docked in Invergordon harbour for routine maintenance, and the special waste and cleaning were managed by Sureclean.

3.2 Methodology

A procedure has been developed specifically for the waste streams discussed in Section 3.1, where Sureclean can analyse these similar waste streams in the future. Based on the information gathered from waste stream analysis and the current environmental legislation, treatment technologies selection and design criteria can be set and provide more confidence towards the treatment efficiency.

Representative samples of various waste streams were collected and analysed. The analytical work was carried out by a Masters (MSc) student, Urenna V. Ekeh in her MSc project that had been instigated by me in order to conduct a comprehensive waste stream analysis. Analytical techniques involved included: pH analysis, particle size analysis, Gas Chromatography/ Mass Spectrometery (GC-MS), COD, TOC, Fourier Transform Infrared (FTIR) Spectroscopy, Molecular Fluorescence Analysis and Inductively coupled plasma atomic emission spectrometry (ICPAES). GC-MS was performed to identify the aromatic and aliphatic hydrocarbons in the waste samples; FTIR was conducted to investigate TPH quantitatively, while Molecular Fluorescence was used to study the concentration of PAHs in the samples. Metal concentrations were analysed using ICPAES. Suspended Solid (SS) measurement was determined by Particle size analysis.

3.2.1   pH

This analysis was carried out to examine the concentration of hydrogen ions in the waste samples. The analysis was performed using pH paper.

For each sample the following procedure was performed in triplicate: pH paper was dipped in 5 mL of each sample that was put into a clean beaker. The decolourised pH paper was then matched with the pH colour chart to obtain the pH value related to the colours and the results recorded.

3.2.2   Particle Size Analysis

This method was used to determine the particle size of suspended solids in wastewater and drilling mud samples and was taken from the environmental method used for monitoring particle size of suspended solids in the laboratory. The instrument used was a Malvern Mastersizer/E utilising the 100 mm Lens, the Malvern particle sizer software, beakers and glass dropping pipettes.

For each sample the following procedure was followed in triplicate:

The beakers were washed with laboratory glassware detergent. The samples of 25 mL each were put into the beakers. The instrument was set up and left to stabilise for 30 minutes. The laser was aligned and the software was programmed to allow the analysis to take place.

3.2.3   GC-MS

The method used for this analysis was based on the US EPA 625 Method (ALS – Columbia 1997) for the qualitative analysis of Hydrocarbons. All of the samples were analysed using a Hewlett Packard HP5890 / 5971 Gas chromatograph /Mass Spectrometer after undergoing a solid phase micro-extraction (SPME) according to the following procedure:

A blank analysis was first carried out to ensure background noise was sufficiently low to run a sample and there was no sign of contamination. Each sample (3 mL) was measured into a clean headspace analysis vial, sealed with a cap lined with Teflon septa, and heated on a heating block for 5 minutes at 60 °C. After which, a SPME extraction was carried out using a 100 µm polydimethylsiloxane (PDMS) coated fibre, as is recommended for analysing non-polar volatile compounds such as hydrocarbons. The needle was then exposed to the sample and the fibre was extended into the headspace above the sample for 15 minutes, after which the fibre was retracted and desorbed immediately into the GC injector port in a similar manner. Headspace sampling was conducted for 45 minutes which included 15 minutes of sorption onto the fibre and 30 minutes of desorption into the

GC. The GC-MS analysis was carried out using a ZB 5, 30 m x 0.25 mm id x 0.25 µm non polar column. Initial oven temperature was 45 °C and was held for 5 minutes, then ramped at 6 °C / minute up to 300 °C, and was held again for 5 minutes. Injector temperature was 250 °C. The carrier gas was helium, and a 5971A mass selective detector was used. The Mass Spectrometer EM Voltage was 1800 volts, scan range 10 - 400 atomic mass unit (A.M.U), and scan rate of 1.97.

3.2.4   FTIR

This method was used for the determination of TPH in each sample and was based on the Department Of Energy and Climate Change (2006) method for the determination of hydrocarbons. This method is based on a tetrachloroethylene (TTCE) extraction followed by infrared quantification at a wavelength of 2930 cm^{-1}.

Each sample was analysed using a Perkin Elmer Spectrum GX FTIR spectrophotometer and analysed by the following procedure:

Standard oil solution (10 cm^3) of perklone (TTCE) of 10, 50, 100, 200, 300 and 400 ppm were prepared from a 1000 ppm stock solution in triplicate. Each solution was transferred into an infrared sample cell and scanned at the region of 3100 to 2700 cm^{-1} region. A calibration graph was then constructed of concentration against peak area (as seen in Figure 3-3). The hydrocarbons from each sample were then extracted by shaking 10 mL TTCE and 1 mL of each sample followed by a Florisil extraction of any fatty acids by running through a column filled with 1.3 g of Florisil. The peak area at 2930 cm^{-1} was then determined using the built in software and the unknown concentrations determined by interpolating the perklone calibration graph generated earlier.
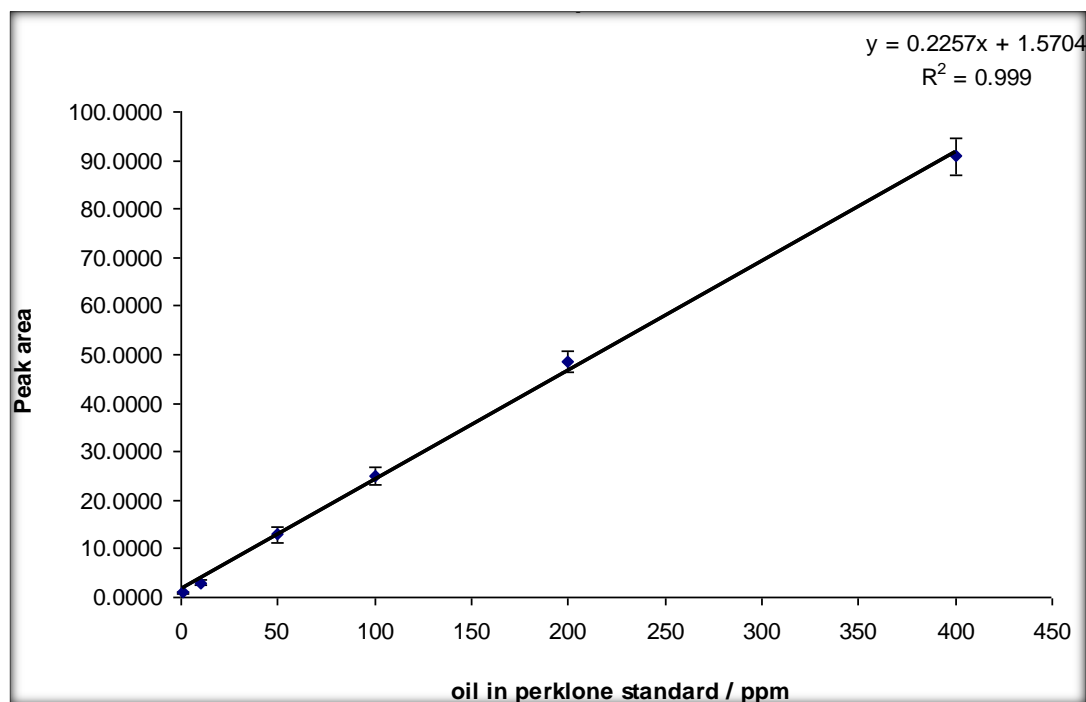
Figure 3-3 shows the FTIR calibration curve of oil in perklone standard solution.

3.2.5   COD

This method was used to determine the COD in wastewater and drilling mud samples. The procedure was obtained from the EPA Method 410.4 of the environmental monitoring systems Laboratory, office of research and development of US EPA (ALS- Columbia 2007a).

A standard calibration graph (refer to Figure 3-4) was generated using solutions of potassium hydrogen phthalate (KHP) to cover the COD range of 3-900 mg/L of the waste samples. Each standard was then analysed for its absorbance, using a Perkin Elmer Lambda 900 UV/Vis spectrophotometer, at 600 nm using deionised water as the blank solution. A digestion solution and a catalyst solution were prepared by dissolving potassium dichromate and (acidified) silver sulphate in 500 mL deionised water respectively.

The graph shows $y = 0.0003x + 0.111$ and $R^2 = 0.9749$, with y-axis "Absorbance / peak area" and x-axis "Concentrations (mg/l)".
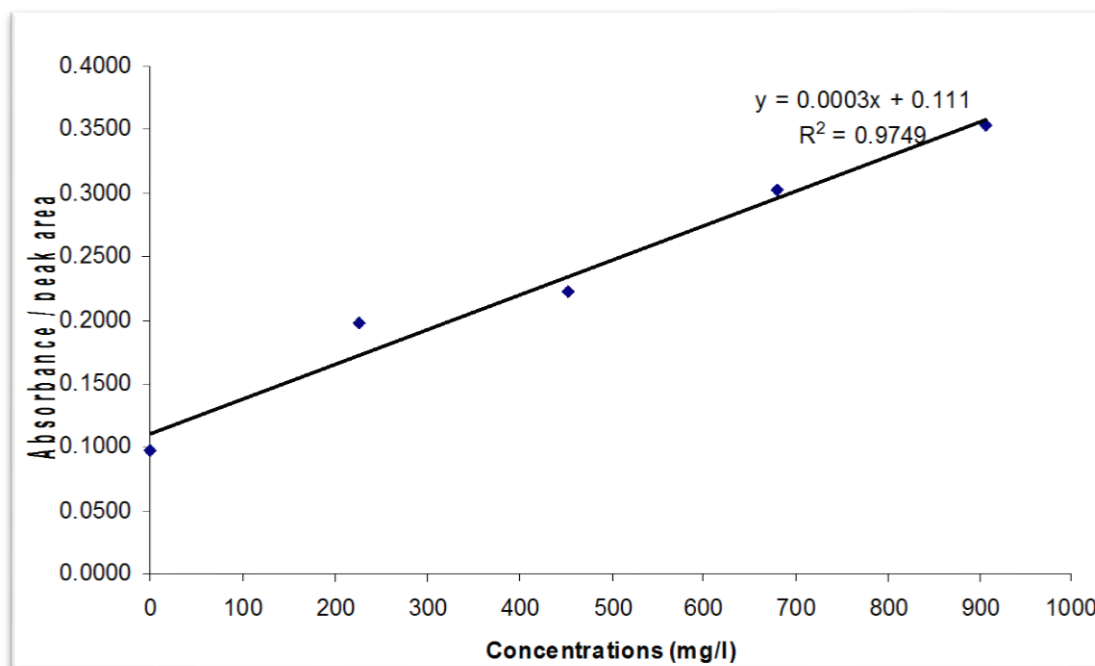
Figure 3-4 COD calibration curve of potassium hydrogen phthalate

Before preparing the sample solutions in triplicate, diluted H_2SO_4 was used to wash all tubes, caps and sample bottles to prevent any contamination. Each sample (2.5 mL) was then pipetted into a COD sample tube before adding the digestion solution (1.5 mL) to the tube and mixed. The catalyst solution (3.5 mL) was then added carefully down the side of the COD vials before the tube was capped and shaken thoroughly. The COD vial was then placed into a digestion block (HACH COD block digester) for two hours. The vials were then removed from the digestion block. With the caps still on, the vials were shaken and was allowed to cool to enable precipitates to settle. The solution was then transferred to a quartz cuvette and its absorbance measured as described for the standard solutions.

The sample concentrations were then computed by interpolating the calibration graph generated with the KHP solutions. Each result was then multiplied by the dilution factor of 2 since samples were diluted with water 50:50 to enable their absorbance values to fit into the calibration curve. The values that fell between the lowest and highest calibration standards were reported in mg/L.

3.2.6 TOC

TOC wastewater and drilling mud samples were analysed based on the EPA Method 415.0 of the environmental monitoring systems Laboratory, office of research and development of US EPA (ALS- Columbia 1999). TOC was analysed using a Shimadzu TOC VCPH TOC Analyser with an attached Shimadzu ASI-V Auto sampler and a TOC – control V software.

For each sample the following procedure was followed in triplicate:

DE, DF, PW and OBM were diluted to the dilution factors of 2.5, 5 and 11.5. The diluted samples (25 mL) and the OBMW sample were transferred to cleaned TOC vials. The TOC analyser was switched on for 30 minutes until the temperature stabilised at 680 °C. The TOC – control V software was programmed so that the analysis could take place.

3.2.7 ICPAES

ICPAES, also known as Inductively Coupled Plasma-Optical Emission Spectrophotometer (ICP-OES) was used to analysed heavy metals in the waste samples. The method used was obtained from US EPA Method 200.7 for the determination of metals (ALS- Columbia 2007b). Heavy metals were analysed using a Perkin Elmer Optima 3300 DV ICPAES with a Perkin Elmer Autosampler AS 9L and a Version 3.4.1 of ICP Winlab software.

For each sample the following procedure was followed in triplicate:

Each sample was digested by adding diluted HNO_3 (50 mL) into 5 mL of sample in a beaker which was subsequently covered with a watch glass and heated on a hotplate to 85 °C; and then the beaker was uncovered to reduce the sample volume to 20 mL. The digested sample was then allowed to cool before the sample was filtered with filter paper. The filtrate was diluted with deionised water to make up 100 mL. Two sets of 100 ppm multi-element standards (As, Cr, Ni, Mo, Co and Zn, Pb, Sn, Cu) were prepared using 1000 ppm standard solutions of the chosen metals. A calibration graph was made by using stock solutions of different dilutions: 0.05, 0.1, 0.5, 1.0 and 5 ppm respectively. A Perkin Elmer 3300 DV ICPAES

was used. The element wavelengths (in nm) chosen from the ICPAES instrument menu were listed as; As – 228.812, Co – 236.892, Cr – 267.716, Cu– 324.752, Mo – 202.031, Ni – 231.604, Pb – 220.553, Sn – 189.980 and Zn– 213.857.

3.2.8   Molecular Fluorescence Spectrometry

The concentration of PAH present in the wastewater and drilling mud samples were determined using a Perkin Elmer LS 55 spectrometer and FL Winlab software.

For each sample the following procedure was followed in triplicate:

The prepared standard solutions of different concentrations were used to construct a calibration curve of marine diesel in dichloromethane with excitation wavelength of 268 nm and emission wavelength of 326 nm. Solvent exchange was carried out using 1 mL of each sample to 25 mL dichloromethane in triplicate. Sample cells were filled and inserted into the instrument for analysis. Measurement of intensity and concentration of the samples were taken using the fluorescence instrument. The results and standard calibration graphs were generated as seen in Figure 3-5.

$$y = 118.55x + 8.2017$$
$$R^2 = 0.9978$$
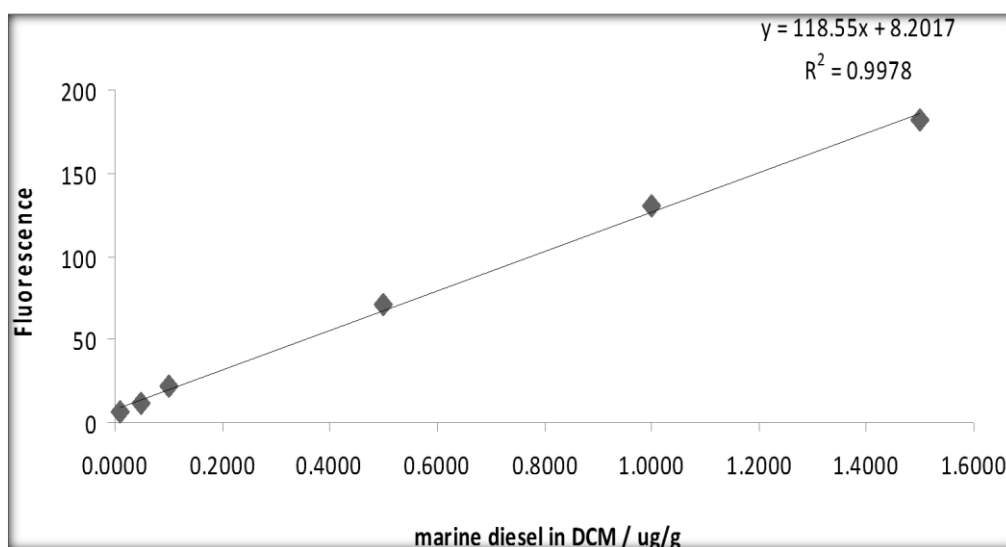
Figure 3-5 Molecular fluorescence calibration curve of marine diesel standard in dichloromethane

3.3 Results and Discussion

A summary of analytical results for Sureclean waste streams can be found in Table 3-1. Six waste streams (DE, DF, IE, PW, OBM, and OBW) were investigated for pH, particle size, TPH, PAH, TOC, COD and heavy metals.

	DE	DF	IE	PW	OBM	OBMW
pH	6	8	7	5	6	5
PSA D[V,0.9] (µm)	45.13	144.55	149.51	141.92	40.80	168.26
PSA D[V,0.5] (µm)	9.23	66.01	63.03	130.03	16.87	109.87
TPH (ppm)	22	11	8	9	54	116
PAH (µg/g)	0.63	1.07	0.49	0.14	0.33	94.27
TOC (ppm)	2070	88	5	2	530	5777
COD (mg/L)	896	842	1053	827	817	1453
As (mg/L)	0.49	<LOD	<LOD	<LOD	<LOD	0.86
Co (mg/L)	<LOD	<LOD	<LOD	<LOD	0.01	<LOD
Cr (mg/L)	0.01	0.49	<LOD	0.01	4.48	<LOD
Cu (mg/L)	2.16	4.61	<LOD	7.89	5.53	5.39
Mo (mg/L)	0.99	2.53	0.72	1.15	1.58	1.35
Ni (mg/L)	0.06	0.5	<LOD	<LOD	1.83	<LOD
Pb (mg/L)	1.89	2.71	0.19	26.23	8.73	5.07

Table 3-1 Analytical data from the characterisation of wastewater and drilling muds (DE: Distillery Effluent; DF: Drilling Effluent; IE: Interceptor Effluent; PW: Produced Water; OBM: Used Oil-Based Mud; OBMW: Used Oil-Based Mud and Water; LOD: limit of detection).

pH

pH was tested to determine the acidity or the alkalinity by measuring the hydrogen ions in the waste streams. pH is an important factor in waste treatment as it can affect the overall efficiencies of the treatment performance especially in chemical or electro-coagulation treatment (World

Health Organization 2006). It can be seen from Table 3-1 that all waste streams were between pH 5-8, therefore pH adjustment would not be part of the waste treatment regime.

Particle size distribution

Particle size analysis was conducted to understand the properties of suspended solids in the Sureclean waste streams. The two main results reported in this study were as follows:

- D [v, 0.5] = n: states that n value has 50% of the distribution above and 50% of the distribution below this value (Volume median diameter).
- D [v, 0.9] = n: states that n value has 90% of the distribution below this value (Volume median diameter).

Particle size distribution of solids in a waste stream is an important factor in physical treatment, as well as the determinant factor for chemical coagulation. It can be seen that D [v, 0.9] of DE and OBM have particle size between the colloids particle range. Solids with size range between 100 to 10^{-4} µm are known as colloid particles (Keily 2007) and generally these particles take a long time to settle from the liquid phase, thus require chemical treatment to enhance the settlement. IE, DF, OBMW and PW have solids with particle sizes between 141.92 - 168.26 µm. Solids with particle sizes that are larger than 63 µm are known to be settleable suspended solids (Hanh 2002) and these solids could be removed by physical treatment such as sedimentation and filtration. The separated oil-contaminated solids could be further treated by mechanical separation, thermal treatment and bioremediation (Al-Ansary and Al-Tabbaa 2004).

TPH

TPH was investigated using FTIR following the DECC method, which also includes the measurement of most light oil fractions. It can be seen from Table 3-1 that OBMW and OBM had two of the highest TPH concentrations among the waste streams. The origins of the hydrocarbons in OBMW could be from a variety of aliphatic hydrocarbons from diesel used in the drilling

muds. The OBM has a lower concentration of TPH than OBMW, which may indicate that the OBMW was contaminated with oil when it was used. The two lowest TPH observed in Table 3-1 were the PW, DF and IE, which was lower that DE. This observation for DE could indicate the presence of other hydrocarbons such as alcohols.

For Sureclean, the discharge consent is set at 100 mg/L. Oily wastewater are generally separated into oil and aqueous phases through gravity settling by an interceptor or clarifier (Noyes 1991). This is evident when comparing the TPH result of IE to the other waste streams. If stringent discharge TPH limit is in place, Sureclean could employ a filtration system, such as ultra-filtration, to further remediate the wastewater.

PAH

PAH was examined using the Molecular fluorescence technique because the unsaturated aromatic ring structure of PAHs have fluorescing ability. PAH contains at least two aromatic rings and these compounds are thought to be carcinogenic and not easily degraded in the natural environment. It can be seen from Table 3-1 that the OBMW sample contained the highest amount of PAH. The PAHs level of all other waste samples i.e. DE, DF, IE, OBM and PW were below 1.07 µg/g. According to González and Ma (2006) PAHs are generally found in mixtures such as soot, crude oil, coal or roofing tar. This may indicate that the OBMW sample was contaminated with one of those mixtures. PAHs could be removed by activated carbon filtration as PAHs were shown to be adsorbed on the activated carbon granule (Bansal and Goyal 2005). González and Ma (2006) also stated that PAHs tend to be absorbed on to particulate matter; therefore removal of suspended solids could potentially also remove PAHs.

TOC

TOC analysis was carried out to determine the amount of organic carbon present in each sample. The TOC results correlated with the PAH and TPH results, in which OBMW showed the highest concentration at 5777 ppm. The

analysis also showed that DE contained 2070 ppm TOC, but the wastewater was relatively low in TPH and PAHs. However, the GC-MS result (as seen in Figure 3-6) of DE showed that there were other organic compounds present and a high percentage of heptanoic, hexanoic and butanoic acid was observed. Thakur (2011) stated that distillery spent wash often contain high concentrations of biodegradable organic compounds such as organic acids and the author recommended biological treatment as a means to remove these compounds. Combined biological and AOP such as ultra-violet (UV) radiation combined with hydrogen peroxide (H_2O_2) and Ozone (O_3) and photocatalysis and anaerobic biological treatment were shown to be effective in reducing the amount of organic compounds in DE (Gogate and Pandit 2004; Oller, Malato and Sánchez-Pérez 2011).
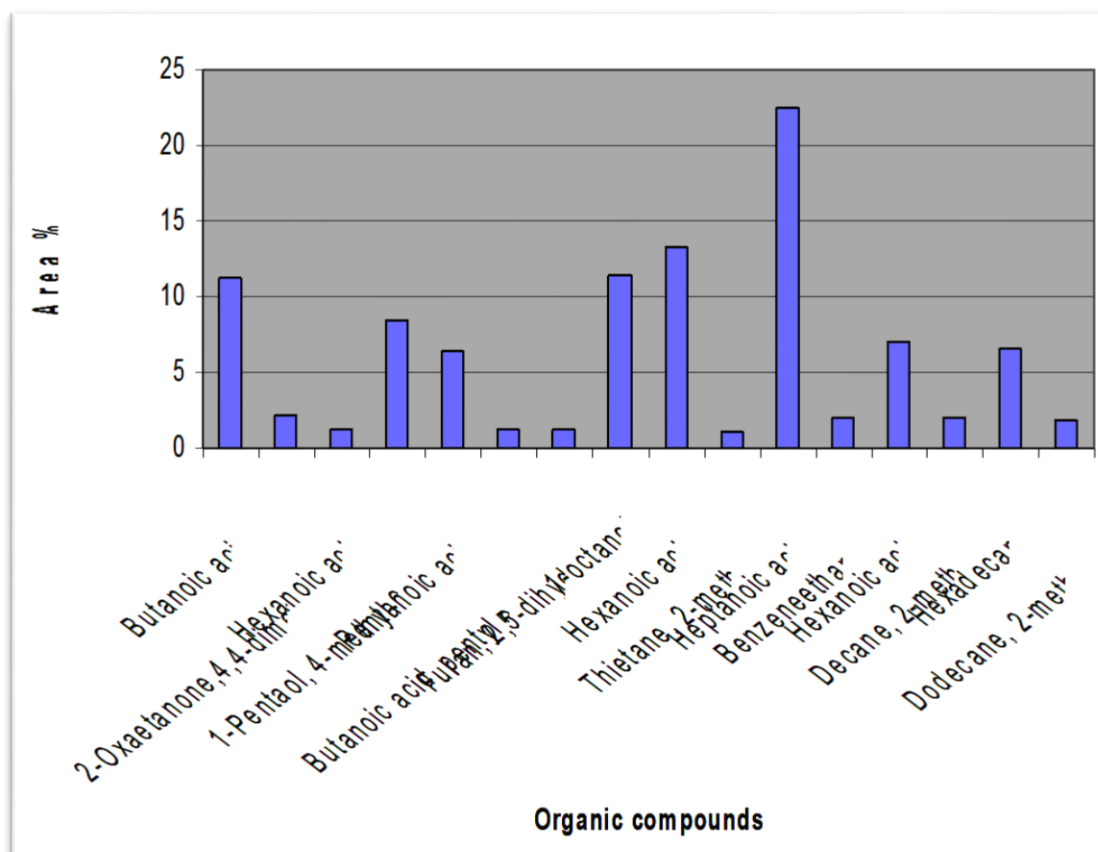
Figure 3-6 demonstrates the GC-MS results for DE.

<u>COD</u>

COD analysis unlike TOC analysis, is an indirect measurement of organic compounds present in the waste streams by measuring the amount of oxygen required to oxidise organic matter that is vulnerable to oxidation by chemical oxidants such as potassium dichromate. COD and BOD are to determine the presence of oxygen demanding wastes (White 1986). However, according to Wang et al. (2004), COD is more commonly used to monitor wastewater quality because the analysis is faster than BOD. BOD analysis takes 5 days as opposed to three hours for COD analysis. The COD results as seen in Table 3-1 shows that OBMW had the highest level at 1453 mg/L, followed by IE at 1053 mg/L. As seen in Figure 3-7, the GC-MS results showed only aliphatic hydrocarbon and from Table 3-1, it can be seen that TPH level for IE was very low at 8 ppm. These results indicate that other inorganic matter that may be present in IE. All the COD levels in the waste streams investigated were within Sureclean discharge consent of 3000 mg/L. However, if Sureclean decided to further improve The COD of Sureclean waste streams could be reduced in a combination of chemical coagulation and filtration for the removal of colloidal particles and heavy metals, followed by AOP to further degrade recalcitrant pollutants.
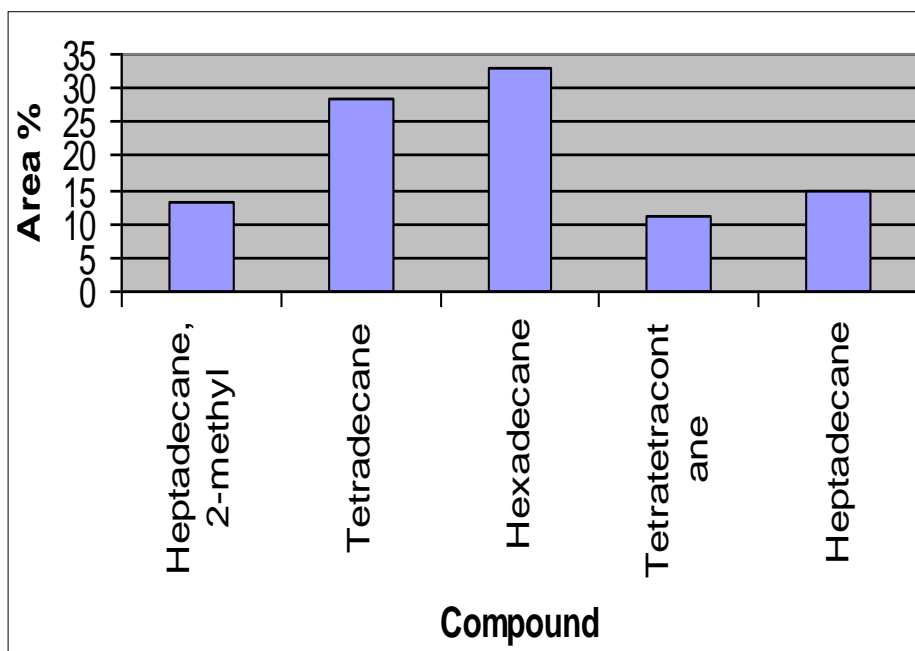
Figure 3-7 demonstrates the GC-MS results for IE.

<u>Heavy metals</u>

ICPAES was conducted to determine the heavy metal concentrations present in the waste samples. It can be seen that the highest concentrations of metal seen in Table 3-1 was noted to be Pb and Cu in PW, OBM and OBMW. The main source of Pb and Cu may have been from vessels or pipe corrosion, as well as additives used in drilling mud (Harrison 2001; Reis 1996). Total heavy metals for each waste stream were as follows: DE- 5.60 mg/L; DF- 10.84 mg/L; IE- 0.91 mg/L; PW- 32.28 mg/L; OBM- 22.16 mg/L and OBMW- 12.67 mg/L. The heavy metals of all the waste streams except IE had exceeded the Sureclean discharge consent. Treatment of these waste streams is pertinent to comply with the PPC license. Physical/chemical treatments such as chemical precipitation and coagulation, electro-coagulation, ion exchange system and membrane filtration have all been found to be effective in removing heavy metals (Kurniawan et al. 2006) and therefore this suggest that Sureclean should employ one of the treatment techniques.

3.4 Conclusions and Future Work

By using combinations of environmental analytical techniques and instrumentations, the chemical, biological, and physical characteristics of wastewater and solid waste streams could be determined. Six waste streams (DE, DF, IE, PW, OBM, and OBW) were investigated for pH, particle size, TPH, PAH, TOC, COD and heavy metals. The significance of the analysis of these waste streams was to provide a comprehensive understanding of major pollutants present in the waste streams.

It can be seen from Table 3-2 that all of these waste samples, apart from IE, exceeded the Sureclean discharge limit and therefore treatment would be required to reduce the contamination level. Mechanical separation processes such as clarifier and filtration treatment could be used to reduce TSS. To reduce the TPH level, gravitational separation such as an interceptor could remove free oil from the wastewater. Chemical or electro-chemical and filtration processes could be used to further reduce TPH as

well as the heavy metals level. To tackle the TOC and COD level of all waste streams, AOP or combined AOP and biological treatment could be used to reduce organic compound in the waste streams.

	pH	TPH (ppm)	COD (mg/L)	Total Heavy Metals (mg/L)
Discharge Consent	5-7	100	3000	2
DE	6	22.1	896	5.60
DF	8	10.67	842	10.84
IE	7	7.96	1052.67	0.91
PW	5	8.57	826.67	35.28
OBM	6	53.85	816.67	22.16
OBMW	5	116.07	1453.33	12.67

Table 3-2 a comparison between Analytical data from the characterisation of wastewater and drilling muds and Sureclean discharge consent (DE: Distillery Effluent; DF: Drilling Effluent; IE: Interceptor Effluent; PW: Produced Water; OBM: Used Oil-Based Mud; OBMW: Used Oil-Based Mud and Water).

Chapter 4   Mechanical Separation

4.1 Introduction

Sureclean Alness WTS licensed by the SEPA receives various types of non-hazardous and hazardous wastes. There is a six-stage interceptor in the waste transfer station that collects wastewater accumulated around the site via a series of drainage systems. The wastewater contains suspended solids, some heavy metals and hydrocarbon. According to the Sewerage Authorities Act under the Sewerage (Scotland) Act 1968, Sureclean was required to obtain discharge consent from SEPA for the treated effluent to be released into the public sewer. Between September 2007 and September 2008, Sureclean discharged a daily average of 12,231 litres of treated trade effluent into the foul sewer. Sureclean intended to expand their waste volume input and thus increase their annual revenue. To improve the treatment efficiency of Sureclean solid and liquid waste streams, the mechanical separation process was investigated.

Mechanical separation, also known as physical methods of waste treatment was described by Woodard (2006) as "the removal of substances by use of naturally occurring forces, such as gravity, electrical attraction, and Van der Waal forces, as well as by use of physical barriers". Generally, this method of treatment does not alter the chemical properties of the waste. The more commonly used mechanical separation technologies in solid waste and wastewater treatment include screening, sedimentation, floating, mixing, filtration and centrifugal or gravitational separation (Ghosal 1993). The selection of the correct mechanical separation unit depends on the aim of the treatment and the waste type.

4.1.1 Types of Mechanical Separation System

There are several types of mechanical separation equipment, which exploit the differences in properties such as density, particle size, viscosity and electrostatic forces (Cheremisinoff 1998). The three main categories of pollutants in Sureclean wastewater are oil, solids and heavy metals. Therefore, the types of mechanical separation systems discussed in this section shall relate to separation of oil, solids and heavy metals in liquid waste streams.

4.1.1.a Solid-liquid Separation

According to Perry and Green (1997), a lot of experience and data that were collected for wastewater treatment had been gained from municipal wastewater treatment plants. In conventional municipal wastewater treatment (shown in Figure 4-1), mechanical separation of large and rigid objects from the wastewater treatment plant is crucial to protect the down flow pipe works (Water Environment Federation 2008a).
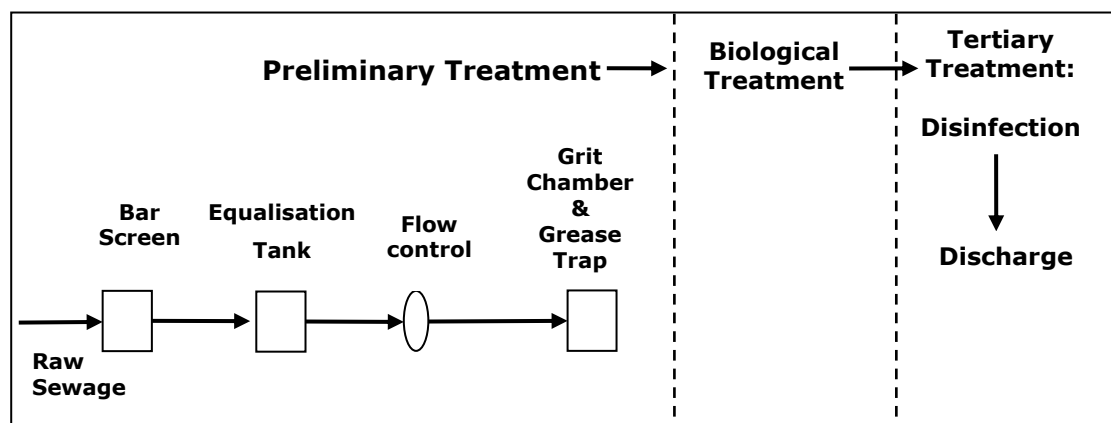
Figure 4-1 displays the mechanical separation units in a conventional municipal wastewater treatment plant.

Screening can remove objects such as debris, floating materials, paper, plastics and wood (Gray 2005). Screens can be classified by the size of their openings into coarse, medium and fine screens (Punmia, Jain and Jain 1998). Coarse screens are usually made from steel bar and the screens are designed as inclined parallel rows, also known as bar screen and each bar is

placed apart with a distance of 50-150 mm (Punmia, Jain and Jain 1998). The bar screens are used to removed rags and large objects to prevent clogging the down-flow pipework or finer screens (Hendricks 2010). Medium screens have openings between 20-50 mm and are used ahead of other devices, as well as improving screening efficiency of the coarse screens (Punmia, Jain and Jain 1998). Fine screens are frequently used in industrial wastewater treatment for removing fine inert and organic solids (Gray 2005). If the wastewater treatment plant uses a membrane biological reactor (MBR), a secondary biological treatment using a membrane as a bio-film to degrade organic matter, then it is crucial to use fine screens of 3 mm or less to prevent clogging of the membrane modules (Judd 2010).

Grit is mainly inert material such as sand, gravel, stones, and metal chippings, as well as some organic material such as coffee grounds and seeds (Vesilind 2003). Vortex separators or hydrocyclone and detritors are the two most commonly used for grit removal (Gray 2005). Vortex separators remove grit by centrifugal force as the inflow enters tangentially into the treatment unit (Gray 2005). Smith and Scott (2005 p.119) defined a detritor as "a square horizontal grit chamber, where the inlet is designed to distribute the flow evenly across the settling tank with a minimum velocity of 0.3 m/s". Removal of grit is necessary to prevent its accumulation within the pipe work, as well as, to prevent abrasion to treatment facilities (Water Environment Federation 2008b).

The process of sedimentation involved settling of particles through gravitational forces (Punmia, Jain and Jain 1998). Sedimentation units are designed to slow the flow of water, which in turn allows sufficient retention time for the solids to settle on the bottom of the units. Examples of mechanical separation units using sedimentation as a means for treatment are grit chambers, oil separators, clarifiers and inclined plate clarifiers. This process is thought to be one of the simplest and economical ways to decrease trade effluent treatment charges as it is thought to reduce solids by up to 50 % (Arundel 2000).

Filtration is a form of mechanical separation of solids from a suspension in a liquid by means of a porous medium or screen (Richardson et al. 2002). A porous medium is composed of solid particles that are generally larger than the solids to be separated (grains such as sand or ceramic beads) with pores that permits the flow of fluid (Coulson, Harker, and Richardson 2003; Espedal, Fasano and Mikelić 2000). Filtration is effective in removing low-level suspended solids (Cheremisinoff 1998). Filtration has been used in the treatment of drinking water since the eighteenth centuries (Agthe, Billings and Buras 2003). Since then filtration has been used in a wide range of industries such as petroleum refineries (Wang et al. 2004), food and beverage industries (Tay and Jeyaseelan 1995; Skeleton 2000) and the textile industry (Sójka-Ledakowicz et al. 1998; De Florio, Giordano and Mattioli 2005). The development of filtration systems have advanced to enable some wastewater reuse especially when the technology is coupled with other treatment systems such as biological, physio-chemical and the AOP; this were reported in academic papers published by numerous researchers (Skeleton 2000; Sójka-Ledakowicz, et al. 1998; De Florio, Giordano and Mattioli 2005; Hamoda, Al-Ghusain and Al-Mutairi 2004; Qi, Wang and Xu 2011).

Mechanical separation methods can also be used for dewatering purposes to reduce moisture content (Svarovsky 2000). This includes the use of a centrifuge. Centrifuge is the use of a high rotating force to separate solids from liquids as well as to separate liquids with different densities (Morris 1991). The centrifuge has been regarded as an extension to a clarifier as it can separate finer particles than a clarifier (Albestson 1991). Its operation is based on a simple idea of a clarifier or a settling tank, in which solids fall to the bottom by gravity. However, clarification is an extremely slow process as particles with a diameter between 0.008 to 0.04 mm can take up to 33 minutes to travel approximately 30 cm (Davies and Scott 2006). Centrifuges have a faster rate of separating solids from liquid, especially for suspended solids, compared to a clarifier.

4.1.1.b   Oil-Water Separation

Oil and grease, which included fatty acids, surfactant, petroleum hydrocarbon, animal or vegetable oil, arise from various industries such as the mining industry, textile, leather or the petro-chemical industry (Kajitvichyanukul, Hung and Wang 2011). Industrial wastewater can be categorised into three types according to the distribution of the oil phase: free-floating oil, unstable oil-in-water emulsions and stable oil-in-water emulsions (Porter 1990). Unstable and stable oil-in-water emulsions are more difficult to treat than free-floating oil and they are generally treated chemically (Porter, 1990). The most common and cheapest oily water treatment equipment relies on mechanical gravity settling to separate free oil droplets from the water fractions based on density differences (Arnold and Stewart 1998; Porter 1990; Coca-Prados, Gutiérrez-Cervelló and Benito 2011). One example of gravity settling equipment is the gravity separator specified by the API used in many refineries. The API separator can remove free oil that is larger than 0.015 cm in diameter (Eckenfelder, Ford and Englande 2009). Other types of gravity settling equipment included the parallel plate separators and corrugated plate separators (CPS), these plate separators were designed to separate oil droplets larger than 0.006 cm (Eckenfelder, Ford and Englande 2009).

4.1.1.c   Heavy Metals Removal

Heavy metals can be removed from aqueous waste streams by biological methods (Wang and Chen 2009), chemical precipitation (Kurniawan et al. 2006), mechanical separation such as reverse osmosis (Liu et al. 2008), ion exchange (Vaca Mier et al. 2001), membrane-filtration (Barakat 2010; Kurniawan et al. 2006), photocatalysis (Barakat 2010), floatation (Kurniawan et al. 2006) and adsorption (Lin and Juang 2002). However, the most widely used removal method in wastewater is chemical precipitation (Russell 2006; Lewinsky 2006; Barakat 2010), with hydroxides precipitation as the most common treatment method (Zhou et al. 1999).

4.1.2   Process Justification

Mechanical separation units currently used in Sureclean Alness WTS to treat interceptor effluent include an oil-skimming device, an inclined plate clarifier and a shale shaker. The oil skimmer, an Abanaki Oil-Grabber Model 4 (as shown in Figure 4-2) was an oil recovery device, which utilised an oleophilic conveyor belt to grab oil from the water surface. The recovered oil was collected into an Intermediate Bulk Container (IBC), where it could then be sold to licensed waste oil recovery companies for further re-use.

Figure 4-2 A photo of the Sureclean Oil Skimmer (painted in blue) that recovers oil from the interceptor underneath the unit; recovered oil was collected in an IBC (white container on the right of the skimmer).

The inclined plate clarifier (Siltbuster HB50) as shown in Figure 4-3, also known as the Sureclean Inclined Plate Clarifier (SIPC), is a type of lamellar clarifier that uses a series of plates tilted to enable heavier solids to settle at the bottom. According to the manufacturer, the clarifier could remove solids down to 2 microns in diameter (Siltbuster 2006).

Figure 4-3 displays (a) a photo of the Sureclean Inclined Plate Clarifier; (b) the mechanism of the clarification: solid accumulate at the bottom of the inclined plate, and the clean water move upwards and out of the plates (Guyer 1998).

The shale shaker (Brandt/EPI LM-3 Full Flo) as shown in Figure 4-4 is equipped with 40 mesh diamond back screens, 40 mesh screens means that there are 40 openings per inch in one direction and its perpendicular direction, this is also known as "40 by 40" screens. Sureclean aimed to expand its waste treatment portfolio; however, these mechanical separation units were not able to handle a wide range of waste. Therefore, there was a need to study other treatment systems to help Sureclean achieve its target.

Figure 4-4 A photo of Sureclean shale shaker (previously known as SWTS 01), which removes heavy solids from liquid waste streams.

Tay and Jeyaseelan (1995 p.33) stated that "the selection of technology depends on the influent characteristics, the required quality on the final product, the cost and ease of production". In order to improve treatment efficiency of Sureclean solid and liquid waste streams, the new processes would ideally complement the existing process to target the three main categories of pollutants: oil, solids and heavy metals. The studies were intended to employ the principles of sustainability and BAT, in which Sureclean intended to re-use the water fraction in the waste stream. This is particularly important because of the increasing cost of fresh water as well as the cost of disposing of trade effluent to sewer. Therefore, cost-effectiveness is also a crucial deciding factor in the selection of the new treatment systems.

As explained in section 4.1.1.c, the most common heavy metal removal method is chemical precipitation. Therefore, chemical treatment was investigated and shall be described in detail in Chapter 5 Chemical Treatment for Oily Waste. For the solid-liquid separation studies, Sureclean selected a decanter centrifuge for the dewatering of sludge and slurry. One of the main benefits of dewatering sludge is to reduce handling and transportation cost after treatment (Records and Sutherland 2001). The Centrifuge was also thought to be a more economical method in comparison to thermal treatment for the dewatering of sludge (Wakeman 2007). Filtration was also investigated as another means of mechanical separation. Filtration units are often installed downstream of a clarifier to further remove suspended solid (Huchler 2007). Filtration also uses less energy in comparison to drying or heating (Coulson, Harker, and Richardson 2003).

4.2 Decanter Centrifuge

There are generally two major types of centrifuge: Sedimenting and Filtrating (also known as perforated bowl centrifuge). The main difference between these two types of centrifuge is that the filtration centrifuge contains gaps that could be replaced with a screen or filters as opposed to a solid bowl in the sedimenting centrifuge. The sedimenting centrifuge can be

a tubular bowl, disk stack or solid bowl (also known as decanter) (Letki 2000). The disk stack and tubular bowl are known to be effective for separating liquid from liquid, i.e. separating liquids that have different density.

A decanter centrifuge, also known as a solid bowl scroll-discharge centrifuge (Records and Sutherland 2001) is comprised of a solid cylindrical bowl with the inside wall fitted with a screw conveyor. The decanter centrifuge have wide spread application in various industries for separating solid from sludge or slurry and it is principally used for clarification of liquids (Records and Sutherland 2001). In oilfield drilling, Bouse (2005) stated that these decanters are generally used in the conditioning of drilling fluids. The application of a decanter centrifuge is not only confined to the oil and gas industry but has also found applications in the petrochemical industry, pharmaceutical industry (Sambamurthy 1998), olive oil mill (Wang et al. 2004), dairy industry, agricultural industry and the paper and pulp industry (Bajpai 2010).

The working principle of a decanter centrifuge lies on the differential speed of the bowl and the screw conveyor, which is provided by the differential gear box (Records and Sutherland 2001). In other words, the bowl and screw conveyor rotates in the same direction but at a different speed. As shown in Figure 4-5, slurry is fed into the decanter centrifuge, particles higher in density (mostly solid) radiate outwards and accumulate towards an area known as the 'drying beach' zone before they are subsequently discharged as dry solid known as 'cake' or 'underflow solids' (Leung 2007). The 'beach' zone also known as the drying area, was designed to curve inwards to allow the opportunity for the solid to accumulate and dry as the liquid sloped down (Records and Sutherland 2001). The same force radiates the liquid counterpart towards the other end of the decanter centrifuge to an area known as the 'pond' zone; and the liquid is subsequently discharged outwards from the effluent discharge point as 'centrate', also known as 'overflow liquids' or 'effluent' (Leung 2007).
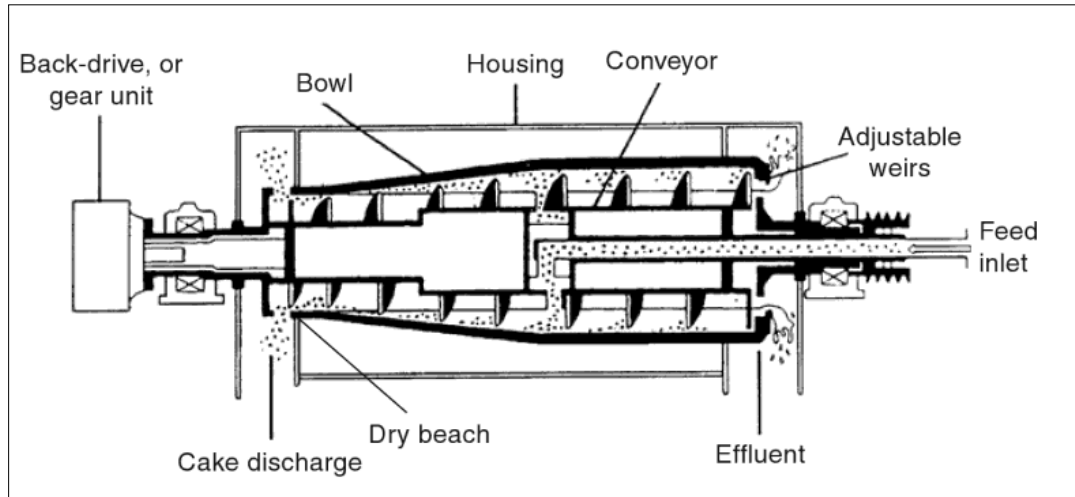
Figure 4-5 shows a schematic diagram of a conventional decanter centrifuge (Leung 2007).

The main advantage of a decanter centrifuge against other centrifuges and mechanical separation equipment is that it can separate solids in a continuous manner and it can be operated unattended for a long period of time (Records and Sutherland 2001). In comparison to gravity sedimentation and hydrocyclone systems, a decanter centrifuge has a faster solid separation, as well as higher slurry handling capacity (Records and Sutherland 2001). The decanter centrifuge can also take higher solid feed rates in comparison to other types of centrifuge (Leung 1998). The use of a decanter centrifuge for drying sludge has also been reported to have lower moisture content as compared to other mechanical separation (Wakeman 2007). The liquid runs around the helical scroll and is discharged over weir plates fitted at the parallel end of the bowl. The solids are moved by the conveying action of the helical scroll up the gentle slope of the conical section, out of the liquid and finally out of the machine.

4.2.1 Optimisation of decanter centrifuge

Axelsson and Madsen (2006 p.1) stated that "the sedimentation rate of solid particles or droplets in the gravity field is a function of the particle (or droplet) size, the density difference, and the viscosity of the suspension (or emulsion)". There are six parameters that affect the success of the decanting process, these can be divided into machine or process parameters

(Shiah 2007); these parameters can be changed in accordance to the incoming waste and the desired output.

4.2.1.a   Machine Parameters

Bowl Speed: Depending on the feed waste and the desired treatment outcome, operation of a decanter can be optimised by changing the bowl speed (revolutions per minute, rpm), also known as the rotational speed. As shown Equation 4-1, the larger the bowl diameter and the bowl speed, the higher the G force (also known as centrifugal force) (Schwarz 2008).

$$G = \frac{n^2 \cdot D_B}{1800} \qquad \text{\textit{Equation 4-1}}$$

Where G= G force; n= bowl speed (rpm), D_B= inner bowl diameter.

Higher centrifugal force acting on the solids in slurry would give rise to higher settling rates; consequently producing drier solids. However, this deduction does not always apply especially with finer solids, which have similar density. Wakeman (2005 p.383) explains that this is due to "adhesion to the contaminants or bubbles to the solid surfaces."

Scroll speed (conveyor speed): The screw conveyor (scroll) rotates at a different speed than the bowl. The scroll scrapes the solids away from the sides of the bowl, and then conveys them in the opposite direction to the dry area of the bowl. By lowering the differential speed, this will increase residence time of the solid, thereby increasing dryness of the sludge cake (Leung 2007).

Weir Height: The drying area is an inclined section of the bowl where further dewatering occurs before it is discharged. Weir plates (also known as the overflow weirs) can be changed for different applications to determine the depth of the pond. A smaller weir height or a shallow pond depth leads to a decrease in residence time for the slurry, which in turn

gives rise to a decrease in centrate quality but a dryer solid (Porteous 1990; Leung 2007).

4.2.1.b Process Parameters

Feed rate: Solid retention time in the bowl can be increased by reducing the feed rate. According to Leung (2007 pp. 96), "decreasing feed rate increases the liquid residence time, and allows more efficient settling of suspended solids". This can be achieved by controlling the feed rate via the feed pump. This allows the solid to remain in the 'beach' area longer, allowing a clearer centrate.

Chemical reaction: Gravitational and centrifugal sedimentation rates can be low when particle size of the feed slurry is very fine (Tarleton and Wakeman 2007). Therefore to improve separation characteristic of liquids that contain a high amount of colloidal fine particles, chemical additives can be used to agglomerate small particles and this acts as a pre-treatment process to centrifugation treatment (Tarleton and Wakeman 2007).

Viscosity of feed slurry: Process optimisation can be achieved by understanding the viscosity of feed waste. Viscosity of the slurry can be reduced by increasing the temperature, which in turn helps particle sedimentation (Wilson and Poole 2009).

4.2.2 Field Trials

Sureclean aimed to expand its service market to include large-scale treatment of oily sludge and water. Dewatering is one of the fundamental treatments to reduce moisture, thereby reducing onwards treatment cost and effort. However, purchasing a decanter centrifuge involved high capital cost; therefore, undertaking a field trial was an important step in determining the suitability of the selected decanter centrifuge towards the target waste. The efficiency of an axial horizontal decanter centrifuge was investigated with waste samples collected at the Sureclean WTS in Alness.

The decanter centrifuge was designed for physical separation of waste material in the oil and gas sector. The main aim of the field trials was to evaluate the dewatering performance of the decanter against different waste materials under different machine parameters without the aid of chemical agents. The field trials were conducted for three waste types: oil based drill cuttings, tank bottom sludge and sludge from a water treatment plant. Samples were collected before and during the treatment process at timed intervals for each trial.

4.2.2.a   Decanter Centrifuge and the Feed Pump

Centrifuges Un-limited supplied the decanter centrifuge reported in this study and the machine type used was the Centrifuges Un-limited Mechanical High Speed Refining (MHSR) 414 FTVB. The decanter was designed for high volume barite recovery and liquid removal via a two-stage separations process. The equipment was suitable to be used in European Standards of hazardous areas Zone 1 and Zone 2. Table 4-1 shows the basic specification of the decanter, a detailed specification can be found in Appendix 1. The decanter consists of a two-inch inlet, a decanter bowl, three sets of control panels, with two drive motors, a gearbox and a radiator. All parts that were in contact with the waste material were made from stainless steel. Two engineers from Centrifuges-Unlimited set up the decanter for optimised operations. The decanter was supported by a 1 m high stand, this was to allow the effluent and discharged solid to be collected from the bottom of the decanter centrifuge. Effluent was discharged on the left side of the decanter and the solid was discharged on the right side of the decanter.

Equipment ID	MHSR 414FTVB
Size (mm)	2400 x 2000 x 1520
Dry weight (kg)	2900
Maximum Bowl Speed (rpm)	4000
Maximum 'G' force (rpm)	3157
Discharge capacity (m³/h)	Maximum 8
Power Requirement:	
Main drive motor	380/460 volt, 3 phase 37 kW
Back drive motor	380/460 volt, 3 phase 13/16 kW
Feed pump motor	380/460 volt, 3 phase 7.5 kW
Electrical power output	54 KW

***Table 4-1 demonstrates the specification of the MHSR 414/FT
decanter centrifuge.***

The main aim of the field trials was to investigate the dewatering capability
of the decanter centrifuge under different operating parameters. The two
important operational parameters were the bowl speed and the differential
speed. The bowl speed was adjustable between 1000 to 3415 rpm by
adjusting the pulley/ hydraulic hand-wheel on the decanter. The initial two
turns of the hand-wheel may not increase the speed of the decanter
immediately, however, after the 2 initial turns; each turn represented an
increase in speed of 400 rpm, i.e. 4 turns in total is 800 rpm with the
maximum speed of 3200 rpm. The differential speed was controlled via the
back drive motor by adjusting this on the main control panel of the
machine. The MHSR 414FTVB had two directions (left and right) and two
speeds (called 1 and 2) with an electrically operated brake. In other words,
the motor gives in total, five different differential speeds at a given bowl
speed: 1 Right, 2 Right, Brake, 1 Left and 2 Left. Photographs of the
decanter centrifuge can be seen in Figure 4-6 and Figure 4-7.
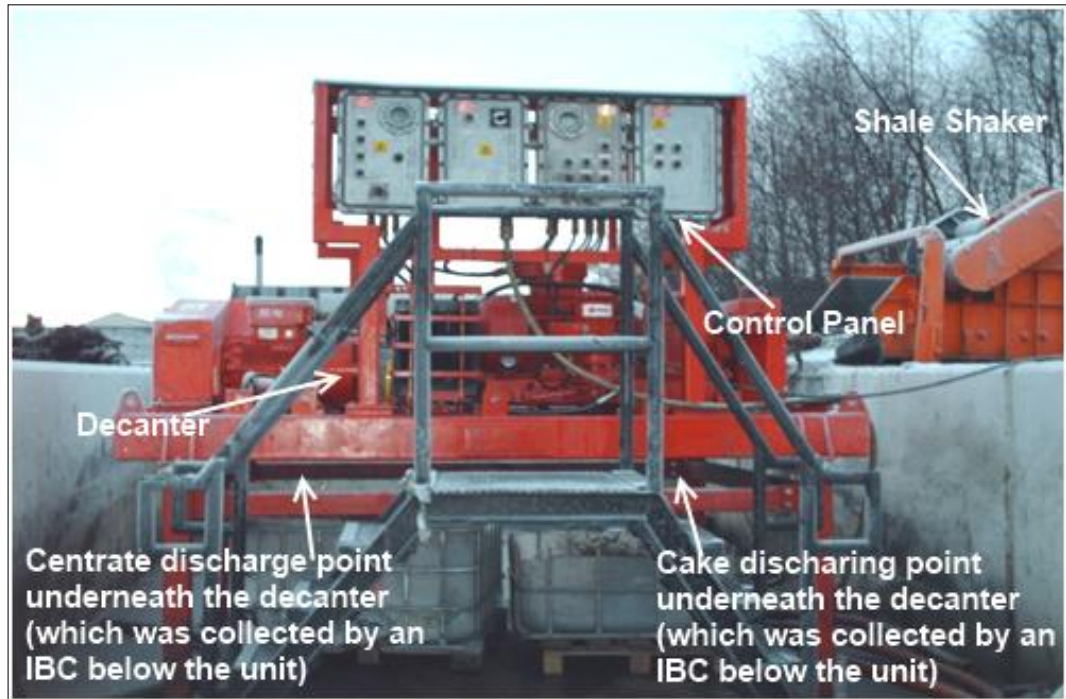
Figure 4-6 demonstrates the front view of the decanter centrifuge used in the field trials.

Figure 4-7 shows the back view of decanter centrifuge showing decanter bowl.

Centrifuges Un-limited also supplied the feed pump used in the field trials. The feed pump was a mono feed pump suitable to be used in European Standard hazardous area zone 2; it consisted of a hand-wheel for controlling pump speed, a four inches inlet, a two inches outlet that feeds into the decanter and a small sampling point (pointing upwards). Flow rate was estimated by the number of turns on the hand-wheel, each turn represented 30 gallons per minute. A water hose was attached to the small sampling point to allow cleaning of the decanter.

4.2.2.b   Waste and Analysis

Three waste types investigated in the field trials were obtained from Sureclean clients: shaker tank oily sludge, OBM and peaty sludge from a water treatment plant. The oily sludge was derived from an oil tank and oil interceptor cleaning operation; the sludge had been screened with Sureclean shale shaker (Brandt/EPI LM-3 Full Flo). The peat sludge was obtained from a water treatment plant in Dornoch, a town in the Scottish Highlands approximately 43 miles north of Inverness. The sludge moisture content was 90 % with more than 95 % of all particles less than 4 mm in size (waste analysis results can be found in Appendix 2). This table shown in Appendix 2 gives an indication of the range of parameters obtained from a typical peaty sludge. The OBM was received from a client that dealt with oil based fluids.

The aim of the field trials was to evaluate the dewatering capability of the decanter centrifuge. After decanter centrifugation, the waste was separated into two parts: solid (also known as the 'sludge cake') and liquid (also known as the 'centrate'). The retort test was used after the field trial for the solid part of the waste sample to analyse the oil, water and solid percentage. According to the guidance note produced by the Marine and Coastguard Agency (Maritime And Coastguard Agency 2004), the retort test had been recommended to test for solid, oil and liquid content within wet bulk waste such as drilling mud (water based and oil based). A retort kit is a form of distillation unit for quantitatively extracting oil and water from samples of drilling fluids or cuttings (Jones and Bagnall 1987). Jones and

Bagnall (1987 p. 243) stated "the oil and water retort provides a simple, direct field method for determining the percentage by volume of oil and water in samples in drilling mud or in core samples of the formation."
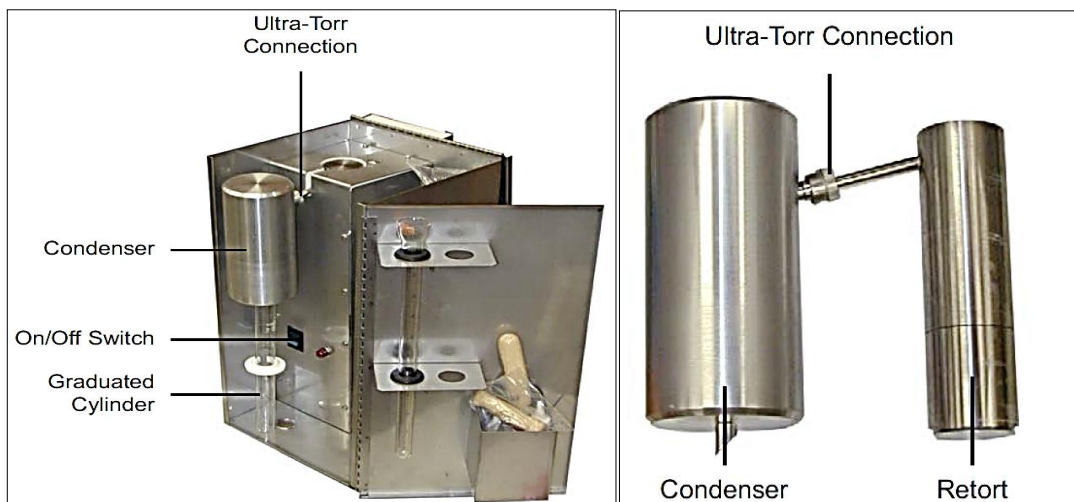
Figure 4-8 demonstrates a 50 mL Retort Kit (left) and a closer look at the ultra-torr connection (right) (Ofite 2011).

The waste sample was placed in a heavy-duty 50 mL steel retort (as shown in Figure 4-8) and was heated until the liquid fractions of the waste vaporised. The vapours were passed through a condenser collected and measured using a graduated cylinder (Lyons, Plisga and Gary 2005). The volume of liquids (water and oil) can be determined directly as volume percentage of the solids. The retort was heated to around 371°C for effective separation of oil and water from the solid (Schlumberger Limited 2011).

For the centrate of the waste sample, the spin-tube test was carried out after the field trials to determine the settle-ability of the suspended solids in the waste sample; as well as to evaluate the supernatant liquid and the solid concentration in the sample (Genck 2008). 50 mL of the waste sample was measured using a measuring cylinder, and the measured sample was transferred into a 50 mL plastic tube. Each waste sample was centrifuged for 5 minutes at the speed corresponding to the field trials, i.e. shaker tank

oily sludge was centrifuged at 3000 rpm; peaty sludge and OBM were centrifuged at 2000 rpm.

In all of the trials, the decanter's weir height used was 127 cm. The bowl speed, which was measured in rpm, was set as shown in Table 4-2. The maximum bowl speed was 3000 rpm. The setting for the scroll position, in other words, the differential speed of the screw conveyor was set to start from 'brake'. A change to the scroll position was made during the trial based on the solid output. Positioning the scroll to the next setting was marked as '1 Right' and increased the differential speed and the final setting '2 Right' which provided an even higher differential speed. According to the manufacturer manual, higher differential speed will give rise to better clarification and higher solid output (Centrifuges Un-limited 2007).

Waste Type	Trial no.	Bowl Speed (rpm)	Scroll Position (changes were based on output waste)
1. Shaker Tank Oily sludge	1	3000	1 Right
	2	3000	2 Right
2. Peaty Sludge	3	2000	1 Right
	4	2000	2 Right
3. Oil Based Mud (OBM)	5	2000	1 Right
	6	2000	2 Right

Table 4-2 Decanter Centrifuge field trials: table shows the operational settlings during field trials.

4.2.2.c   Results and Discussion

The centrifuge decanter that was used in the field trial was hired for 2 weeks to carry out all the trials. This utilised a constant feed rate to the decanter centrifuge of 30 gallons per minute (gpm) and the effect of change to the scroll position (which controls the differential speed of the centrifuge) was investigated using three different Sureclean waste types. The main aim

of the study was to evaluate the dewatering performance of the decanter centrifuge against the waste.

Precise total treatment time of the shaker tank oily sludge could not be noted because the pump was not running at maximum capacity at the start of the trial. Figure 4-9 shows the retort test results of the shaker tank oily sludge treated with three different decanter differential speeds; the graph demonstrates the percentage breakdown between liquid and solid fractions of the sludge. The higher the percentage of the liquid fractions reveals that the sludge was wetter, which indicated that the dewatering capacity was less effective against drier sludge. It can be seen that the dewatering capacity of the shaker tank oily sludge changed from 67 % in 'Brake' to 11 % for the highest scroll position ('2 Right'). By increasing the differential speed (scroll position from '1 Right' to '2 Right'), reduction in moisture content in the solids was noted. This may suggest that the optimum operational setting of the decanter centrifuge for the treatment of shaker tank oily sludge was the highest position (2 Right), as this produced the driest sludge.
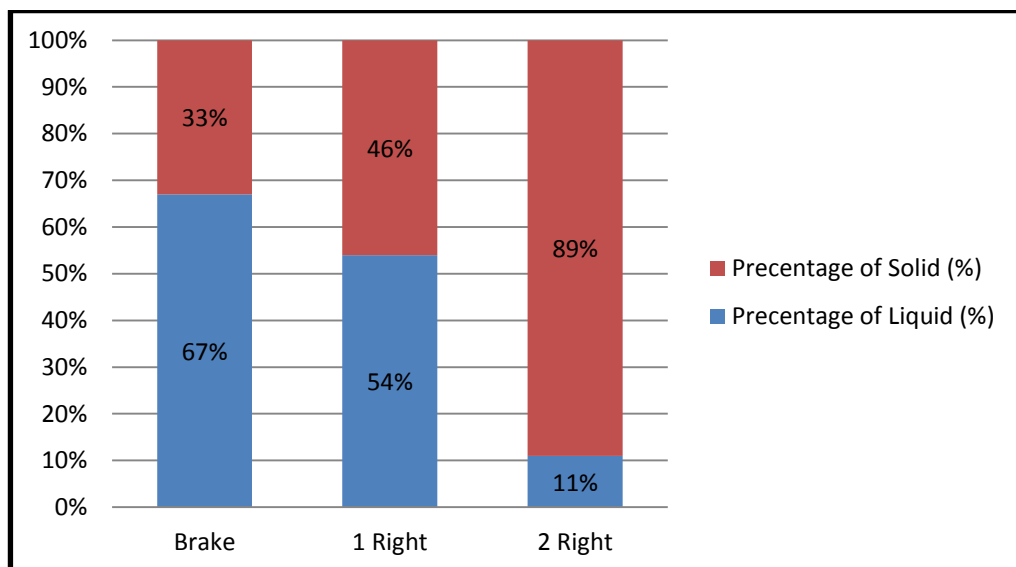
Figure 4-9 Decanter Centrifuge field trials: Retort test results of the shaker tank oily sludge showing the percentage breakdown between liquid and solid fractions of the sludge post-treatment.

A Spin test was also carried out on the separated liquid from the decanter trial, using a laboratory centrifuge. The waste was centrifuged at 3000 rpm. In trial 1, the solid percentage was recorded below the lowest indicated level on the centrifuge tube. However, in trial 2 and trial 3, higher solid levels were recorded in the centrate (i.e. liquid fractions from the separation) for both trials. This suggests that increasing the differential speed gave rise to a drier solid but a more turbid centrate. Also from Table 4-3, it can be seen that the percentage of oil recovered from the decanter decreased from 3 % in the 'Brake' to 1 % in the highest scroll position ('2 Right'). In contrast, the percentage of the liquid fractions decreased from 64 % to 10 % from Control to '2 Right'; this indicated that water removal increased as the differential speed increased.

Trial	Scroll Position	Feed rate (gpm)	Retort test for the cake to determine the liquid fraction (50 mL)	Spin test for centrate (50 mL)
1	Brake	30 gpm	64 % of water and 3 % of oil were yielded from 50 mL of sludge (A total of 67 %)	Solid level below lowest indicating line
2	1 Right	30 gpm	52 % of water and 2 % of oil were yielded from 50 mL of sludge (A total of 54 %)	20 mL solid
3	2 Right	30 gpm	10 % of water and 1 % of oil were yielded from 50 mL of sludge (A total of 11 %)	20 mL solid

Table 4-3 Decanter Centrifuge field trials: Retort and spin test results of the shaker tank oily sludge treated with three different decanter differential speeds.

A second field trial was carried out with peaty sludge as the waste stream. Unfortunately, no separation was achieved using any of the differential speeds of the decanter centrifuge. Peaty sludge is a very different waste type to the oily tank sludge. The most probable reason for achieving no separation was that the decanter centrifuge was not powerful enough to separate out the very fine particles of the peaty sludge. Leung (2007) also stated if the finer solids had a density close to the liquid they would not settle despite increased centrifugal force. This is exactly what was observed with the peaty sludge. Also as suggested in section 4.2.1.a, Wakeman (2005 p.383) explains that this is due to "adhesion to the contaminants or bubbles to the solid surfaces." Chemical treatment could be used to improve settling of the solids.

The third Sureclean waste stream investigated was OBM. Using the slowest centrifuge position, 8 % of the water was separated from the mud but very little oil was measured before the freezing conditions stopped the centrifuge working. The test only lasted 30 minutes rather than the 3 hours. Therefore, a second attempt was made, however, the weather conditions deteriorated and after 10 minutes, the centrifuge failed. Therefore due to freezing weather conditions, no conclusive results were available for the treatment of OBM.

4.2.2.d   Performance Recommendation

The freezing weather conditions were the main challenge for optimising the process during the trial; the equipment had to be stored and operated taking into account the freezing conditions which occurred during the field trials that included frozen pipework and ice-formation on the waste. Additionally, agitation of the feed waste was a crucial step to maintain viscosity of the feed waste. Several difficulties with the feed pump were encountered, as it was required to have flooded suction for a more efficient pumping action. Hence, the feed tank had to be situated higher than the pump.

Full understanding of waste characteristics, set up of the decanter and decanter parameters for process optimisation were crucial for successful operation. To improve the overall performance of the decanter centrifuge, the feed tank must have a four inch gravity outlet to connect it to the decanter feed pump. Therefore, waste such as OBM that arrived in skips can be tipped into the feed tank. The waste can be agitated to give rise to a more homogeneous feed prior to the decanter. Hoses and pipes may be frozen in winter time; therefore, at the end of the operation of the decanter, all hoses and the feed pump were cleaned and drained to prevent water freezing inside them. To enhance the decanter lifetime, a screen or a shaker was recommended to be installed prior to the decanter. The screen or shaker could remove any solids such as metal bolts that may potentially be invasive to the decanter. A recommended set up of decanter treatment is shown in Figure 4-10.
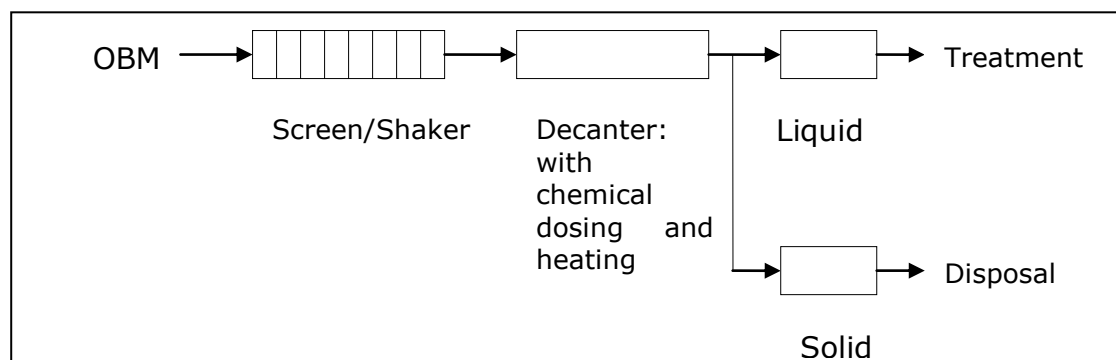
Figure 4-10 demonstrates the recommended set up for the treatment of OBM.

A chemical dosing system and heating system can be incorporated into the treatment system. As mentioned in 4.2.1.b, chemical treatment can lead to solid agglomeration and this enhances separation efficiency. According to the Water Environmental Federation (2008a), one effective way to separate solids and oils in oily sludge is by first heating the oily sludge in a tank between 82 and 93 °C, this should then be followed by decanter centrifugation treatment to separate the oil, water, and dewatered solids. Separated liquid can then be treated with the Sureclean treatment process available in the Alness waste transfer station.

4.2.2.e   Cost-benefit Analysis

At the time of the trials, the current waste treatment at Sureclean for oily sludge was processed by the shaker to remove larger solids or grits. The remaining sludge with high moisture was left in tanks to settle for a period. Subsequently, oil was skimmed off from the top layer; water was siphoned and treated via the interceptor. The remaining solids/sludge as well as untreated OBM and drill cuttings were both sent to third party waste companies for further treatment and disposal. The cost of disposal for both OBM and oily sludge differ year by year, this was because Sureclean did not use the same treatment company every year. As shown in Table 4-4 and Figure 4-11, the average annual OBM and oily sludge disposal cost were £8,210.92 and £3,566.55 respectively, which total to £11,777.47 (based on oily sludge and OBM disposal in 2007-2009). Note the economic recession has affected the amount of waste disposal being sent to Sureclean for clean-up.

Year	2007		2008		2009		2010 (projection)/ Average of 07-09	
	Oily sludge	OBM	Oily sludge	OBM	Oily sludge	OBM	Oily sludge	OBM
Total Quantity (tonne)	44.16	0	124	57.04	40	12	69.39	23.01
Cost per unit	£185	0	£100	£150	£70	£160	£118.33	£155.00
Total cost	£8,169.60	0	£12,400	£8,556	£2,800	£1,920	£8,210.92	£3,566.55

Table 4-4 2007-2010 annual oily sludge and OBM disposal; 2010 figure was based on projection
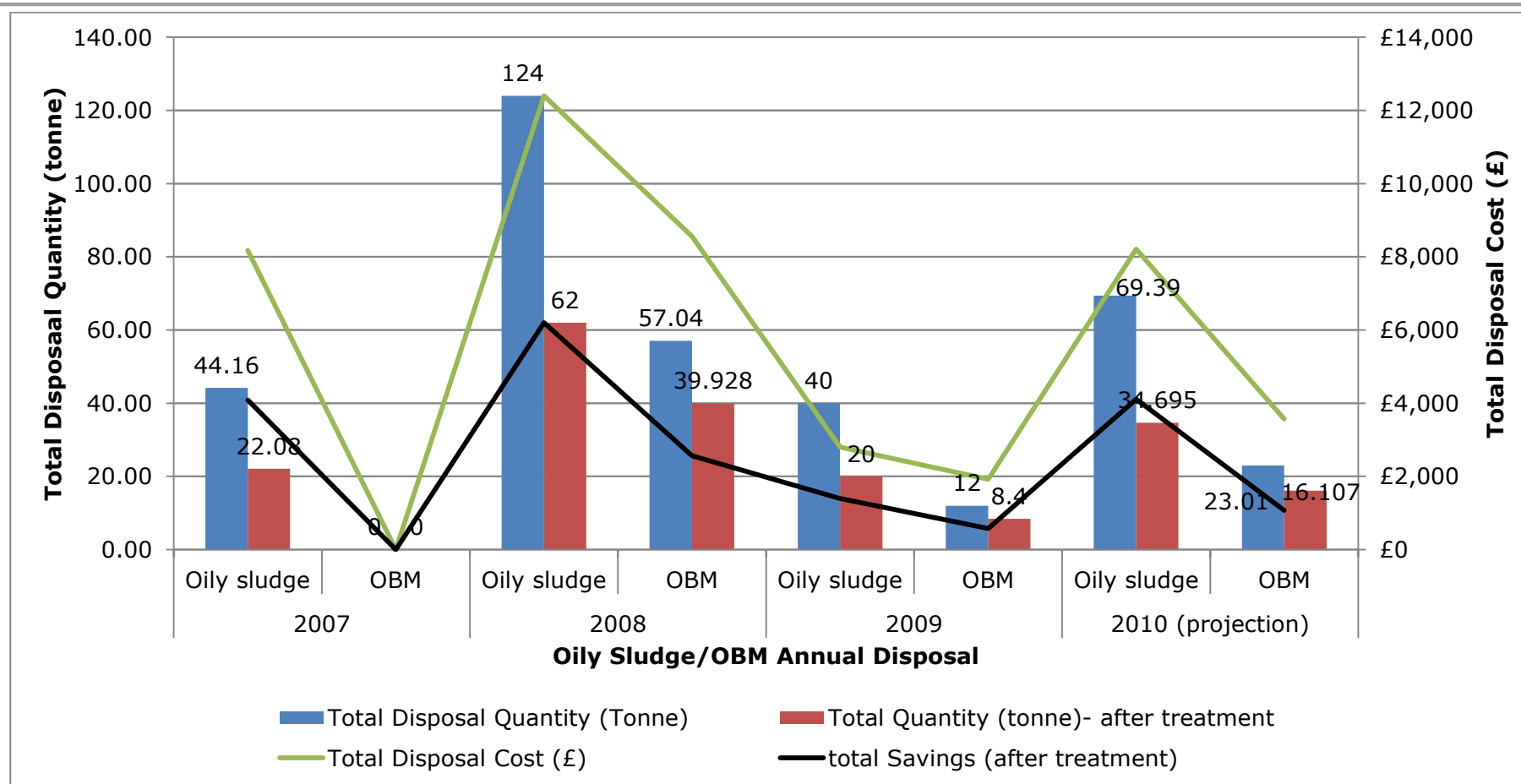
Figure 4-11 2007-2010 Annual Oily Sludge and OBM Off-Site Disposal

Purchase quotations for a decanter centrifuge with a specification similar to the unit used in the field trials were obtained from three different suppliers (as shown in Table 4-5).

Company/Unit	Unit Description	Cost (all cost based on quote received in 2008)
GEA Bespoke Unit	2 gear drive system, variable speed drive and ATEX zone 2 motor, up to 3000 rpm	Approximately £165,500
Alfalaval Lynx 20-200 or Lynx 20-700	2 gear drive system, variable speed drive, ATEX zone 1, up to 3,250 rpm	Approximately £160,000
Centriquip CQ3000	Variable speed drive, Up to 2,940 rpm drive motor ,flow rate at 0.5 and 15 m^3/h	Approximately £160,000
Average estimated cost:		**£161,800**

Table 4-5 indicates a summary of three decanter specifications and costs from different suppliers.

Based on the field trials, by using a decanter centrifuge, a potential 56 % moisture reduction in oily sludge could be achieved, which equates to a potential annual saving of £4,064.64 (as shown in Table 4-6). However, the savings obtained does not pay off the annual capital and maintenance cost, which is a sum of £22,504.91 per year (Table 4-7). To obtain a return from the capital cost, a net profit of 231 tonnes or more of oily sludge/oil based mud (at £80 per tonne) would be required (Table 4-8).

	Total tonnage	Cost per tonne	Total cost
Average Sureclean oily sludge disposal (tonne), taken from Table 4-1.	69.35	£118.33	£8,206.19
Disposal of oily sludge less 56 % moisture	35	£118.33	£4,141.55
		Total savings:	£4,064.64

Table 4-6 Potential cost savings by using a decanter in Sureclean WTS.

Capital cost of a decanter of £161,800 + 20 %)[1]	£194,160.00
Discount Rate	10%
Depreciation Rate for Decanter (years)	10
Annual capital cost (cost of decanter divided by 10 years)	-£17,650.91
Annual maintenance cost @ 2.5 % of capital cost[2]	-£ 4,854.00
Total	-£22,504.91
Annual savings (from Table 4-6)	£ 4,064.64
	-£18,440.27

The cost was based on quotation received in 2008; an allowance of 20% increment was added for purchase in 2010.

[2] This does not include labour and electricity cost

Table 4-7 demonstrates the cost benefit analysis of a decanter purchase.

Assumed that net profit for treating oil based mud/oily sludges is £80 per tonne:		
To get a net return from the purchase of the decanter, total annual tonnage of oily waste required (£18,440.21 divided by £80.00):	**231**	tonnes
Monthly tonnage	19	tonnes

Table 4-8 Calculation of the total oily sludges require to be treated in order to cover the cost of a decanter centrifuge.

4.2.2.f   Conclusion

It was a great challenge to conduct the decanter centrifuge field trials due to sub-zero conditions. Preparation work for the field trials such as setting up of the equipment, defrosting frozen pipe works and breaking surface ice on the OBM proved to be time consuming and challenging. This highlighted the technical difficulties that would be encountered by Sureclean if they purchase a decanter centrifuge. Sureclean could set up a facility to enhance the decanter centrifuge performance, but that would require even more capital investment on top of the cost of a decanter centrifuge. During the current recession, it is not economically viable for Sureclean to purchase a decanter centrifuge for oily sludge and OBM.

4.3 Filtration

The separation of solids from a suspension in a liquid by using a porous medium is termed filtration (Coulson, Harker, and Richardson 2003). Filtration has been used after clarification in which larger solids are removed prior to filtration. Chemical conditioning of the waste stream can be employed as a pre-treatment to improve solid-liquid separation. Chemical conditioning techniques such as coagulation and flocculation have been regarded as a filtration aid to increase suspended solids and turbidity removal in water and wastewater treatment (Svarovsky 2000). The use of

chemicals for suspended particle coagulation and flocculation shall be covered in Chapter 5 and Chapter 6 respectively.

There are several mechanisms that can be described for water filtration i.e. straining, sedimentation and adsorption (Alley 2007). Straining is thought to be the primary mechanism in filtration; however in granular filters, each void between the media is considered a sedimentation basin and the filter media could have some adsorption properties to remove contaminants (Alley 2007). According to Coulson, Harker, and Richardson (2003), some of the main factors affecting filtration selection include the properties of the liquid including its viscosity, density and corrosive properties; the particle sizes of the solids; and the quantity of the liquid to be handled.

According to Green and Perry (2008), filtration systems can be classified according to the driving force, which is created by a pressure difference across the filter medium to force the wastewater through the filter media. According to Sutherland (2007) this can be achieved either by means of fluid pressure upstream of the medium (pressure filter) or by means of suction downstream (vacuum filter). Pressure filter, in an enclosed steel tank, which can be either horizontal or vertical, is best applied in swimming pool water filtration, as well as for iron and manganese removal in industrial wastewater (Agardy, Nemerow and Salvato 2003). Cartridge filter is a type of pressure filter with a cylindrical housing, usually operated under pressure or sometimes under vacuum conditions, in which a cartridge, also known as replaceable filter element, sits (Sutherland 2007). The cartridge can be made with a vast diversity of material such as paper, woven fabrics, thin felts, plastics and woven wire mesh (Sutherland 2007). The cartridge must be replaced once it is plugged with solids from the wastewater (Frankel 2010).

Filtration can also be achieved by gravitational force to create the pressure difference across the filter media (Sincero and Sincero 2003); slow sand filtration is one example of this type of filtration. Slow sand filtration, a type

of depth filtration removes particles in water by percolation at slow speed; this type of filtration is often employed in drinking water treatment to remove turbidity (Cheremisinoff 1998). These filters are mainly made from granular and crushed media such as sand, garnet, ilmenite, alumina, anthracite and quartz (Purchas and Sutherland 2002).

Filter media can be made of various materials including polypropylene, fibre glass or polyester and enable particle removal ranging from 0.5 to 50 μm (Frankel 2010). Other examples of filter media include inorganic materials, carbon or charcoal, glass, metals, metal oxides or ceramics, natural organic fibres, synthetic organic fibres and synthetic sheet materials (Purchas and Sutherland 2002). These filter media have different pore sizes; the smaller the pore size, the finer and more solids it retains; however, these can lead to faster choking of the filter (Purchas and Sutherland 2002).

4.3.1   Laboratory Trials

A granular media filter was investigated in the laboratory using Sureclean interceptor wastewater samples. A student, Hezekiah Ayodeji Adesina, under my direction, conducted the laboratory-based trials in 2009. The filter media used in the trial was a commercially available media called Activated Filter Media (AFM). The media was made from mainly recycled brown and green-graded waste glass. The manufacturer (Dryden Aqua Limited) claimed that the media has a higher zeta potential as compared to sand, the current recommended and more commonly used granular filter media (Cheremisinoff 1998). This enables the media to have higher potential to interact with the organic particles in the water, which should thereby improve the retention of the organic particles (pollutant) onto the filter bed (Dryden Aqua 2007). The justification of choosing this filter media for the trial was based on the sustainability of the filter media as well as its performance demonstrated by the manufacturer in other wastewater types such as sewage and swimming pool water (Dryden Aqua 2011). The media used in the trials was grade 1 AFM media with an average media size of 0.6 mm. The effluent that was used for the trials was obtained from the last

stage interceptor of the Sureclean WTS. The feed wastewaters used in the trials were obtained from Sureclean Alness WTS Interceptor 6.

4.3.1.a   Methodology

The laboratory trial was set up by placing a known weight (100 g) of filter media into a separating funnel (as shown in Figure 4-12). The bottom of the funnel was plugged with cotton wool to hold the filter media; a layer of pea gravel was placed on top of the filter media to prevent disturbance of the media layers.   Three samples of 100 mL of the feed wastewater were passed through the filter media under gravity. The effluent samples were analysed before and after the filtration with the following parameters: TOC, particle size and heavy metals.
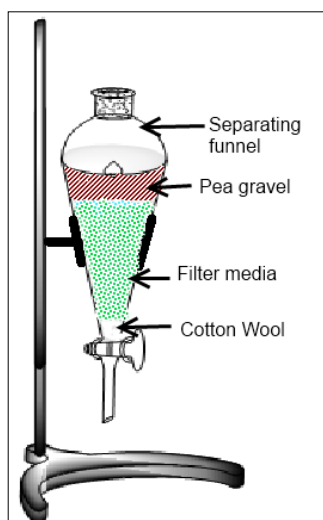
Figure 4-12 Schematic representation of the filtration laboratory trials using AFM filter media

TOC Analysis

TOC is a measurement of organically bound carbon by oxidising the organic compounds to carbon dioxide (Eaton and Franson 2005). TOC was measured as it has been regarded as a more direct measurement of the total organic in the wastewater as compared to BOD and COD; however, TOC measurements cannot replace COD or BOD (Eaton and Franson 2005; Alley 2007), although the results can be correlated to those of BOD and

COD (Stander and Theodore 2008). Effluent samples before and after treatment were analysed for TOC using a Shimadzu TOC VCPH analyser. 25 mL of each sample was measured, placed into cleaned vials and analysed in triplicate to improve accuracy of the results.

Particle Size Analysis

The analysis of suspended solids was carried out by particle size determination. The Particle size of the effluent samples was measured using a Malvern Mastersizer E. The cell of the analyser was filled with distilled water and stirred continuously. This was done to prevent formation of bubbles within the cell. Three drops of effluent sample were then added into the cell. The instrument was set up and left to stabilise for 30 minutes before analysis was carried out.

Heavy Metals Analysis

Heavy metals were analysed using the ICPAES (Perkin Elmer Optimer 3300DV ICP-AES). A stock solution was prepared by diluting the 1000 ppm multi-element standard for Al, Cu, Ti and Zn with deionised water in a 50 mL volumetric flask. Standard solutions of 0.05, 0.1, 0.5, 1.0 and 5.0 ppm of each metal were then prepared using the stock solution in a 50 mL volumetric flask diluted by deionised water. These standard solutions were used to generate the calibration graphs in order to determine the concentrations of the unknown effluent samples. Effluent samples before and after filtration treatment were digested using 20 mL of 1.1 M HCl in a steam bath for 4 hours. The digested samples were filtered 541-hardened Ashless Circle 110 mm diameter filter paper. 30 mL of the prepared effluent samples were placed in 50 mL sample vials. The chosen analysis wavelengths for detection in nm of metals were Al- 396.153, Cu– 324.752, Ti- 336.121 and Zn– 206.2. Triplicate samples were analysed. Data was recorded given in mg/L.

4.3.1.b   Results and Discussion

TOC analysis

The TOC concentration before treatment was 3448.60 ± 89.66 mg/L and the TOC concentration after treatment with AFM was 2281.93 ± 70.79 mg/L as shown in Figure 4-13. The lab-based filtration treatment of Sureclean interceptor wastewater was calculated to have reduced TOC by an average of 33.9 %. The RSD for the concentration of TOC before and after treatment with AFM were 2.8 % and 3.1 % respectively, which indicated the instrumental results have small errors and the results were reproducible.
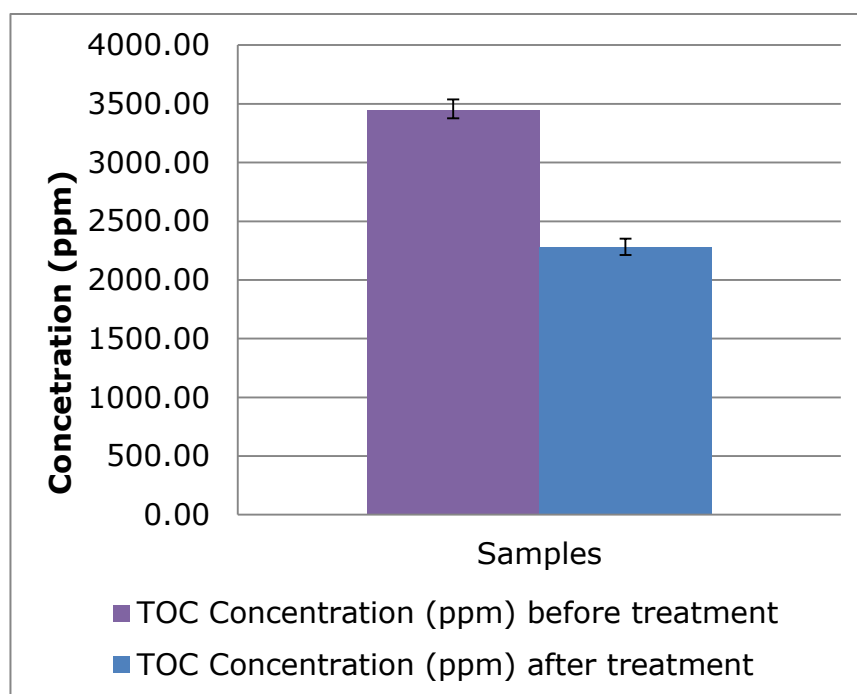
Figure 4-13: Filtration Laboratory trial: TOC results of the Interceptor 6 before and after treatment using the AFM.

Since TOC measured all organically bound carbon, the reduction of TOC could indicate that these organic particles were retained on the filter media. The filter media manufacturer claimed that the media has a high negative zeta potential that draws the particles and holds them within the filter bed (Dryden 2007). The manufacturer also claimed that the filter media has catalytic activity in the presence of oxygen that increases the oxidation potential thus degrading organic compounds (Dryden 2007).

TOC was used to measure the organic compounds in wastewater, some of these compounds can be oxidised further by chemical or biological treatment (Eaton and Franson 2005). To improve TOC removal efficiencies in filtration systems, the American Water Works Association (2011) suggested that chemical coagulants could react with dissolved natural organic material (NOM) to form a solid phase that can be further removed by filtration or clarification. AOP such as photocatalysis can also improve TOC removal, further studies on the latter can be found in Chapter 7 Physio-chemical Treatment.

Particle size analysis

Particle size of the effluent was measured, as it is one of the crucial factors controlling the filtration rate of the feed (waste) (Wakeman 2005). Figure 4-14 demonstrates the concentration distribution of varying particle sizes for both pre- and post-treated wastewater. An overall reduction of concentration for all particles size less than 600 µm can be seen after the treatment using AFM. This suggested that the filter was effective in reducing suspended solids.
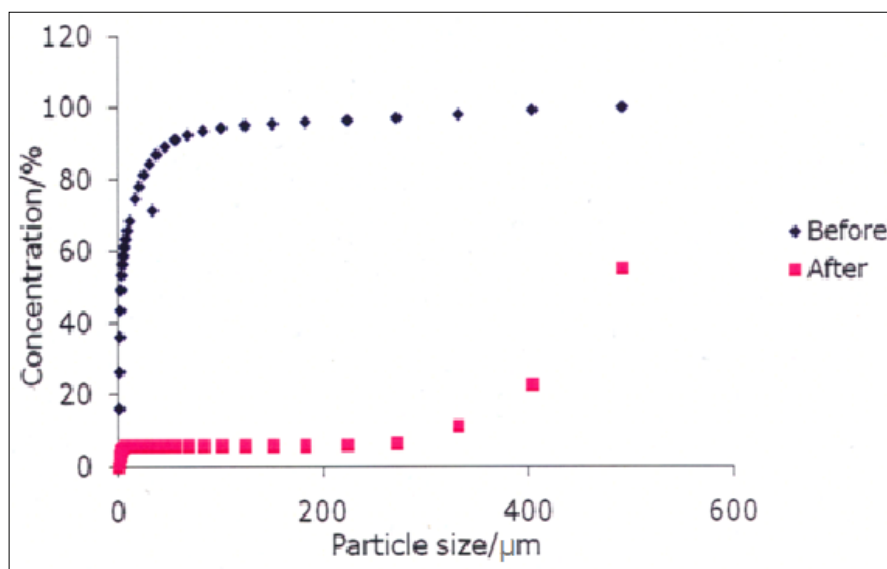
Figure 4-14 displays the Filtration Laboratory trial results: concentration distribution of varying particle sizes for both pre- and post-treated wastewater using the AFM.

Heavy Metals Analysis

Zn, Ti, Cu and Al were analysed before and after treatment using AFM with the average of triplicate samples reported in Table 4-9. Only Cu was listed in Sureclean discharge consent; therefore, the results could only provide an indication of the filtration efficiency towards heavy metal in Sureclean interceptor effluent. The results showed that AFM reduced the heavy metals tested. The reduction of these metals can be due to the zeta potential on the surface of the filter media. According to Dryden (2007), a high charge density was generated on the surface of the filter media that attracted positively charged particles such as heavy metals. Dryden (2007) also claimed that the particles (heavy metals) would not bind permanently onto the surface of the media, which would allow them to be washed off by backwashing the media after several usages. It can be seen from Table 4-9 that Ti had the highest removal efficiency in comparison to other metals that were investigated in this study.

Heavy metal	Percentage Reductions	RSD
Zn	21.3 %	0.29 %
Ti	68.2 %	4.06 %
Cu	31.6 %	14.69 %
Al	42.3 %	1.1 %

Table 4-9 Filtration laboratory trial heavy metals results: Percentage Reductions of Zn, Ti, Cu and Al of the Interceptor 6 before and after treatment using the AFM.

	Zn	Al
Before Treatment (mg/L)	1.318	0.496
After Treatment (mg/L)	1.037	0.286
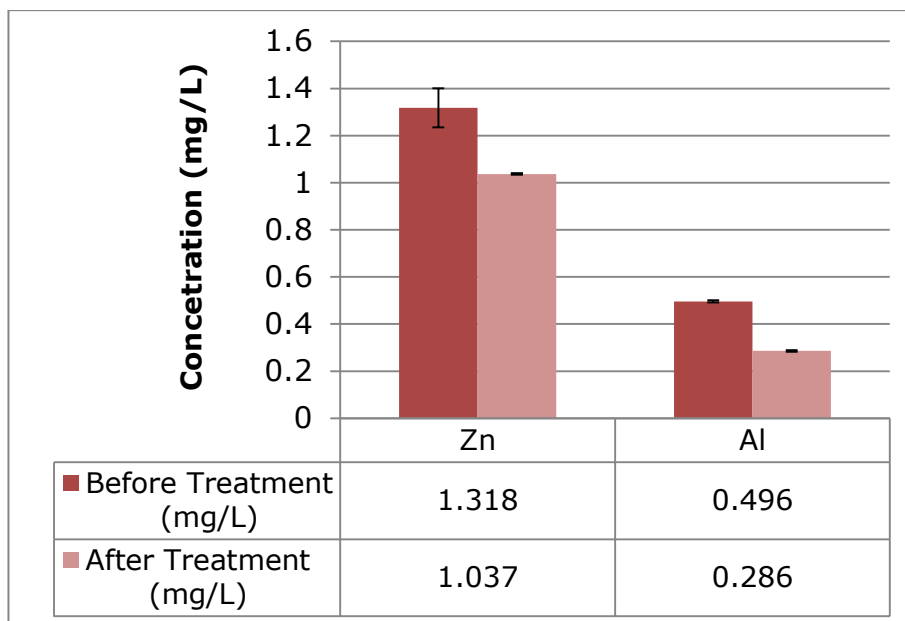
Figure 4-15 Filtration Laboratory trial results: analysis of heavy metals (Al and Zn) for both pre- and post-treated wastewater using the AFM.

	Ti	Cu
Before Treatment (mg/L)	0.022	0.038
After Treatment (mg/L)	0.007	0.026
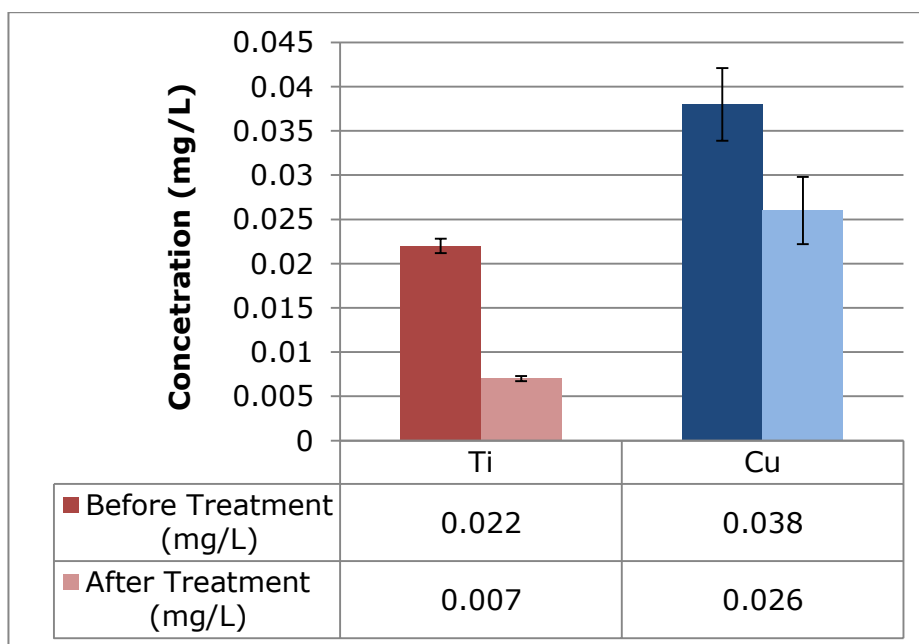
Figure 4-16 Filtration Laboratory trial results: analysis of heavy metals (Ti and Cu) for both pre- and post-treated wastewater using the AFM.

4.3.1.c   Conclusion

The results of the laboratory filtration trials showed a decrease in TOC by approximately 33.9 %, of all particles had a size less than 600 µm and there was a reduction of Al, Cu, Ti and Zn concentrations. The preliminary results suggested that the filter media, AFM may be suitable for Sureclean interceptor wastewater for the removal of TSS. Field trials using the filter media on interceptor wastewater may further prove the effectiveness of the treatment system.

4.3.2   Field Trials

Extending from the laboratory trials, two different types of prototype filtration systems were investigated: granular filtration and a cartridge filter. The granular filter consisted of a cylindrical tank filled with AFM, a type of recycled glass media, is also known as Glass Bead Filter (GBF); it was composed of layers of recycled glass with gravel as a supporting media. Effluent from Sureclean interceptors was transferred to the filter by gravity feed. The cartridge filter was a type of pressure filter where the filter media was deposited on rings mounted on a fluted rod. Application of these types of filters is usually used for trapping finer suspended solids (Frankel 2010). A cartridge filter was reported to be compact and relatively easy to operate (Tarleton and Wakeman 2007). The trial aimed to investigate the performance of these two filters in relation to the treatment capability to make recommendation for further development.

4.3.2.a   Material and Methodology

The GBF was designed by Sureclean, by using a bespoke stainless steel cylinder tank, as shown in Figure 4-17 (dimension as per Table 4-10). The tank was filled with grade 1 AFM (same material used in the laboratory trials in section 4.3.1) supplied by Dryden Aqua.

Absolute height (cm)	147
Height of cylinder tank (cm)	96
Diameter of cylinder tank (cm)	178

Table 4-10 Dimensions of the glass media filter cylinder tank.

Figure 4-17 Shows a photograph of the Glass bead filter (GBF).

Sureclean acquired the cartridge filter (CF) from a purchased asset; the filter was bespoke and built for the previous owner (as shown in Figure 4-18). Table 4-11 demonstrates the specification of the unit. The filter unit consisted of two air-operated diaphragm pumps with a two-inch camlock hose connection; as well as two stainless steel housings. This is also a type of pressure filter where the filter media was deposited on rings mounted on a fluted rod. Each of the steel housing could contain up to four 1 meter long cartridge filters of different materials depending on the treatment requirement. The type of cartridge filter used for this trial had a pore size of 20 µm and was made of spun-wound polypropylene.

Dimensions	Height (cm)	170
	Length (cm)	95
	Width (cm)	177
Tare Weight (kg)		800
Gross Weight (kg)		800
Water Inlet and Outlet		2" camlock
Maximum operating pressure		5 bar

Table 4-11 Specification of Sureclean CF

Figure 4-18: A photograph of the Sureclean cartridge filter (CF).

4.3.2.b   Waste and Analysis

The waste streams investigated in this field trial were obtained from the Sureclean WTS in Alness. Waste stream 1 was wastewater collected from Interceptor 5, this refers to effluent that was mainly contaminated with hydrocarbon and suspended solids. The effluent was pre-treated with the clarifier and the oil skimmer to remove heavy solids and surface oil. Waste stream 2 was collected from Interceptor 6, a stage after Interceptor 5. Due to longer clarification and oil removal, effluent from Interceptor 6 had a

lower solid and oil content than that of interceptor 5. The trial assemblies can be seen in Table 4-12.

Trial	Trial Assembly
Trial 1	Interceptor 5→glass bead filter
Trial 2	Interceptor 6→glass bead filter
Trial 3	Trial 2 liquid→cartilage filter

Table 4-12 Filtration Field Trials: trials set up.

The setup of Trial 1 and Trial 2 can be seen in Figure 4-20 and Figure 4-19; wastewater was collected directly from the waste transfer station interceptors to an IBC. The waste was fed to the filter via gravity. Effluent samples were collected before and after the treatment to be analysed.
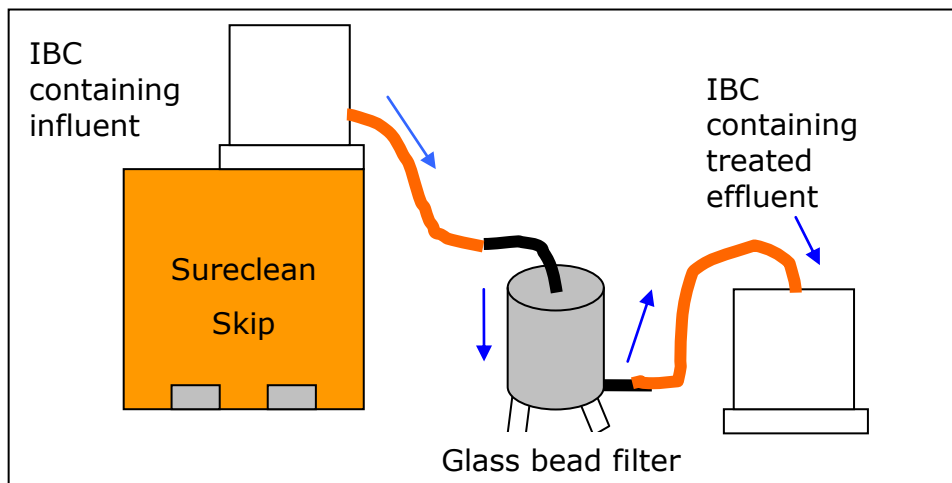
Figure 4-19 Filtration Field Trials: a schematic representation of the trial set up.

Figure 4-20 Filtration Field Trials: trial set up of the GBF in the Alness WTS.

The wastewater was analysed with the following analysis: TSS, COD and BOD. Due to time and labour constrains within the company, only one set of data was obtained. The effluent samples were sent to an independent UKAS accredited laboratory, AlControl Limited for analysis. The following methodology was obtained from AlControl Limited method statements. AlControl stated that all analysis below conforms to ISO 17025 - Competence of Testing and Calibration Laboratories.

TSS Measurements

The TSS was gravimetrically measured using the AlControl in house method reference TM 022 that was based on the British Standard (BS) 2690: Part 120:1981. A known volume of homogenised wastewater sample was filtered through a pre-washed and weighed GFC Grade Glass Fibre Filter (with a pore size of 1.2 μm). The filter paper was dried in an oven at 105 °C for two hours and was re-weighed on a 5-figure balance. The total suspended solids content of the sample was calculated from the difference in the two weights

following Equation 4-2. A blank and an analytical quality control were also performed with the batch. The limit of detection was 2 mg/L for every 200 mL of sample and the range of application was between 2 – 2000 mg/L.

$$\text{TSS} = \frac{(\textbf{weight of sample + paper}) - (\textbf{weight of paper}) \times 10^6}{\textbf{Volume of wastewater sample}}$$

Equation 4-2

COD Analysis

The chemical oxygen demand in water samples was determined by using sulphuric acid and potassium dichromate in the presence of a silver sulphate catalyst to oxidize the samples. The COD of the wastewater samples was measured using AlControl in house method reference TM 107 using a Hach Lange Xion 500 Spectrophotometers (range between 340 -900 nm). The Hach Lange COD test follows the ISO 15705 Determination of the chemical oxygen demand index (ST-COD) – Small-scale sealed tube method. To perform the test, 2.0 mL of wastewater sample was pipetted into a Hach Lange COD cuvette containing sulphuric acid and potassium dichromate in the presence of a silver sulphate catalyst (method reference LCK 514) and the cuvette was capped. The closed cuvette was inverted to mix the inside contents. The cuvette was heated in a heating block for two hours at 148 °C. At the end of this period, the sample was cooled and the change in colour intensity of the solutions can be measured with the factory programmed spectrophotometer. Sediment had been settled before evaluation was carried out. The detection range was between 7 – 1000 mg/L and the limit of detection was 7 mg/L.

BOD Analysis

The BOD was measured using the AlControl in house method reference TM 045 based on MEWAM BOD5 2nd Edition 1988/AWWA/ APHA. The sample was first neutralised and an aliquot of the sample was mixed with aerated deionised water and was seeded with bacteria. Allyl thiourea (ATU) was also added to suppress nitrification during the course of the test. The test was performed in triplicate using varying sample aliquots to cover as large a range of the result as possible. The samples were well shaken before

analysis. The dissolved oxygen concentration was measured (DO_0). The samples were incubated at 20 °C for five days after which the dissolved oxygen was measured again (also known as DO_5). The difference in the dissolved oxygen content from Day 0 to Day 5 was used to calculate the amount of oxygen used by the bacteria as they digested the sample and it is expressed as BOD in mg/L O_2. The limit of detection was 1 mg/L.

Dissolved Heavy Metals Analysis

Dissolved heavy metals were analysed with the Thermo X series Inductive-Coupled Plasma Mass Spectrometry. The method used was Alcontrol In-house method reference 080W. All samples were conducted neat so that low levels of detection could be achieved. 10 mL of the wastewater sample before and after treatment were filtered through a 0.459 m filter cartridge and were placed into a 12 mL polypropylene test tube. The samples were then acidified with 0.2 mL high purity nitric acid. The samples were loaded into an autosampler, and the instrument was set up to run the samples. The range of application or the working range of each metal can be found in Table 4-13. The limits of detection for all metals were 0.005 mg/L except Co which was at 0.001 mg/L.

Metals	Range of application (μg/L)	Limit of detection (mg/L)
As	0 - 1000	0.005
Co	0 - 1000	0.001
Cr	0 - 1000	0.005
Cu	0 - 1000	0.005
Ni	0 - 1000	0.005
Mo	0 - 1000	0.005
Pb	0 - 1000	0.005

Table 4-13 Filtration Field Trials: Range of applications and limit of detection for Dissolved As, Co, Cr, Cu, Ni, Mo and Pb in SWTS field trials.

4.3.2.c Results and Discussions

<u>BOD,COD and TSS results</u>

In trial 1, the wastewater was collected from Sureclean Alness WTS Interceptor 5. From Table 4-14, it can be seen that the TSS, BOD and the COD levels were decreased by 76.6 %, 78.5 % and 63.8 % respectively after treatment with GBF. Figure 4-21 shows that there was an overall reduction of all the parameters measured, i.e. BOD, COD and TSS. It took 20 minutes for a 1000 litre batch of effluent to pass through the filter, flow rate of 40 L/min (litres/minute). Based on this preliminary trial data, the treatment of wastewater with the GBF has reduced BOD, COD and TSS of interceptor 5 wastewater. In Trial 2, Interceptor 6 effluent was treated with GBF with a flow rate of 4.88 L/min and the duration was 205 minutes. From Figure 4-22, it can be seen that the treatment of wastewater with GBF improved water quality of interceptor effluent in trial 2.

Parameters	Reduction after treatment with GBF (%)
BOD	78.5%
COD	63.8%
TSS	76.6%

Table 4-14 Filtration Field Trial results: Trial 1, BOD, COD and TSS results of Interceptor 5 before and after treatment using the GBF.

	BOD (5 day)	COD	TSS
■ Before treatment (mg/L)	70.1	334	154
■ After treatment using GBF (mg/L)	15.1	121	36
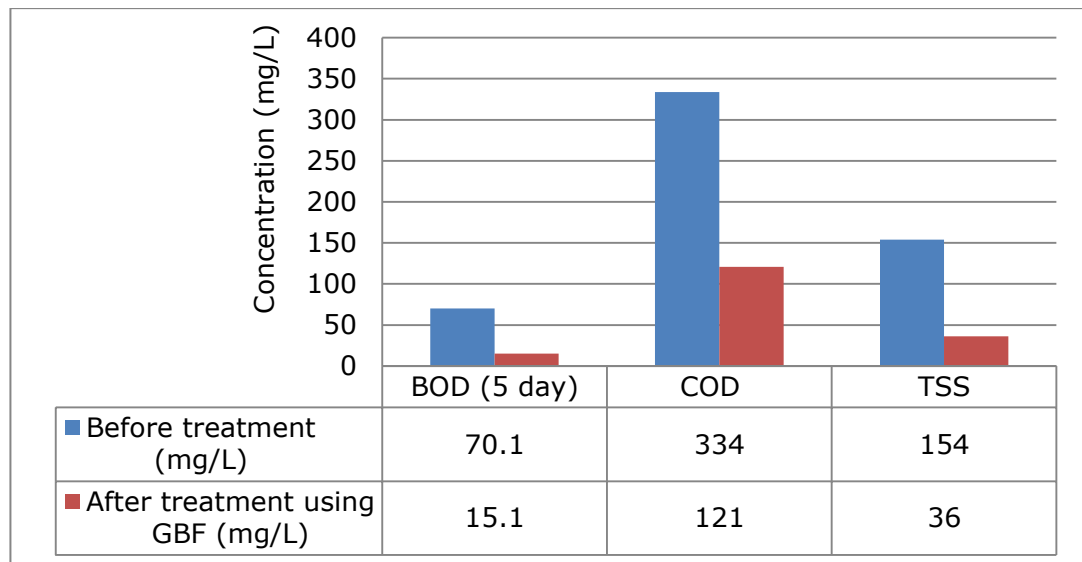
Figure 4-21 Filtration Field Trial results: Trial 1, analysis results of Interceptor 5 before and after treatment using the GBF.

	BOD (5 day)	COD	TSS
■ Interceptor 6 before treatment (mg/L)	20.7	113	73
■ Treated effluent (GBF) (mg/L)	37.6	80.6	43
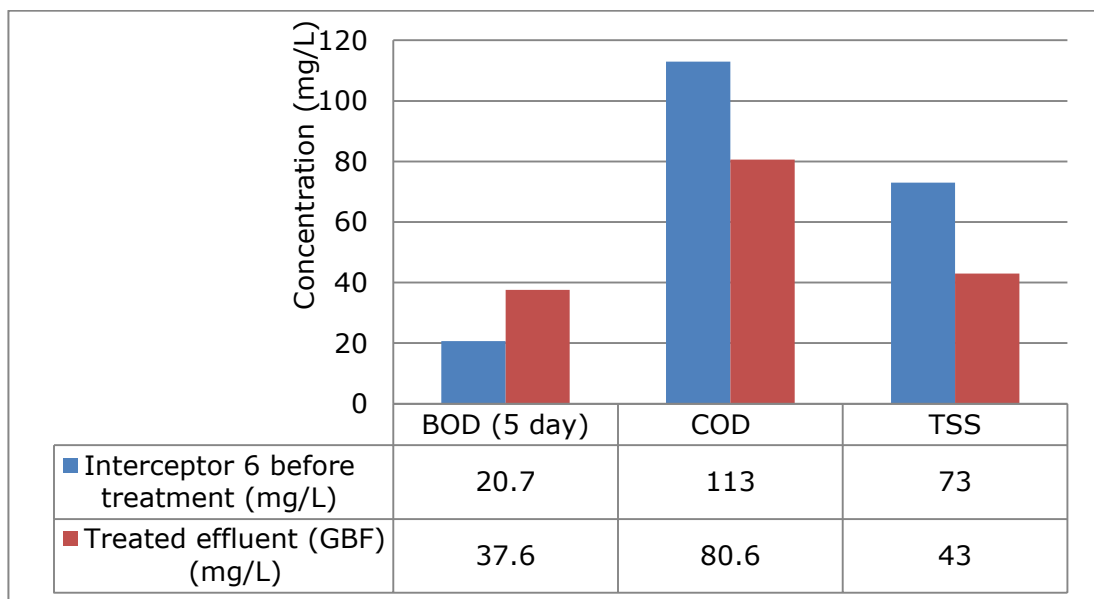
Figure 4-22 Filtration Field Trial results: Trial 2 analysis results of Interceptor 6 before and after treatment using the GBF.

The preliminary results suggest a reduction in COD (29 %) and TSS (41 %). However, there appears to be an unexpected increase in BOD level for trial 2-treated effluent, which may be due to sampling error or contamination of the sample bottles and this would therefore have to be repeated to confirm the result. Interestingly Cheremisinoff (1998) also observed such increases and suggested that this may be due to accumulation of particles on the

filter media, as particles have to attach on to the surfaces of the filter media for the removal to occur. Alley (2007) also stated that during gravitation filtration, particles may retain their positions on the filter media, and this could be an opposing effect to that of maximum efficiency.

In Trial 3, the input effluent was the GBF treated interceptor 6 effluent in Trial 2. The flow rate for CF was 100 L/min and the total treatment time was 10 minutes. This was significantly faster than GBF treatment in Trial 2, however just marginally faster than GBF treatment of Interceptor 5 in Trial 1. It can be seen in Figure 4-23, which COD increased after the treatment; this may be due to the sampling error and would have to be repeated. In trial 3, BOD and TSS were decreased by 11 % and 30 % respectively.

	BOD (5 day)	COD (total)	TSS
Influent (mg/L)	37.6	80.6	43
Treated effluent (mg/L)	17.8	90	22
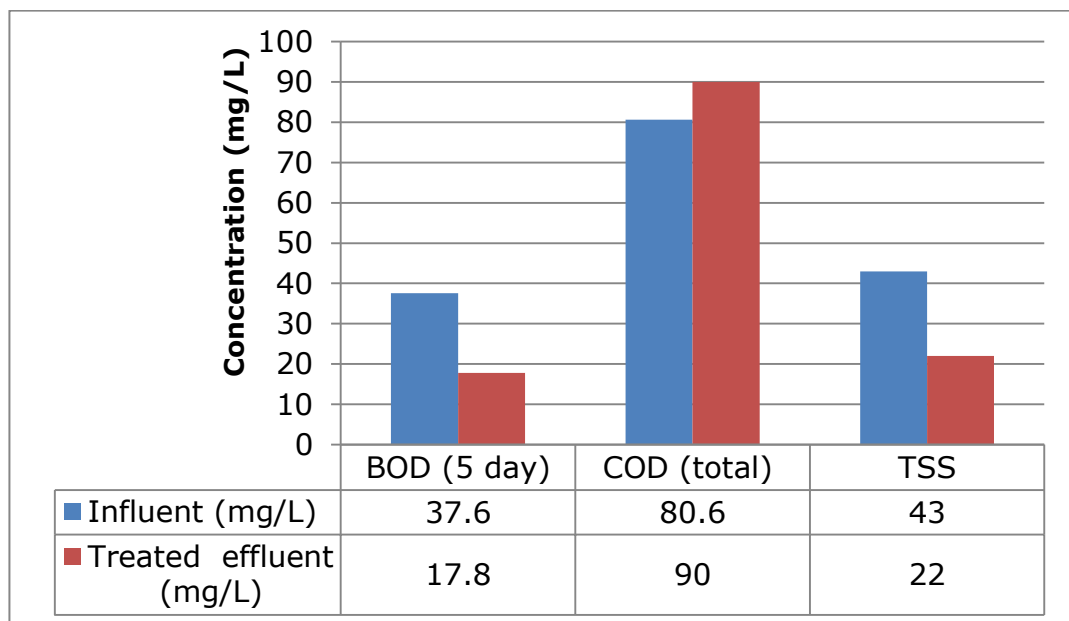
Figure 4-23 Filtration Field Trial results: Trial 3 analysis results of effluent from Trial 2 and after treatment using the CF.

Heavy metals Results

In Trial 1, 2 and 3 (as shown in Table 4-15), all metals apart from Ni, Mo and Co were found to be below the limits of detection before treatment commenced. This suggests that Interceptor 5 and 6 effluent was low in heavy metals at the onset of the trial although this was not known at the

time of the trial, so no conclusions can be drawn for the treatment of heavy metals.

Metals	As	Cr	Co	Cu	Mo	Ni	Pb
LOD (mg/L)	0.005	0.005	0.001	0.005	0.005	0.005	0.005
Trial 1							
Interceptor 5 before treatment (mg/L)	< LOD	< LOD	< LOD	< LOD	< LOD	0.007	< LOD
Treated effluent (GBF) (mg/L)	< LOD	< LOD	< LOD	< LOD	0.005	0.02	< LOD
Trial 2							
Interceptor 6 before treatment (mg/L)	< LOD	< LOD	0.001	< LOD	0.005	0.031	< LOD
Treated effluent (GBF) (mg/L)	< LOD	< LOD	0.001	< LOD	0.006	0.027	< LOD
Trial 3							
Effluent from Trial 2 (mg/L)	< LOD	< LOD	0.001	< LOD	0.006	0.027	< LOD
Treated effluent (CF) (mg/L)	< LOD	< LOD	0.001	< LOD	< LOD	0.02	< LOD

Table 4-15 Filtration Field Trial results: Trial 1, 2 and 3- dissolved heavy metals results of Interceptor 5 and 6 before and after treatment using the GBF or CF.

4.3.2.d   Conclusions and Recommendations

From the preliminary studies, it can be seen that the treatment of the prototype filtration system GBF can improve the effluent quality of Sureclean Interceptor effluents. The TSS appeared to be reduced in Trial 1,

2 and 3. However, there were no significant changes in the heavy metals level after treatments with both GBF and CF. The preliminary results did not appear to correlate with the results in the laboratory trials found in section 4.3.1. However, this may also suggest that Interceptor 6 effluent was very low in heavy metals at the onset of the experiments. GBF treatment may have used less energy than the CF treatment as in this trial the influent was passed through by gravity feed rather than pumped. However, the treatment time for the CF was faster than the GBF treatment.

To optimise the performance of the GBF, the effluent could be pumped into the tank rather than using gravity feed, thus making it a pressure filter. The filter can also benefit by installing a valve on the inlet to control the incoming flow. A flow meter could also be installed to determine the flow rate. To prevent large objects, such as stones and plastics, blocking the incoming pipe work of the filter, a course screen could be used as a pre-treatment.

Back washing of the filter system could be one method to improve the overall treatment efficiency. Hucher (2007) stated that backwashing could be achieved by reversing the flow through the filter media, thus lifting and fluidising the media. This removes the solids from the surface of media and carries them to the upper part of the media bed, finally out of the filter with the backwash water.

In order to evaluate the significance (i.e. with replication) and compare the performance differences to the GBF and CF, further work involving replication of analysis for each sample and performing statistical analysis on the data should be carried out. In terms of performance evaluation of the filtration, future work can be carried out to investigate the enhancement of filtration that may be achieved by changing the media size or media type. This was not investigated during this set of trials due to lack of time. In addition, the backwash mechanism, frequency, backwash liquid handling and the life time of the filter media investigation can also be carried out.

4.4 Filtration: Development of Sureclean Water Treatment System

Based on the laboratory and field trials conducted in section 4.3.1 and 4.3.2, Sureclean decided to incorporate the GBF and a clarifier to develop a filtration-based technology to recycle and reuse wastewater for use in both onshore and offshore industrial wastewater treatment. One of Sureclean core services is to provide high-pressure water jetting services to the oil and gas industry. Water jetting is an effective and fast tool to remove paint coatings and clean surfaces on oil platforms by using water with pressure of up to 2,750 bar (40,000 pound per square inch (psi)) (Sureclean 2011). With the view of sustainability, the primary aim of this technology is to reuse water during jetting operation, thereby reducing raw water requirement. A high pressure jetting (HP) unit can use between 40-70 litres per minute, with an average running time of 4-5 hours per day. For an Ultra High Pressure jetting (UHP) unit, the average water usage has been estimated to be 12 litres per minute. The technology was also designed to include treatment of wastewater contaminated with oil and/or suspended solids for water re-use. Sureclean appreciated that this application would be a vital commercial approach to expand in the oil industry, especially to the oil-rich Middle-east countries, where water is scarce.

The prototype was called Sureclean Water Treatment System (SWTS01). The SWTS01 has a small footprint (modular) and it can be transported by a lorry or inside a standard shipping container (mobile). The system was designed to have "slot-in" ability to increase the flexibility of the unit. Sureclean aimed to expand its waste treatment profile by using this innovative design to provide immediate wastewater treatment support for clients anywhere in the world.

4.4.1 Specification

The design of the modular plant was carried out for the following process parameters: pneumatic control, stainless steel, housed in a 10″ by 8″ ISO shipping container (as shown in Figure 4-24). SWTS was designed to be

completely pneumatically driven so the unit could be operated in a different industrial environment (onshore, inshore and offshore).

Figure 4-24 Photograph of the Sureclean Water Treatment System in a container (SWTS01).

The SWTS01, with the facility to contain three main elements (as shown in Figure 4-25):

- Inclined Plate Clarifier – for the removal of heavy solid material (such as silt and sand) and oil
- GBF – for the removal of suspended solids and some oil.
- Bag filters – a type of pressure-driven filter that contains filter housing, a holding basket and the replaceable filter bag (1-10 µm ratings). The bag filters were used for the final polish of the wastewater.

The inclined plate clarifier was purchased from a company called Siltbuster Limited (HB10). The GBF were bespoke made and designed based on understanding from a literature review and results from the laboratory and field trials in section 4.3.1 and 4.3.2 above. A local welder in Alness performed the welding work.

Figure 4-25 photograph of the individual components inside the SWTS01: an inclined plate clarifier (left) and the glass media filter (right) with two bag filters and two holding tanks).

The filter system adopted in SWTS was a pressure filter fed by a diaphragm pump from the first holding tank (as shown in Figure 4-26). The treated effluent was transferred to second holding tank by a second diaphragm pump. The other two sets of diaphragm pumps were used to reverse the process to backwash the filter media. The filter was fitted with an actuator that is air operated and a stainless steel butterfly valve for automatically stopping / starting flow. Table 4-16 shows the specification of the two treatment units within SWTS.

Figure 4-26 shows a 1 inch air operated diaphragm pump installed on SWTS01.

	Inclined Plate Clarifier	Glass Media Filter
Dimensions (Height x Length x Width):	2100 mm x 1900 mm x 900mm	2500mm x 1000mm x 1670mm
Weight:	800kg	510 kg
Descriptions:	Skid mounted inclined plate clarifier with 1 inch crowfoot inlet and 2 inches Bauer outlet	Contains: • 2 holding tanks (each with a dimension of 1000 mm x 550 mm x 1520 mm), • 1 cylindrical vessel containing glass media • 2 bag filters • 4 pumps (1 inch diaphragm pumps)

Table 4-16 SWTS01: Specification of the inclined plate clarifier and the glass media filter.

4.4.2 SWTS Field Trials-Leachate Treatment

Field trials were carried out on the Sureclean WTS and the waste was leachate collected from Cell 4 of the Nether Dallachy Municipal Landfill site, which was operated by Moray Council in Scotland. Leachate is water that has infiltrated sediments in a landfill site that contains high dissolved contaminants (Cheremisinoff 2002). These trials were carried out to investigate the efficiency of the newly designed SWTS01 to treat the leachate to comply with the Sureclean discharge consent. The leachate was passed through the SWTS01 at different flow rates. One sample was taken from the raw leachate, and then a sample was subsequently taken after treatment with SWTS at different flow rates. The flow rate examined was at 1, 1.5, 2 and 3 m^3/h (cubic meter per hour). The SWTS01 was backwashed

with clean water in between changes of flow rates to ensure that the filter media were not contaminated. Hoses and other connections were also flushed with clean water to prevent cross contamination.

The main parameters examined were the removal efficiency of TSS and heavy metals. The overall reduction of BOD and COD were also examined, however, a significant decrease of these two parameters was not expected as leachate typically contains high dissolved organics (Mcardle, Arozarena and Gallagher 1988). However, some decrease in TSS and heavy metals was expected. The leachate was pumped from one of the storage tank in Sureclean Alness WTS directly into the first holding tank in SWTS01 via a diaphragm pump. Samples were collected before the treatment from the SWTS01 first holding tank and after the treatment from the second holding tank for analysis.

4.4.2.a   Analysis

The wastewater was analysed before and after the filtration with the following analysis: heavy metals, TSS, COD and BOD. Due to cost and time constrains within the company, only one set of data was obtained. The effluent samples were sent to an independent UKAS accredited laboratory, STL Limited for analysis. The following methodology was obtained from STL Limited for the various analytical method statements.

TSS Measurements

Suspended matter was removed from a measured volume of sample by filtration under reduced pressure through a pre-treated, pre-weighed glass fibre filter paper and determined gravimetrically after washing and drying at $105 \pm 5°C$. The reporting range was equal or more than 2 mg/L and the limit of detection was 1.815 mg/L.

COD Analysis

Samples were oxidised by heating in vials with sulphuric acid and potassium dichromate. Mercuric sulphate was added to suppress chloride interference. The dichromate was reduced to chromate during the digestion and the chromate produced was measured colorimetrically. The range of application was between 20-2000 mg Oxygen/L without dilution, and the limit of detection was 12 mg/L as Oxygen.

BOD Analysis

The BOD is defined as the mass of dissolved oxygen required by a specific volume of liquid for the process of biochemical oxidation over a 5 day period at 20°C in the dark. The result was expressed as milligrams of oxygen per litre of sample. The range of application was 1 mg/L and above, and the limit of detection was 0.88 mg/L.

Heavy Metals Analysis

The heavy metals examined were As, Co, Cu, Mo, Ni and Pb. The heavy metals were determined by ICPMS or ICPAES depending on the metal. The metals were reported as a total concentration of the elements which included the concentration of the dissolved element. Total As was determined by ICPMS after dissolution by a boiling nitric acid digestion. The digestion was used to bring as much of the sample into solution as possible, prior to analysis. This included organometallic compounds of the analytes and elements in their oxidation states. The reporting range was between 1 – 250 µg/ L and the limit of detection was 0.238 µg/L.

All of the other metals were determined by ICPAES after dissolution in the presence of nitric acid. The pretreatment ensured that any metals in suspended or colloidal forms were converted to soluble forms. The range of applications and the limit of detections for the metals are shown in Figure 4-17.

Metals	Range of application (mg/L)	Limit of detection (mg/L)
Co	0.0005 - 10	0.00054
Cr	0.001 – 10	0.00071
Cu	0.001 – 10	0.00100
Ni	0.0009 - 10	0.00090
Mo	0.0013 – 1.0	0.00130
Pb	0.0019 – 10	0.00190

Table 4-17 SWTS01 Field Trials: Range of applications and limit of detection for Co, Cr, Cu, Ni, Mo, Pb, Ti and Zn in SWTS field trials.

According to STL Limited, spectral interference may occur from the presence of other elements, therefore the spectral lines were chosen so that overlap was minimal. Elements within standards were chosen to minimise chemical interference. Internal standards were used to compensate for interference from plasma anomalies caused by high dissolved solids content.

4.4.2.b   Results and Discussion

Cell 4 leachate was treated at different flow rates using SWTS01. The analytical results for the effluent before treatment can be seen in Table 4-18. Although all of the parameters were within the current Sureclean discharge consent, Sureclean was keen to improve the effluent quality to allow for any tightening of the discharge limits in the future. Therefore, it was important to investigate new treatment methods in order to improve on these values.

The flow rate examined was at 1, 1.5, 2 and 3 m^3/h. Samples were collected before and after treatment. As seen in Figure 4-27, BOD, COD and TSS appears to be reduced when the SWTS01 was running at 2 m^3/h.

However, this reduction was not observed in other flow rates. The BOD, COD and TSS were reduced by 46 % 25 % and 38 % respectively.

Parameter	Raw Effluent (mg/L)	Sureclean Discharge Consent (mg/L)
BOD	140	1000
COD	2260	3000
TSS	308	1000
As	0.064	
Co	0.17	
Cr	0.023	
Cu	0.097	
Ni	0.029	
Mo	0.002	
Pb	0.21	
Total Metals	0.595	2.000

Table 4-18 SWTS01 Leachate Field Trials: Analytical results of raw leachate prior to treatment using SWTS01.
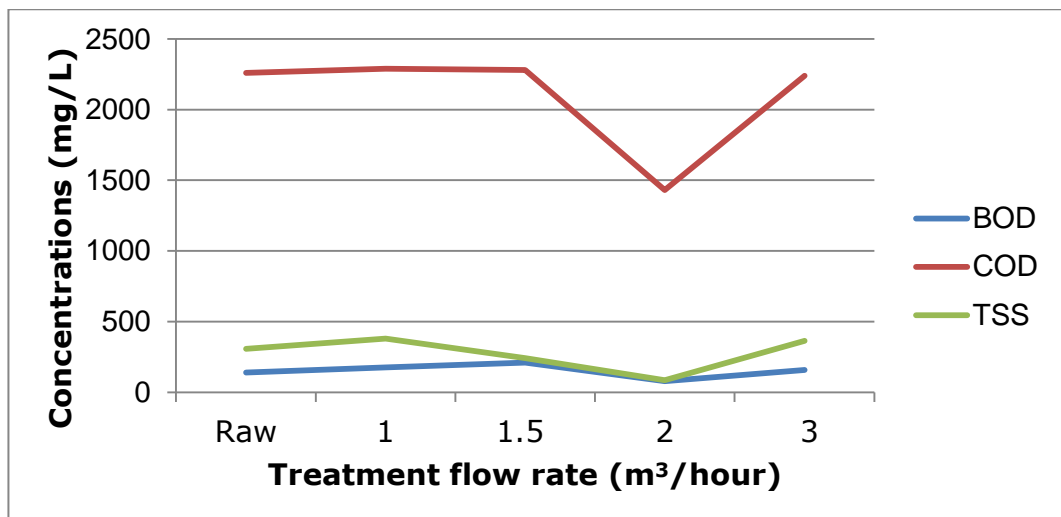
Figure 4-27 SWTS01 Leachate Field Trials results: Cell 4 wastewater sample analysis of BOD, COD and TSS before and after treatment using SWTS01.

Heavy metals included in the discharge consent are As, Cr, Co, Cu, Mo, Ni and Pb. ICPMS was used to measure As and ICPAES was used to measure Cr, Co, Cu, Mo, Ni and Pb all of which are reported in Figure 4-28, which shows that not all heavy metals were noted to be reduced. In conclusion, the results appears to confirm the findings seen in the field trials conducted in Section 4.3.2, which showed that filtration would not reduce heavy metals significantly.
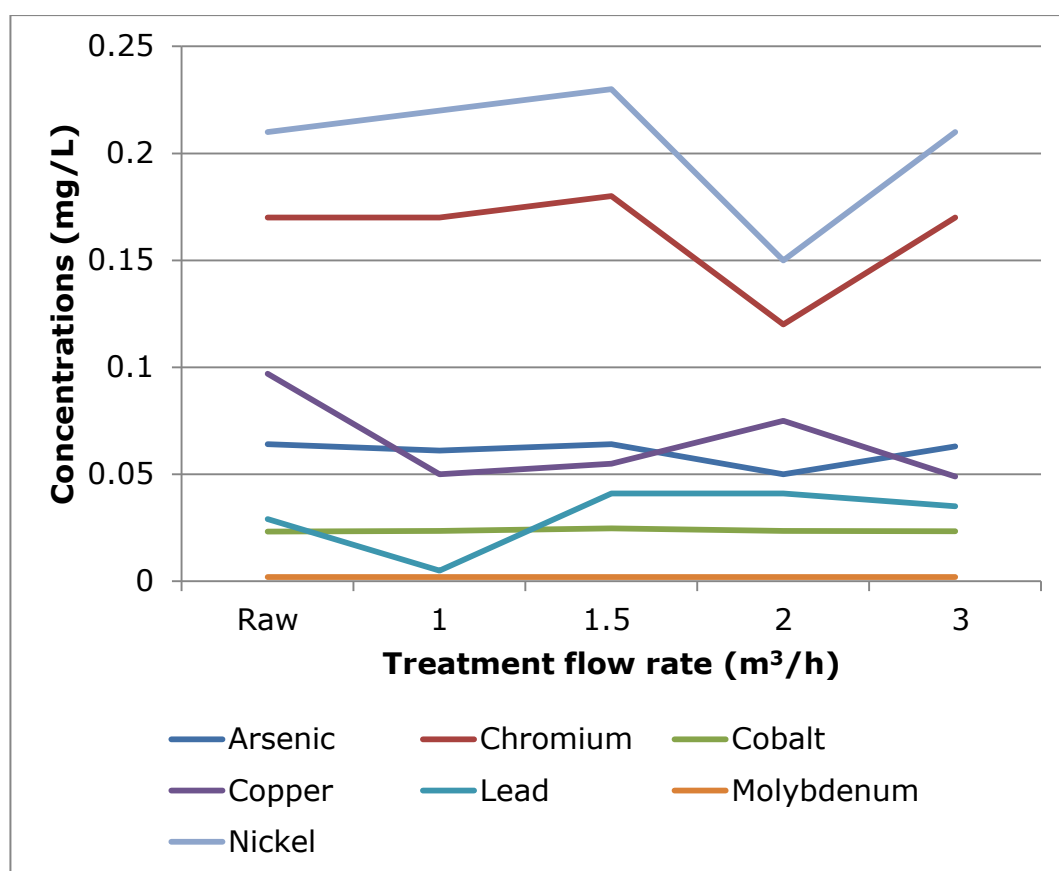
Figure 4-28 SWTS01 Leachate Field Trials results: Heavy metal analysis results of the Cell 4 leachate before and after treatment using SWTS01 in different flow rates.

4.4.2.c   Conclusion

The preliminary studies conducted in this section have demonstrated that 2 m^3/h was the optimum flow rate for SWTS01 in the treatment of leachate. In order to evaluate the significance (i.e. with replication) and compare the performance of SWTS01 in the treatment of leachate, further work involving

more replication of analysis for each sample should be carried out which would then allow the performance of statistical tests on the data to be carried out. The choice of the type of method used to analyse Sureclean waste streams rely on the type of wastes, time limitation as well as cost consideration. However, on this occasion cost was one of the limiting factors as each set of analysis for one sample cost as much as £130. According to Prichard and Barwick (2007), cost is one of the factors that have to be considered when choosing the types of method use for analysis. Sureclean could consider other factors that influence the choice of analytical methodology such as instrument detection limit, interferences, time and sample volume required for one analysis. For example, in heavy metal analysis, ICPOES and ICPMS could analyse multi elements simultaneously as compared to single-element instrument such as Flame Atomic Absorption Spectroscopy. However, multi-elemental instruments are considerably more expansive that Flame Atomic Absorption Spectroscopy (Broekaert 2005).

Other future work should include using different types of filter media, as stated by Cheremisinoff (1998 pp. 14), "effectiveness of the particle removal is determined by several variables, including type of filter media (size, depth, material)". Based on this recommendation, Sureclean has started to investigate different types of filter media including coconut based-granular activated carbon, crushed stone and garnet sand. More investigation on other types of wastewater could also be conducted using different filter media.

4.5 Chapter Conclusion and Future Work

The treatment efficiency of different mechanical separation units were investigated for Sureclean waste streams. The new process was expected to complement the existing process in Sureclean WTS. For the solid-liquid separation, a decanter centrifuge was selected for the dewatering of sludge and slurry. Preliminary results appear to show that the decanter centrifuge could reduce 56 % of the moisture content in oily sludge. However, the decanter centrifuge failed to separate peaty sludge from a water treatment

plant and OBM from an offshore platform. By using a decanter centrifuge, a potential annual saving of £4,064.64 could be achieved. However, the savings obtained does not pay off the annual capital and maintenance cost, which is a sum of £22,504.91 per year. Therefore, it was concluded that a decanter centrifuge would not benefit Sureclean at this stage.

Filtration was also investigated as another means of mechanical separation. The laboratory trials suggested that filtration using a filter media called AFM could decrease TOC by approximately 34 %, as well as the reduction of all particles size less than 600 μm and also some heavy metals. In the field trials, which involved a GBF, a prototype bespoke steel tank filled with AFM and a CF, the TSS appeared to reduce in all three trials. However, there were not significant changes in the heavy metals level after treatments with both GBF and CF.

Based on the laboratory and field trials conducted in section 4.3.1 and 4.3.2, Sureclean decided to incorporate the GBF and a clarifier to develop a filtration-based technology to recycle and reuse wastewater and they called the unit SWTS01. The landfill leachate was treated with SWTS01 and the samples were analysed before and after the filtration with the following parameters: TOC, particle size and heavy metals. The BOD, COD and TSS results appeared to reduce by 46 %, 25 % and 38 % respectively. To enhance the knowledge of SWTS01, more work could be conducted to evaluate the treatment efficiency of the SWTS01 using a range of Sureclean waste streams for the removal of TSS, TPH, COD, BOD and heavy metals. This type of results may be able to provide Sureclean further information on the selection of flow rate and filter media types in accordance to the waste type.

Chapter 5   Chemical Treatment of Oily Waste

5.1 Overview of Chemical Treatment

Colloidal particles in wastewater which are not readily settable can be chemically enhanced to coagulate. These particles typically have a particle size range of 10^{-7} to 10^{-1} mm, and they have almost negligible settling velocity (Keily 1997). Fine and dispersed colloid particles with particle size of 10^{-2} mm would take up to 20 hours to settle 100 mm (Bratby 2006). Therefore, colloid particles in wastewater are difficult and not economical to be removed by conventional physical and mechanical means due to their long settling velocity. These particles contribute to the turbidity of the wastewater and consist of micro-organisms, NOM and inorganic clay particles. The interaction of these colloidal particles with water molecules and other solutes, through their surface charge, allow the formation of stable dispersions.

In wastewater treatment, coagulation is the reaction where chemical destabilisation of particles occurs to form aggregation (Cosgrove 2010). Coagulation can be defined as the initial colloid destabilisation that is mainly caused by charge neutralisation; whereas flocculation can be defined as the aggregation of these colloid particles through hydrogen bonding or Van der Waal forces that followed the destabilisation into flocs (Jiang 2001; Wakeman and Tarleton 1999). Coagulants and flocculants such as short-chained polymers and polyelectrolytes promote floc formation (Tchonobaglous, Burton, and Stensel 2002). Coagulants can also precipitate soluble salts by chemically or physically combining the salts into the floc (Blake 1989). Chemical coagulation/flocculation is an important pre-treatment process for water and wastewater treatment. The main purpose of chemical treatment is to remove suspended solids and heavy metals, thereby aiding in mechanical separation (by filtration or centrifuge decanter) (Spellman 2011; American Water Works Association 2011).

5.1.1   Coagulation-The mechanism

The mechanism of the surface particles interaction is fundamental to understanding chemical treatment of water or wastewater. According to Lyklema (1977 pp.3), colloid stability is a term widely used to include "interaction of dispersed particles, their aggregation or their resistance to aggregation". Colloid particles are charged and the charge could arise from a number of different ways: absorption of ions from the bulk solutions, ionisation of particles in solutions or lattice imperfections, which refers to atom replacement in a lattice structure by atoms with a different valence (American Water Works Association 2011; Wilkinson and Lead, 2007; Benefield, Judkins, and Weand 1982). Colloid particles are usually negatively charged in solutions (HDR Engineering 2001; Alley 2007) and they are held apart from each other by electrical charges (Wilkinson and Lead 2007; Lyklema et al. 1991). However, a colloidal dispersion does not have a net electrical charge, in other words, the net charge of the solution is neutral (Benefield, Judkins, and Weand 1982). The charge on the particles is offset by ions of opposite charge, also known as 'counter ions' to form an outer layer of the colloid particles (Benefield, Judkins, and Weand 1982). The interactions formed are due to the surface charges on the particles and the 'counter ions' in the solutions, together they are collectively known as the "electrical double layer" or "electrostatic double layer" (Elimelech et al. 1995; Ghosh 2009). The electrical double layer is an important concept in understanding colloid interactions (Ghosh 2009; Butt, Graf, and Kappl 2003). Destabilising this electrical double layer can lead to the agglomeration of particles; this can be achieved by using chemical coagulants or flocculants (Elimelech et al., 1995).

There are four main mechanisms behind chemical coagulation/flocculation in wastewater treatment (Benefield, Judkins and Weand 1982; Pani 2009; Bache and Gregory 2007): Double-layer compression; Adsorption and charge neutralisation; Enmeshment in a precipitate; and Adsorption and interparticle bridging. These mechanisms also demonstrate the different types of chemical conditioner in waste and wastewater treatment.

1. Double-layer compression

As mentioned earlier, disruption to the "electrical double layer" would lead to the de-stabilisation of the interaction between colloids and solutes, and consequently lead to agglomeration of the two counterparts (Benefield, Judkins and Weand, 1982). According to Alaerts and Haute (1982), the double layer is compressed by particles that have high ionic strength (counter ions) in the solution, thereby neutralising the surface charge nearer to the isoelectric point (IEP). If the concentration of these counter ions continues to increase to cause sufficient double layer compression, the particles attractive forces will exceed the repulsion and thus leads to coagulation (Sincero and Sincero 2003).

2. Adsorption and charge neutralisation

The charge of the colloids can be neutralised by ions of the opposite charge (Pani 2009). Metal salts such as ferric chloride ($FeCl_3$) and aluminium sulphate (more commonly known as Alum, Al_3SO_4) can be directly absorbed on to the colloid particles and cause destabilisation. However, at higher dosage, coagulation occurs because of enmeshment of colloids in the precipitated metal hydroxides (HDR Engineering 2001). $FeCl_3$ and Al_3SO_4 have been used historically to remove colour caused by NOM in wastewater (Amirtharajah and O'melia 1999). Destabilisation through adsorption is stoichiometric; therefore the required chemical dosage depends on the concentration of colloids (Benefield, Judkins and Weand 1982). Overdose of coagulants beyond neutralisation to the point of charge reversal can occur and lead to re-stabilisation of colloids (Benefield, Judkins and Weand 1982; American Water Works Association 2011).

3. Enmeshment in a precipitate

Precipitate can be formed by addition of certain metal salts, oxides or hydroxides in water, which would enmesh colloid particles as they settled (HDR Engineering 2001). This process is called enmeshment in a precipitate or sweep floc coagulation. The mass of precipitate depends on pH and the dose of the chemicals (Bache and Gregory 2007). The combined

precipitates, also known as the coagulant floc can then be removed easily through settling or filtration (American Water Works Association 2011).

4. Adsorption and interparticle bridging

Natural compounds such as starch and cellulose as well as synthetic polymer such as polyacrylamide are often large in molecular size and contain multiple electrical charges along the molecular chain (HDR Engineering, 2001; Benefield, Judkins and Weand 1982). These polymers are categorised based on the molecular weight, charge type (Cationic, Anionic and Non-ionic) and density (Bache and Gregory 2007). Figure 5-1 demonstrates the bridging mechanisms for particle destabilisation using polymers. At the initial stage, the polymer binds to the colloid particles at one or more sites. Bridging is commonly observed in cases where non-ionic polymers are used or in cases where the polymers and the particles have similar charges (Bache and Gregory 2007).

Polyelectrolyte is a type of polymer (Holmberg et al, 2003) where the charged groups are attached covalently to the polymer backbone (Eagland, 1989). Polyelectrolyte as compared to uncharged polymers are highly water soluble, binds strongly with opposite charged surfaces or macromolecules and have a high tendency to swell and bind a large amounts of water (Stuart, De Vries and Lyklema 2005). However, excess dosage of polymer in water treatment may result in restablisation of colloid particles, thus no coagulation occurs (Bache and Gregory 2007).
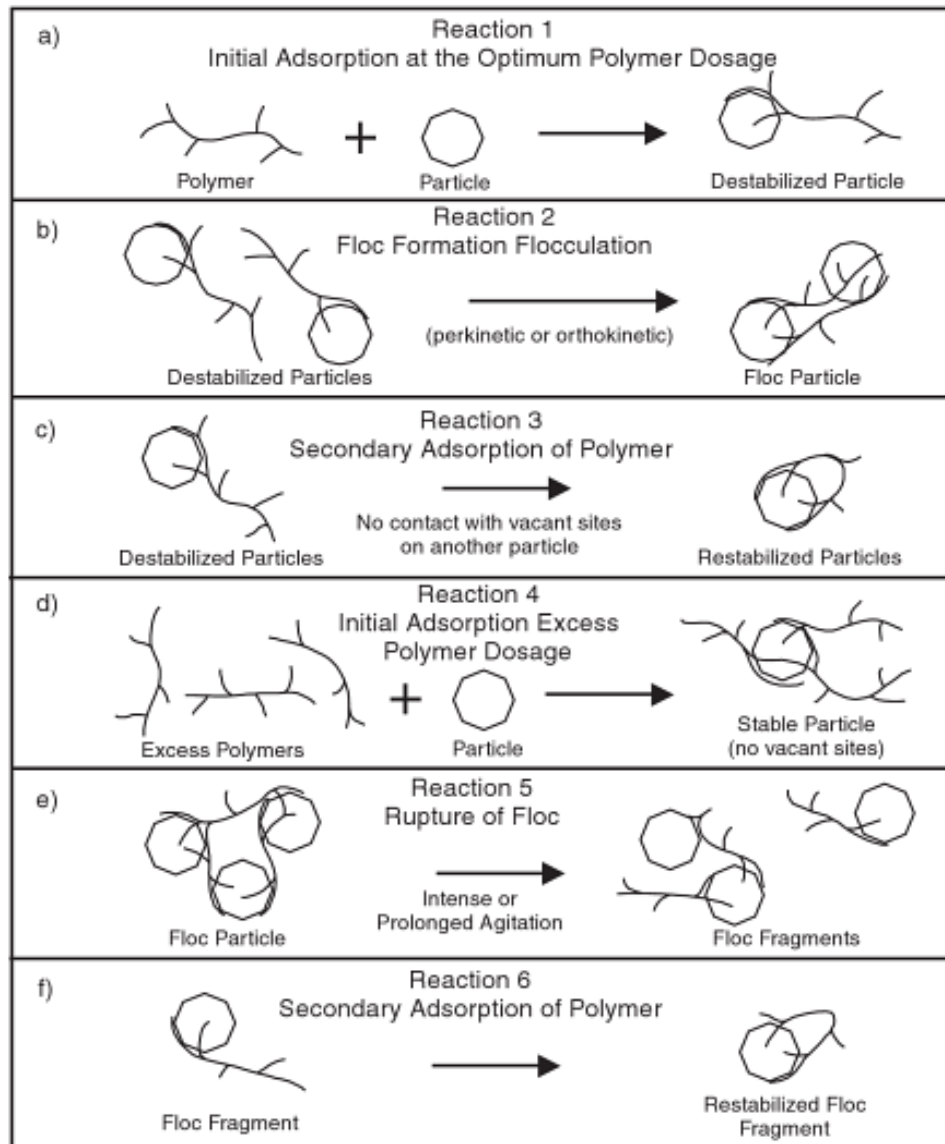
Figure 5-1 shows the schematic representation of the bridging model for destabilisation of colloids (HDR Engineering, 2001).

5.1.2   Types of Coagulants and Flocculants

Among the inorganic coagulants, the multivalent metal salts such as $AlCl_3$, $FeCl_3$ and Al_3SO_4 are more commonly used than electrolytes (Wakeman and Tarleton 1999) as they are thought to be more effective and cheaper in comparison to polymers (Bratby 2006). The mechanism by which metal salts cause coagulation is through charge neutralisation, double compression and enmeshment in precipitates (Binnie, Kimber and Smethurst 2002). However the nature of the aggregation formed using metal salts is thought to be pH dependant (Bratby 2006). Moreover, if the

coagulation process is based on charge neutralisation, overdosing could lead to charge reversal and re-suspension (Binnie, Kimber and Smethurst 2002).

Polyaluminum chloride (PAC) and Polyaluminum Silicate Sulfate are types of manufactured prehydrolysed coagulant that aims to produce the correct type of hydrolysed metals (Bratby 2006). These high molecular weight polymerised metal salts are thought to be more effective in terms of floc formation and they perform in a wider range of pH and at lower dosage than non-polymerised metal salts (Binnie, Kimber and Smethurst 2002). Although the price of PAC may be more expansive than Alum, PAC can achieve faster floc formation, requires lower dosage rate than Alum and operates over a wider pH range (Binnie, Kimber and Smethurst 2002).

Flocculants or coagulant aids such as acids or alkali and polyelectrolytes are often used where the flocs are slow settling and easily fragmented by hydraulic shear in the settling tank (Sincero and Sincero 2003). As mentioned in section 5.1.1, polyelectrolyte is a type of high molecular weight, charged organic polymer. The polymer could be synthetic or natural, and it can be non-ionic, anionic or cationic (Gregory and Barany 2011). Polyacrylamides (PAM) are the basis for all commercial flocculants (Rabiee 2010). Although PAM is nominally non-ionic, they can easily be hydrolysed to form anionic sites or the acrylamide can be co-polymerised with a cationic monomer to become cationic (Gregory and Barany 2011). Although the cost of polymers are higher than non-polymeric chemicals, polymers are thought to be effective even at low dosage and they work over a wider pH range; therefore, the advantages offset the cost of using the polymers (Binnie, Kimber and Smethurst 2002). The typical polymer dosage is between 0.01 and 0.5 mg/L (Binnie, Kimber and Smethurst 2002).

5.1.3   Chemical Treatment- The Process

The process of coagulation consists of three main stages: chemical adding and mixing, colloid destabilisation and floc formation. The first step of a chemical treatment process is to add the coagulant into the desired wastewater and mix it together. Mixing is a form of mechanical agitation and this could provide energy to the solution thereby forcing the particles closer together (Moody and Norman 2005). Rapid mixing also helps to disperse the chemical throughout the mixing tank (Sincero and Sincero 2003) and it is considered to be one of the most important stages since particle destabilisation occurs at this stage (Bratby 2006). The rapid mixing enhances colloid particle destabilisation under different mechanisms according to the chemical used (as described in Section 5.1.1). The flocs formed in the treated wastewater could be removed by floatation, sedimentation, filtration or centrifugation (Svarovsky 2000; Logsdon 2008). Therefore, chemical treatment is thought to be a pre-treatment to mechanical separations (Svarovsky 2000).

5.2 Chemical   Coagulations   in   Oily   Wastewater Treatment

The aim of using chemical treatment was to increase the effective particle size of colloids thereby assisting in its removal (Moody and Norman 2005). As described in Chapter 4 Mechanical Separation, Sureclean wastewater contains suspended solids, some heavy metals and hydrocarbon. Coagulation and flocculation could act as a pre-treatment process for advanced treatment such as photocatalysis (Shon et al. 2007), which was of Sureclean interest. Two separate laboratory trials were conducted to study the suitability and feasibility of chemical treatment towards Sureclean waste streams. The first trial investigated the use of different chemicals in the removal efficiencies of heavy metals and oil; two main parameters in the Sureclean discharge consent (refer to Table 1-1). The second trial looked into the effectiveness of chemical conditioning towards dewatering Sureclean oily sludge.

5.2.1   Laboratory Trial-Heavy metals and oil removal

Heavy metals, such as As and Pb, can cause intoxication and bioaccumulation in organisms; and these compounds can be introduced to the water bodies usually through industrial effluent such as effluent from a paint manufacturing plant, a petroleum refinery or an industrial machinery manufacturing plant (Meltzer 1990). The conventional method of heavy metals removal is through precipitation as metallic bases by changing the pH (Warey 2006). In the discharge consent that was set out by SEPA, Sureclean was required to discharge no more than 100 mg/L of TPH and 2 mg/L of heavy metals, which include As, Co, Cr, Cu, Mo, Ni, Pb, Sn and Zn. Sn and Zn were tested in this study as they were included in the total heavy metals count in the old Sureclean discharge consent (before 2009). According to Sincero and Sincero (2003), the optimum pH and dose rate can be determined by a jar test. The objective of the laboratory trial was to investigate the effectiveness of four different coagulants on the removal efficiencies in heavy metals and TPH. The removal efficiencies of two commercial coagulants were also investigated over a range of pH with Sureclean Interceptor 6 effluent. The laboratory based trials were conducted by an MSc student, Findlay Bryce. The parameters that were investigated were pH, heavy metal concentration and oil content.

5.2.1.a   Waste and Analysis

Heavy Metals Analysis

Heavy metals were analysed using ICPOES (Perkin Elmer Optimer 3300DV ICPOES). Two multi-elemental standards were prepared: standard #1 consisted of Ni, Sn, Zn, Cr and Al; standard #2 consisted of Co, As, Mo, Pb and Cu. These standards were prepared over a concentration range of 0.01, 0.05, 0.1, 1, 5, and 10 ppm at 25 mL each. A calibration curve was created using these standards enabling the heavy metal content of the samples and controls to be determined. Prior to analysis, the wastewater samples were acidified by adding 600 μL of 6 M nitric acid to each of the 6 mL samples whilst 2.5 mL was added to each of the 25 mL standards. The chosen measurement wavelengths in nm of the metals were Al- 396.153, As –

228.812, Co – 236.892, Cr – 267.716, Cu– 324.752, Mo – 202.031, Ni – 231.604, Pb – 220.553, Sn- 283.998 and Zn– 213.857. Triplicate samples were analysed.

TPH Analysis

FTIR spectroscopy was the method used for the determination of TPH in the wastewater samples. The extraction procedure was conducted by adding 6 mL of TTCE to 6 mL of wastewater samples. The sample was then transferred to a separating funnel and was shaken vigorously for 60 seconds and then left for the layers to settle. The bottom layer containing TTCE was then collected. A calibration curve of absorption peak area (Acm^{-1}) against a range of marine diesel concentrations: 0, 5, 10, 50, 100 and 500 ppm was generated using known concentration of diesel standard. The concentration of TPH present in this study was calculated from the regression line of the calibration curve. The peak area of the region 3100 to 2700 cm^{-1} was recorded.

5.2.1.b   Experimental Procedure

Four chemicals were studied in this trial: FloQuat R100 (a commercial PAC based product, supplied by SNF, UK); FloPam (a commercial cationic PAM based product, supplied by SNF, UK), FeCl$_3$ and AlCl$_3$. FeCl$_3$ and AlCl$_3$ are some of the most commonly used coagulants in wastewater treatment (Water Environment Federation 2006). All dosages were prepared in six replicates with both heavy metal and TPH analysis carried out in triplicate.

FloQuat R100 Dosage Determination Trials

The overall procedure for FloQuat R100 conditioning of Sureclean interceptor wastewater was as follows:

1. A dosing range of 0, 15, 20, 25, 30 and 35 µL/L was investigated, with the 0 µL/L as the control dosage.

2. Stock solutions were prepared by adding deionised water to FloQuat R100, so that the concentrations of the solutions were 10 times of the

required concentration for the dosage trials, i.e. at 150, 200, 250, 300 and 350 µL/L.

3. 9 mL of interceptor water was pipette from the stirred sample into 15 mL plastic centrifuge tubes. 1 mL of each stock solution was then added to the samples to provide the appropriate dosage.

4. The samples were mixed at 120 rpm for 15 minutes; then 50 rpm for 45 minutes by using a Stuart Scientific Orbital Incubator SI50 at ambient temperature to control the mixing before allowing the samples to settle for 60 minutes.

5. Following the settlement period, 6 mL of liquid was sampled from each tube from below the liquid-air interface, to be used for analysis. Care was taken to ensure that any sludge that had settled at the bottom of the test tube was not disturbed when sampling.

6. This sampled liquid was then used for the heavy metal and oil content analysis.

7. The pH of the treated samples was measured using Whatman Narrow Range pH 4-6 paper.

Coagulant Trials

Four coagulants were investigated for their oil and heavy metal removal efficiencies at a dosage reported to be of their highest efficiency: 20 µL/L FloQuat (results from section 5.2.1.c part 1); 5 µL/L FloPam (Neilson, A., 2008, personal communication by email. 11 April 2008); 120 µg/L $FeCl_3$ (El Samrani, Lartiges and Villiéras 2008); and, 60 µg/L $AlCl_3$ (El Samrani, Lartiges and Villiéras 2008), as well as control samples to allow removal efficiencies to be calculated. All dosages were prepared in six replicates with both heavy metal and oil content analysis carried out in triplicate (as per Section 5.2.1.a). The preparation, dosing, and mixing procedures were the same as the procedures used in FloQuat R100 Dosage Determination Trials. Thus, the removal efficiencies of the following coagulants were investigated:

1. 20 µL/L FloQuat
2. 5 µL/L FloPam

3. 120 µg/L FeCl$_3$

4. 60 µg/L AlCl$_3$

5. 20 µL/L FloQuat and 5 µL/L FloPam

6. 120 µg/L FeCl$_3$ and 5 µL/L FloPam

7. 60 µg/L AlCl$_3$ and 5 µl/L FloPam

pH Adjustment Trials

pH of wastewater is one of the key factors affecting the effectiveness of chemical coagulation. This is because the changes in pH would affect the surface charge of the particles as well as the solubility of the coagulants (Pernitsky and Meucci 2002). Hydrolysis occurs when metal coagulant is added into water, and depending on pH, positive ions are formed. These ions will attach to the negative ions (colloid particles) and destabilise particles interaction (American Water Works Association 2011). However, as the pH of the wastewater is increased, the coagulants tend to lose protons that leads to the oxide surface becomes more negative (Elimelech et al. 1995). The more common coagulants (alum and ferric salt) were shown to provide the optimal organic matter removal at pH less than 6 (Yan et al. 2008).

Due to the diversity of Sureclean wastewater input, the pH of the interceptor effluent varied. Therefore the impact of pH with different types of coagulant was investigated. All of the interceptor water samples and previous experimental samples with added coagulants were of pH > 4 and < 5, therefore only sodium hydroxide (NaOH) was used to increase the pH.

The pH adjustment trials used 100 mL of interceptor water (pH ~4.3). 50 µL aliquots of 1 M NaOH were added to the interceptor water and the pH noted after each addition until pH 11 was surpassed. The NaOH dosage and pH were then correlated to provide a linear graph allowing the appropriate doses for an experimental range of pH 5, 6, 7, 8, 9, 10 and 11 to be calculated (as seen in Figure 5-2). The pH values were checked using Whatman® Narrow Range pH paper to ensure the NaOH dosing was

appropriate. These results provided a linear trend line with a high R^2 value of 0.9956, allowing for dosages to be calculated for smaller 10 mL samples. Equation 5-1 was used to calculate the required amount of NaOH to achieve different pH as seen in Table 5-1.
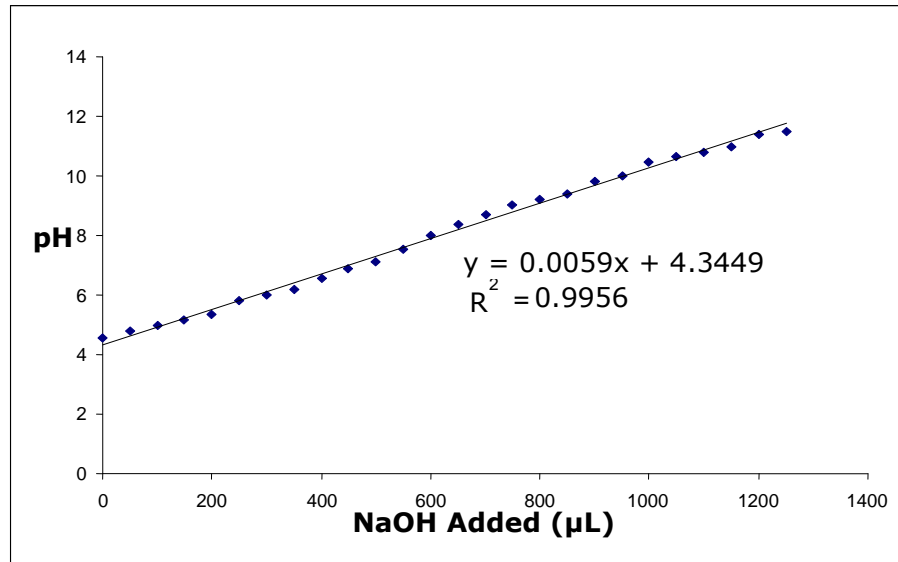
Figure 5-2 shows the pH adjustment trial against NaOH volume.

pH = 0.0592 * volume of 1.000 M NaOH required (µL) + 4.3349

Equation 5-1

pH	1 M NaOH required (µL)
4.3	0
5	11.1
6	28.0
7	44.8
8	61.7
9	78.6
10	95.5
11	112.4

Table 5-1 Table of the volume (µL) of 1 M NaOH required to increase the pH of 10 mL of interceptor water (batch#3) to predetermined values.

5.2.1.c   Results and Discussions

1. FloQuat R100 Trial

An initial trial was carried out to establish the optimal dosage for FloQuat in the removal of heavy metals and oil for Sureclean Interceptor water (batch #1; pH ~4.5), the experiment was performed in triplicate. It can be seen from Figure 5-3, that the optimal removal efficiency for both total heavy metals and oil removal was at 20 µg/L of FloQuat. Although the highest oil removal efficiency was at 30 µg/L of FloQuat the removal efficiency of total heavy metals was reduced. At 20 µg/L FloQuat, 9.2 % heavy metal removal efficiency and 37.9 % oil removing efficiency were achieved. The increase dosage of FloQuat after 20 µg/L did not show significant improvement in oil and heavy metals removal but a reduction of removal efficiencies was observed. As discussed in Section 5.1.1, the results may suggest that restablisation of colloid particles occurs, thus coagulation activity cease.
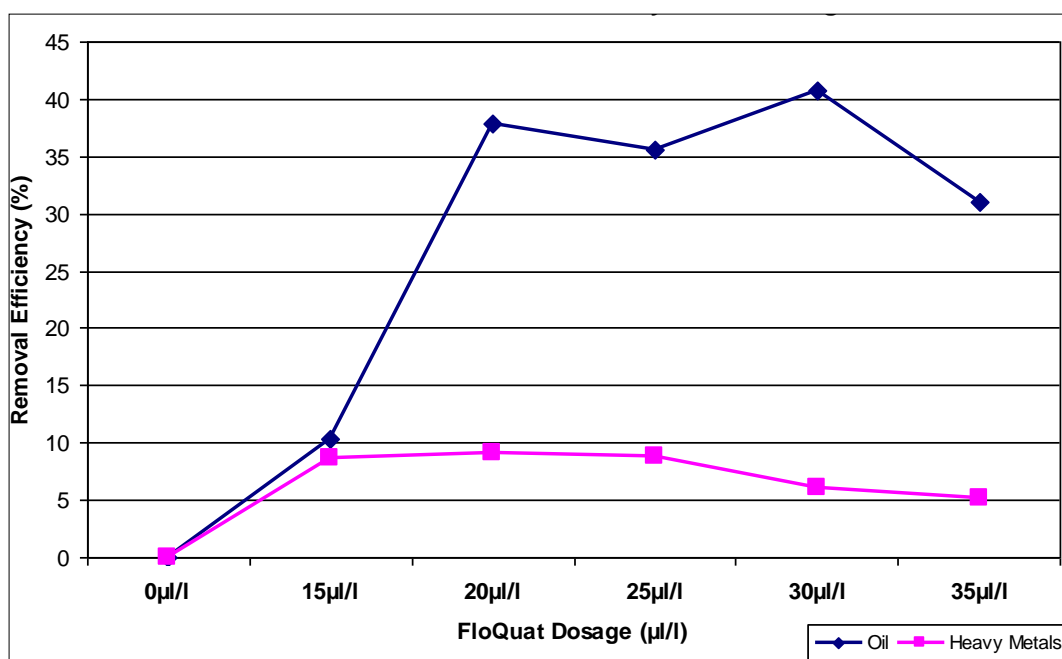
Figure 5-3 Chemical Treatment for Oily Waste Laboratory Trials: Total heavy metal and oil removal efficiencies of FloQuat R100 coagulant in wastewater various dosages.

2. <u>Coagulants Trial</u>

The removal efficiencies of the following coagulants were investigated on Sureclean Interceptor water (batch #2; pH ~4.5) with FloQuat, FloPam, FeCl3 and $AlCl_3$, as well as a combination of FloQuat, $FeCl_3$ and $AlCl_3$ with FloPam. As discussed in Section 5.1.2, polymerised metal salts such as PAC are thought to be more effective than metal salts. From Figure 5-4, it can be seen that the performance of FloPam, FloQuat, $FeCl_3$ and $AlCl_3$ had relatively similar heavy metal removal efficiency at approximately 10 %. The results indicated that the polymerised metal salt FloQuat or polyelectrolyte FloPam alone were not more effective than $FeCl_3$ and $AlCl_3$ as suggested by some literature in heavy metal removal.
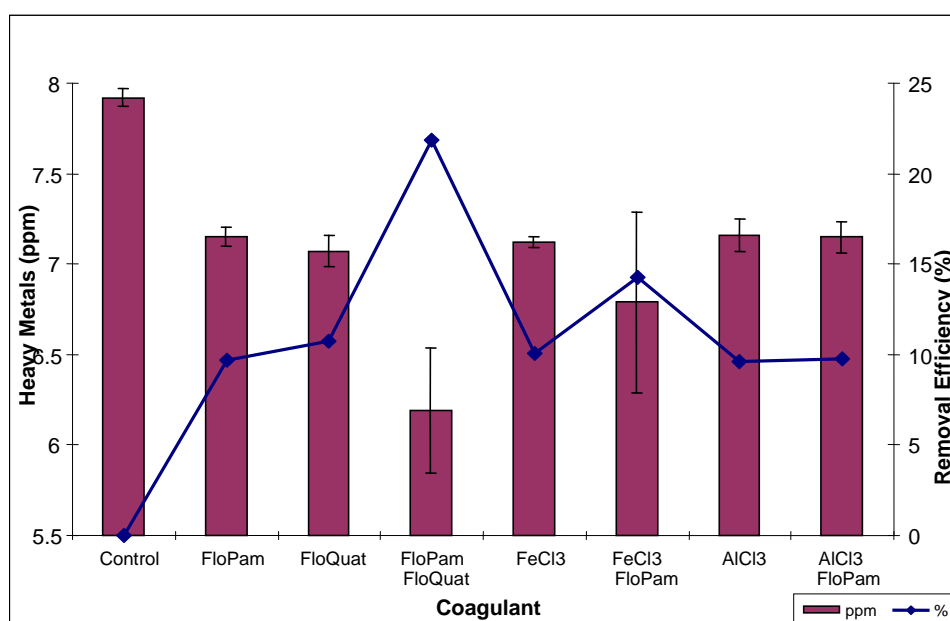
Figure 5-4 Chemical Treatment for Oily Waste Laboratory Trials: Total heavy metal (excluding aluminium) removal from wastewater by, and total heavy metal removal efficiency of various coagulants.

However, when FloPam was combined with FloQuat, the removal efficiency improved to 21.8 %. However, a similar trend was observed for the $FeCl_3$ and FloPam combined treatment, but the observed result was not as good as the FloQuat and FloPam combination. Both results (FloPam/FloQuat and FloPam/$FeCl_3$ combinations) have large error bars which indicate that the

results may not be reproducible. FloPam, a polyelectrolyte was thought to have an auxiliary bridging and linking effect when used after a metal coagulant (Binnie, Kimber and Smethurst 2002). The interceptor effluent had a pH of 4.5, whilst the recommended optimum pH for $FeCl_3$ dosing was thought to be between pH 4-5.5 (Nieuwenhuijzen and Van Der Graaf 2009); therefore, this explained the high removal efficiency seen in the total heavy metal removal efficiency. No marked improvement was observed for the $AlCl_3$ and FloPam combination. According to Versilind (2003 pp.13.3), the primary difference between Aluminium and Ferric chemistry are their difference in relative solubility. This may explain the performance difference between $FeCl_3$ and $AlCl_3$ when combined with FloPam.

Chemicals are frequently used for treatment of oily wastewaters and are used to improve mechanical treatment and separation (Flynn 2009). Oil carries electrostatic charges and addition of chemicals especially cationic coagulants could destabilise the oil droplets, thus aggregation can be induced. In this study, oil was analysed using FTIR, and it can be seen from Figure 5-5 that all chemical coagulants were able to reduce the oil level in Sureclean interceptor effluent. The highest oil removal efficiency of 40.1 % was seen as a result of $FeCl_3$ treatment. $FeCl_3$ treatment was significantly more effective than the other coagulant especially $AlCl_3$ and the $AlCl_3$ and FloPam treatments. Whilst care had been provided to ensure the homogeneity of the effluent by mixing the effluent using a magnetic stirrer, large error bars can be seen for all results. In future, the treated wastewater samples could be filtered to remove the coagulated sludge, thereby reducing the chances of samples inhomogeneous.
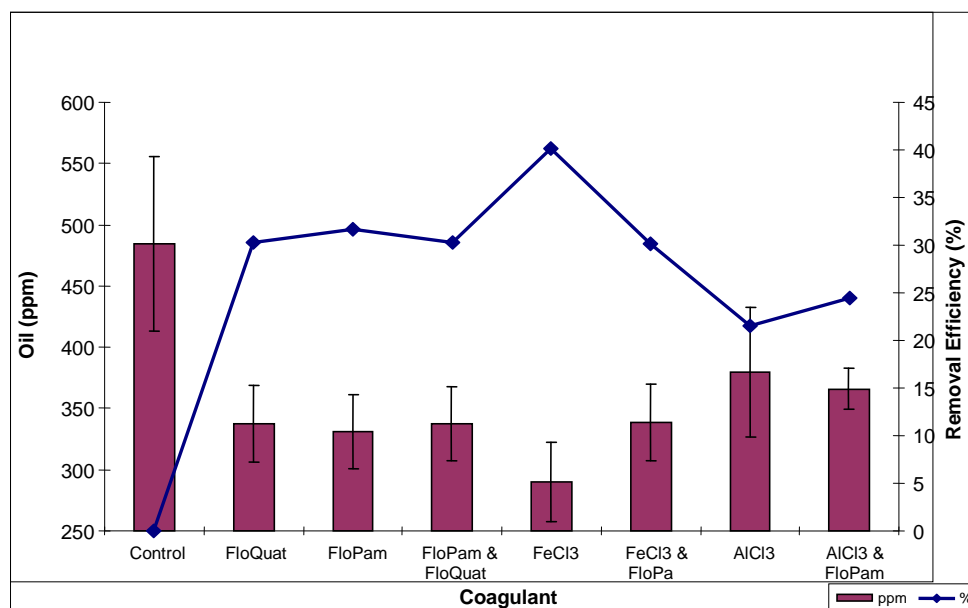
Figure 5-5 Chemical Treatment for Oily Waste Laboratory Trials: Oil removal from wastewater by, and oil removal efficiency of, various coagulants.

3. pH Adjustment Trials

Coagulation pH is important to control the precipitation of metal hydroxides (Binnie, Kimber and Smethurst 2002). In Figure 5-6 total heavy metal content can be seen to reduce both the treated and untreated samples. At the lowest pH of 4.3, the treated sample had significantly lower heavy metal concentrations than the untreated sample, with a removal efficiency of 45.8 %. The treated samples at pH 5 also showed a significantly improved heavy metal removal efficiency of 46.1 % compared to 30.5 % observed in the untreated samples.

For the samples exposed to pH $\geq$ 6, there was no significance found between the treated and untreated samples, although the general trend of the removal efficiency continued to increase (although at a decreased rate) from pH 6-8 with the removal efficiency rate levelling out at pH 9. The highest removal efficiency recorded, 72.0 %, was for the treated sample at pH 10. These results showed that in acidic conditions the addition of FloPam and FloQuat coagulants was a useful tool in heavy metal removal, however, if the pH was increased to neutral or above then the addition of NaOH is a

far more effective method. According to Water Environment Federation (2008b), "many of the common heavy metals form insoluble hydroxides at pH 11, so lime coagulation reduces these metal concentrations."
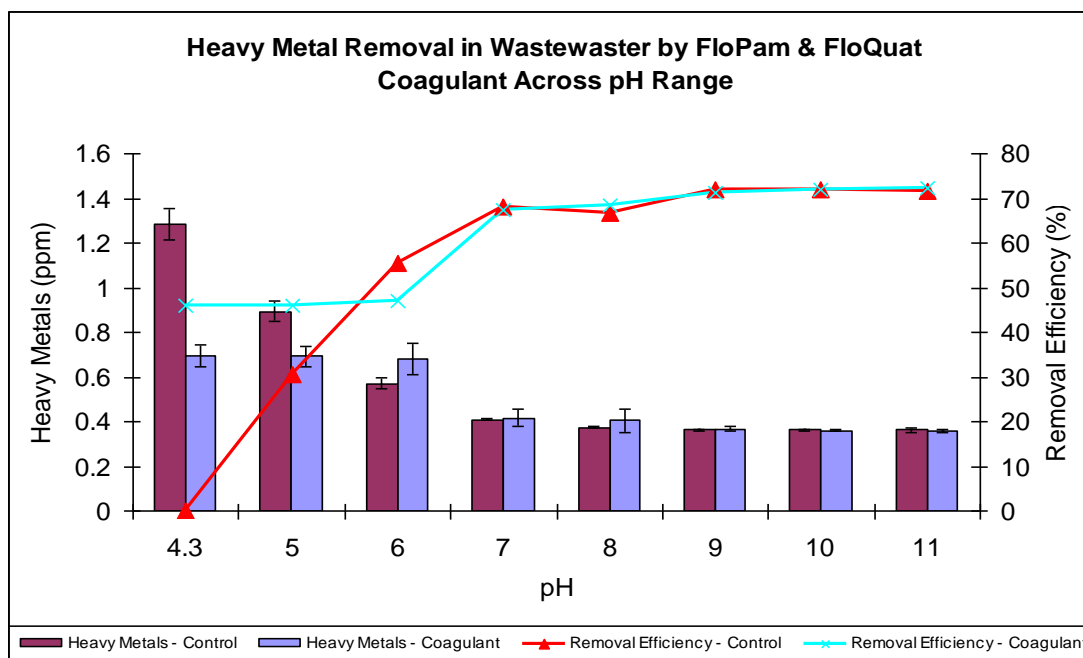
Figure 5-6 Chemical Treatment for Oily Waste Laboratory Trials: Total heavy metal removal from wastewater by, and total heavy metal removal efficiency of FloPam & FloQuat treatment across pH range.

It can be seen from Figure 5-7, the oil removal efficiencies were better when the effluent was treated with FloQuat and FloPam as compared to the untreated samples at pH 4.3 and 5. At pH between 6 to 10, the oil removal efficiencies of both treated and untreated samples were very similar of 36.2 – 39.1 %, whilst the most alkali untreated sample of pH 11 showed a slightly poorer removal efficiency of 29.8 %. The removal efficiencies of the treated samples did not differ significantly from pH 4.3 through to pH 10 with a range of 26.7 to 42.1 %, but the most alkali sample of pH 11 showed a large increase in removal efficiency to 59.6 %. This large increase in oil removal at pH 11 was in contrast to the untreated sample which showed a decrease at this pH.
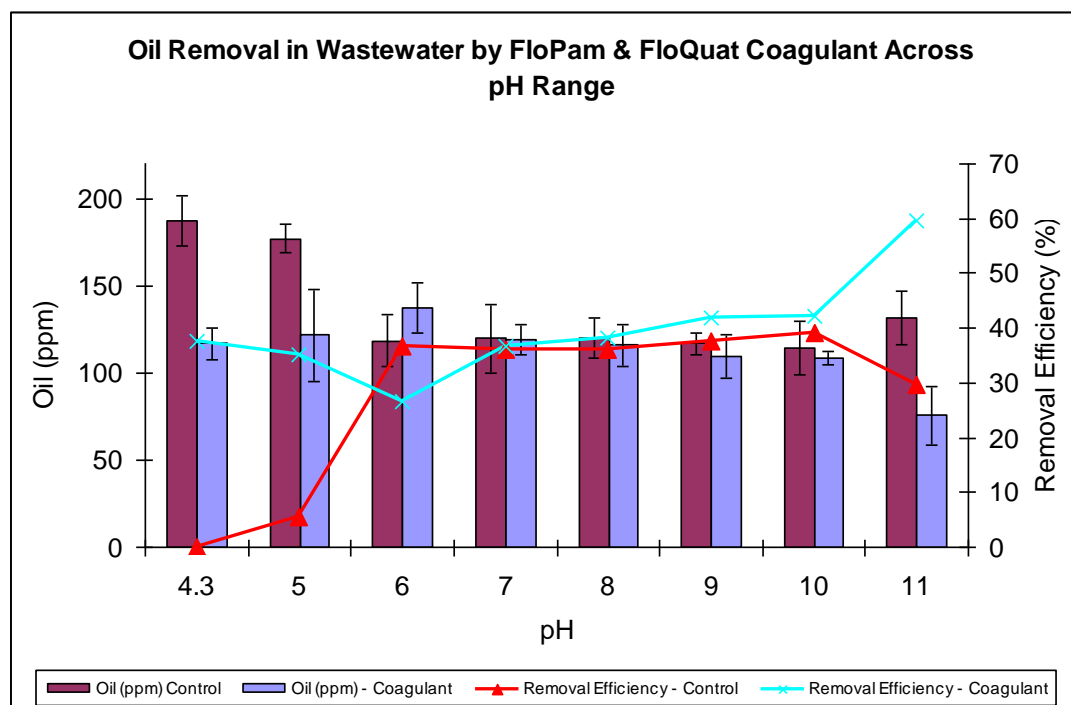
Figure 5-7 Chemical Treatment for Oily Waste Laboratory Trials: Oil removal from wastewater by, and oil removal efficiency of FloPam & FloQuat treatment across pH range.

5.2.1.d   Conclusion

As stated by Nieuwenhuijzen and Van Der Graaf (2009), the two significant factors for achieving optimal coagulation were the chemical dosage and coagulation pH. It was concluded that without pH adjustment, the FloQuat and FloPam combined treatment had the highest total heavy metal removal, whereas $FeCl_3$ had the highest oil removal efficiency. $FeCl_3$ is a corrosive chemical and needs to be handled with care (Rushton, Ward and Holdich 1995). However, simply raising the effluent pH to neutral state (pH 7) by using NaOH, the oil and heavy metal removal efficiency were almost as effective as adding FloQuat and FloPam alone. The outcome of the trials suggested that by employing NaOH to raise the overall pH in the effluent, oil and heavy metal removal can be achieved and that the outcome would be as good as using coagulants.

5.2.2 Field Trial- Heavy metals and oil removal

The laboratory results shown in Section 5.2.1 indicate that there was a decrease oil and total heavy metals level in Sureclean interceptor effluent after treatment with chemicals, and most notably the commercial chemicals FloQuat R100 and FloPam EM640. A field trial (1000 L) was conducted simultaneously with the laboratory trial to investigate the efficiency of the commercial chemicals with Sureclean interceptor effluent. Due to time and labour constrains, only one trial was able to be performed. The main purpose of the field trial was to compare the findings obtained in the laboratory trials. Other parameters such as TSS, COD and BOD were also investigated.

5.2.2.a Waste and Analysis

The chemicals used in the field trials were FloPam and FloQuat; both were supplied by SNF (UK) Limited. The wastewater was analysed before and after the chemical coagulation with the following analysis: TPH, heavy metals, TSS, COD and BOD. Due to cost and time constrains within the company, only one set of data was obtained. The effluent samples were sent to an independent UKAS accredited laboratory, STL Limited for analysis. The following methodology was obtained from STL Limited for the various analytical method statements. Analytical methods for heavy metals, TSS, COD and BOD have been described in Section 4.4.2.a.

TPH

The wastewater samples were first extracted with pentane. The extracts were analysed by capillary gas chromatography with flame ionisation detection (GC-FID). Any co-extracted material which had a retention time in the carbon range of C6 to C40 and which elicits a detector response would cause interference to the spectra. The range of application was between 10 - 20000 µg/L and the normal reporting level was 10 µg/L.

Experimental Procedure

For the dilution of FloPam (to 5 ppm as recommended by manufacturer), two batches of 500 mL diluted solutions were prepared as followed: 2.5 mL of FloPam was measured using a 5 mL syringe and was then added to 500 mL of water in a beaker; the mixture was mixed for 10 minutes using a magnetic stirrer, the overall steps were repeated for the second batch of diluted FloPam. For the diluted FloQuat (to 15 ppm as recommended by the manufacturer as a starting point), 15 mL of the neat polymer was added into a beaker with 500 mL of water. The mixtures were mixed using a magnetic stirrer for 10 minutes.

In the transfer station, two 1000 litres IBC were filled with Sureclean interceptor effluent using an electrical submersible pump. The first IBC was used as the control with no chemical addition. In the second IBC, the submersible pump was used to re-circulate the effluent and as the mixing energy for the chemical additions.

500 mL of the diluted FloQuat (15 ppm) was added and was allowed to mix for 15 minutes in the second IBC. This was then followed by the addition of 1000 mL of the diluted FloPam; the mixture was allowed to mix for 10 minutes. After 10 minutes, the submersible pump was turned off and the contents were allowed to settle for 30 minutes.

A second trial was conducted using the same concentration of FloPam but 20 ppm of FloQuat. Therefore to prepare the diluted FloQuat (to 20 ppm), 20 mL of the neat polymer was added into in a beaker with 500 mL of water. The mixtures were mixed using a magnetic stirrer for 10 minutes. Wastewater samples before and after treatment were collected for analysis from the middle of the IBC without the disturbing the layers of settles sludge or floating scum.

5.2.2.b   Results and Discussions

Sureclean Interceptor 5 effluent was treated using FloQuat and FloPam. For the optimum removal efficiency, the concentration of FloPam was recommended by SNF at 15 ppm. The concentration of FloQuat was trialled at 15 and 20 ppm to compare the effectiveness of both concentrations. After the addition and mixing of chemicals, the treated effluent was allowed to settle for 10 minutes before samples were collected for analysis. The colloid particles were seen to grow in size during the mixing stage; and the solid was observed to be settling to the bottom of the IBC during the settling stage. Therefore at the end of the settling stage, there was a clear layer of sludge observed at the bottom of the IBC. Wastewater samples were then taken from the middle of the IBC without disturbing the bottom sludge layer.

Using GC-FID, TPH was measured and the results were recorded according to the carbon length of the hydrocarbons. It can be seen from Figure 5-8 that there was an overall reduction in TPH after treatment with FloQuat and FloPam. Treatment with FloQuat at 15 ppm and 20 ppm showed removal efficiencies of 11.3 and 44.6 % respectively. This result also correlated with the laboratory trial findings from Section 5.2.1. The polymers form aggregates by the bridging mechanism as well as changing to the surface charge of the oil droplets leading to coalescence of the oil droplets (Coca-Prados, Gutiérrez-Cervelló and Benito 2011). The coalesced oil droplets could be removed by mechanical separation systems such as oil skimmer or inclined plate clarifier.
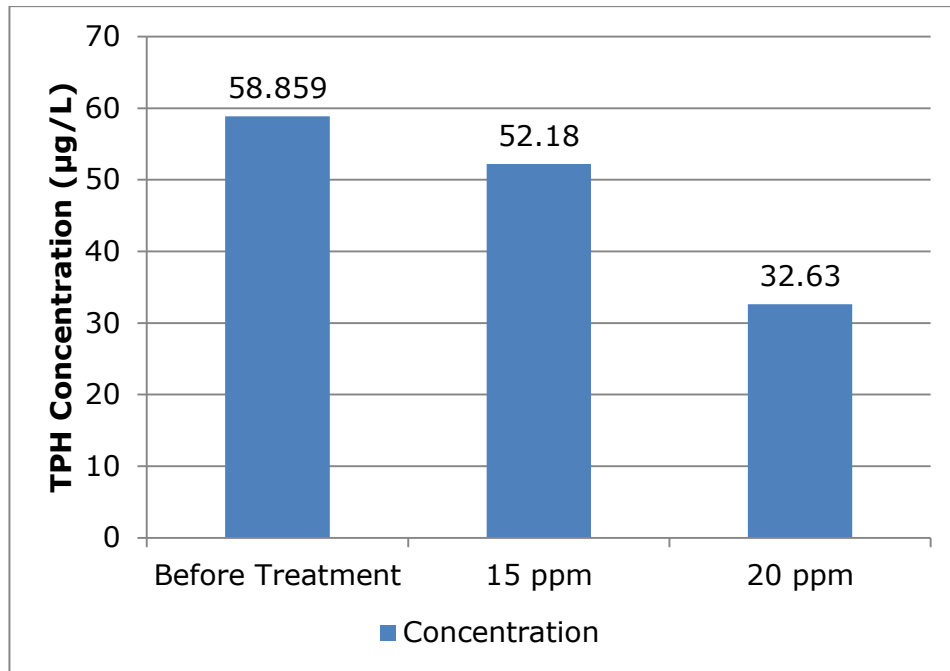
Figure 5-8 Chemical Treatment for Oily Waste Field Trials: TPH results of Interceptor 5 wastewater before and after treatment with FloQuat and FloPam.

For BOD, COD and TSS results, it can be seen from Table 5-2 that there were little or no changes seen in the level of BOD and COD. The BOD results after treatment with 20 ppm of FloQuat R100 could not be determined precisely by AlControl because the analyst under-diluted the sample, therefore the results represented an indication of greater than 1160 mg/L. No statistical calculation was able to be made as only one set of analysis was performed. Therefore, in order to verify the significance of the results, the analysis could be repeated again in triplicate.

From this preliminary assessment, with 15 ppm and 20 ppm FloQuat treatments, it appears that there were high removal efficiencies for TSS of 73.8 and 89 % respectively. This shows that the polymers were destabilising the colloid particles that lead to aggregations. As the colloid particle aggregated, the density of the aggregates increased and therefore sinks to the bottom of the IBC.

Parameters	Before Treatment (mg/L)	15 ppm of FloQuat and 5 ppm of FloPam (mg/L)	20 ppm of FloQuat and 5 ppm of FloPam (mg/L)
COD	1940	1940	1926
BOD (5 day)	1310	1310	>1160
TSS	320	84	64

Table 5-2 Chemical Treatment for Oily Waste Field Trials: COD, BOF and TSS results of Interceptor 5 wastewater before and after treatment with FLOQUAT and FLOPAM.

Heavy Metals Analysis

The heavy metals were determined by ICPMS or ICPAES depending on the metal. Total As was determined by ICPMS after dissolution by a boiling nitric acid digestion. The reporting range was between 1 – 250 μg/ L and the limit of detection was 0.238 μg/L. All of the other metals were determined by ICPAES after dissolution in the presence of nitric acid. The details of the analytical methodology could be found in Section 4.4.2.a. It can be seen in Table 5-3 that there was no significant changes in the heavy metal level between treated and untreated Interceptor 5 effluent. The preliminary results were inconclusive in terms of heavy metal removal.

Heavy metals	Before Treatment	Treatment with 15 ppm of FloQuat and 5 ppm of FloPam	Treatment with 20 ppm of FloQuat and 5 ppm of FloPam	Limit of detection (mg/L)
ICPAES (for method refer to 4.4.2.a)				
Co (mg/L)	<0.005	0.006	<0.005	0.00054
Cu (mg/L)	0.015	0.008	<0.005	0.00071
Cr (mg/L)	<0.005	<0.005	<0.005	0.00100
Pb (mg/L)	0.022	0.008	0.008	0.00090
Mo (µg/L)	<0.005	<0.005	<0.005	0.00130
Ni (mg/L)	0.022	0.021	0.024	0.00190
ICP-MS (for method refer to 4.4.2.a)				
As (mg/L)	0.006	0.004	0.004	0.00054

Table 5-3 Chemical Treatment for Oily Waste Field Trials: heavy metals results of Interceptor 5 wastewater before and after treatment with FLOQUAT and FLOPAM.

5.2.2.c   Conclusions and Future Work

The performance of the commercial coagulant and flocculants FloQuat and FloPam were investigated with Sureclean interceptor effluent. The field trial used 1000 litres of Interceptor 5 effluent. Due to time and labour constrains, only one set of data could be obtained. Therefore the results suggest a preliminary indication of the chemicals performance. It can be seen from Figure 5-9 that the removal efficiency of 20 ppm dosage of FloQuat was higher in TPH, BOD, COD and TSS than at dosage of 15 ppm. The highest removal efficiencies were the TSS at 73.8 % and 80.0 % for 15 ppm and 20 ppm respectively whilst 27.7 % of heavy metal was removed from both 15 and 20 ppm of FloQuat. The preliminary results suggested that the optimum dosage for FloQuat was at 20 ppm as concluded in Section 5.2.1.c.
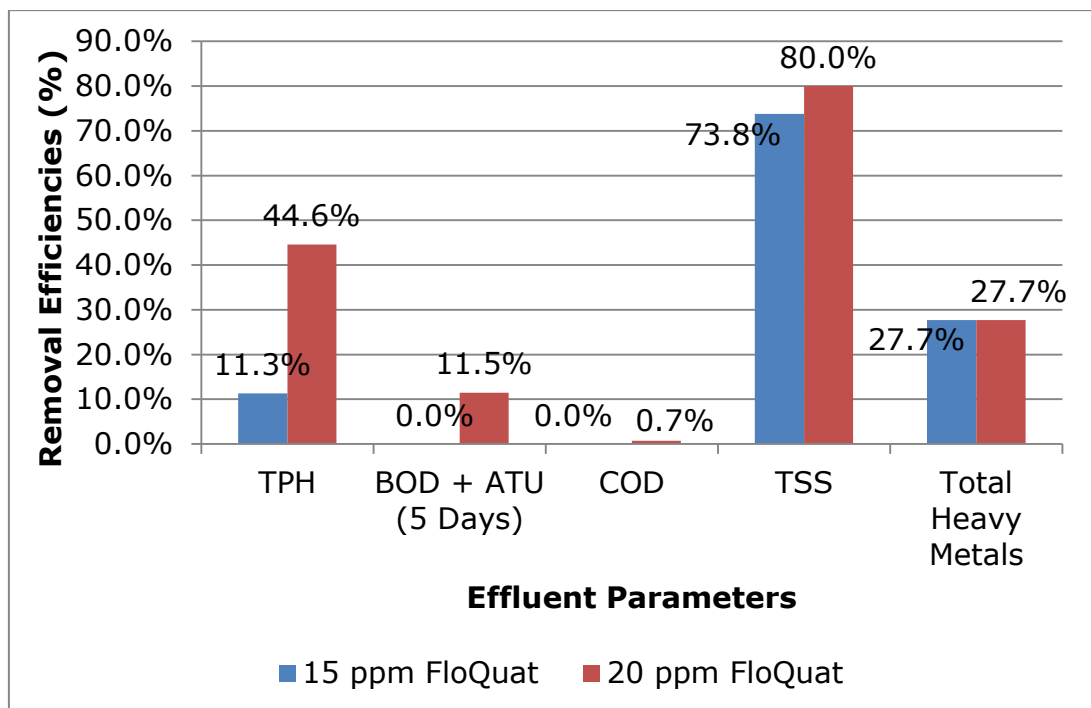
Figure 5-9 Chemical Treatment for Oily Waste Field Trials: Comparison of the removal Efficiencies of TPH, BOD, COD and TSS.

Chemical treatment would improve treatment of Sureclean interceptor effluent based on the findings seen in the laboratory and field trials (as seen in Section 5.2.1 and 5.2.2). The outcome of the laboratory trials suggested that by raising the pH of the interceptor effluent to pH 7 using NaOH, the oil and heavy metal removal efficiency were almost as effective as adding FloQuat and FloPam alone. Large scale trials (1000 L or more) could be performed at Sureclean WTS using NaOH. A chemical dosing system as described in Section 5.1.3 that include dosing pump and chemical mixing system could be introduced to Sureclean WTS.

5.3 Dewatering of Oily sludge Using Chemicals

As described in Chapter 4, the waste treatment at Sureclean for oily sludge was processed by simple mechanical means to remove larger solid or grits and surface oil. The remaining solids/sludge was sent to third party waste companies for further treatment and safe disposal. According to the Landfill Directive WAC, "any waste load containing a free-draining liquid substance

that is more than 250 litres or 10% of the load volume, whichever represents the lesser amount" are prohibited to be landfilled (Environment Agency 2010, pp.13). Therefore oily sludge has to be treated prior to landfill. As concluded in Chapter 4, it was not economically viable yet for Sureclean to purchase a decanter centrifuge for the dewatering of oily sludge and OBM, therefore, other means of treatment were investigated.

The treatment of oily sludge can be achieved by chemical or biological methods. Composting and bioremediation are two biological methods used in oily sludge remediation. Although composting of oily sludge has low capital cost and low maintenance, however, this treatment method could not meet current environmental legislation (Kuznetsov and Zaikov 2006). Oily sludge with a high pollution level could also limit the performance of bioremediation (Kuznetsov and Zaikov 2006).

Several studies have shown that chemical conditioning can improve oily sludge dewatering capacity (Hwa and Jeyaseelan 1997; Buyukkamaci and Kucukselek 2007; Guo et al. 2011). According to Zall, Galil and Rehbun (1987), chemical conditioning can improve aggregation of sludge by binding the small gel-like sludge particles into bigger and stronger aggregates, in which they have less affinity for water. The use of sludge conditioners such as polymers helps dewatering and achieves higher solid content (Wang et al. 2004). The conditioned sludge could improve sludge dewatering when used with a solid bowl decanter centrifuge (Liu and Liptak 1997). Carbon-based materials such as charcoal, lignite and fly ash are used as physical conditioners for sludge, which are also known as skeleton builders (Qi, Thapa and Hoadley 2011). Lignite is the lowest rank of coal, often known as brown coal due to its colour (Cleveland and Morris 2009). According to Thapa et al. (2009), the use of lignite could increase the calorific value of the sludge when the treated sludge is sent for incineration. Fly ash is the remaining waste of the incineration process and it can be considered as a zero-cost raw material to destabilise sludge (Buyukkamaci and Kucukselek 2007). Polyethylenimine (PEI) is a positively charged water soluble polyelectrolyte manufactured from aziridine (ethylenimine), and has also

been used in the paper industry as a retention aid (Grace 2007). According to Wu, Wu and Huang (2003), polymer conditioning has been widely used to improve dewatering of sludge.

This laboratory study aims to investigate chemical techniques to reduce moisture content in the sludge, which would potentially reduce the final disposal cost and sludge handling (Andreoli 2007; Punmia, Jain and Jain 1998). The chemical or compounds used were lignite, PEI, FloQuat R100, fly ash and Charcoal.

5.3.1 Laboratory Trial- Dewatering of Oily sludge Using Chemicals

5.3.1.a Waste and Analysis

The laboratory trials instigated were performed by a Masters Student, Partha Talukdar under my direction. The main methods used in this study to evaluate the performance of chemical conditioning to the oily sludge were: TSS Analysis and Moisture Content Analysis by weight loss.

Sludge Moisture Determination

According to Keily (1997), total solid (TS) in water or wastewater refers to solid residues after evaporation at 105 °C in a furnace. However, it was not possible to perform this analysis in the normal manner as there was no furnace available; instead a hotplate was used in a fume hood. The conditions of these tests were between 70 to 80 °C. To help in quality assurance, the sludge was subject to a cycle of drying, cooling and weighing until a constant weight was produced (Eaton and Franson 2005). In addition, every experiment was done in triplicate. The following procedure was used to obtain TS in suspension: The sludge sample was centrifuged for 25 minutes to remove excess water. A petri dish was weighed, before a known amount of centrifuged sludge was placed on the petri dish; and this was then dried in the fume hood using a hotplate at approximately 80 °C

until a constant weight was achieved. The increased weight in the petri dish represented the TS in suspension of blank sludge.

Dewaterability can be expressed as the increase in Moisture Content (MC) (Negulescu 1985). Unfortunately, as explained previously, it was not possible to achieve total dryness; therefore an estimate of the sludge moisture content was calculated using Equation 5-2.

$$\text{Sludge Moisture Content (MC)(\%)} = \frac{100 \times \text{W}}{\text{I}}$$ **_Equation 5-2_**

Where W= weight of dried solid obtained (in g)

I= weight of sludge obtained (in g)

From Equation 5-2, total solid content can be deduced from the equation below:

$$\text{Total Solid in Sludge (\%)} = 100\% - \text{MC}$$ **_Equation 5-3_**

5.3.1.b   Experimental Procedure

Four experiments were conducted using four different chemical conditioners: lignite (Sigma-Aldrich, granulated material ~1 mm, type darco 12×20, from lignite, activated), fly ash (supplied by Sureclean Limited), Polyethylenimine or PEI (Sigma Aldrich) and FloQuat R100 (supplied by SNF UK Limited) to evaluate their dewatering efficiencies in Sureclean oily sludge.

The overall procedure for chemical conditioning of Sureclean oily sludge was as followed:

1. 20 mL of sludge was added to each beaker.
2. A known volume or weight of conditioner was added in the sludge.
3. The sludge was stirred with a magnetic stirrer for 25 minutes.
4. TS analysis was performed as described in Section 5.3.1.a for all experiments and MC was then calculated using Equation 5-2.

5.3.1.c   Results and Discussions

Four oily sludge samples were originated from Sureclean Alness WTS interceptor sludge. Figure 5-10 shows the MC of four untreated oily sludge samples. The MC was calculated using Equation 5-2. The small error bar for each sludge sample indicated that the results were was reproducible. The average MC for all four samples was 54.2 %. Therefore, as discussed in Section 5.3, this sludge was not fit to be landfilled without treatment.
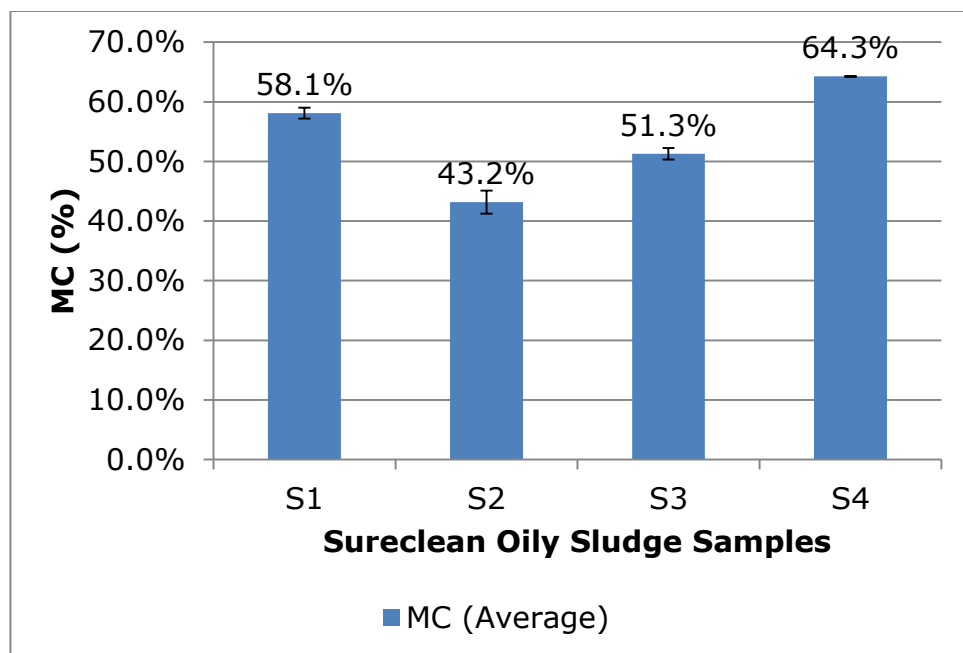
Figure 5-10 Chemical Conditioning of Oily Sludge Laboratory Trials: MC of the four oily sludge samples.

Chemical Conditioning

Four chemical compounds were investigated as a chemical conditioner for Sureclean oily sludge: lignite, fly ash, PEI and FLOQUAT to evaluate their dewatering efficiencies in Sureclean oily sludge. Each conditioner was tested over a range of dosage concentrations and the best performance of each conditioner is summarised in Figure 5-11. It can be seen that the highest MC reduction was seen in the PEI and Lignite combined treatment of 17.1 %. When PEI and Lignite were used alone, their performances were very poor with a MC reduction of -3.8 and -0.2 % respectively. This clearly shows that sludge conditioned with lignite and polyelectrolyte provided a

better dewatering capability than the polyelectrolyte flocculation, which correlated with the findings obtained by Thapa et al. (2009). Fly ash and FloQuat reduced the sludge MC by 6.7 % and 5.0 % respectively.
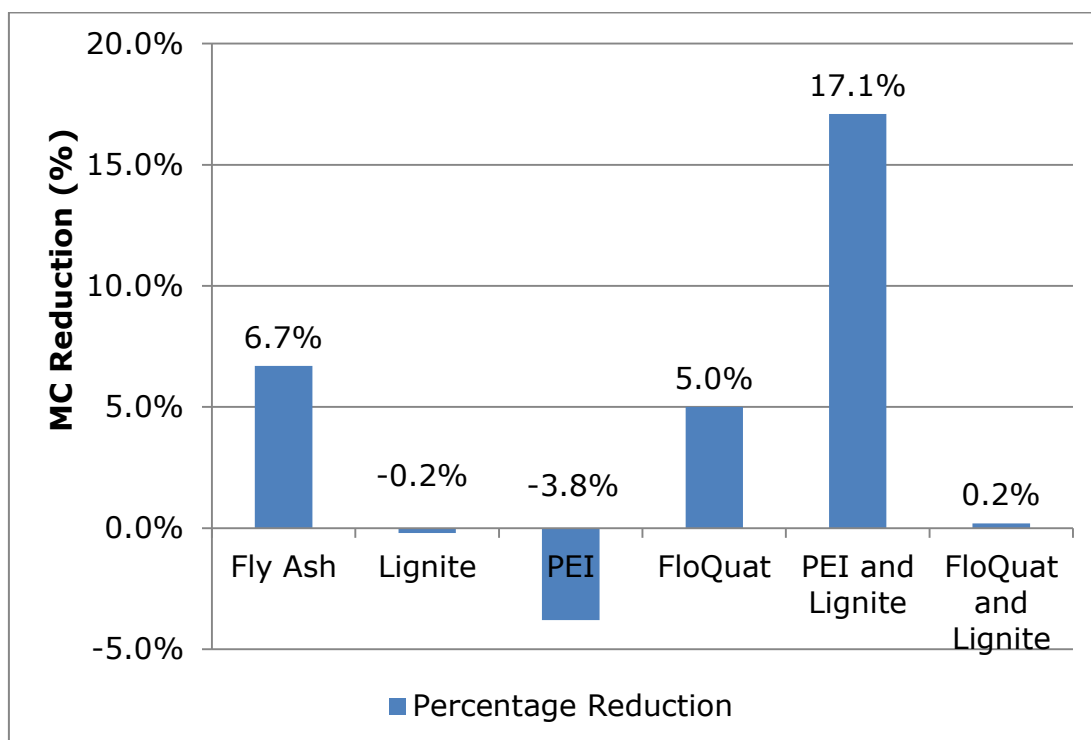
Figure 5-11 Chemical Conditioning of Oily Sludge Laboratory Trials: analysis of the MC reduction in the Chemical Conditioning of Oily Sludge.

Dewaterability can be expressed as the increase in TS Content expressed as a percentage of TS (Pandey and Carney, 2003; Sperling, 2007). However, as explained in Section 5.3.1.a, it was not possible to achieved 105 °C. Therefore, for the quality and visual range of particles, it was best to use a centrifuge and heat. Also, in this study, there cannot be a guarantee that all of the water in the sludge had evaporated and achieved complete dryness because the sludge was dried on a petri dish that was heated by an 80 °C hotplate. However, as the heating was continued until the petri dish achieved constant weight, it can be taken that it was near to total dryness. Drying may not be uniform since the petri dish that was at the centre of the

hotplate would get a higher heat than those nearer the outside boundary of the hotplate. Therefore temperature control could be poor.

5.3.1.d   Conclusion

The laboratory trials aimed to investigate the suitability of chemicals for the conditioning of Sureclean oily sludge to assist dewatering. From the results demonstrated in Section 5.3.1.c, it can be concluded that the best dewatering performance for the oily sludge was to condition with PEI and Lignite of 17.1 %. However, based on these preliminary results, the overall performance of each chemical was not good enough to convince Sureclean to embark on chemical treatment for oily sludge. Therefore, additional work to verify the results is required to provide further recommendation to Sureclean.

5.4 Chapter Conclusion

Coagulation is the reaction where chemical destabilisation of particles occurs to form aggregation. The main purpose of chemical treatment is to remove suspended solids and heavy metals, thereby aiding in mechanical separation for sludge dewatering. Laboratory and field trials were conducted to study the suitability and feasibility of chemical treatment towards Sureclean waste streams. The first laboratory trial was to investigate the effectiveness of four different coagulants on the removal efficiencies in heavy metals and TPH. The removal efficiencies of two commercial coagulants were also investigated under different pH conditions. It was concluded that without pH adjustment, the FloQuat and FloPam combined treatment had the highest total heavy metal removal, whereas $FeCl_3$ had the highest oil removal efficiency. However, at pH 7 the oil and heavy metal removal efficiency were almost as effective as adding FloQuat and FloPam alone. A field trial was also conducted for Sureclean interceptor effluent using a FloQuat and FloPam combined treatment with two different FloQuat dosages. The performance of 20 ppm FloQuat out performed by the 15 ppm of FloQuat in TPH, BOD, COD and TSS removal.

In the second trial, the effectiveness of chemical conditioning towards dewatering of Sureclean oily sludge was investigated. Four chemical compounds were investigated as potential chemical conditioners for Sureclean oily sludge: lignite, fly ash, PEI and FloQuat R100. However, it was not possible to heat the sludge samples to 105 °C in a furnace; a petri dish heated on an 80 °C hotplate was used instead to dry the sludge until the samples achieved complete dryness. Drying may not be consistent as it depends on the location of petri dish relative to the heat source on the hotplate and therefore temperature control could be poor. Based on the preliminary results, it can be concluded that the best dewatering performance for the oily sludge was conditioning with PEI and Lignite. However, the overall performance was not noteworthy enough for Sureclean to proceed further in this direction.

Chapter 6 Chemical Treatment of Sewage Sludge

Sureclean is one of the main septic tank and sewage wastes removal companies in the Scottish Highlands. Septic tank is a watertight container that receives sewage waste and separates the solid component from liquid through gravity (Massey 1998). The liquid is then drained from the tank through an outlet pipe; whereas the settled solid, also known as septic tank sludge, is required to be emptied from the septic tank at regular intervals (Olsson 2011). Sureclean collected the wastes from a number of sites, which were stored onsite and subsequently disposed of to a third party with no prior treatment. These waste streams were characterised by their high solid and BOD content; and it was not possible to use Sureclean existing facilities to treat these waste streams. Sureclean saw this as a significant opportunity for cost saving to their business in septic tank and sewage wastes, as well as an opportunity to improve the wastewater treatment facilities in the WTS. There was also an opportunity for the new wastewater treatment plant (WWTP) to be used at client's site. Therefore, the main treatment criteria for the WWTP were: the system would be small enough to be transported to a client's site if desired and therefore it would be relatively mobile, although it would primarily be located at their Alness facility. In addition, the WWTP would require minimal operator input and maintenance to allow it to be left unsupervised at a client's site if required.

6.1 Sureclean Wastewater Profile

In order to choose a suitable WWTP, an initial study was conducted to evaluate Sureclean wastewater profile that included wastewater analysis and volume analysis. The volume of incoming wastes was collected everyday between the months of June to October 2007. Wastewater samples were also collected from Sureclean tanker upon arrival to the WTS. The samples were then analysed by Alcontrol Limited using the methods presented in Section 4.3.2.b. There were two main types of biological

wastewater which the new WWTP would treat. Firstly, there was waste from septic tanks, to be known as Waste Stream 1 and secondly, there was a combination of sewage and grey water, to be known as Waste Stream 2. The proposed new WWTP should be able to treat these two waste streams to within Sureclean discharge consent and the Alness WTS PPC license.

Analysis of Waste streams 1 and 2 can be seen in Figure 6-1 and Figure 6-2. The wastes were collected from Sureclean tankers. Upon their arrival to the WTS, a sample was collected immediately. This was because the transportation caused the waste to be agitated within the tanks and therefore it was hoped that a representative sample could be obtained. A comparison of the analysis results for Waste Stream 1 and 2 can be seen in Figure 6-1. The result shows that Waste Stream 1 was significantly higher than Waste Stream 2 in BOD, COD and TSS. Visual inspection showed that the consistency of Waste Stream 1 was thicker and denser than Waste Stream 2. It can also be seen from Figure 6-1 that BOD, COD and TSS level in Waste Stream 1 were above the Sureclean discharge limits. Therefore treatment would be required before the waste could be discharged to the public sewer. As for Waste Stream 2, all of the parameters were within Sureclean discharge consent; therefore the wastewater could be discharged to the public sewer without prior treatment. From Figure 6-2, it can be seen that both waste streams were within Sureclean total heavy metals discharge limit. However, the large error bar for Waste Stream 1 may suggest that the result is not reproducible. Detailed analysis results for Waste Stream 1 and 2 could be found in Appendix 3.
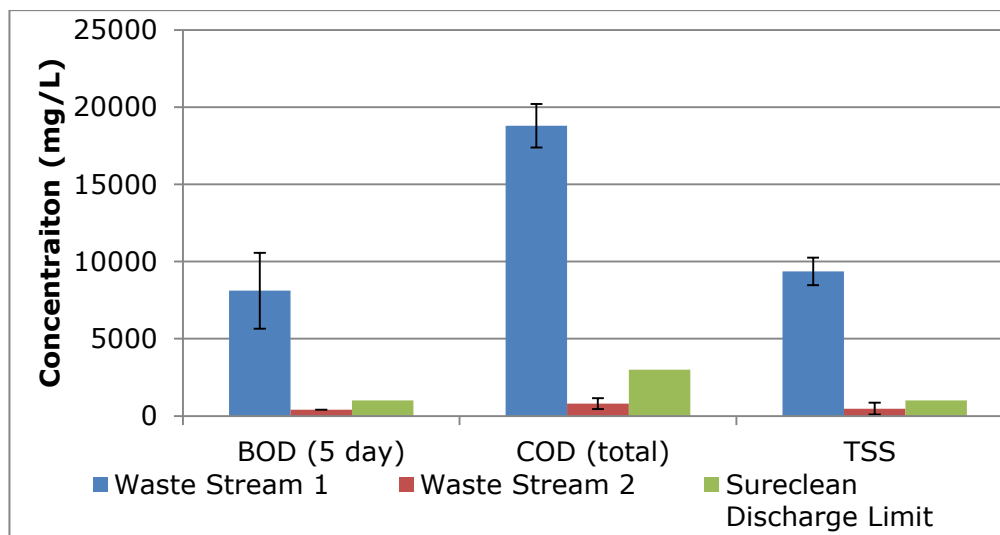
Figure 6-1 Biological Wastewater analysis results: A comparison of BOD, COD and TSS results of Waste stream 1 and 2 to Sureclean discharge limit.
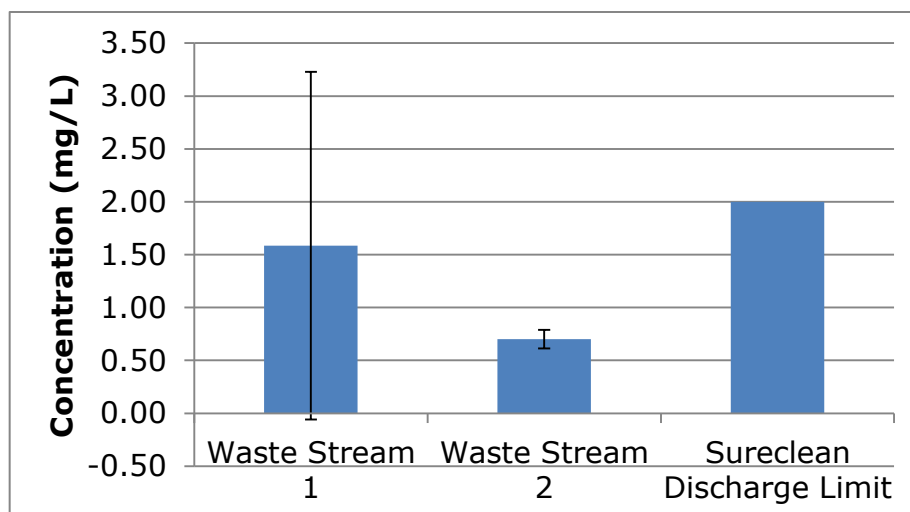
Figure 6-2 Biological Wastewater analysis results: A comparison of total heavy metal results of Waste stream 1 and 2 to Sureclean discharge limit.

From Figure 6-3 and Figure 6-4, it can be seen that the quantity of Sureclean incoming wastewater fluctuated daily. The maximum volume that Sureclean received in one day was 188.8 m^3 i.e. 58 m^3 of Waste Stream 1 and 130.8 m^3 of Waste Stream 2. In Figure 6-3, it can be seen that the highest wastewater intake was in August 2007 with higher quantity of

Waste Stream 2 than Waste Stream 1. Large error bar in Figure 6-3 also showed that the results are consistent every month.
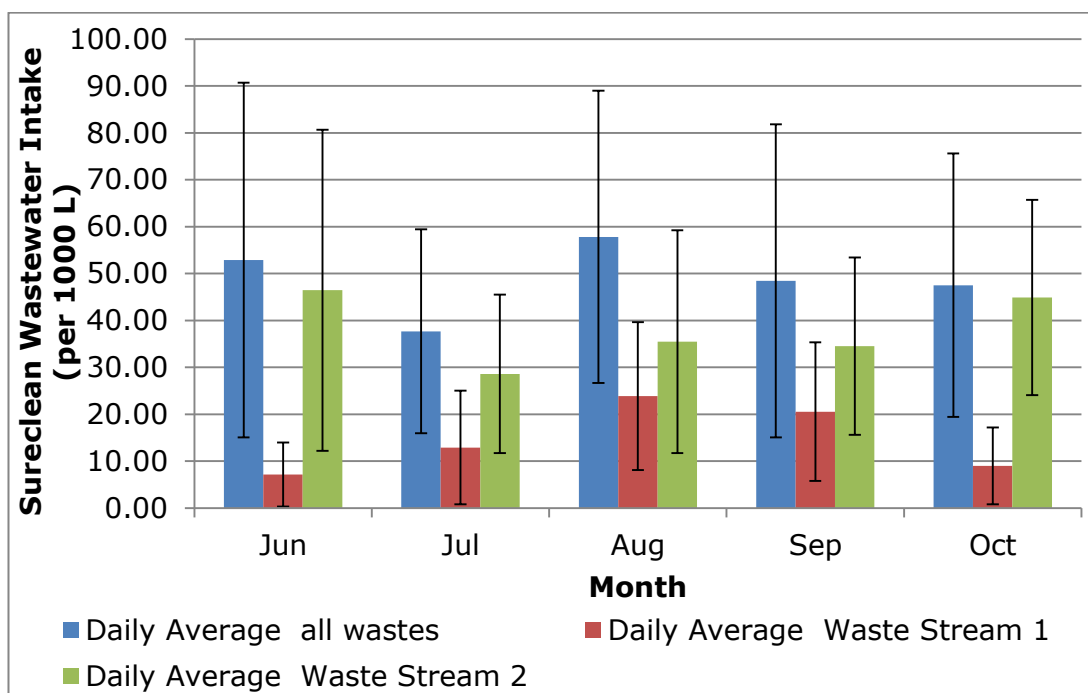
Figure 6-3 Wastewater Profile: Daily Average of Waste Stream 1 and 2 from June to October 2007.
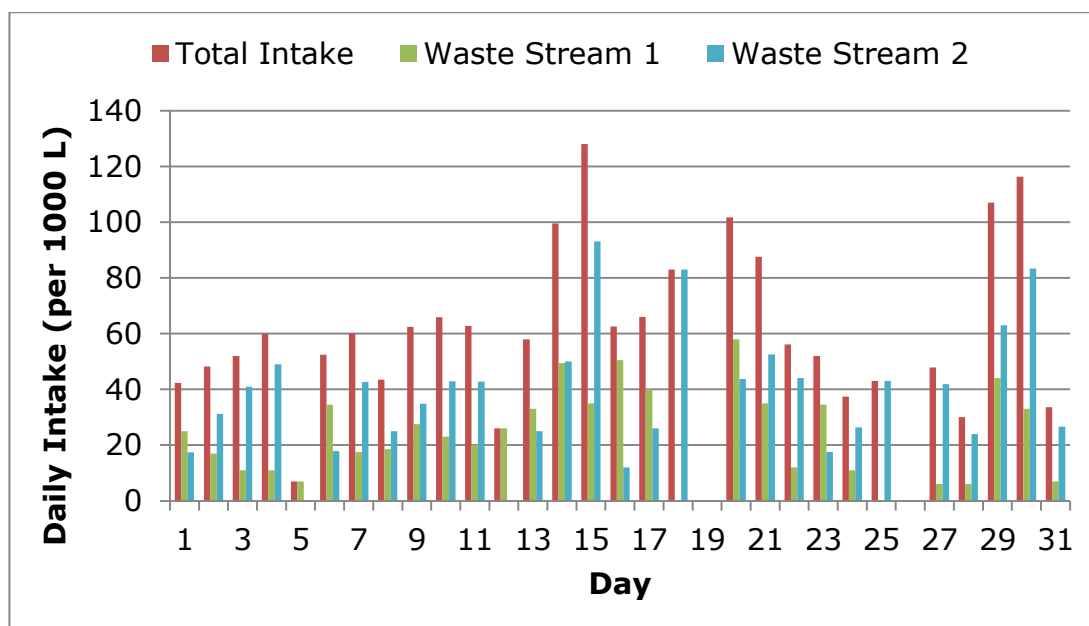
Figure 6-4 Wastewater Profile: Daily Average of Waste Stream 1 and 2 in the month of August 2007.

In conclusion, Waste Stream 1 was more heavily contaminated than Waste Stream 2 with respect to every parameter analysed. In general, Waste Stream 2 was larger in volume as compared to Waste Stream 1; however, Waste Stream 2 may not require further treatment prior to discharge.

6.2 Technology Selection

For Waste Stream 1 to meet the discharge consent, the levels of BOD, COD and TSS should be reduced significantly. The main treatment aims for sewage sludge are to reduce its water content and reduce pollutant level (Barceló and Petrović 2011). Typical sewage sludge treatment included biological, chemical or physical treatment (Cheremisinoff 2002). Biological treatment can be divided into aerobic (with the presence of oxygen) or anaerobic (without the presence of oxygen). Anaerobic treatment systems are thought to be a more favourable method for treating waste with high organic content (BOD > 1000 mg/L) and has smaller footprint, in comparison to an aerobic treatment system (Şengil and Özacar 2006; Akunna and Clark 2000). However, both biological treatment systems in general do not favour fluctuation of waste streams (Wang et al. 2006) and could be affected by certain heavy metals present in industrial wastewater (Arceivala and Asolekar 2007).

Physical or mechanical treatment of sewage sludge is usually employed after chemical treatment to increase the solid concentration by removing the water content (Diaper et al. 2001). These treatment units include centrifuge, dissolved air flotation (DAF) thickener and filter press (Williams 2007).

Chemical treatment or chemical conditioning of sewage sludge can be used to improve dewatering property of the sludge (Diaper et al. 2001; Chen et al. 2010). Flocculation of sewage sludge with chemicals such as polyelectrolytes, lime, organic polymers, $FeCl_3$, and physical conditioners (also known as skeleton builders) such as carbon-based materials and fly ash have shown to improved dewatering (Qi, Thapa and Hoadley 2011;

Thapa et al. 2009; Böhm and Kulicke 1997). However, Williams (2007) stated that prior to physical treatment for dewatering; sludge is usually condition with cationic polymers (typically a PAM) or occasionally with two polymers applied in succession, or an inorganic chemical such as Alum followed by a polymer. Cationic polymers such as a PAM are widely used in sludge dewatering because most sludge carried negative charge (Albertson 1991).

There were a few challenges faced by Sureclean in the selection of the correct treatment process; one of the main requirements was that the new WWTP needed to have mobility where a truck could easily lift the unit without any disruption to the system. One major concern regarding the overall design of the new WWTP was the volume fluctuation of the incoming waste, the volume differences as seen in Figure 6-3 and Figure 6-4 could be vast. Moreover, there were no permanent contractual agreements with clients and therefore the volume of the waste could differ from current estimation. There were other issues such as space constraints and the PPC license in Alness WTS. Odour could be a potential concern as the WTS is located near offices and houses. Cost and budget were also main constrains to this study.

In August 2007, a technologies evaluation was conducted involving liaisons with wastewater suppliers to obtain prices and specifications for different biological wastewater treatment plants. Most suppliers showed concern about the fluctuation of incoming waste and the high level of BOD and COD of Waste Stream 1 and therefore Sureclean was uncertain if a biological treatment system could be able to handle the waste streams. In November 2007, Mabbett & Associate (M & A), (an environmental consultancy firm) was appointed by Sureclean to conduct an engineering feasibility study. In March 2008, M & A concluded that due to all the constraints discussed earlier, they provided a feasibility report that suggested chemical/physical treatment that would cost approximately £200,000 (Mabbett & Associate Limited 2008). M & A suggested that Sureclean install a wastewater screening process, followed by raw wastewater equalisation to ensure

control of flow could be achieved. The raw wastewater would then be dosed with a chemical from a chemical dosing tank (with a capacity of approximately 3 m^3). The dosed sludge would be further treated by a DAF process to remove suspended solids, oils and other contaminants via the use of air bubble flotation (with a capacity of 26 m^3). M & A stated that they would require a laboratory trial to further ascertain the chemical and the process involved.

To evaluate the feasibility of the treatment system for Sureclean, a cost benefit analysis was conducted. At the time this study was undertaken, Sureclean transported the sewage waste to Aberdeen for final disposal. However, with a capital outlay of £200,000 and an annual operating cost in the region of £60,000, a cost benefit analysis was conducted as seen in Table 6-1 and it can be seen that it would require 19 months to recover the cost of the WWTP.

Profits		
Clients	Commercial	Private household
Effluent charges to client (per m^3)	£39.00	£24.99
Estimated future average volume per day	10	10
Turnover per year	£101,400.00	£64,974.00
Total Turnover		£166,374.00

Cost			
Disposal to third party		**In-house treatment**	
Cost of delivering waste to Aberdeen (per trip)	£220.00	Capital cost of WWTP	£200,000.00
Annual delivery cost (Estimated two journey per week)	£22,880.00	operating cost per year	£60,000
Cost of sewage disposal (per m^3)	£28.50		
Annual cost of sewage disposal	£148,200.00		
Total cost	£171,080.00	Total cost	£260,000.00

Payback	
Annual Turnover	£166,374.00
Total cost of a WWTP- year 1	£260,000.00
Payback time (months)	19

Table 6-1 shows the cost-benefit analysis of installing a WWTP for Sureclean sewage waste.

6.3 Prototype Field Trial

In September 2008, M & A submitted a scope of work letter to help Sureclean move the project forward. However the recession in 2009 meant that Sureclean would have to consider a cheaper alternative. Sureclean decided to design a sewage sludge dewatering prototype using chemical treatment based on evidences from literature searches conducted in Section 6.2.

A prototype plant with a simple chemical dosing of wastewater (septic tank sludge) was investigated. The sludge dewatering container consisted of a 5 m³ offshore open top mud skips that were modified to equip the inside with filters to dewater the flocculated sludge. The main objective of this trial was to evaluate the effectiveness of chemical dosing of septic tank waste and to evaluate the feasibility of in-house built treatment system.

6.3.1   Material and Methodology

The wastewater used in this pilot plant trial was obtained from Sureclean clients. The waste was delivered by a Sureclean tanker. The chemical used in this study was FloPam DW2160, a cationic PAM supplied by SNF. The chemical was diluted using 1.3 L of neat product with 200 L of tap water in a 45 gallon drum. 2000 L of septic tank waste from a caravan camping site was treated with the diluted chemical. A cleaned IBC was used as a container to enable maximum waste and chemical contact before the coagulated waste was allowed to be separated. As seen in Figure 6-5, the waste was pumped straight from the tanker into the "holding tank". 2 inch air driven Wilden pumps were used to pump the chemicals and coagulated waste. The dewatering unit consisted of a wooden frame with mounted membrane bag (AUTOWAY 90 Membrane) that was installed inside a 5 tonne offshore open top mud skip. The membrane acts as a sieve to retain the coagulated solids, allowing only liquid to drain. The frame had a 150 mm clearance gap between the walls and floor of the skip. Tarpaulin was used as the lid to prevent odours escaping. The configuration of the pilot plant can be seen in Figure 6-6.
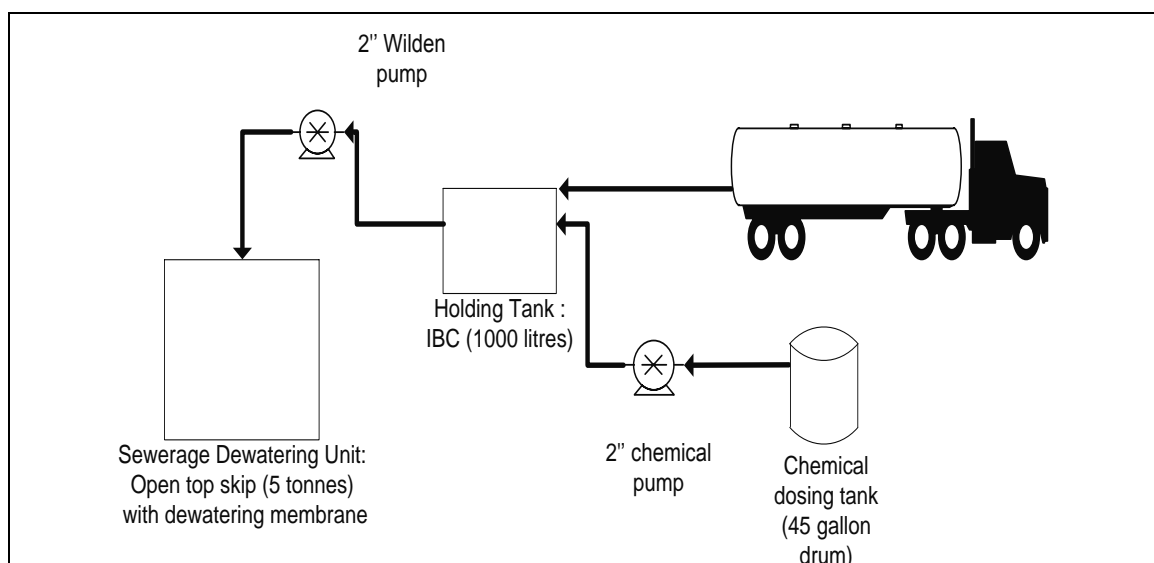
Figure 6-5 shows a schematic diagram of the prototype field trial.
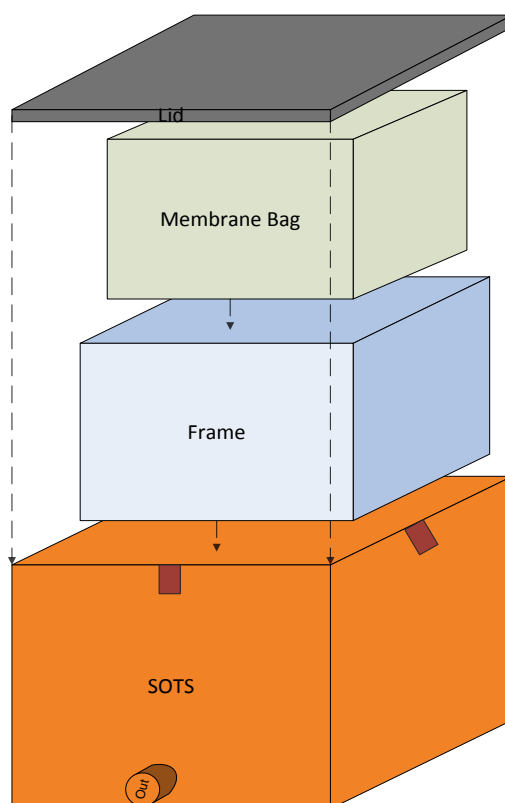
Figure 6-6 Configuration of the prototype sewage sludge dewatering unit (SOTS: Sureclean Open Top Skip).

6.3.2   Results and Discussion

Unfortunately, no sample analysis was conducted due to time constrains. However, visual inspection showed that coagulation of the septic tank wastewater could be observed instantaneously. The sludge was retained on the membrane and the separated effluent appeared to be clear (as seen in Figure 6-7). However, the amount of chemical used appeared to be too high as clumps of chemical was found underneath the holding tank. This may also suggests that contact time between the chemical and the waste may not be sufficient due to poor agitation.
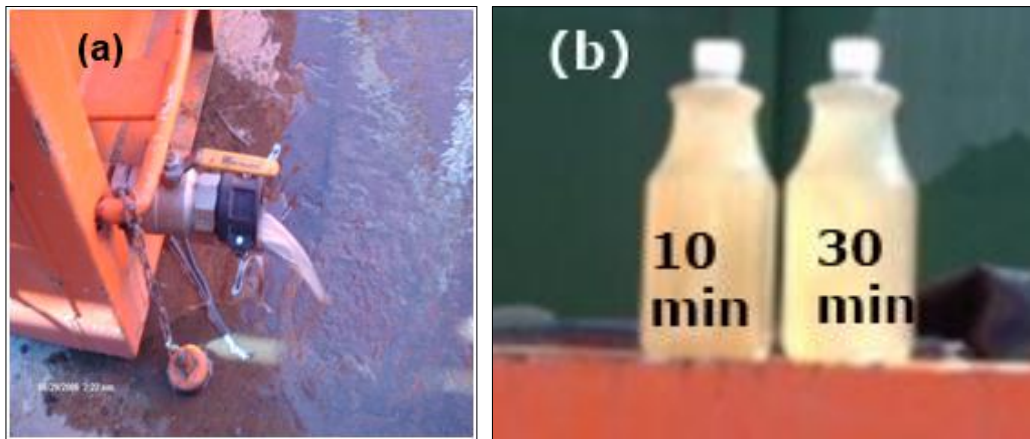
Figure 6-7 (a) The discharged effluent appeared to be clear; (b) Left bottle: effluent collected after 10 minutes and Right bottle: effluent collected after 30 minutes.

6.3.3   Conclusion

Septic tank waste disposal has historically contributed significant income generation to Sureclean Alness. Therefore it is business critical that a robust and cost effective treatment system is in place. The prototype trial was conducted using an offshore open top mud skip with a wooden supported filtration frame. However, due to limitation in time, no analysis was conducted.

Based on this preliminary observation, it was suggested that a more durable and less elastic material for the filtration membrane would be used for

further treatment system development. More support to the membrane would be required especially the central weight bearer, this could be done by making a mesh frame to support the membrane. An evaluation on the feasibility of using the Sureclean storage tank would also be required.

6.4 Sureclean Sludge Separation Treatment System (SSSTS) Field Trial

Based on the preliminary results shown in Section 6.3, Sureclean decided to design and build a sewage dewatering unit called the Sureclean Sludge Separation Treatment System (SSSTS). The technology is a simple chemical dosing of wastewater (septic tank sludge) to give rise to a liquid and solid phase. The sludge dewatering container consisted of a closed container, equipped inside with filtration screens to drain and dewater the flocculated sludge. Dewatering of sludge is achieved by injecting polymer into the sludge through the pumping unit. Using the dosing pump the correct amount of polymer is adjusted in order to achieve the best sludge flocculation possible. As flocculation occurs the solids draw together and separate from the water. After separation the water flows through a special set of filter nets inside the sludge dewatering container and out of drain ports located on the side, or at the front of the sludge dewatering container. Solids remain inside the container until accumulation requires dumping and disposal.

6.4.1 Specification

The main dewatering unit was a modification of an Offshore Storage Tank (OFT). The tank specification can be seen in Table 6-2. The front of the tank was fitted with a stainless steel door fitted with a 3 inch diameter ball valve as the unit inlet; each side of the tank adjacent to the door was fitted with an outlet valve (as seen in Figure 6-8). The length of the inner tank was mounted with screen mesh supported by stainless steel panels (Figure 6-9). The screen mesh (purchased from Boddingtons Limited) which acts as sludge retention element was made from polypropylene with a thickness of 0.482 mm and mesh count of 90 strands per 100 mm.

Capacity	
Tank Capacity	23,845 L
Fluid Design Density	2 kg/L
Dimension	
Length x Width x Height	4,440mm x 2,700mm x 5,475mm
Area	12 m^2
Weight	
Dry	7,800 kg
Operating	55,500 kg

Table 6-2 demonstrates the technical specification of the offshore storage tank (before modification was conducted).
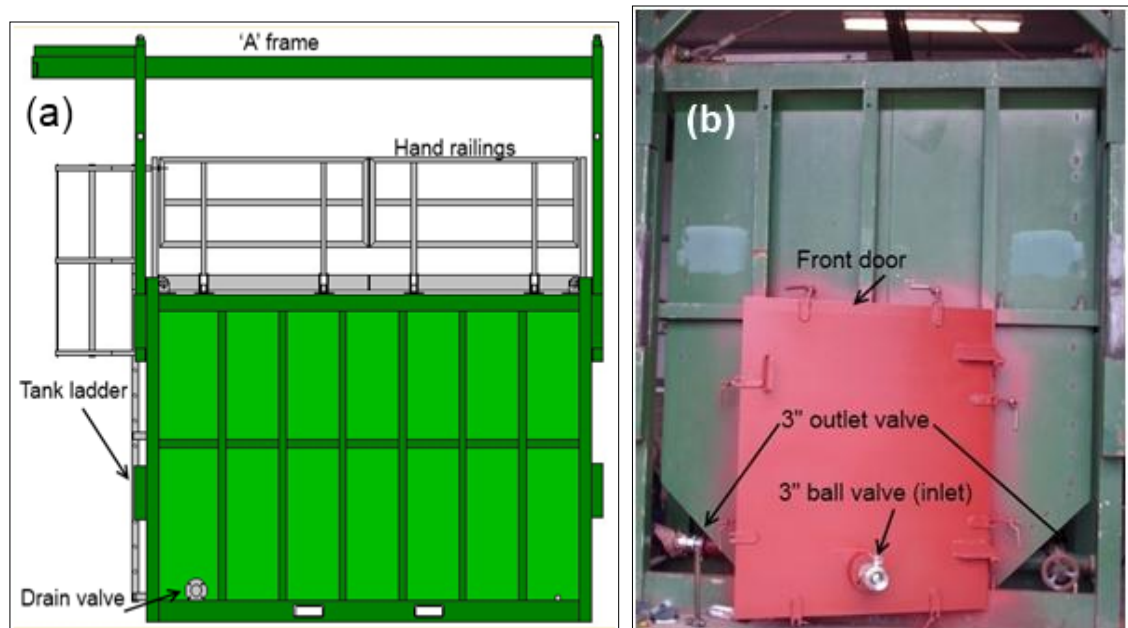
Figure 6-8 (a) Picture showing the side view of the Sureclean Sludge Separation Treatment System (SSSTS); (b) Front view of SSSTS.
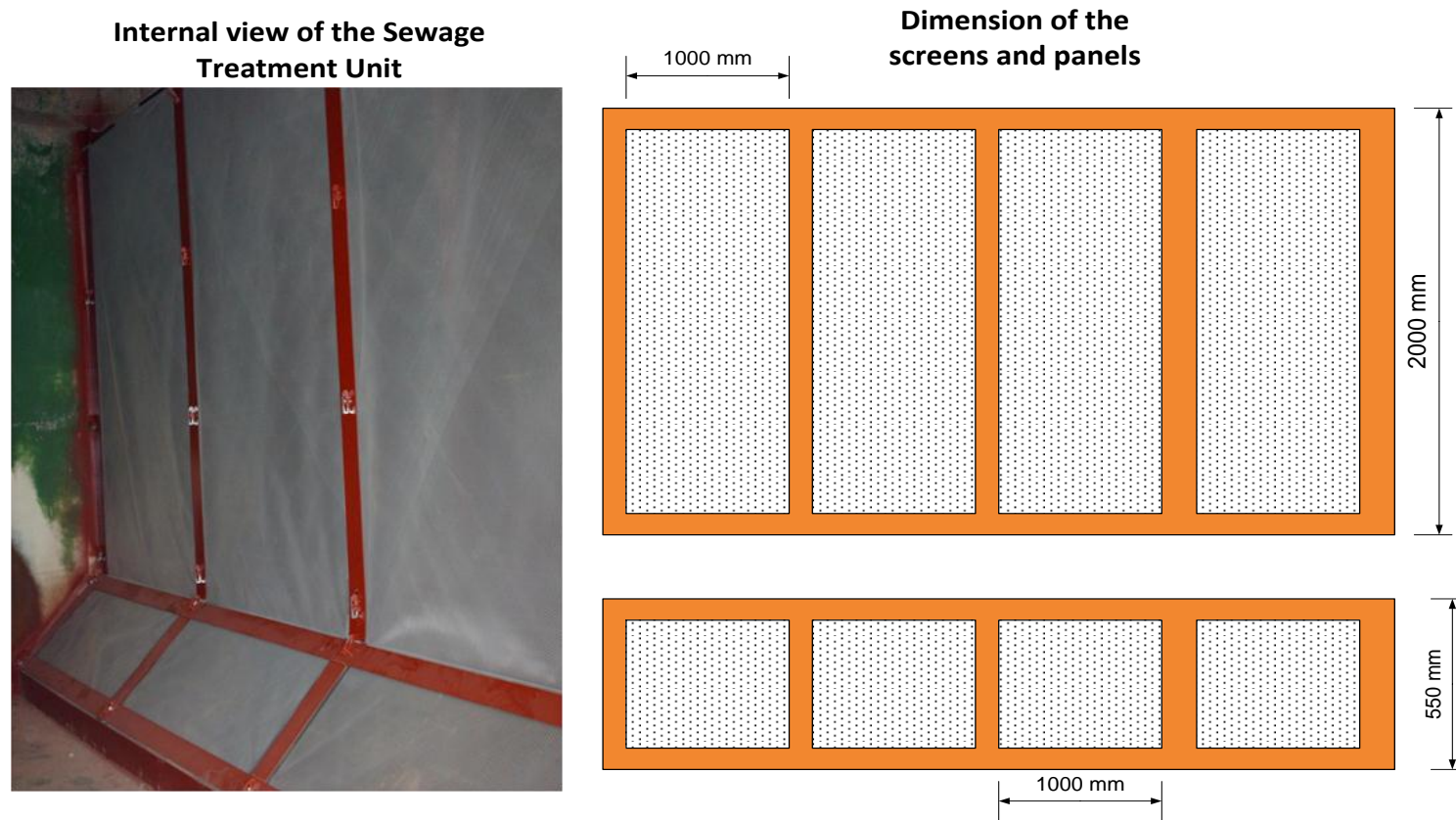
Figure 6-9 shows the internal component of the sewage treatment unit.

6.4.2 Material and Methodology

This section describes the methodology to evaluate the effectiveness of the newly fabricated sewage treatment unit in the treatment of septic tank waste with volumes of up to 15,000 L. The waste samples were collected before and after chemical treatment, and these samples were subsequently sent to STL Limited for further analysis (heavy metals, TSS, COD and BOD) as seen in Section 5.2.2.a. Due to cost and time constrains within the company, only one set of data was obtained.

Three trials took place in the Alness transfer station. The waste was collected from household septic tanks. The set-up of the trial was as per Figure 6-10 and Figure 6-11. Flopam DW 2160 (supplied by SNF Limited) was used as the dosing chemical. The chemical was diluted to 0.5 % by mixing 1.5 litres of chemical to 300 litres of water in an IBC as recommended by the chemical supplier. The chemical was mixed with water vortex from a water hose and was pumped, using a 2 inch Wilden pump, into a T piece, where the chemical and sewage mixed. The SSSTS inlet was fitted with a T-connection for the chemical to be pumped (via a 2 inch chemical Wilden pump) and the incoming septic tank waste discharged from the tanker. The tanker discharge valve was opened to match the dosing rate or a maximum of 50 % of the valve capacity. The inlet valve was opened fully to allow the tanker to discharge into the SSSTS. The outlet valve was then shut until all of the waste had been loaded into the unit. Treated effluent could be seen from the outlet valve after an initial wait of approximately 10 minutes. A 2 litre sample of the treated effluent was collected for analysis by STL Limited.
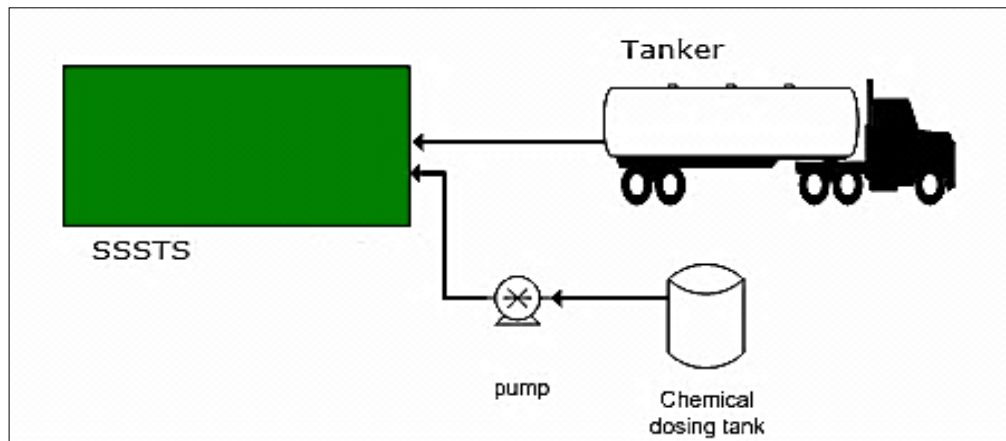
Figure 6-10 SSSTS Field Trial: Schematic representation of the Trial set up.
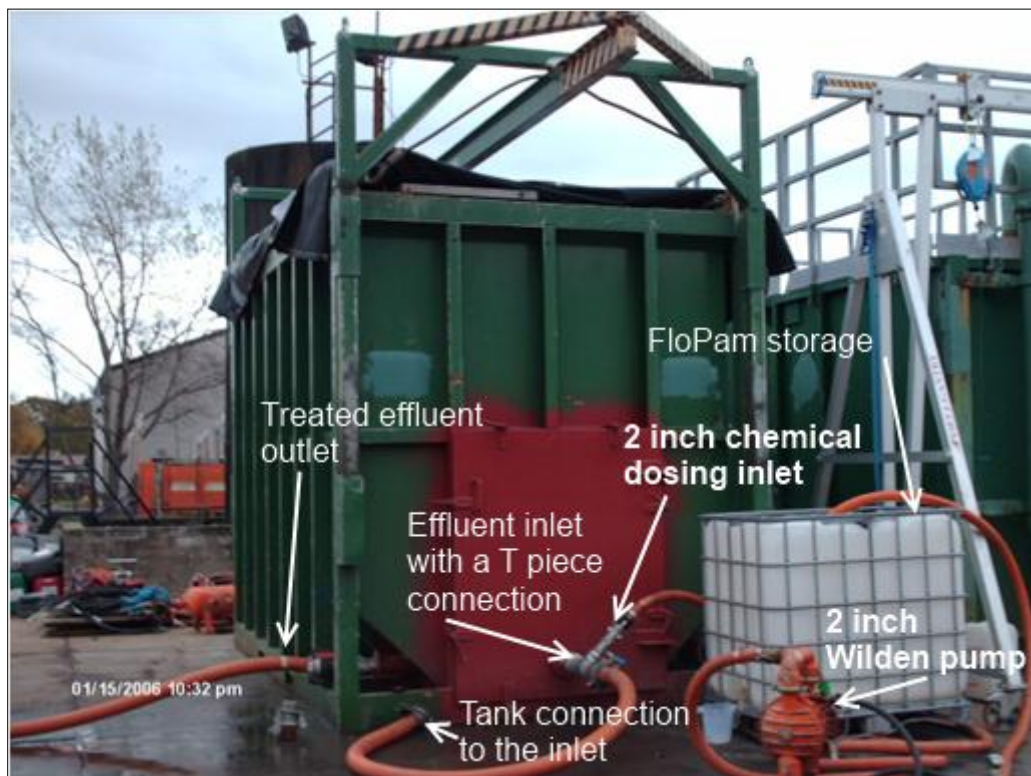
Figure 6-11 SSSTS Field Trial: Photo of the Trial set up.

6.4.3   Results and Discussion

Visual observations showed that the incoming wastewater was concentrated and thick; the treated effluent was clear with slight discolouration and had very little solids, which demonstrated that the SSSTS was able to contain most of the solid materials. It can be seen from Figure 6-12 that a volume

reduction was observed for all three trials. The average percentage reduction was 52.6 % with a standard deviation (SD) of 17.7 %. The fluctuation of volume reduced may indicate that the chemical dosing was not always effective.
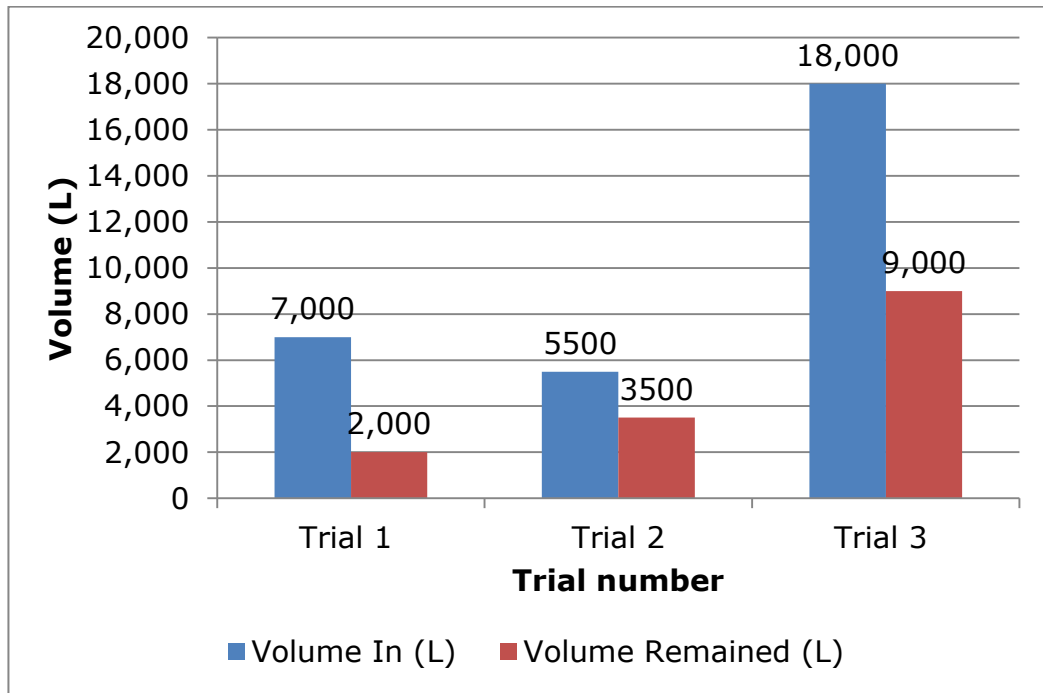
Figure 6-12 SSSTS field trial results: Volume reduction of the septic tank waste.

From Figure 6-13, it can be seen that TSS, BOD and COD were reduced. The SD for all parameters was small, which showed that the results were reproducible. The percentage reduction of the TSS, BOD and COD were 94.8, 94.4 and 88.6 % respectively. Not all of the treated effluent complied with Sureclean discharge consent; therefore further treatment such as filtration should be employed to reduce the pollutant loads.
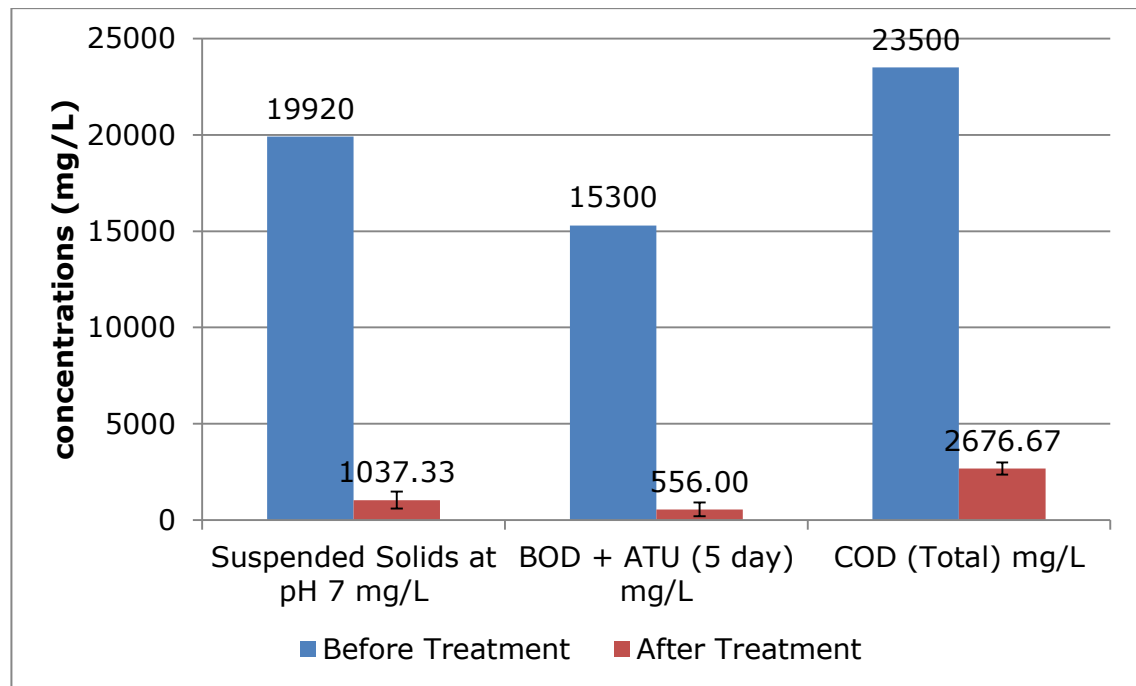
Figure 6-13 SSSTS field trial results: incoming and treated waste parameters results.

6.4.4   Conclusion and Future Work

To secure Sureclean business in septic tank waste disposal in the Highlands region, it is inevitable for Sureclean to have a treatment of septic tank waste in place. As seen in the field trials, SSSTS could reduce septic tank waste volume by up to 52.6 %, as well as reducing the pollutant load of the waste streams. However to enable the system to be more effective, a future upgrade of the whole process would be required as seen in Figure 6-14. Other units such as a sludge screen, an odour control system as well as a dosing unit could improve the overall treatment process.

According to the European Union (2010), sewage sludge contains valuable materials that would benefit the agriculture sector. The Sewage Sludge Directive (86/278/EEC) stated that the sewage sludge must be treated before applying to land (with the exception that if it is injected or worked into the soil). According to the same Directive, treated sludge is defined as sludge that have undergone "biological, chemical or heat treatment, long-term storage or any other appropriate process so as significantly to reduce

its fermentability and the health hazards resulting from its use". Therefore, the conditioned sludge could be further treated by either mechanically, chemically or biologically. The choice of treatment for Sureclean would depend on availability of space, volume of sludge and the cost-benefit associated with the treatment.
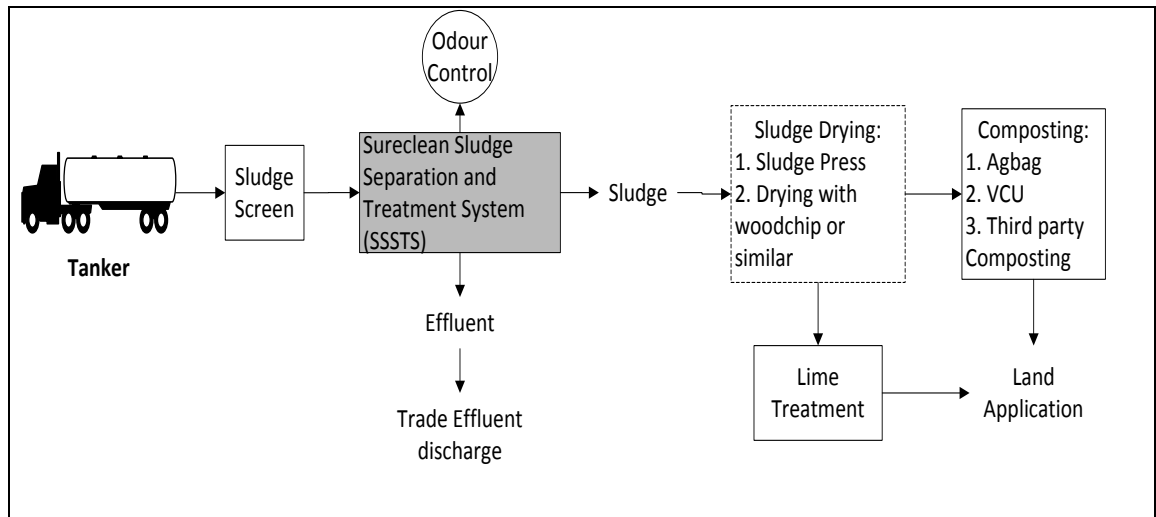
Figure 6-14 A schematic representation of a complete process for the treatment of Sureclean septic tank waste

Sludge Screen for large solid:

Screening removes objects such as rags, paper, plastics or any large objects to prevent damage and clogging of downstream equipment and piping. This enables the treated sludge to have higher recyclable value by retaining valuable nutrients. Bar screen for large objects as discussed in Section 4.1.1.a could be employed to remove those large objects.

Odour Control

According to Arundel (2000), filtrate produced by dewatering of organic sludge could be very odorous. Identification of potential odour sources at a wastewater treatment plant is important as these odours could cause serious complaints from local residents or offices in the vicinity of wastewater treatment plants. As a result, odour control has become a key

issue facing wastewater treatment plant management (Stuetz 2001; Water Environment Federation, and American Society Of Civil Engineers, 1995). There are a few technologies in odour abatement such as AC filtration, bio-filters, scrubbers and ozone treatment (Adams et al. 2003) that would reduce odour release.

Chemical Dosing System for Sewage sludge

As described in Section 5.1.3, rapid mixing helps disperse the chemical throughout the mixing tank and it is considered to be one of the most important stages since particle destabilisation occurs at this stage. Therefore it is essential to optimise the dosing and mixing system of SSSTS with the incoming waste. A chemical dosing system that consisted of a sludge pump and a chemical dosing pump for transporting the sludge and adjusting the amount of polymer respectively (refer Figure 6-15). The polymer amount can be adjusted by means of a dosing pump. Polymer addition is optional before or after the sludge pump. In addition to this it is possible to dilute the polymer mixture with water through an injector.

Figure 6-15 Chemical dosing system with cyclone sludge pump (unit above is owned by H&E Trotter, Cumbria).

Chapter 7   Physico-Chemical Treatment

7.1   Introduction

Physico-chemical treatment involved the combined chemical and physical processes in the treatment system. Sureclean has always been striving to use innovative and unconventional technologies in their waste treatment system. In this chapter, two wastewater treatment methods shall be discussed and investigated: EC and Semiconductor Photocatalysis.

7.2   Electrocoagulation (EC)

EC is a wastewater treatment whereby sacrificial anodes corrode to release active metal ions that act as coagulants for colloid particles with simultaneous hydroxyl ions and hydrogen gas released at the cathode (Holt, Barton and Mitchell 2005; Moreno et al. 2009). Electrolytic oxidation of a metal anode such as aluminium or iron could generate coagulants in situ in the EC system with simultaneous formation of hydroxyl ions and H_2 gas at the cathode (Arvanitoyannis 2008). The mechanism of an EC system in the destabilisation of colloid particles is similar to chemical coagulation using metal hydroxides (refer to Chapter 5).

EC was reported for the use of sewage treatment in London in the nineteenth century, and the first recorded patent for an EC system was found in the US in 1906 that treated bilge water from ships (Moreno et al. 2009; Vik et al. 1984). Commercialisation of EC before the late twentieth century had been hindered by high capital cost and operational complexities (Gu et al. 2009). Moreover, the EC designer often used EC as a chemical dosing system but failed to make use of the H_2 gas produced in the EC process (Gu et al. 2009). According to Vik et al. (1984), a boost in EC sold in the last decade of the twentieth century showed that there were renewed interests in the use of EC systems. One of the major advances in the EC development was in minimising electrical usage and improved effluent

throughput (Vik et al. 1984). The EC system could be a robust and compact system (Mollah et al. 2004).

EC has been used for the remediation of wastewater containing heavy metals, non-metals, TSS, organic compounds, COD and BOD (Moreno et al. 2009). Research and applications of wastewater and water treatment using EC have been used in various industries that included potable water (Holt, Barton and Mitchell 2005), food and drink (Barrera-Díaz et al. 2006; Kannan, Karthikeyan and Tamilselvan 2006), textile (Kobya et al. 2006; Can et al. 2006) and petrochemical (Abdelwahab, Amin and El-Ashtoukhy 2009) industries. EC has also been reported extensively for the treatment of oily wastewater, in particular oil-in-water-emulsion (Cañizares et al. 2007a; Asselin et al. 2007; Yang 2007). Sureclean could therefore exploit the wide applications of EC treatment to expand its service market.

Sureclean discharged approximately daily average of 12,231 litres of trade effluent into public sewer between 2007 and 2008, with a discharge limit of 100 mg/L of TPH; therefore, treatment of oily wastewater could be an important technology for Sureclean to optimise its oily waste treatment. Current treatments for oil-in-water emulsion include chemical treatment, DAF and filtration and biological treatment (Wang et al. 2004; Cañizares et al. 2007a). Asselin et al. (2007) reported that EC removed up to 95 % of oil and grease removal in oily bilge water. Cañizares et al. (2008) reported that the EC and the conventional chemical coagulation using aluminium salts could be used to degrade oil-in-water emulsions. Yang (2007) reported that EC demulsified oily wastewater and removed up to 96 % of wastewater turbidity in 4 minutes. Therefore this shows that EC system could be a rapid and effective system in the treatment of oily wastewater.

7.2.1   Mechanisms

Mollah et al. (2004) stated that there are three main stages in the EC systems. First of all metal ions are formed by electrolytic oxidation of the sacrificial electrode (the ions formed depend on the electrode used). The

metal ions could then destabilise colloid particles including oil emulsion and induce aggregation to form flocs. Equation 7-1 and Equation 7-2 show the electrochemical reaction in the anode; Equation 7-3 and Equation 7-4 show the reaction on the cathode. It can be seen that when a direct current is supplied to water or wastewater through electrodes (cathode and anode), water molecules are broken down into oxygen (O_2) and H_2 gas (Yang 2007). The most common materials to be used as electrodes are aluminium (Al) and iron (Fe) due to abundance, low cost and proven effectiveness (Mollah et al. 2001; Larue et al. 2003).

The main reactions at the anode:

$$M_{(s)} \rightarrow M_{(aq)}^{n+} + ne^- \hspace{3cm} \textbf{\textit{Equation 7-1}}$$

$$2H_2O_{(l)} \rightarrow 4H^+_{(aq)} + O_{2\,(g)} + 4e^- \hspace{2cm} \textbf{\textit{Equation 7-2}}$$

The main reactions at the cathode:

$$M_{(aq)}^{n+} + ne^- \rightarrow M_{(s)} \hspace{3cm} \textbf{\textit{Equation 7-3}}$$

$$2H_2O_{(l)} + 2e^- \rightarrow H_{2\,(g)} + 2OH^- \hspace{2cm} \textbf{\textit{Equation 7-4}}$$

However, EC has been known to be a complex process (Holt et al. 2002; Kobya, Can and Bayramoglu 2003; Moreno-Casillas et al. 2007). The chemical reactions above had been described as the primary or the main reactions that occur in an EC reactor. However, there are arrays of secondary reactions as shown in Figure 7-1, which operate simultaneously to eliminate pollutants (Holt et al. 2002).
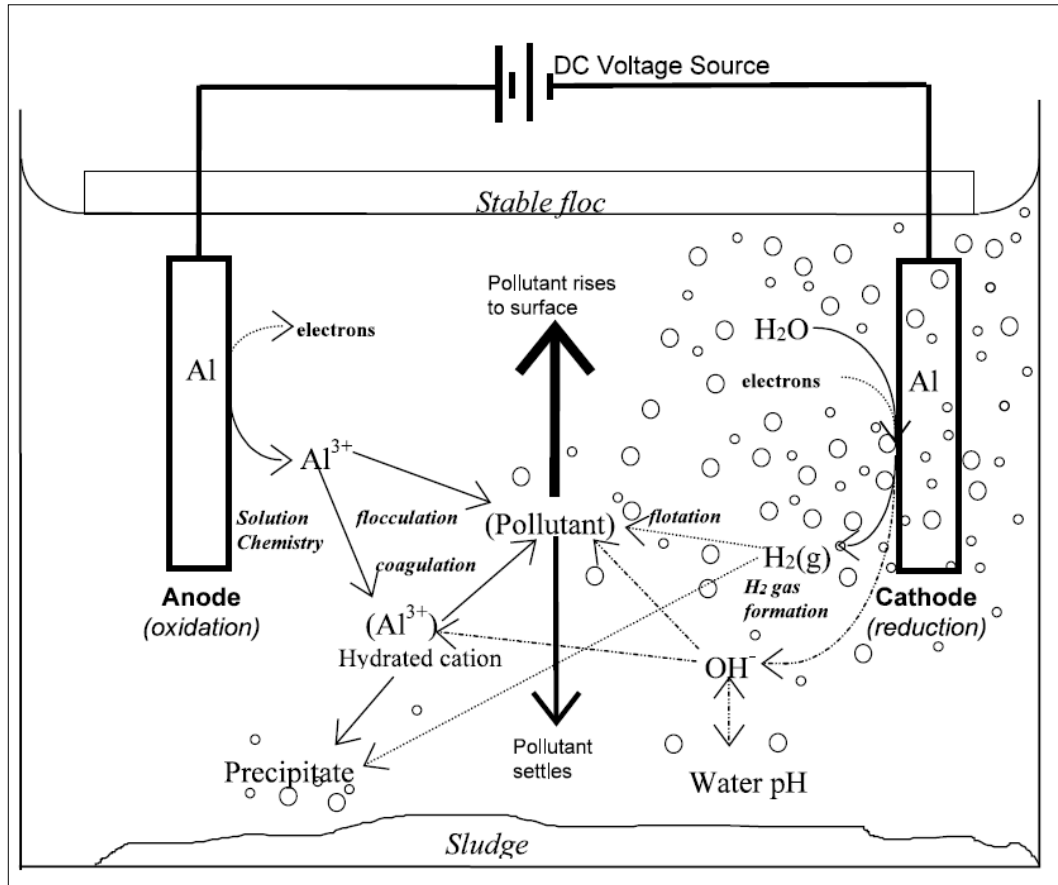
Figure 7-1 shows a schematic diagram of an Electrocoagulation cell (Holt et al. 2002).

The performance of EC reactions could be affected by several factors which include current density, pH, and conductivity. EC coagulating performance depends on the concentration of metal ions released during the electrochemical process. The concentration of metal ions in turn depends on the amount of electricity that is passed through the sacrificial electrode. The amount of metal dissolved could be related to current density by Faraday's law as shown in Equation 7-5.

$$m = \frac{itM}{zF}$$ ***Equation 7-5***

Where: m = mass of metal ion introduced; i = electrical current or current density; t = run time; M = relative molar mass of the electrode concerned; z = number of electrons transferred and F = Faraday's constant (96486 C/mol).

The power consumption P (W) and the operating cost of an EC reactor is controlled by the applied amperage I (A) or voltage U (V), and their relationship can be described in Equation 7-6 (Koren and Syversen 1995).

$$P = U \times I$$ **Equation 7-6**

The amount of gas released in the EC system depends on the current flowing across the electrodes (Koren and Syversen 1995). Chen, Chen, and Yue (2002) also stated that "The electrolysis voltage is one of the most important variables. It is strongly dependent on the current density, the conductivity of the water/wastewater to treat, the inter-electrode distance, and the surface state of electrodes."

Conductivity of the wastewater is one of the factors limiting the performance of an EC reactor. When the electrolytic conductivity is low, the current efficiency will decrease (Liu, Zhao and Qu 2007). Generally table salt or sodium chloride (NaCl) is added to increase conductivity (Wang, Pereira and Hung 2004). Therefore, EC is thought to be effective in the treatment of produced water or bilge water as it contains sea water. According to Chen (2004), "The effects of pH of water or wastewater on EC are reflected by the current efficiency as well as the solubility of metal hydroxides." The best removal results for Al and Fe plates were noted to be with an initial pH of near 7 (Chen 2004).

EC treatment is thought to have several advantages against chemical treatment including the fact that an EC system does not require chemicals. Rajeshwar and Ibanez (1997) stated that EC could be more effective than chemical treatment in the removal of the smallest charged colloids as they can mingle with the dissolved metal ions easily within the electrical field. The sludge produced by EC is dryer and the amount is smaller in comparison to chemical treatment (Mollah et al. 2001; Rajeshwar and Ibanez 1997). Moreover, the production of H_2 gas bubbles during electrolysis could bring lighter flocs to the surface for easy removal (Mollah et al. 2001). However, EC has some draw backs. The sacrificial electrodes

get eroded; a higher voltage would be required to compensate the increased spaces between the electrodes (Russell 2006). Similarly, an oxide layer could form on the external surfaces of the electrodes (also known as passivation), which would limit metal dissolution and thereby restrict the chemical coagulation performance in the solution (Chen and Comninellis 2007).

7.2.2   Prototype Trial

Based on the literature research in Section 7.2 it can be seen that EC could be a compact and fast-action system for the treatment of oily wastewater. The efficiency of an EC prototype system was investigated in December 2008. The EC prototype system designed by Sivex Engineering Limited was investigated with Sureclean interceptor oily wastewater collected from the WTS in Alness. The main aim of the prototype field trials was to evaluate the efficiency and suitability of the EC reactor using Sureclean waste streams. The prototype system had previously been trialled for produced water but had never been trialled with oily wastewater.

7.2.2.a   The EC Prototype

Figure 7-2 shows the main components of the EC prototype, which include a pH balancing system, an EC reactor or cell, a set of control panels with adaptive software, a controllable power supply and a holding tank. The pH balancing system also included one dosing pump for each acid and base, a pH monitor and a mixing tank with in-tank agitation. The system was also equipped with an electrically controlled delivery pump. The whole system was housed in a converted camper trailer for ease of transport to different sites. The system required a 380 – 460 V, 3-phase, 60 - 70 A power supply and a 5-pin power cable with a female connection. The wastewater feed pipe could be connected to either a two inches female British Standard Pipe (BSP) threaded coupling or two inch male camlock coupling. The electrodes used in these trials were mild steel.
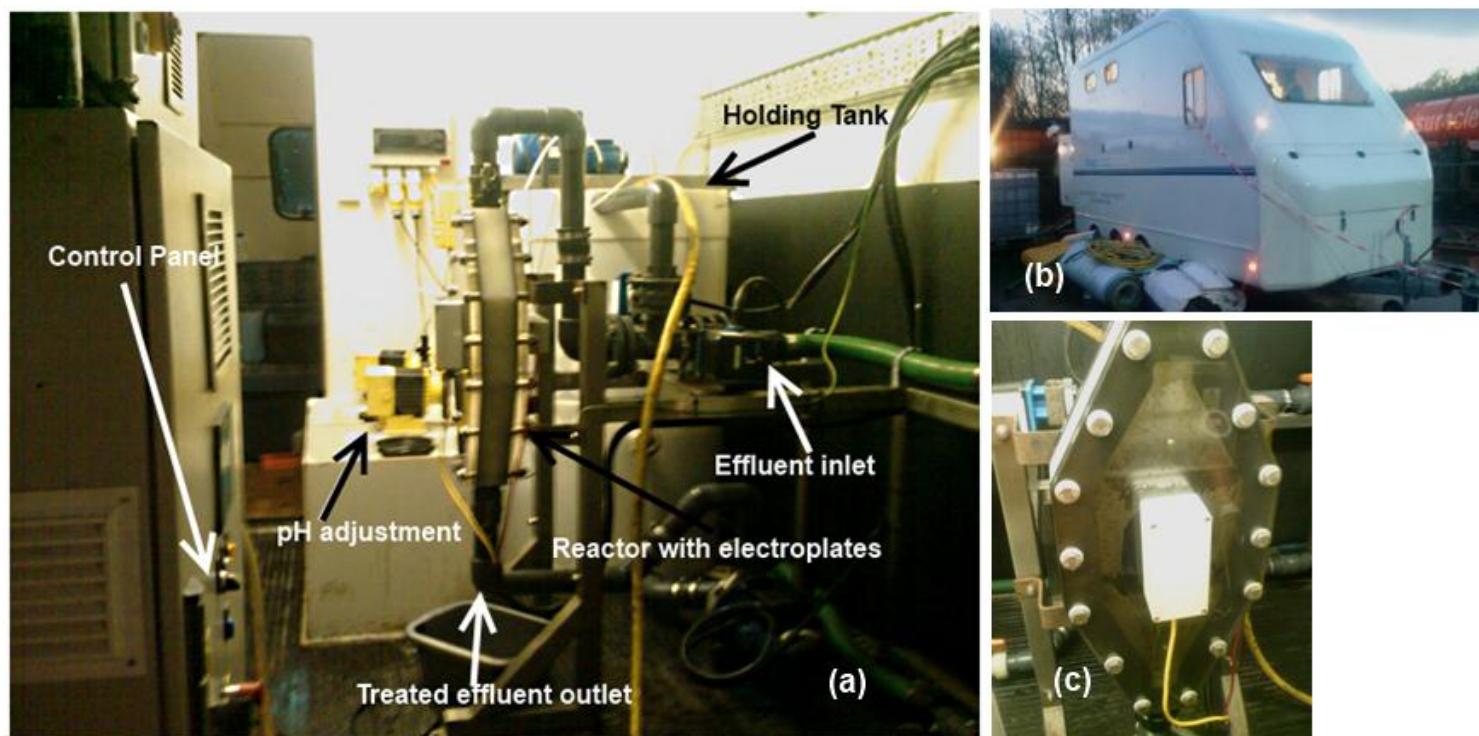
Figure 7-2 showing the EC Prototype System designed by Sivex Engineering Limited. (a) The EC system was equipped with a pH adjustment system, control panel and a balancing tank; (b) the EC system was housed in a converted camper trailer; (c) the EC cell

7.2.2.b   Waste and Analysis

Four waste samples were investigated in the prototype field trials. Sample 1 was the oily wastewater obtained from the shaker tank (for details of waste origin refer to Section 4.2.2.*b*)*.* Sample 2 was oily wastewater collected from Sureclean dewatering of waste oil tanks; the waste oil was a mixture of oil-water emulsion collected from garages, ships or boats and oil platforms. Sample 3 was oily wastewater collected from interceptor 5 and Sample 4 was oily wastewater collected from interceptor 6 (for details of waste origin refer to Section 4.3.2.*b*). A summary of the waste samples used in the prototype trials can be seen in Table 7-1.

Samples	Oily Wastewater Location
Sample 1	Shaker Tank
Sample 2	Waste Oil Tanks
Sample 3	Interceptor 5
Sample 4	Interceptor 6

Table 7-1 EC Prototype Trials: Oily Wastewater Samples from different sources.

The wastewater was analysed before and after treatment with the EC prototype system with the following analysis: TPH, heavy metals, TSS, COD and BOD. Due to cost and time constrains within the company, only one set of data was obtained. The effluent samples were sent to an independent UKAS accredited laboratory, STL Limited for analysis. The analytical methodology can be found in Section 5.2.2.*a*.

7.2.2.c   Results and Discussion

500 L of oily wastewater samples from Sureclean Alness WTS were pumped straight from the interceptor to the EC prototype. Treatment operational parameters can be found in Table 7-2. The wastewater samples were pumped into the EC reactor and immediately after that, the treated effluent

was transferred into an IBC with the top cut open. The IBC was used as a clarifier or a settling tank for the arrays of EC to take place. Visual inspection showed that all treated wastewater produced a scum layer on top of IBC after 10-15 minutes of settling time. The settling time could also be seen as the actual reaction time as metal ions were dissolved when the wastewater passed through the electrodes, and during the settling time, metal hydroxides formed and destabilised the colloid particles. Bubbles could be observed when in the IBC during the settling period. The pH of all samples appeared to be 5 – 7.

Samples	Oily Wastewater Location	Current (A) and Voltage (V)	pH
Trial 1	Shaker Tank	35 A, 300 V	Not recorded
Trial 2	Waste Oil Tank	40 A, 300 V	5.7
Trial 3	Interceptor 5	35 A, 300 V	6.8
Trial 4	Interceptor 6	35 A, 300 V	Not recorded
Trial 5	Interceptor 6	30 A, 37 V	6.89
Trial 6	Interceptor 6	20 A, 24 V	6.92
Trial 7	Interceptor 6	35 A, 27 V	6.88
Trial 8	Interceptor 6	45 A, 21 V	5.96

Table 7-2 EC Prototype Trials: Trials treatment parameters.

The wastewater samples were collected before and after treatment, then the samples were sent to STL Limited for analysis. Details analytical method could be found in Section 4.4.2.a and 5.2.2.a. According to STL Limited, BOD of all the samples was not able to be performed due to the oily nature of the samples.

TPH Removal

No results were obtained for TPH of shaker and waste oil tanks before treatment. According to STL, this was because oil was absorbed onto the PET bottle used for sampling. However, from Figure 7-3 and Figure 7-4, it can be seen from the chromatograms generated by a GC-FID that there was a mixture of weathered diesel fuel oil and lubricating oil (as seen in Figure 7-3 and Figure 7-4). These results may suggest that samples from the shaker and waste oil tank were very oily in nature.
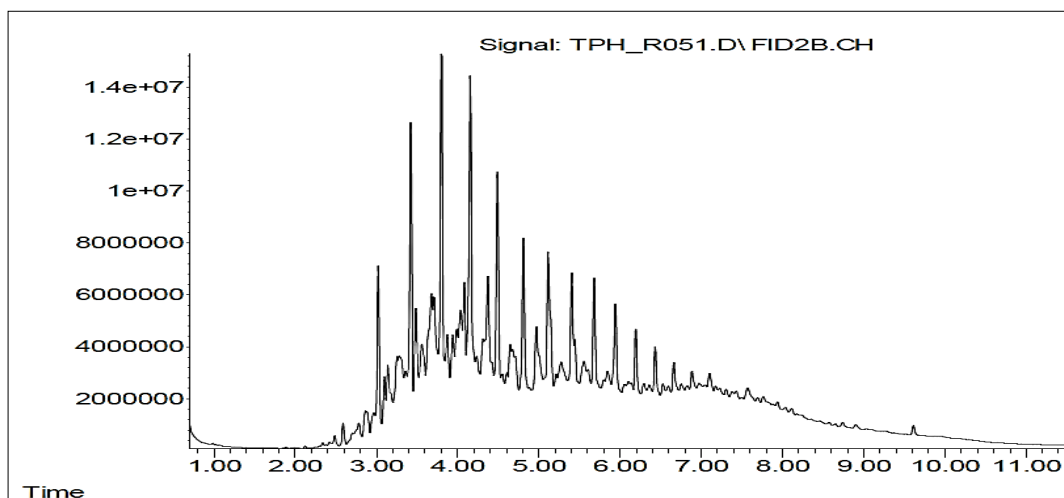
Figure 7-3 EC Prototype Trials: GC-FID chromatogram of sample from shaker tank.
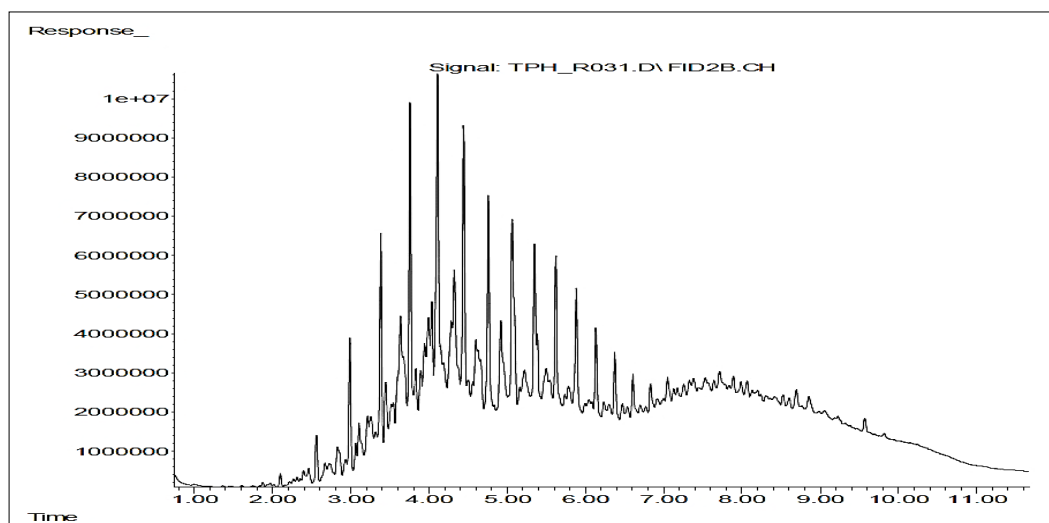
Figure 7-4 EC Prototype Trials: GC-FID chromatogram of sample from shaker tank.

The TPH results after treatment showed that TPH level were within the Sureclean discharge limit. Treatment of Interceptor 5 marked improvement. Almost 100 % of TPH were removed from Interceptor 5 after EC treatment. One of the main reasons for this improvement (as compared to the waste oil tank) may be due to the fact that Interceptor 5 effluent had a pH closer to pH 7 and therefore the steel plates performed better as stated by Chen 2004.

Oily Wastewater Samples	TPH before treatment (mg/L)	TPH after treatment (mg/L)	Removal Efficiency (%)
Shaker tank	No result	25.2	
Waste oil tank	No result	32.21	
Interceptor 5	58500	11.1	100.0%

Table 7-3 EC Prototype Trials: TPH of oily waste samples before and after treatment using an EC reactor.

It can be seen in Figure 7-5 that the TPH level for Interceptor 6 effluent samples was within Sureclean discharge limits of 100 mg/L. However, from the preliminary results seen in Figure 7-5, 35 A produced the best removal efficiency of 65.9 %. All samples showed a decrease in TPH level from the initial sample apart from the sample at 30 A. The level of increase was not significant (1.27 mg/L) and could be attributed to cross-contamination from the EC reactor. However, as only one set of samples were taken for each waste stream, certainty was not possible.
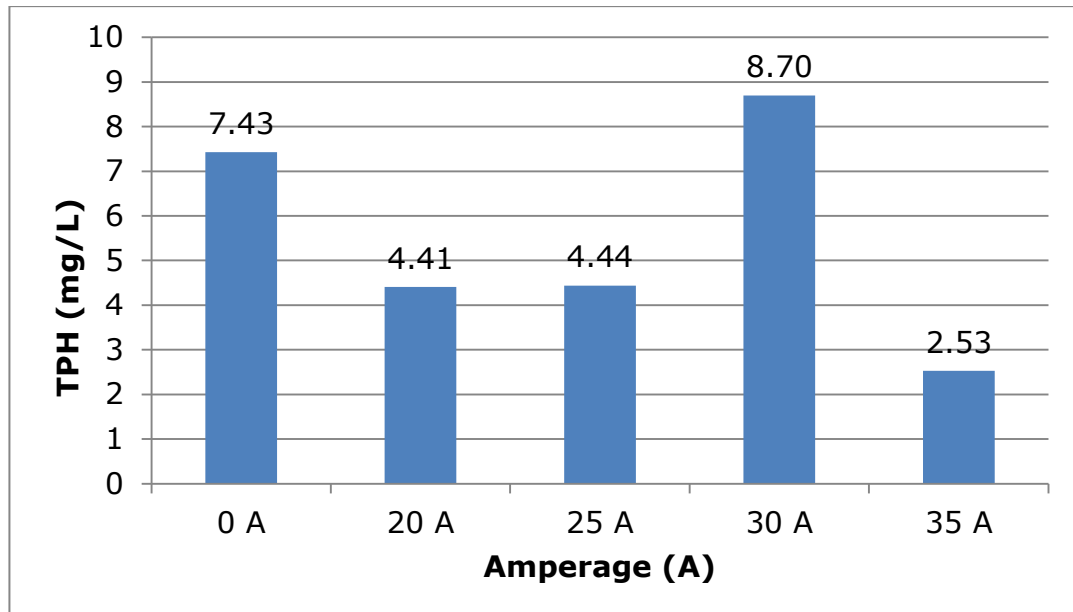
Figure 7-5 EC Prototype Trials: TPH of interceptor 6 samples before (at 0 A) and after treatment using an EC reactor at different amperage.

From the preliminary results, it can be seen that EC was able to reduce the TPH level. The scum on the surface of the IBC suggested flocs were formed and the production of electrolytic gases may have pushed the flocs to the surface of the treated effluent. The coalesced oil droplets could then be removed by mechanical separation systems such as an oil skimmer.

TSS Removal

It can be seen from Table 7-4 that the TSS of all waste samples was reduced after the treatment of EC. All the treated samples were within the discharge consent apart from the shaker tank waste sample. However, the best removal efficiency of TSS was seen in shaker tank waste (97.5 %). Further treatment with the clarification and filtration system in SWTS01 could improve the effluent quality. For interceptor 6 effluent, the result obtained at 35 A showed the best removal efficiency of 57.5 % as compared to other amperage of the same waste as shown in Table 7-2.

Oily Wastewater Samples	Before treatment (mg/L)	After treatment (mg/L)	Removal Efficiency (%)
Shaker tank	54000	1360	97.5%
Waste oil tank	1470	221	85.0%
Interceptor 5	6960	98	98.6%
Interceptor 6 (35 A)	146	62	57.5%

Table 7-4 EC Prototype Trials: TSS of four oily waste samples before and after treatment using an EC reactor.

COD Removal

It can be seen from Table 7-5 that the COD for all waste samples were reduced after the treatment of EC apart from shaker tank waste. Only treated samples of interceptor 5 and 6 were within the discharge consent level of 3000 mg/L. The result for shaker tank showed that COD was increased after treatment with EC; this could be due to cross contamination or sampling error. However, Moreno-Casillas et al. (2007) stated increase COD is possible especially if compounds within the wastewater react with the iron ions Fe (II) to form soluble produces and stay behind in the solution. As only one analysis was performed, the certainty of the result could not be verified.

Oily Wastewater Samples	Before treatment (mg/L)	After treatment (mg/L)	Removal Efficiency (%)
Shaker tank	3350	4220	-26.0%
Waste oil tank (30 A)	8540	3430	59.8%
Interceptor 5	5880	840	85.7%
Interceptor 6 (30 A)	985	630	36.0%

Table 7-5 EC Prototype Trials: COD of four oily waste samples before and after treatment using an EC reactor.

Heavy metals Removal

It can be seen from Table 7-6 that the total heavy metals content in all waste samples was shown to be reduced after the treatment of EC. All the treated samples of interceptor 5 and 6 were within the discharge consent levels at 2 mg/L. The preliminary results may suggest that EC could be effective in heavy metal removal in Sureclean waste streams. The analysis for As was not able to be carried out for Shaker tank and Waste oil tank samples because these two samples were too oily. Pb result in Interceptor 6 after treatment with EC was the only result that was below the LOD. The full heavy metals results for the EC prototype trial could be found in Appendix 4.

Oily Wastewater Samples	Before treatment (mg/L)	After treatment (mg/L)	Removal Efficiency (%)
Shaker tank	23.74	0.30	98.8%
Waste oil tank (30 A)	7.63	0.40	94.8%
Interceptor 5	3.24	0.12	96.4%
Interceptor 6 (30 A)	0.49	0.11	78.4%

Table 7-6 EC Prototype Trials: Heavy metals (As, Cr, Co, Cu, Pb, Mo and Ni) of four oily waste samples before and after treatment using an EC reactor.

7.2.2.d   Conclusion

Treatment of Sureclean oily wastewater was investigated using a prototype EC system designed by Sivex Engineering Limited. The preliminary results may suggest that EC could be effective in TPH, TSS and heavy metals removal in Sureclean waste streams. Treatment trials carried out for the treatment of interceptor wastewater in the Alness waste transfer station showed almost 100 % of TPH removal for Interceptor 5 effluent. It can be seen that 35 A was the best operational amperage for Interceptor 6 (pH 6.88). Total heavy metals levels in all treated waste samples were all well within the discharge consent and achieved over 90 % reduction for all waste

streams apart from Interceptor 6. For some waste samples such as the shaker tank sample, post treatment using filtration or advanced oxidation treatment may be required to meet discharge consent.

7.2.3 Sureclean Electro-coagulation Water Treatment System (SEWTS) Field Trial

Based on the prototype trial outcome seen in Section 7.2.2, Sureclean decided to design and build an EC reactor for the treatment of its waste streams. A cost-benefit analysis was carried out to evaluate the viability of the treatment unit. Sureclean charged £39.00 per 1000 L for oily wastewater treatment from various origins such as garage, petrol station forecourt and oil tank cleaning. The estimated fabrication and build cost of an EC system with a flow rate of 5 m^3 was £33,220.00 with a 20 kVA power supply (maximum output of 100 A, at 100 V). The cost included a contingency of 10 % of the actual fabrication cost. The cost also included a reactor cell where sacrificial electrodes that would be housed in a polypropylene cartridge, a 2,000 L capacity polypropylene buffer tank, a 5 m^3/h capacity electrical positive displacement pump, polyvinyl chloride (PVC) electrically actuated ball valves and PVC pipe-work.

As seen in Table 7-7, there were five main annual costs to consider in the analysis: trade effluent charges, power cost, maintenance cost and sludge removal cost. The trade effluent cost was estimated based on trade effluent bills received from Scottish Water. Approximately 832 plates per annum would be consumed and at the cost of £4.50 per plate (Kemartek 2010). The power consumption of the EC reactor was estimated to be £2.55 per 50 m^3 of wastewater treated (Kemartek 2010). Based on projection and estimation, Sureclean estimated that they would treat approximately 20 m^3 of oily wastewater per day at £39.00 per 1 m^3. Based on Table 7-7, the payback period was estimated to be five months. The numbers and figures that are quoted in Table 7-7 has been based on information received in 2009.

Effluent charges to client per m^3		£39
Estimated average volume per day (m^3)		20
Annual Turnover (5 days/week for 52 weeks)		**£202,800**
Estimated Total cost of whole WWTP (+10 % contingency)	£33,220	
Trade effluent Charge (£60.00 per day)	£15,600	
Annual Power cost (£0.05 per m^3)	£10,400	
EC electrode plates (annually)	£3,744	
Annual cost	**£62,964**	
Annual profit		**£139,836**
Payback period		**5 months**

Table 7-7 demonstrates the cost-benefit analysis of purchasing an EC.

7.2.3.a   SEWTS- Reactor Specifications

The Sureclean Electrocoagulation Water Treatment System (SEWTS) was developed collectively by Sureclean and Kemartek Technologies Limited. Kemartek Technologies Limited is a sister company of Sivex Engineering that designed the prototype. The system operational flow rate was between 2.5 - 7.5 m^3/h. The system footprint allows the unit to be transported and operated inside a standard 10' x 8' ISO container. The system can operate outside these containers: for example, in an enclosed 'weather-proofed' space or within a building. The effluent inflow, power cable, compressed air input (the valves being pneumatically actuated) and the treated outflow are sited close together to facilitate ease of operational connections. The entire system is skid-mounted as seen in Figure 7-6. A polypropylene bund with Glass Reinforced Plastic (GRP) open mesh grating was installed at the base of the unit. The rigid steel frame fixed to the skid provides mounting points for the hydraulic and electrical components.

Figure 7-6 Photo of the SEWTS that could be housed in a 10′ x 8′ ISO container.

7.2.3.b   Waste and Analysis

The electrodes used in this study were mild steel plates with a dimension of 3 mm x 200 mm x 208 mm. Four waste types obtained were investigated in the SEWTS field trials. Sample 1 was the oily wastewater obtained from Sureclean Interceptor from Alness (Interceptor 4 effluent)*.* Sample 2 was oily wastewater collected from interceptor effluent in Sureclean Aberdeen WTS and Sample 3 was oily bilge water collected by a waste oil collection company called Northburn Industrial Services.

The wastewater was analysed before and after treatment with the SEWTS with the following analysis: TPH, heavy metals, TSS, COD and BOD. Due to cost and time constrains within the company, only one set of data was obtained. The effluent samples were sent to an independent UKAS accredited laboratory, STL Limited for analysis. The analytical methodology can be found in Section 4.4.2a and 5.2.2.a.

7.2.3.c   Results and Discussions

Approximately 1000 L of oily wastewater samples were treated with SEWTS. Treatment operational parameters and settling time can be found in Table 7-8.   The wastewater samples were pumped into the buffer tank and through the EC reactor and the treated effluents were transferred into an IBC with on open top immediately after treatment with EC. Visual inspection showed that all treated wastewater produced a scum layer on top of IBC after 10-15 minutes of settling time as shown in Figure 7-7. The high voltages of Sample 1 before salt addition suggested that conductivity was low and that caused high energy consumption as described in Equation 7-6. Therefore 2 kg of table salt bought from a supermarket was added and the voltage decreased from the initial 173 V to 35 V.

Samples	Oily Wastewater Location	pH	Amperage (A)	Voltage (V)	Settling time (min)
Sample 1	Alness WTS Interceptor Effluent	7	25	173	15
Sample 1	Alness WTS Interceptor Effluent (with addition of 2 kg of salt)	7.9	30	35	15
Sample 2	Aberdeen WTS Interceptor Effluent	7.9	30	185	12
Sample 3	Bilge Water	6.7	40	21	6

Table 7-8 SEWTS Field Trials: Oily Wastewater Samples from different sources.

Figure 7-7 shows (Left) Formation of contamination 'float' in IBC; (Right) before and after effluent samples of Alness Interceptor Effluent.

The TPH, heavy metals, TSS, COD and BOD results before and after treatment with the SEWTS is shown in Table 7-9. All waste samples show a reduction in TPH with the highest removal efficiency observed in bilge water (99.9 %). This may be due to the fact that bilge water had the highest TPH level and the high salt content in the wastewater may help improve the separation of oil and water. The highest TSS reduction was observed in Alness interceptor effluent (68.1 %). The low conductivity could be the reason for the relatively low TSS removal efficiency for Aberdeen interceptor effluent as compared to the other waste streams.

The presence of organic and inorganic compounds in the wastewater influenced the level of COD. COD results for treated Alness and Aberdeen interceptor effluent were seen to be reduced by 68.4 % and 97.2 % respectively; however, the reduction of COD in bilge water was only 6.7 %. The small reduction in COD for bilge water may indicate that the iron ions from the electrodes reacted with the pollutants in the solutions to form more soluble than insoluble compounds as only insoluble iron-complex could be removed by EC (Moreno-Casillas et al. 2007).

Total heavy metals for Alness interceptor effluent were seen to increase; however, the increase may not be significant because only one result was obtained. The heavy metals reduction was seen to be reduced by 94.1 % in Aberdeen.

When compare to Sureclean discharge consent (as shown in Table 1-1), the TSS results for the treated bilge water and Aberdeen interceptor effluent did not complied with the discharge limit. COD result of the treated bilge water did not complied to the discharge consent as well. This implied that further treatment using filtration or clarifier could further reduce SS in the treated effluent. The COD could be further reduced by AOP (which would be discussed in Section 7.3.

Parameters	Oily Wastewater Samples	Before treatment (mg/L)	After treatment (mg/L)	Removal Efficiency (%)
TPH	Alness Interceptor Effluent	10.20	3.67	64.0%
	Aberdeen Interceptor Effluent	5.52	2.60	52.9%
	Bilge Water	2890	2.97	99.9%
TSS	Alness Interceptor Effluent	204	65	68.1%
	Aberdeen Interceptor Effluent	4050	3350	17.3%
	Bilge Water	11900	9000	24.4%
COD	Alness Interceptor Effluent	940	297	68.4%
	Aberdeen Interceptor Effluent	5910	163	97.2%
	Bilge Water	165000	154000	6.7%
Total Heavy metals	Alness Interceptor Effluent	0.08	0.18	-130.8%
	Aberdeen Interceptor Effluent	2.2697	0.118	94.8%
	Bilge Water	1.49	1.24	16.9%

Table 7-9 SEWTS Field Trials Results: TPH, TSS, COD and Total Heavy metals results.

7.2.4 Conclusion and Future Work

The system's operations and capabilities were demonstrated and proved using a variety of Sureclean generated waste streams (from the Alness and Blackdog interceptor systems) with varying degrees of contamination. Treatments were also undertaken on samples of bilge water. The bilge water demonstration successfully removed 99.9 % of petroleum hydrocarbons given the very high level of organic chemicals.

Sureclean as a waste treatment company could apply the EC system to sewage sludge as a pre-treatment process for dewatering as proven by Shin and Lee (2006) in Korea where pressure was applied to the coagulated solids after treatment with EC. Sureclean could also expand its client bases to other industries such as those described in Section 7.2. In Chapter 4, distillery effluent was examined and the study revealed that EC could remediate this type of waste (Kannan, Karthikeyan and Tamilselvan 2006). A combination of the EC and floatation processes could improve the effluent quality as reported by various studies (Wang et al. 2010; Pouet and Grasmick 1995). Boroski et al. (2009) presented that there was a reduction of COD from 1753 mg/L to 160 mg/L after EC and 50 mg/L after EC and heterogeneous photocatalysis treatment for wastewater collected from the pharmaceutical and cosmetic industry. The treated effluent could be further processed using the AOP that will be discussed in Section 7.3. Therefore, Sureclean could incorporate both the EC and AOP treatment together to enhance the efficiency in future. The cost-benefit analysis shown in Table 7-7 clearly shows that the EC would benefit Sureclean if the unit is fully utilized.

7.3 Advanced Oxidation Process (AOP)

Most harmful pollutants such as aromatics, dioxins, pesticides, hormones, haloaromatics, dyes and other pollutants which are mainly found in industrial effluents are usually chemically stable and not easily bio-degraded in natural water bodies. These compounds account for the TOC and COD in the wastewater. Conventional biological and physical treatments fail to degrade these harmful compounds effectively and economically. Also, alternative treatment routes such as incineration of these harmful pollutants would be more expensive (Andreozzi et al 2008).

The AOP has great potential to effectively degrade toxic compounds under ambient temperature and pressure (Andreozzi et al. 2008; Munter 2000). AOP are characterised by the formation of hydroxyl radicals (OH·) to achieve mineralisation of potential toxic organic compounds through

oxidation processes. The hydroxyl radical is a powerful oxidant and is highly reactive, it has an oxidation potential, E^0 of 2.80 V, stronger than ozone (E^0= 2.07 V), chlorine oxide (E^0= 1.50 V) and chlorine (E^0= 1.36 V) (White 2010). The hydroxyl radical is a strong electrophile that reacts rapidly and non-selectively towards electron rich organic compounds (Stasinakins 2008). Oxidation processes in environmental treatment technologies can be performed either by using chemicals or physical methods or a combination of both. Chemical oxidising agents, such as potassium manganese, chlorine and sodium hypochlorite have been widely used, in particular for disinfection purposes; however, these chemicals mainly target specific pollutants and therefore are not effective if a combination of pollutants is present in the wastewater (Wang and Yan 2006). Comparatively, hydroxyl radicals are far better oxidising species than most acids. There are different AOPs in wastewater treatment, some of the more technically promising AOPs are semiconductor photocatalysis using ultra-violet (UV) light (UV/TiO_2), Fenton's reagent (H_2O_2/Fe^{2+}), Ozone under UV radiation or combined with hydrogen peroxide (O_3/UV or H_2O_2/O_3) and UV and hydrogen peroxide (UV/ H_2O_2) (Catalkaya and Kargi 2008).

Applications of AOP has been reported in the treatment of wastewater effluents from a variety of industrial sectors; examples of industries include textiles (Kos and Perkowski 2009; Perez et al. 2002), paper and pulp mills (Pirkanniemi, Metsarinne and Sillanpaa 2007; Catalkaya and Kargi 2008), olive mills (Cañizares et al., 2007b), agricultural wastewater (pesticides/herbicides contaminated wastewater) (Catalkaya and Kargi 2009; Hequet, Gonzalez and Le Cloirec 2001), municipal wastewater treatment (Antoniadis 2007; Ormad et al. 2008) and pharmaceutical wastewater treatment (Klavarioti, Mantzavinos and Kassinos 2009; Esplugas et al. 2007). AOP has gained huge interest due to the fact that most harmful pollutants that are present in industrial effluents were poorly degraded by conventional wastewater treatment processes such as biological treatment (Andreozzi et al. 2008). However, most AOPs have limitations:

1. AOPs are not effective in treatment of industrial effluent with COD of more than 5 g/L) (Andreozzi et al. 2008); and

2. The hydroxyl radical oxidation effect is inhibited by the presence of radical scavengers such as the chloride ion (Cl^-) (Hoigne and Bader 1979; Kiwi, Lopez and Nadtochenko 2000).

Therefore combinations of treatment processes such as biological, chemical and physical treatment coupled with pre- or post-treatment using AOPs have been widely proposed (Cañizares et al. 2007b; Andreozzi et al. 2008).

Chemical species with oxygen bonds (O-O) are strong oxidants similar to those in hydrogen peroxide (H_2O_2). However H_2O_2 itself is not effective for high concentration contaminants due to its low rates of reaction (Neyens and Baeyens 2003). Fenton's reagent was first discovered by H.J.H Fenton in 1894 by using ferrous salt (Fe^{2+}); but it was Haber and Weiss who discovered this technique. Basic chemical reactions can be demonstrated in the equations below (Patai 1983):

$$Fe^{2+} + HO\text{-}OH \rightarrow (Fe\text{-}OH)^{2+} + \cdot OH \qquad \textbf{\textit{Equation 7-7}}$$

$$OH\cdot + CH_2\text{=}CHR \rightarrow HO\text{-}CH_2\text{-}CHR\cdot \rightarrow HO\text{-}(CH_2\text{-}CHR)_n\cdot \qquad \textbf{\textit{Equation 7-8}}$$
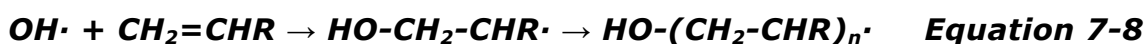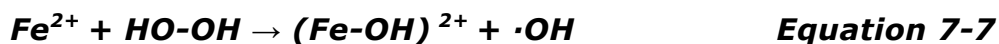
pH plays a significant role in the treatment efficiency; the optimum operating pH is between 2 and 4 (Klavarioti, Mantzavinos and Kassinos 2009; Gogate and Pandit 2004). Therefore all forms of Fenton wastewater and water treatment processes include pH adjustment as shown in Figure 7-8.
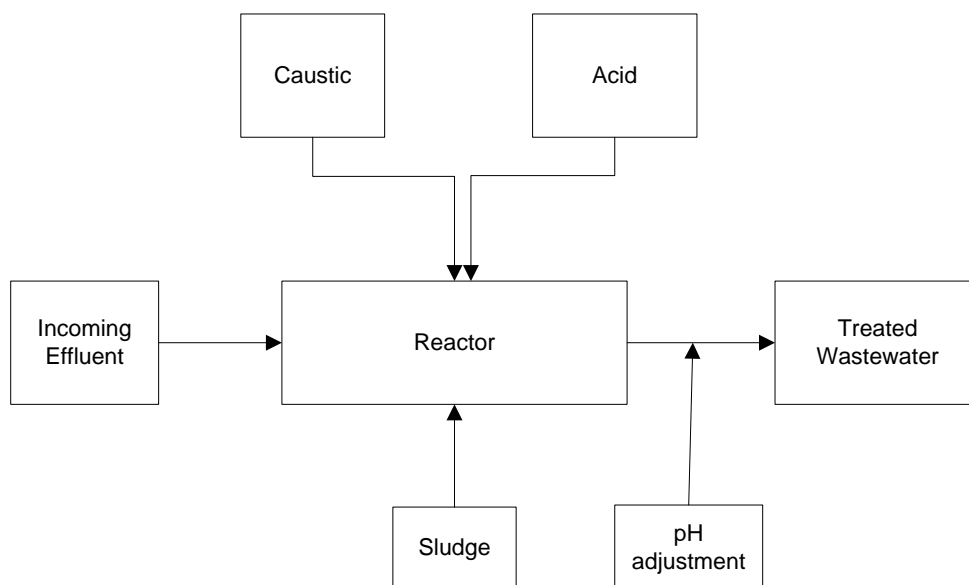
Figure 7-8 shows a typical design of a Fenton wastewater treatment (Gogate and Pandit 2004).

The oxidation effects of Fenton's reagent can be enhanced with UV exposure as the rate of OH· formation is markedly increased by photoreactions of H_2O_2 ($\lambda <$ 360 nm) (Pignatello, Liu and Huston 1999). However, like all photo-assisted reactions, the oxidative reaction for effective mineralisation of pollutants is inhibited by the presence of radical scavengers such as the chloride ion (Kiwi, Lopez and Nadtochenko 2000). Similarly to Fenton's reagent, the optimum operating pH for photo-assisted Fenton's reagent must be maintained at or slightly above pH 3 throughout the oxidative process (Machulek et al. 2007). Due to its high efficiencies, photo-assisted Fenton's reagent is more economically favourable towards Fenton's reagent itself (Goi and Trapido 2002). Moreover, evidence has suggested that in the presence of UV light, Fe^{2+} ions are regenerated as per Equation 7-9 (Andreozzi et al. 2008):

$$Fe(OH)^{2+} \xrightarrow{hv} Fe^{2+} + HO\cdot$$

Equation 7-9

Ozone is a powerful oxidant (oxidation potential, $E^0 =$ -2.07 eV); ozonolysis has been well known in the research of organic chemistry as ozone also acts as an electrophile (Fox and Chen 1981). Ozone molecules attack unsaturated carbon-carbon (C=C) double bonds by means of electrophilic

reaction (Keinan and Varkony 1983). This is due to the fact that ozone exists as a zwitterion (as shown in Figure 7-9).
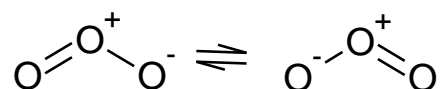
Figure 7-9 Resonance structure of ozone

Boncz (2002) concluded that there are three main pathways for ozone decomposition in water:

1. initiated by hydroxide ion (OH⁻);
2. by hydroperoxy ion (OOH⁻);
3. By UV illumination as demonstrated in Equation 7-10 and Equation 7-11 (Boncz 2002).
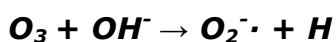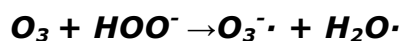
$$O_3 + OH^- \rightarrow O_2^{-}\cdot + H \qquad \text{Equation 7-10}$$
$$O_3 + HOO^- \rightarrow O_3^{-}\cdot + H_2O\cdot \qquad \text{Equation 7-11}$$

Ozone is an unstable gas; therefore an ozone generation reactor is usually present on-site and generates gas in-situ. The first commercial wastewater treatment was reported in Houston as early as the 1970s for the oxidation of cyanides chlorinated solvents and other difficult to treat pollutants; subsequently, development was focussed on ozone coupled with UV on the treatment of drinking water (Glaze, Kang and Chapin 1987).

7.3.1   Semiconductor Photocatalysis (UV/Ti O_2)

Semiconductor photocatalysis is classified as an AOP (Andreozzi et al. 2008; Han et al. 2009). When a semiconductor is illuminated with light of energy greater than its bandgap, electrons and holes are formed as illustrated in Figure 7-10; the bandgap energy is the energy difference between the valence band and the conduction band. The electron and holes formed are

highly charged and initiate reduction and oxidation reactions, thereby mineralising the aqueous pollutants (Anpo 2000; Herrmann 1999).
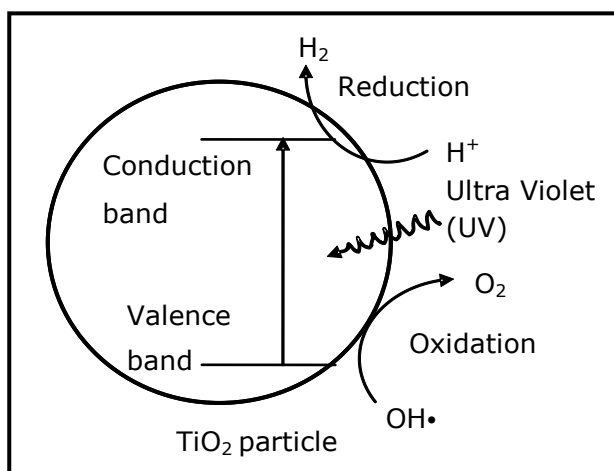
Figure 7-10 demonstrates a schematic representation of an energy band of a TiO$_2$ particle (Anpo 2000; Herrmann 1999).

Titanium dioxide (TiO$_2$) has been reported to be the most efficient catalyst in semiconductor photocatalysis environmental remediation (Mills and Lee 2002). The main advantages of using titanium dioxide as the catalyst are: TiO$_2$ is a relatively abundant resource and relatively inexpensive, moreover TiO$_2$ is chemically very stable and generates electrons that are highly oxidising (Fujishima, Rao and Tryk 2000; Fan and Yates 1996). Additionally, semiconductor photocatalysis is also a relatively less energy intensive technology for the destruction of complex chemical pollutants when compared to other technologies such as incineration (Andreozzi et al. 2008). The research on heterogeneous semiconductor photocatalysis in environmental applications has been reported extensively, in particular for treatment in water and gas phases in the mineralisation of organic compounds such as alkenes (Fox and Whitesell 2004), aromatics (Robertson et al. 2005; Shukla, Dorris and Chikkaveeraiah 2009) and organohalides (Hoffman et al. 1995; Ollis 1985).

There are a variety of photochemical reactors that have been reported in the literature e.g. fixed-bed reactors, slurry reactors and fluidised bed reactors (Kabra, Chaudhary and Sawhney 2004). Fixed-bed reactors

immobilise the catalyst onto a fixed surface such as glass beads (Ferguson and Hering 2006), silica (Kostedtiv et al. 2005), stainless steel support (Fernandez et al. 1995) or quartz (Mills and Lee 2002); however, manufacturing these materials on a larger scale may be expansive. Slurry reactors are characterised by using powdered catalyst, which can provide a large surface area for photocatalysis (Ollis and Turchi 1990), however, the catalyst must be separated from the liquid phase after treatment by filtration, thus increasing costs and reducing the ease of use (Hoffman et al. 1995). Fluidised bed reactors offers high-throughput and efficient reactant-catalyst contact (Dibble and Raupp 1992). Mills and Lee (2002) reported several companies worldwide which promote semiconductor photocatalysis for commercial application, but a lot of these products are still in the developmental stage.

7.3.2   Photocatalysis Field Trial

The efficiency of a Sureclean owned industrial scale photocatalytic reactor was investigated. The photocatalytic reactor was designed for the remediation of industrial effluent, oil and gas wastewater and other waste streams such as contaminated groundwater. Methylene blue (MB) was chosen as it is widely reported as an indicator for photocatalytic degradation (Mills and Wang 1999). Toluene, also known as methyl-benzene, was chosen as a hydrocarbon analogue because of its presence in industrial solvents and crude oil. The contaminated groundwater was collected as part of a groundwater monitoring exercise from an ex-gas works. Mineralisation of TPH in laboratory based trials has been reported (Herrmann et al. 2007).

7.3.2.a   Reactor

The photocatalytic reactor reported in this research is a twin tank water treatment system purchased from UVPS Limited, Aberdeen, UK. The photocatalytic reactor is called Sureclean Advanced Water Treatment System (SAWTS) and it was composed of a structural steel base frame with inbuilt forklift pockets, a full drip tray with drainage point and a crash protection frame for the tanks. The reactor was designed to support on-site

and off-site treatment; hence it is relatively mobile and modular. The two fabricated stainless steel tanks; (Tank A and Tank B) were manufactured from 3 mm 316 grade stainless steel with removable lids. Inside each tank there were TiO_2 pellet catalyst suspended in four metal trays with uniform holes on the bottom of the tray and seven black ultra violet light tubes (wavelength output of 345 to 400 nm, 120 cm length and 36 Watt, reactor power supply was 110 Volts Alternating Current). The TiO_2 pellet catalyst used in the reactor was Hombikat C obtained from Sachtleben Chemie, Germany. Approximately 40 kg of catalyst was placed in each tank (approximately 10 kg in each basket). Compressed air was supplied by a compressor into the tanks with a pressure of between 0.5 and 1.5 bars as the air supply forms bubbles which provide oxygen as the oxidising element and a form of agitation inside the reactor. The total capacity of the twin tanks is 1000 litres (500 litres in each tank). The trials were conducted at 12.5 litres per gram of titanium dioxide. A photo of the reactor and a summary of the reactor specifications can be found in Figure 7-11 and Table 7-10 respectively.

Figure 7-11 Photocatalysis Field Trial: Photo of SAWTS 01-a photocatalytic reactor.

Dimensions	Height:	2000mm
	Length:	1400mm
	Width:	2140mm
Weight	Dry (shipping):	2150 kg
	Wet (operation):	6150 kg
Rating	Flow:	7 m³ / hour
Power		110v

Table 7-10 Photocatalysis Field Trial: Summary of the specification of the investigated photocatalytic reactor.

The trials were conducted in the Sureclean transfer station and the analyses of the wastewater were conducted by a MSc student, Hermina Mafe in RGU. Wastewater was stored in an external tank and was transferred via a 240 V electrical pump with flow control. The operation of the photocatalytic reactor was batch or continuous mode. In batch mode the effluent was treated using either Tank A or Tank B, whereas in continuous mode, effluent was treated using Tank A followed by Tank B. Figure 7-12 demonstrates the effluent flow regime on the reactor: wastewater was pumped from the bottom of the tanks, and treatment occurs while the effluent fills the tank; treated effluent was then discharged near the top of the tank through stainless steel pipework.
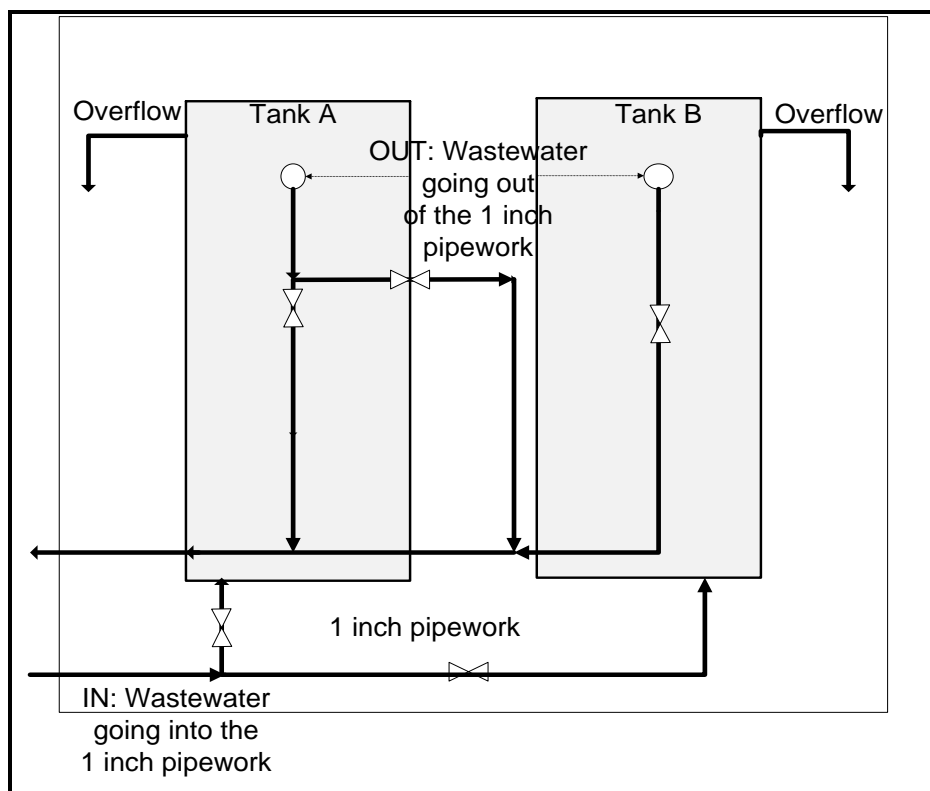
Figure 7-12 Photocatalysis Field Trial: Flow diagram of the advanced water treatment reactor.

7.3.2.a   Waste and Analysis

Chemicals and Waste

Both MB and toluene (99.5 %) were laboratory reagent grade from Fischer chemicals. Neat chemicals were added into water to make up the wastewater samples. Contaminated groundwater was collected as part of a groundwater monitoring exercise from an ex-gas works in Nairn, North of Scotland. No pre-treatment was carried out prior to treatment with the photocatalytic reactor. The groundwater was used as a step-up from MB and toluene. Potential organic contaminants that may be associated with a gas works are derived from coal tar such as aromatic hydrocarbons including benzene, toluene and xylenes, PAHs, hydroxyl substituted phenyls and other hydrocarbon based pollutants (Department of The Environment 1995). The degradation process of these compounds was studied by quantitative and qualitative analysis of MB and toluene using Ultra Violet/Visible (UV/Vis) absorption Spectroscopy (Perkin Elmer Lambda 900

UV Spectrophotometer), TOC analysis (Shimadzu TOC VCPH analyser), and FTIR spectroscopy (Perkin Elmer Spectrum GX).

The MB and toluene concentrations in the effluent were measured by the UV-Vis spectrophotometer at λ_{max} of 664 nm and 261 nm respectively. FTIR analysis was carried out on the contaminated groundwater. The wastewater samples were extracted and the TPH was analysed. Extraction procedure was conducted by adding 1.1 M hydrochloric acid (HCl) to the wastewater samples followed by 30 mL of tetrachloroethylene (TTCE) before the sample was stirred for 30 minutes. The samples were then transferred to a separating funnel where the bottom layer containing TTCE was collected. The TTCE extract was run through a 1.3 g Florisil column. A calibration curve was established using known diesel standards as recommended by the Department Of Energy and Climate Change (Department Of Energy and Climate Change 2006). The peak area of the region 3100 to 2700 cm^{-1} was recorded. Methodology for TOC analysis could be found in Section 3.2.6.

Photochemical experiments

The experiments were all carried out in the waste transfer station at Sureclean Ltd. Samples were collected before and during the treatment process at timed intervals for each trial. The trial regime was set up as in Figure 7-13 and Table 7-11.
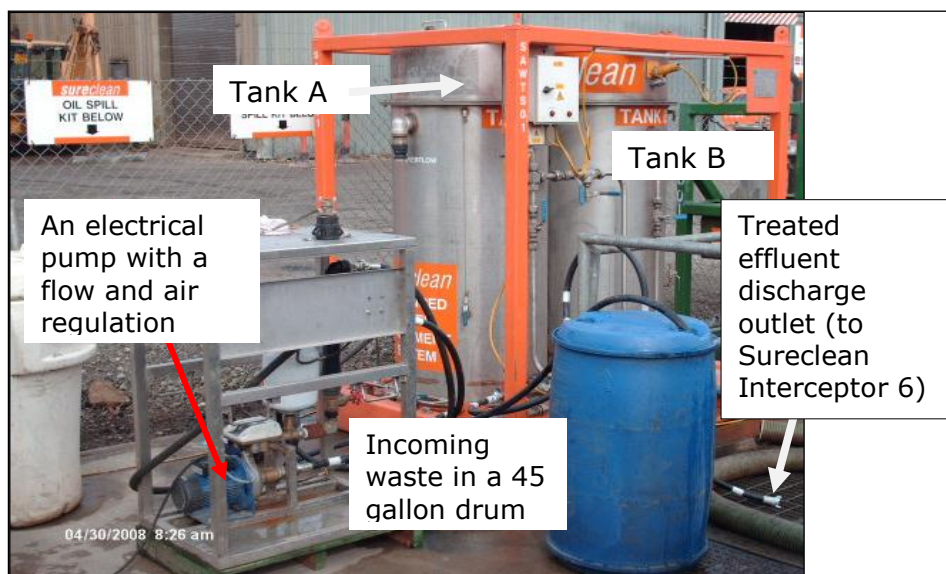
Figure 7-13 Photograph of the photocatalysis Field trial set-up.

(a) MB trials		Condition
1	Tank A	t=0-20 minutes: (1)
		t=20-40 minutes: (2)
		t=40-60 minutes: (3)
		t=60-150 minutes: (4)
2	Tank A	(4)
3	Tank B	(4)
4	Both Tanks	(4)
(b) Toluene trials		
5	Tank A	t=0-20 minutes: (1)
		t=20-40 minutes: (2)
		t=40-60 minutes: (3)
		t=60-150 minutes: (4)
(c) Contaminated groundwater trials		
6	Batch	(4)

Table 7-11 displays the Photocatalysis Field trial schedule for (a) MB; (b) toluene; (c) contaminated groundwater. Operational conditions: 1. absence of air and UV lights; 2. absence of air and presence of UV lights; 3. presence of air and absence of UV lights; and 4. presence of both air and UV lights.

7.3.2.b   Results and Discussion

In Trial 1, MB was treated using the photocatalytic reactor under four different conditions as shown in Table 7-11**.** The main purpose of this trial was to establish the efficiency of the reactor under different conditions. It can be seen from Figure 7-14 that the intensity of the colour reduced from the left hand side of the figure to the right hand side. This may suggest that there was degradation of MB using SAWTS. As shown in Table 7-12, the treatment of MB with Tank A of the photocatalytic reactor had 97.89 % removal efficiency and TOC reduction of 84.40 % after 150 minutes of treatment in the reactor.
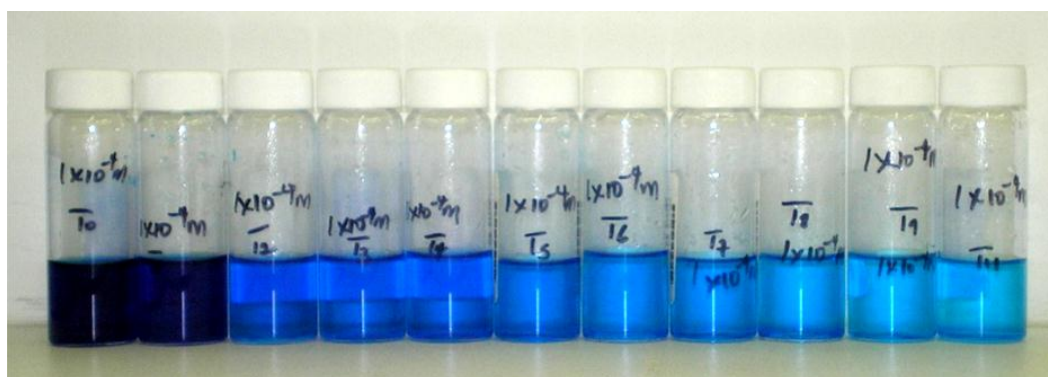
Figure 7-14 Photocatalysis Field Trial: Samples collected every 10 minutes from the SAWTS during the field trial.

Parameters	Concentration before treatment (Molar)	Concentration after treatment (Molar)	Percentage Reduction (%)
MB Concentration	$8.16 \times 10^{-5} \pm 5 \times 10^{-8}$	$1.72 \times 10^{-6} \pm 5 \times 10^{-9}$	97.89
TOC	22.94 ± 0.04	3.58 ± 0.00	84.40

Table 7-12 Photocatalysis Field Trial: MB and TOC concentrations of the wastewater samples before and after treatment by the SAWTS.

From Figure 7-15, it can be seen that the intermediate samples at 10 minute intervals of Trial 1, UV/Vis absorption spectroscopy and TOC analysis results demonstrated that the concentration of MB in Trial 1 decreased during the first 20 minutes (t=20) when the reactor was operated without air and UV light (Figure 7-15). This is attributed to the adsorption of MB onto the TiO_2 catalyst (Goi and Trapido 2002; Kiwi, Lopez and Nadtochenko 2000; Machulek et al. 2007). A sharp drop was observed for the subsequent 20 minutes (t=40) when the reactor was being operated with air only, after which the decrease continued gradually when it was operated with only UV light for a further 20 minutes (t=60).
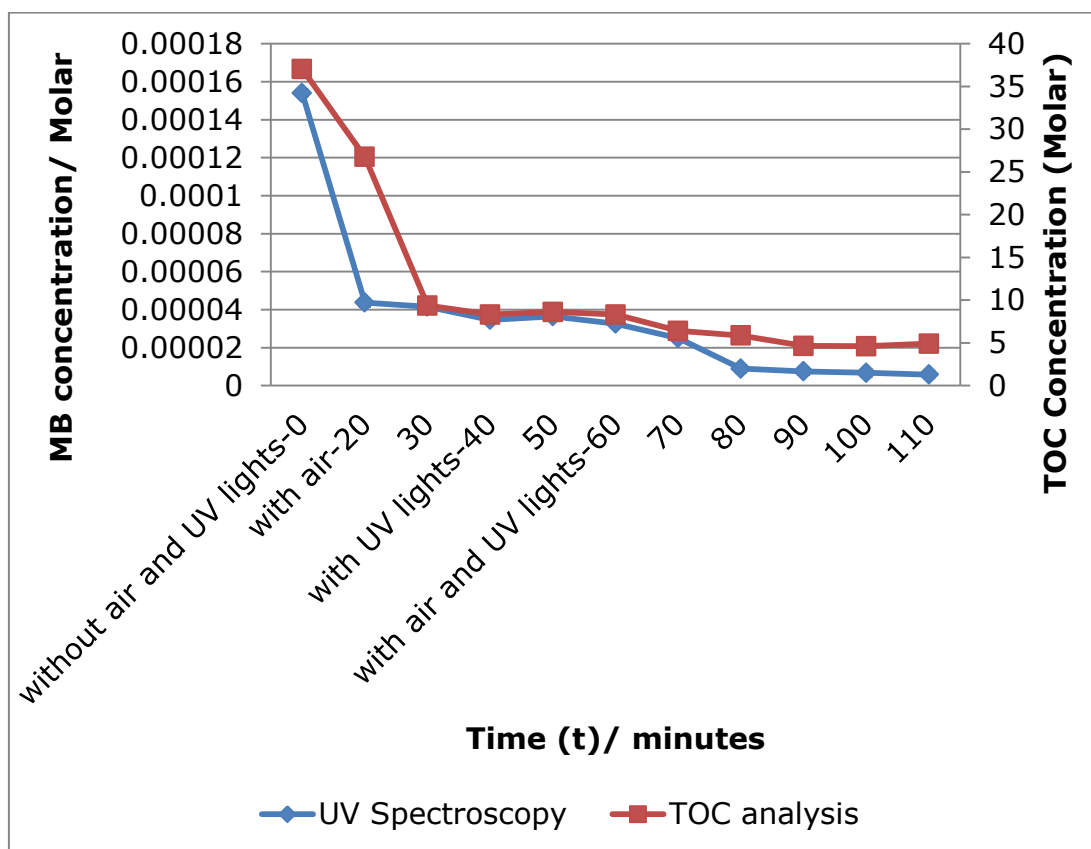
Figure 7-15 Photocatalysis Field Trial: Trial 1- UV/Vis absorption spectroscopy and TOC analysis results of MB of the wastewater samples with the treatment of SAWTS.

TOC analysis shows a similar degradation curve to the UV/Vis absorption spectroscopy degradation curve as shown in Figure 7-15. Further investigation of the data revealed that the decrease in MB concentration

was also observed even without the presence of UV lights. This could be due to holes which are the minority carriers forming in the dark as reported by Fujishima, Rao and Tryk (2000). Fujishima, Rao and Tryk (2000) also explained that the bare TiO_2 surface could reduce O_2 to either the superoxide O_2^- or H_2O_2 even in dark because it is an n-type material. Rajeshwar, De Tacconi and Chenthamarakshan (2001) also stated that "oxidation of an organic hydrocarbon has a negligible rate on a semiconductor surface in the dark, although thermodynamically it is feasible (i.e., has a negative Gibbs free energy)". The reactor was subsequently operated with UV light and air for an additional 90 minutes during which there was a noticeable decrease in MB concentration. After 90 minutes of this treatment, the MB and TOC concentration plateau. The UV/Vis absorption spectroscopy and TOC analysis results both had small errors which indicated that the results were reproducible.

In Trial 2, 3 and 4, the efficiency of the reactor in the treatment of the MB wastewater in the batch mode using Tank A, Tank B and then both tanks (continuous mode) in the presence of air and UV light were investigated with a treatment time of 130 minutes. From Figure 7-16, it can be seen that the MB remaining in the effluent after treatment with Tank A and Tank B in batch mode was 42.27 % and 57.58 % respectively. This shows that Tank A had better removal efficiency than Tank B as the concentration of MB in Tank A was lower than Tank B. The difference of the efficiency was due to a faulty UV light bulb in Tank B, which was subsequently discovered by Sureclean operatives on a routine maintenance on the reactor. In continuous mode, the removal efficiency was 96.20 % (3.80 % of MB remained in the effluent as shown in Figure 7-16) after treatment of 130 minutes. When comparing the results of the treatment using batch mode and continuous mode with the same treatment time, the treatment of MB wastewater using both tanks was significantly more efficient than batch mode because in continuous mode, the wastewater had a longer exposure time with the catalysts than in batch mode.
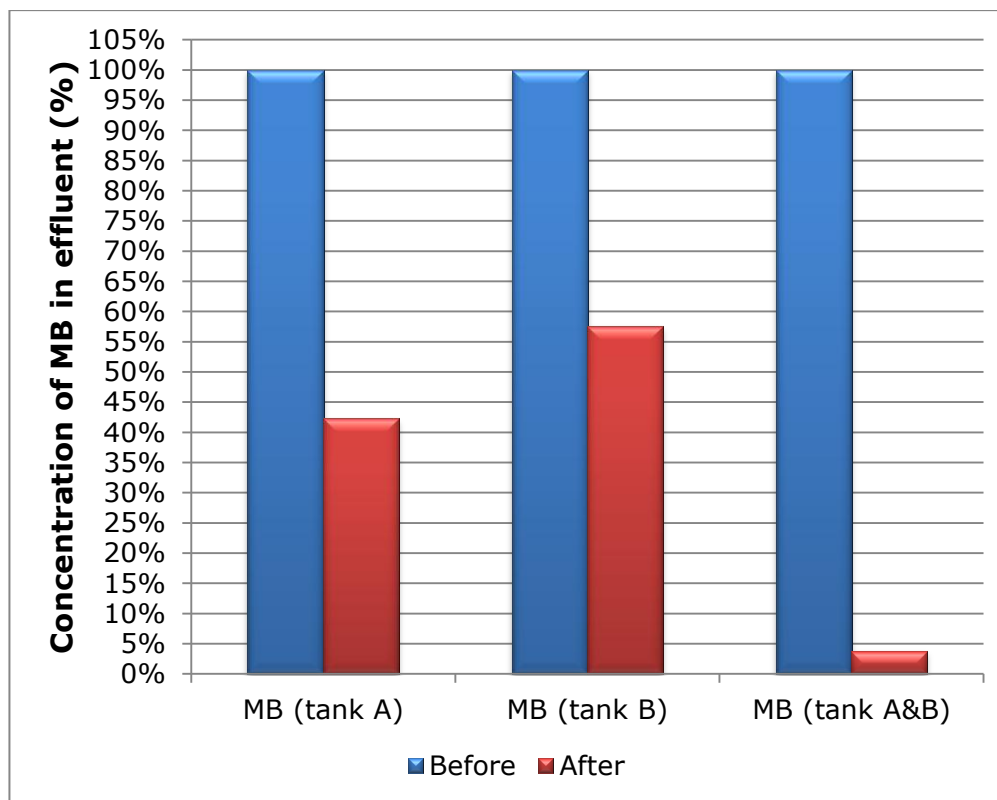
Figure 7-16 Photocatalysis Field Trial: Trial 2,3 and 4 results: A comparison between the before and after MB concentrations of the samples collected from Tank A, Tank B and Tank A and B of the reactor monitored by UV spectroscopy.

toluene degradation

In Trial 5, toluene was treated with the photocatalytic reactor using Tank A under four different conditions as demonstrated in Table 7-11. As shown in Figure 7-17, the trend for toluene reduction could be seen to be similar to MB reduction in the first 40 minutes (t=40). At t=20, the toluene level in Tank B dropped significantly and increased again at t=30. This result did not follow the trend like MB and was attributed to a sampling error. Analytical measurement error could be eliminated as triplicate analyses were performed and the errors were small. At t=60, a sharp drop in concentration was observed when the reactor was operated with air and light until t=70, by which time the toluene concentration levelled off at about 5 % for Tank A. A separate study on Tank B showed a similar trend

but with a lower efficiency compared to Tank A as evident in the treatment of MB (Trial 2 and Trial 3).
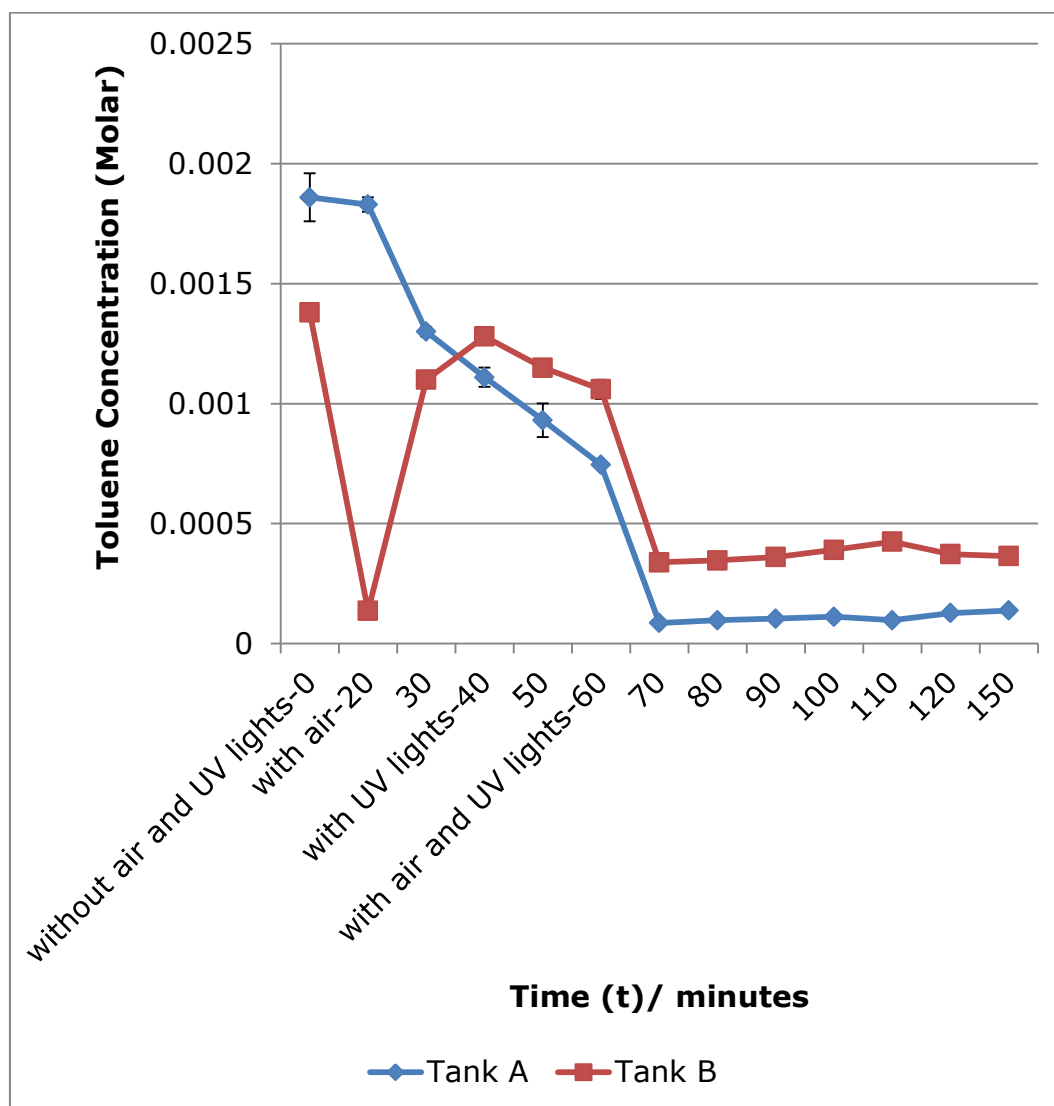
Figure 7-17 Photocatalysis Field Trial: Trial 5 results:

Degradation efficiency curve of toluene in samples obtained

from Tank A of the reactor monitored by UV/Vis Spectroscopy.

It can be seen from Figure 7-18 that the TOC removal efficiency in the initial sample from Tank A decreased gradually during the first 20 minutes (t=20) then increased slightly for a further 10 minutes. A gradual decrease was observed when the reactor was operated with air and UV light until t=150. All the samples were analysed simultaneously and in triplicate to reduce analytical instrumental error. Therefore, the fluctuations of TOC

concentration at t=20 are most likely due to contaminants from the storage containers, pipework and flow meters during the treatment process. The UV/VIS absorption spectroscopy and TOC analysis results both had small errors which indicated that the results were reproducible.
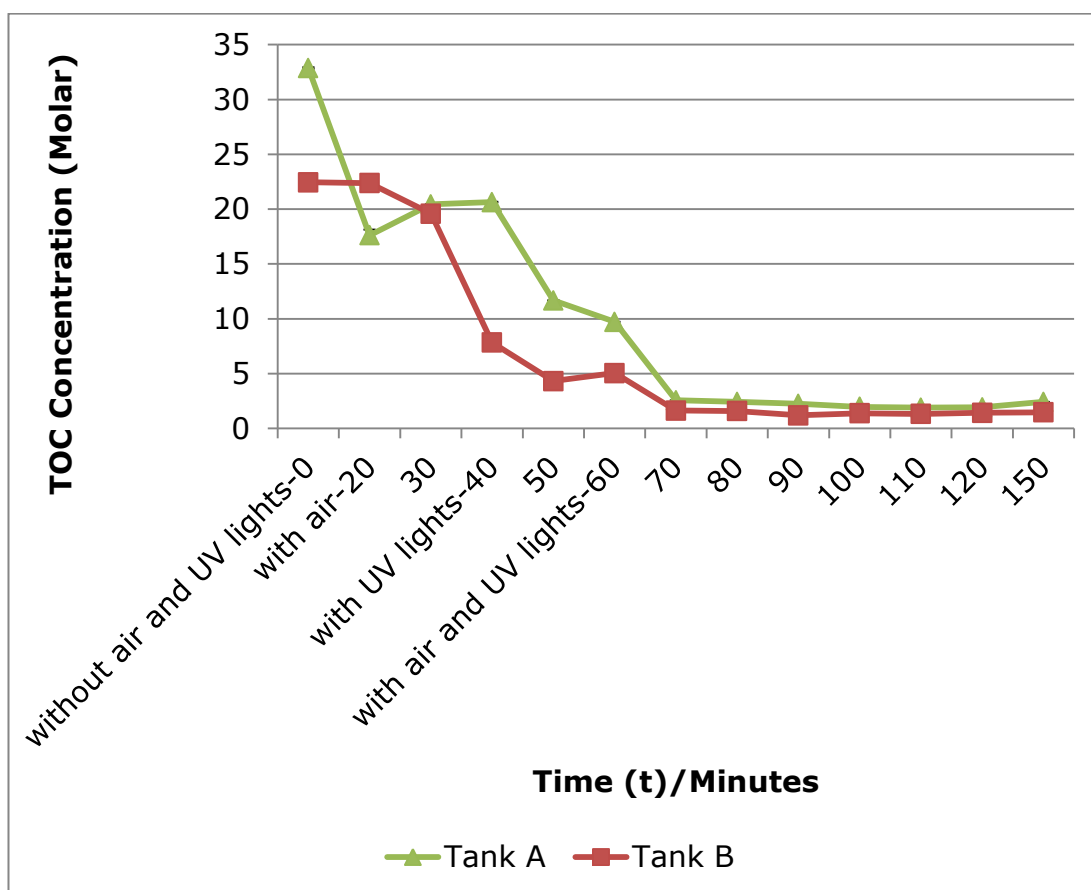
Figure 7-18 Photocatalysis Field Trial: Trial 5 results: Degradation efficiency curve of toluene in samples obtained from Tank A of the reactor monitored by TOC Analyser.

From Table 7-13, it can be seen that the percentage reduction of toluene in Tank A and Tank B were 92.58 % and 73.55 % respectively. It is clear that the efficiency of Tank A was slightly better than Tank B as was also seen in Trial 1. However, Tank B had higher removal efficiency for TOC in comparison to Tank A. This could be due to the lower starting TOC concentration in Tank B than Tank A. In conclusion, SAWTS had similar removal efficiencies for both MB and toluene.

Tank	Concentration before treatment (Molar)	Concentration after treatment (Molar)	Percentage Reduction (%)
toluene Concentrations			
A	$1.86 \times10^{-3}\pm1 \times10^{-4}$	$1.38\times10^{-4} \pm3\times10^{-6}$	92.58
B	$1.38 \times10^{-3}\pm2\times10^{-6}$	$3.65 \times10^{-4}\pm1 \times10^{-6}$	73.55
TOC Concentrations			
A	32.86±0.07	2.42±0.01	92.64
B	22.11±0.42	1.47±0.03	93.43

Table 7-13 Photocatalysis Field Trial: toluene and TOC concentrations of the wastewater samples before and after treatment by the SAWTS.

Remediation of contaminated groundwater

In Trial 6, contaminated groundwater remediation using the reactor was investigated and the TPC content in the contaminated groundwater samples was monitored using FTIR spectroscopy. FTIR analysis of these samples clearly showed the presence of bands between 3100- 2700 cm^{-1}, which indicated the presence of hydrocarbons (Loconto 2006). A calibration curve of peak area (Acm^{-1}) against diesel concentration (ppm) was generated using a series of known concentration of diesel standards. The peak area of the sample before treatment was 9.55 Acm^{-1} and after treatment was 0.91 Acm^{-1} showing a significant decrease in TPHs. The concentration of TPH present calculated from the regression line of the calibration curve (Figure 7-19) demonstrated that TPHs in the sample before and after treatment with the photocatalytic reactor for 300 minutes was 40.74 ppm and 3.50 ppm respectively demonstrating 91.41 % of TPH removal efficiency (Figure 7-20).
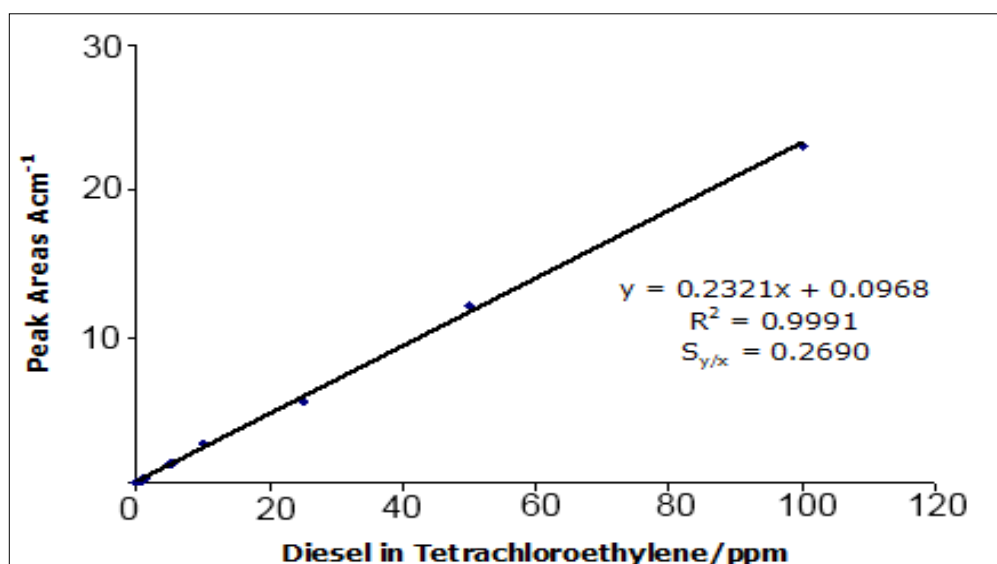
Figure 7-19 shows Photocatalysis Field Trial 6: a calibration graph for the determination of TPH using FTIR Spectroscopy.
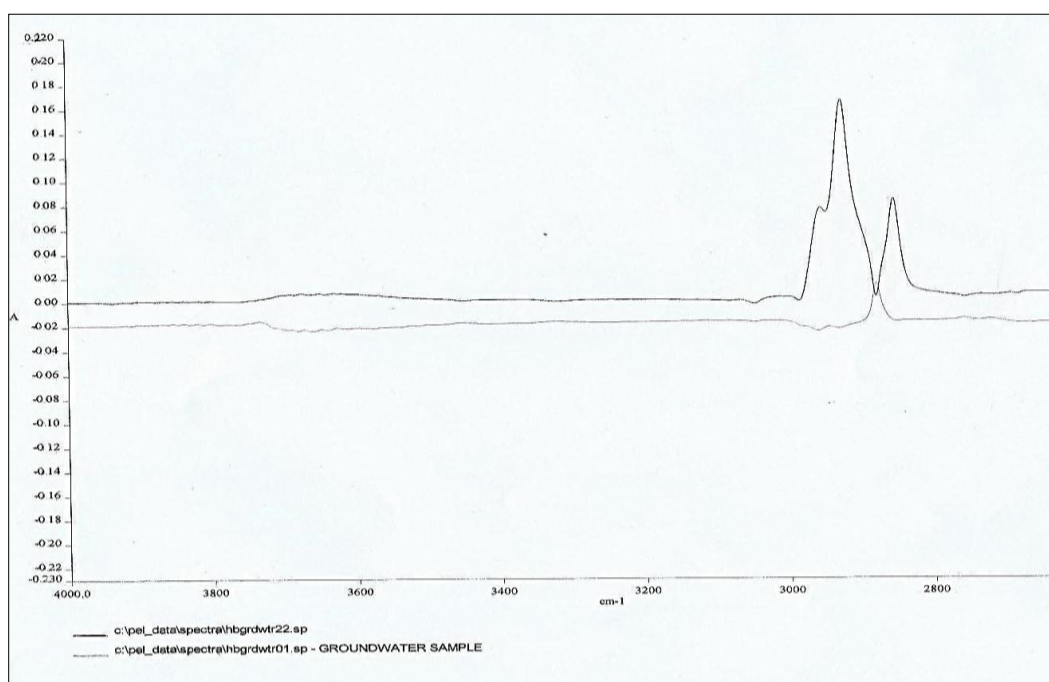
Figure 7-20 Photocatalysis Field Trial 6 result: Spectra of the contaminated groundwater before (top line) and after (bottom line) treatment by the photocatalytic reactor.

Trial Difficulties and Recommendations

There were several technical difficulties encountered during the field trials (as shown in Figure 7-21). One of the biggest challenges encountered was the overflowing of the effluent from the overflow outlet on both Tank A and Tank B. The treated effluent outlet was drained by gravity and cannot be pumped out. Therefore, the influent flow was faster than the outgoing treated effluent. The incoming flow could be adjusted to suit the outflow, however, that would restrict the reactor treatment capacity. The outlet of the tanks could be enlarged to enhance drainage of the treated effluent.

As stated in Section 7.3.2.a, wastewater was transferred via a 240 V electrical pump with flow control into SAWTS. The 240 V pump was powered by the transfer station office mains voltage (230 V). The pump was old and did not function properly during the trials despite multiple repairs by a Sureclean technician. Also, according to the Health and Safety Executive (2011), "where mains voltage (230 V) is used, the risk of injury is high if equipment, tools, or leads are damaged or there is a fault". The Health and Safety Executive (2011b) also stated that, "As a general rule the lower the voltage used the lower the risk of a serious electric shock. Where reasonably practicable hand held equipment should operate at 110V, or less". Although the pump was not hand held, the 240 V pump had a comparative higher voltage than 110 V and may have higher electrical injury risks than 110 V, and this would be unsuitable if SAWTS is to be used in an industrial setting. Alternatively, a pneumatic diaphragm pump may be used in place of an electrical pump.
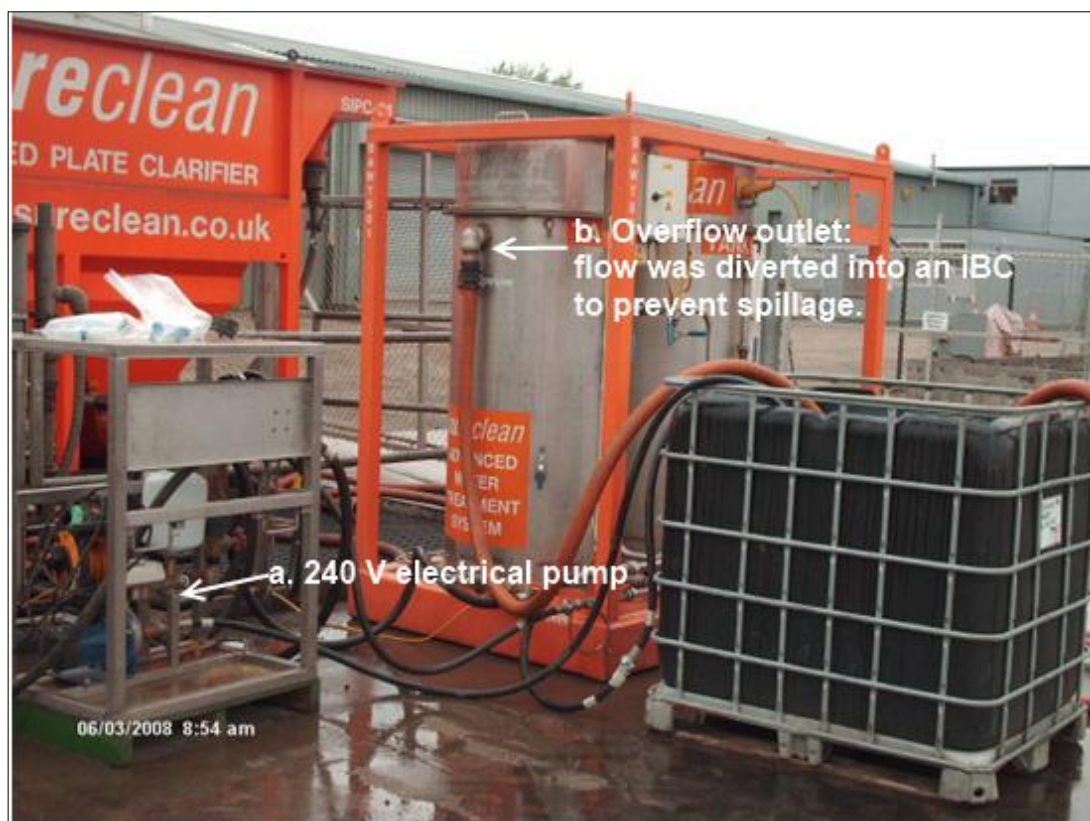
Figure 7-21 Photocatalysis Field Trial: A photo showing two main technical difficulties encountered during the field trials, a. 240 V electrical pump failed to function; b. constant spillage from SAWTS overflow that lead to hoses being fitted on the overflow outlet.

7.3.2.c   Conclusion

The industrial scaled semiconductor photocatalytic reactor discussed in this research employs a low cost and simple mechanism in water treatment by using TiO_2 pellets in the effluent. Moreover, the reactor was designed to be mobile and modular to support treatment on-site as well as off-site or even remote areas. The wastewater remediation of MB, toluene and contaminated groundwater was investigated. The results reveal that the reactor was capable of removing up to 97.89 % of MB by treatment with Tank A for 150 minutes. In the efficiency test, Tank A was demonstrated to have higher efficiency than Tank B with the removal efficiency of 57.73 % and 42.42 % respectively. When the reactor was operating under continuous mode (Tank A and Tank B together), 96.20 % removal efficiency

for MB was achieved. For toluene contaminated effluent, Tank A achieved 92.58 % and 92.64 % of toluene and TOC removal efficiency respectively. The results from the treatment of the contaminated groundwater collected from an ex-gas work site indicated that the reactor was capable of removing more than 90 % of the total hydrocarbon. These results also demonstrated promising prospect for the reactor in environmental remediation. In conclusion, the continuous mode using both Tank A and Tank B was proven to be better than batch mode. Depending on pollutants, the recommended time for the treatment of toluene, MB and the contaminated groundwater was 60 minutes. The remediation time corresponded to a study conducted by McCullagh et al. (2010) using a continuous flow reactor, where degradation of 1×10^{-5} M of MB took 60 minutes.

The trials also showed that there were some technical difficulties when operating SAWTS which included the electrical pump and the overflow outlet of SAWTS. The efficiency of SAWTS could be further improved if these problems were solved.

7.3.3   SAWTS Refurbishment

Based on the field trials conducted in Section 7.3.2, it was concluded by Sureclean that a few adjustment to the SAWTS was required. The flow diagram before refurbishment can be seen in Figure 7-22. The changes proposed to Sureclean were shown in Figure 7-23. The key changes required were to source a pump with pressure gauge and flow meter, as well as to alter the pipework the outflow pipes so that the treated effluent could be diverted quickly out of SAWTS via the extra tank by using a pump. A diaphragm pump and a bag filter prior to SAWTS were also proposed to be added. A pneumatic driven diaphragm pump was chosen instead of the electrical pump because it was thought to have a smaller chance of leakage and would be more suitable for handling hazardous wastewater (Sahu 2000; Noyes 1992). A pulsating damper could be installed with the diaphragm pump, as this type of pump causes sharp pulsation. The bag filter prevents debris and suspended solids to enter the reactor. A digital or

mechanical flow meter and a check valve could be installed at the inlet of the reactor. The flow meter would provide accurate readings to the incoming flow. The extra tank enables fluid to be pumped to a designated storage facility, which would not be possible in the original version of SAWTS as the fluid could only be drained by gravity. The overall adjustment would improve SAWTS performance and further field trials could be carried out to validate the efficiency.
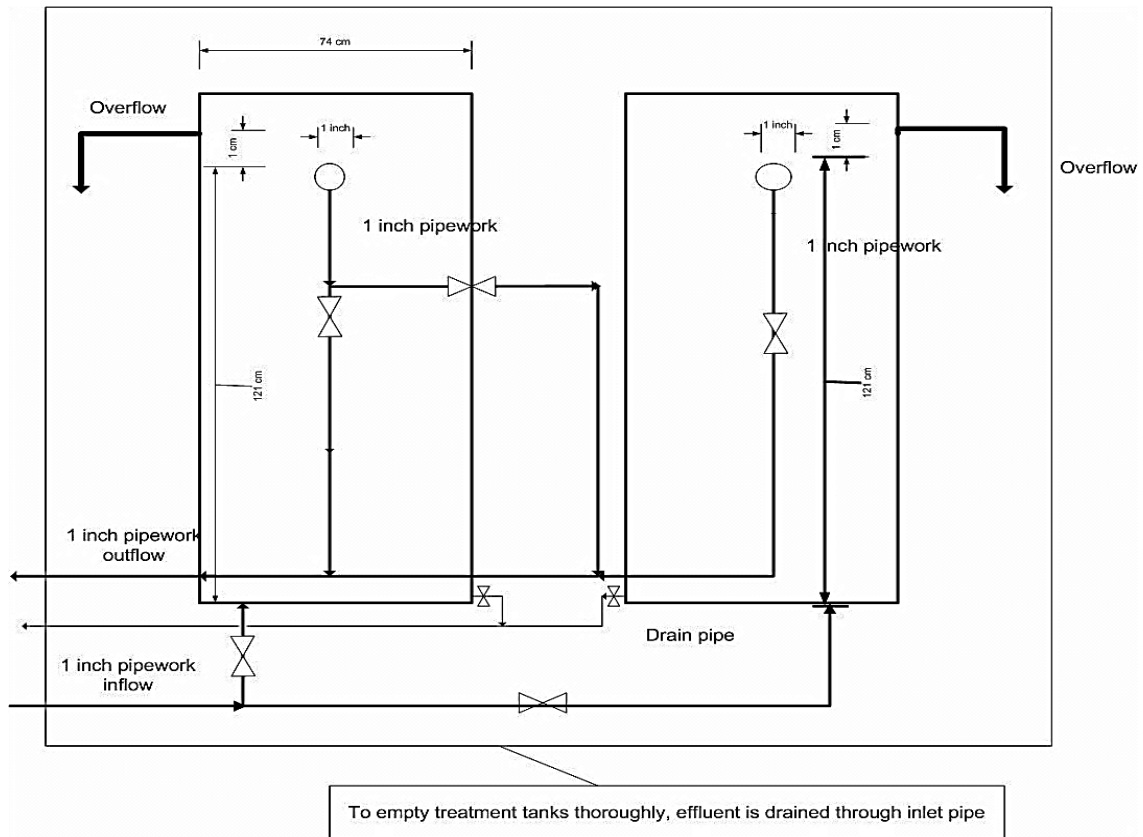
Figure 7-22 demonstrates the original flow design of SAWTS.
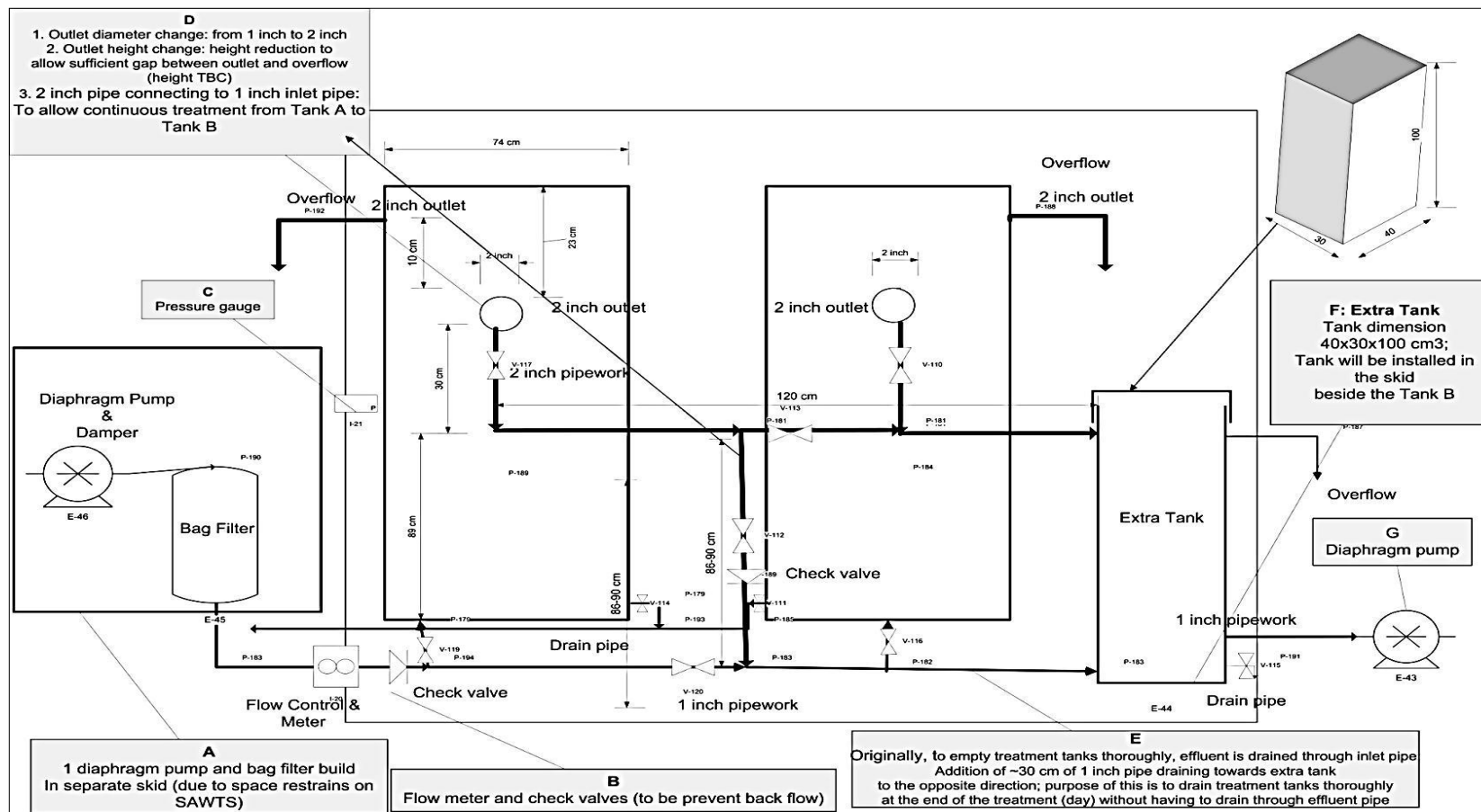
Figure 7-23 demonstrates the schematic representation of SAWTS after the refurbishment- new design.

7.3.3.a   Outcome and Future Work

Due to time and budget constraints, the refurbishment has only been partly completed. As shown in Figure 7-24, the pipe-work of the outlet adjustment and an extra tank were installed on SAWTS. The outlets for both Tank A and Tank B were enlarged from the original 1 inch diameter to 2 inches diameter, so that the treated effluent was prevented from overflowing. The extra tank as seen in Figure 7-24 (b) was installed so the treated fluid could be pumped to a designated storage facility.

Figure 7-24 shows the refurbished SAWTS with (a) an enlarged outlet for SAWTS and (b) extra tank to enable effluent collection.

In the future, the refurbishment could be continued, to include the flow meter and a damper. Further field trials could be carried out to validate the efficiency of the unit by evaluating the degradation of other chemicals such as 4-chlorophenol.

7.4 Overall Conclusion

Sustainable development of environmental technologies is vital to sustain future needs for clean water and land. Environmental legislation remains one of the key drivers for the development of treatment technologies. EC was demonstrated to be effective in a variety of Sureclean generated waste streams (from the Alness and Blackdog interceptor systems) with varying degrees of contamination. Treatments were also undertaken on samples of bilge water. The bilge water demonstration successfully removed 99.9 % of petroleum hydrocarbons given the very high level of organic chemicals.

AOPs have been proven to be an effective treatment for toxic compounds. AOPs such as Fenton's (Fe^{2+}/H_2O_2), photoassisted-Fenton ($Fe^{2+}/H_2O_2/UV$) and ozone ($O_3/UV/\ H_2O_2$) and semiconductor photocatalysis are more effective in combination with more traditional methods such as biological or chemical treatment to enhance treatment efficiency. Semiconductor photocatalysis advanced water treatment is a clean technology. Research has been actively carried out on semiconductor photocatalysis; however, commercial application is still limited.

The treated effluent could be further processed using advanced oxidation process as discussed in Section 7.3. Therefore, future work could incorporate both the EC and AOP treatment together.

Chapter 8 General Conclusions and Recommendations for Future Work

Sustainable development is the development of our current needs without compromising the need of future generations. Businesses unquestionably impose impacts on the three pillars of sustainable development: environmental, economic and social issues. With the increasing need for CSR, Sureclean recognised the importance of the greater impact of their actions; therefore they integrated business and environmental matters into their operation and core strategy. Sureclean was treating its waste streams from a diverse range of sources. To battle the challenges of ever stringent environmental, regulatory and legislative requirements, as well as to maximise its operation efficiencies to attract new markets, integration of Sureclean solid and liquid waste is paramount to Sureclean future growth.

8.1 The Research Aims

This project aimed to investigate conventional and new technologies for the combined treatment of Sureclean solid and liquid waste streams. The treatment technologies were intended to have a small footprint (modular) so that transportation could be achieved either by lorry or inside a shipping container (mobile). The treatment solution was aimed to have a minimum start up time, ease of operation and low maintenance. The ideal design operation flow rate was 5 m^3 per hour. The resulting final discharge effluent had to meet the Sureclean Alness Waste Transfer Station discharge consent (as shown in Table 1-1) whilst any solid waste was expected to be fit for inert landfill disposal in the UK. To achieve this, the following objectives were completed:

8.1.1   MRes Objectives

1. Literature review on environmental legislation
2. Conduct market research on treatment technologies of solid waste and wastewater.

3. Review Advanced Oxidation Processes in treatment of contaminated water and soil.

4. Conduct a comprehensive analysis of the waste streams of the Sureclean waste treatment company, thus identifying major pollutants.

8.1.2   PhD Objectives

5. Conduct laboratory and field based technology trials with identified waste streams.

6. Evaluation and interpretation of results from field trials.

7. Evaluation and interpretation of results from laboratory trials.

8. Implementation of processes based on field trial reports and cost-benefit analysis.

8.2 Conclusion on Environmental Legislation

Legislation plays a vital role in managing waste safely and efficiently. It can be seen that regulation of waste is fundamental to protect public wellbeing and protection as well as to safeguard the environment. The Waste Framework Directive (WFD) (2006/12/EC) (amended as the WFD 2008/98/EC) on waste is considered one of the most important legislation for waste. The Directive endorses the prevention of waste by adopting a hierarchy of waste management selections (waste prevention, re-use, recovery, recycling and safe disposal). Traceability by means of labelling, record keeping and monitoring of waste from production to final destination (disposal) and control of hazardous waste is mandatory according to the Directive. The Landfill Directive (99/31/EC) requires the pre-treatment of waste before landfilling. In Scotland, hazardous waste is regulated according to the Special Waste Regulation 1996 and Special Waste Amendment (Scotland) Regulations 2004. For trade effluent discharges in Scotland, the Sewerage Authorities Act under the Sewerage (Scotland) Act 1968 requires licensing of discharges to the sewer. Under the Pollution Prevention and Control (Scotland) Regulations SI 2000/323, a stationary technical unit where one or more activities listed in Part 1 of Schedule 1 are carried out requires a PPC Part A. As Sureclean operates a hazardous waste

transfer station, they obtained a PPC Part A permit that set out conditions for their operations to prevent pollution. Regular monitoring and recording of data such as environmental releases are required by permit. Other supporting regulations that govern Sureclean waste treatment and disposal include the Offshore Petroleum Activities (Oil Pollution Prevention and Control) Regulations 2005 and the Offshore Petroleum Activities (Oil Pollution Prevention and Control) (Amendment) Regulations 2011 that control all deliberate oil discharges such as produced water (Great Britain 2009) and the Paris Convention (PARCOM) which set the discharge limit for water contaminated with oil at 40 mg/L. A knowledge and understanding of this large body of regulations was required to ensure that this research was directed towards waste treatment solutions that complied with the most up to date regulations.

8.3 Conclusion on Waste Streams Analysis

Waste streams classification and characterisation are essential to achieve an effective design of a solid waste and wastewater treatment system. Six typical Sureclean waste streams were investigated in this study: distillery effluent (DE), drilling fluid (DE), interceptor effluent (IE), produced water (PW), used oil based mud (OBM) and used oil based mud and water (OBMW). The waste streams were analysed with the following analyses: pH, particle size, GC-MS, COD, TOC, FTIR, Molecular Fluorescence and ICPAES. By using this combination of environmental analytical techniques and instrumentation, the chemical, biological, and physical characteristics of wastewater and solid waste streams were determined. A summary of the characterisation results can be found in Table 3-3. The results revealed that apart from IE, all other wastes exceeded the Sureclean discharge limit and therefore treatment would be required to reduce the contamination level.

8.4 Conclusion on Mechanical Separations

The treatment efficiency of different mechanical separation units were investigated for Sureclean waste streams. For the solid-liquid separation, a

decanter centrifuge was selected for the dewatering of sludge and slurry. Preliminary results appeared to show that the decanter centrifuge could reduce up to 56 % of the moisture content in oily sludge. However, the savings obtained did not pay off the annual capital and maintenance cost, which was a sum of £22,504.91 per year. Therefore, it was concluded that a decanter centrifuge would not benefit Sureclean at this stage.

Filtration was also investigated as another means of mechanical separation. The laboratory trials suggested that filtration (using a filter media called AFM) decreased TOC by approximately 34 %, all particles after treatment had a size less than 600 µm and there was also a reduction in the Al, Cu, Ti and Zn concentrations. Not all the heavy metals were analysed in this study, therefore the results could not be compared to Sureclean discharge consent. In the field trials, which involved GBF (a prototype bespoke steel tank filled with AFM and a CF) the TSS appeared reduced in all three trials.

Based on the results of the laboratory and field trials conducted in section 4.3.1 and 4.3.2, Sureclean decided to incorporate the GBF and a clarifier to develop a filtration-based technology to recycle and reuse wastewater and they called the unit SWTS01. The effluent samples were analysed before and after the filtration with the following parameters: TOC, particle size and heavy metals. Field trials were carried out to evaluate the efficiency of the SWTS01 treatment of landfill leachate. The best removal efficiencies results for BOD, COD and TSS appeared to be 46, 25 and 38 % respectively at a flow rate of 2 m^3/h. However, all heavy metals concentrations appeared to be unchanged. Therefore 2 m^3/h was recommended as the optimum flow rate for SWTS01 for the treatment of landfill leachate.

8.5 Conclusion on Chemical Treatment of Oily Waste

Coagulation is the reaction where chemical destabilisation of particles occurs to form aggregation. Therefore, colloid particles in wastewater are

difficult and not economical to be removed by conventional physical and mechanical means due to their long settling times or low velocities. Among the inorganic coagulants, the multivalent metal salts such as $AlCl_3$, $FeCl_3$ and Al_3SO_4 are more commonly used than electrolytes (Wakeman and Tarleton 1999) as they are thought to be cheaper than polymers. However, high molecular weight polymerised metal salts such as PAC are thought to be more effective in terms of floc formation and they perform in a wider range of pH and at lower dosage than non-polymerised metal salts. Two sets of laboratory trials were conducted to study the suitability and feasibility of chemical treatment towards Sureclean waste streams. The first trial investigated the use of chemicals for the removal efficiencies of heavy metals and TPH in Sureclean interceptor effluent; the second trial focussed on the evaluation of chemical conditioning towards dewatering Sureclean oily sludge.

The removal efficiencies of the FloQuat, FloPam, $FeCl_3$ and $AlCl_3$, as well as a combination of FloQuat, $FeCl_3$ and $AlCl_3$ with FloPam were investigated on Sureclean Interceptor wastewater. The results from Chapter 5 concluded that the treatment efficiency of oil and heavy metal removal with these chemicals was equal to that of simply raising the effluent pH to 7 by using NaOH.

In a separate field trial, the efficiencies of FloQuat and FloPam were investigated on a larger scale (1000 L of interceptor effluent). The preliminary results showed that the highest removal efficiencies were achieved on the TSS at 73.8 % and 80.0 % for 15 ppm and 20 ppm doses respectively whilst 27.7 % of heavy metal was removed from both 15 and 20 ppm of FloQuat. The preliminary results suggested that the overall optimum dosage for FloQuat and FloPam were at 20 ppm and 5 ppm respectively in the treatment of Sureclean interceptor effluent.

In the second laboratory trial, the effectiveness of chemical conditioning towards dewatering of Sureclean oily sludge was investigated to reduce

moisture content in the sludge, which would potentially reduce the final disposal cost and sludge handling. The chemicals or compounds used were lignite, PEI, FloQuat R100, fly ash and Charcoal. TSS Analysis and MC Analysis by weight loss were two parameters that were used to determine the condition performance. In this research it was found that the best dewatering performance for the oily sludge was conditioning with PEI and Lignite. However, due to poor temperature control during the sludge drying process to obtain the MC of the sludge; therefore, additional study is required for further recommendation to Sureclean. This study should also include an investigation of raising the pH to 7 in order to establish the optimum conditions.

8.6 Conclusion on Chemical Treatment of Sewage Sludge

To secure Sureclean business in septic tank waste disposal in the Highlands region, it was found to be imperative for Sureclean to treat the septic tank waste. A chemical sludge dewatering system that gave rise to a liquid and solid phase was developed; the system is called SSSTS. The sludge dewatering container consisted of a closed container, equipped inside with filtration screens to drain and dewater the flocculated sludge. Dewatering of sludge was achieved by introducing a cationic polymer such as PAM into the sludge through the pumping unit. As seen in the field trials, by dosing the sewage sludge with 0.5 % FloPam DW2160, followed by separation in the SSSTS, the sludge volume was reduced by 52.6 %. The treated wastewater pollutants such as COD were reduced from the initial value of 23,500 mg/L to 2676.67 mg/L. The TSS of the treated effluent was 1037.33 mg/L, which exceeded Sureclean discharge limit; therefore further treatment such as filtration should be employed to reduce the pollutant loads. Further trials involving different concentrations of chemical could be conducted to further verify the optimum dosage of FloPam DW2160 for the dewatering of sewage sludge.

8.7 Conclusion on Physico-Chemical Treatment

Physico-chemical treatment involved the combined chemical and physical processes in the treatment system. Two wastewater treatment methods were investigated: EC and Semiconductor Photocatalysis. The process of EC involved the electrolytic oxidation of a metal anode, such as aluminium or iron, which could generate coagulants in situ in the EC system with simultaneous formation of hydroxyl ions and H_2 gas at the cathode (Arvanitoyannis 2008). In the initial EC prototype trial (EC operated at amperage between 30 -45 A at pH near neutral) was shown to reduce TPH, TSS, COD and total heavy metals of four different Sureclean oily wastewater streams, with the exception of the COD result for oily wastewater collected from the shaker tank. The COD was shown to increase which would be possible if compounds in the shaker tank reacted with the Fe (II) ions from the electrodes to form soluble products which stay behind in the solution. Based on the preliminary results obtained from the prototype trials, Sureclean and Kemartek Technologies Limited developed the SEWTS. Field trials were performed to evaluate the performance of SEWTS with three waste streams: Alness and Aberdeen WTS interceptor effluent and Bilge water collected from Aberdeen harbour. The preliminary results showed that EC could reduce TPH for all three waste streams, with the best result seen in bilge water (99.9 % removal efficiency). However the results of TSS and COD for treated bilge water were seen to have little improvement after the treatment by EC. For Aberdeen interceptor effluent, all the treated parameters were within the Sureclean discharge consent except TSS. Therefore it can be seen that EC can be effective in the treatment of TPH. The type of pollutant presents in the wastewater was seen to influence the removal efficiencies of COD; because iron ions from the electrodes reacted with the pollutants. More trials could be conducted to improve the certainty of these results.

The efficiency of a twin tank (Tank A and Tank B) photocatalytic reactor (SAWTS01) was investigated using MB, toluene and contaminated groundwater samples from an ex-gas works. The reactor was designed to use a pellet form of titanium dioxide catalyst which was directly submerged

in the effluent. A series of trials were carried out and it was found that treatment in continuous mode was more effective than single tank batch mode with approximately 95.88 % removal of MB demonstrated in continuous mode. Tank A achieved 92.58 and 92.64 % removal of toluene and TOC respectively. FTIR analysis suggested that 91.41 % of TPH removal was achieved after the treatment of contaminated groundwater using the photocatalytic reactor. These promising results demonstrated a desirable prospect in an environmental application. However, there were several technical difficulties encountered during the field trials. One of the biggest challenges encountered was the overflowing of the effluent from the overflow outlet on both Tank A and Tank B, therefore SAWTS01 was refurbished so that the pipework of the outlet was adjusted and an extra tank was installed on the SAWTS. In the future, a diaphragm pump, a bag filter prior to SAWTS, a pulsating damper, a digital or mechanical flow meter and a check valve could be installed at the inlet of the reactor to improve the overall effectiveness of the reactor.

8.8 Conclusion of the Integration of the Developed Waste Treatments

As seen in Figure 8-1 (page 220), this research expanded Sureclean waste treatment capabilities and an integrated system was developed to treat different waste streams and to improve the treatment efficiency. Oily wastewater could be treated with SEWTS01 followed by SIPC to remove colloid particles and oil. Studies shown in Section 7.2.2.c and 7.2.3.c demonstrated that some of the treated effluent complied with Sureclean discharge consent. The treated effluent could be discharged to Sureclean interceptor where further treatment using SWTS01 could further reduce the TSS and TPH levels as a final polish of the effluent. For sewage sludge, SSSTS was developed to dewater the sludge thereby reducing the water content and save cost on transport. The study conducted in Section 6.4 showed that although COD and TSS in the treated effluent exceeded the discharge consent, further treatment using SWTS01 and SAWTS01 could improve the quality.

Mechanical separation using a decanter centrifuge was shown to be effective in shaker tank oily sludge (as seen in Section 4.2.2), however, the cost-benefit analysis demonstrated that it was not economically sound for Sureclean to purchase a decanter centrifuge. The SSSTS could dewater the sewage sludge, however, further work would be required to enable the sludge to be used in agriculture as land application (thereby diverting from landfill).

In conclusion, three new systems were developed in this research: SWTS01, a mechanical separation system for the removal of solids from liquid wastewater; SEWTS, an EC system that agglomerate colloid particles and demulsify oil; and SSSTS, a sludge dewatering system that separates chemically flocculated sludge and the aqueous part of the sludge. Based on this research and the data gathered so far, Sureclean could also further develop chemical treatment of oily waste, as well as investigate the use of a decanter centrifuge for oily sludge separation. Sureclean could integrate these three new systems into their existing treatment regime to enhance their in-house waste treatment; the company could also introduce these treatment systems to their clients.
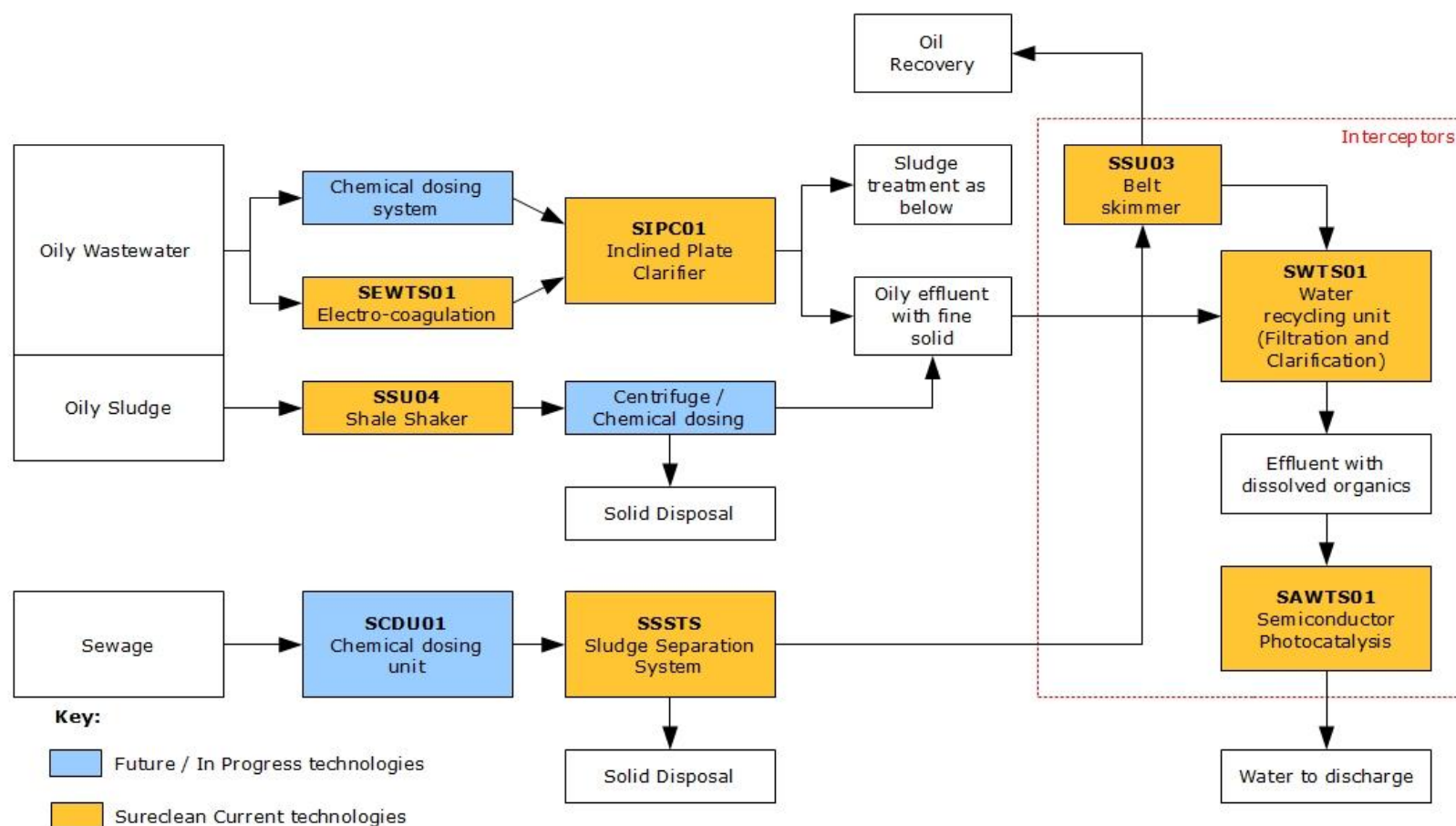
Figure 8-1 Schematic representation of Sureclean Integrated Solid and Liquid waste treatment

8.9 Recommendations for Future Work

8.9.1 Mechanical Separation

The treatment efficiency of the SWTS01 using a range of Sureclean waste streams could be further evaluated. In order to evaluate the significance (i.e. with replication) and compare the performance of SWTS01 in the treatment of leachate and other waste streams, further work involving replication of analysis for each sample should be carried out to improve certainty. Further trials could also be conducted with different wastewaters and different filter media types (such as coconut based-granular AC, crushed stone and garnet sand).

8.9.1 Chemical Treatment of Oily Waste

Chemical treatment was shown to improve the quality of Sureclean interceptor effluent based on the findings seen in the laboratory and field trials (as seen in Section 5.2.1 and 5.2.2). The outcome of the laboratory trials suggested that by raising the pH of the interceptor effluent to pH 7 using NaOH, the oil and heavy metal removal efficiencies were almost as effective as adding FloQuat and FloPam alone. Therefore, large scale trials (1000 L or more) could be carried out at Sureclean WTS using NaOH to alter the pH. A chemical dosing system as described in Section 5.1.3 that involves a dosing pump and a chemical mixing system could be introduced to Sureclean WTS.

8.9.2 Conclusion on Chemical Treatment of Sewage Sludge

The preliminary results showed that SSSTS could reduce sludge volume. Further trials to identify the optimum dosage of FloPam DW2160 for the dewatering of sewage sludge could be conducted. A future upgrade of the whole sludge dewatering process would be required as seen in Figure 6-14. A sludge screen, an odour control system and a dosing unit is pertinent to the dewatering unit and could improve the overall treatment process. Screening could remove objects such as rags, paper, plastics or any large objects to

prevent damage and clogging of downstream equipment and piping. This also enables the treated sludge to have a higher recyclable value by retaining valuable nutrients. Odour control is a crucial step when using SSSTS, therefore a system such as a scrubber should be installed. It is also essential to optimise the dosing and mixing system of SSSTS with the incoming waste, therefore Sureclean could either purchase (as seen in Section 6.4.3) or design their own sludge dosing and mixing system. Further studies could be performed to improve the sludge quality for land application.

8.9.3   Physico-Chemical Treatment

Sureclean as a waste treatment company could apply the SEWTS system to sewage sludge as a pre-treatment process for dewatering as reported by Shin and Lee (2006) in Korea where pressure was applied to the coagulated solids after treatment with EC. Sureclean could also expand its client bases to other industries such as those described in Section 7.2. In Chapter 4, distillery effluent was examined and various studies showed that EC could remediate this type of waste (Kannan, Karthikeyan and Tamilselvan 2006). A combination of the EC and floatation processes could improve the effluent quality as reported by various studies (Wang et al. 2010; Pouet and Grasmick 1995). Boroski et al. (2009) proved that there were reductions of COD from 1753 mg/L to 160 mg/L after EC and 50 mg/L after EC and heterogeneous photocatalysis treatment for wastewater collected from the pharmaceutical and cosmetic industry. The treated effluent could be further processed using the AOP as discussed in Section 7.3. Therefore, Sureclean could incorporate both the EC and AOP treatment together to enhance the efficiency in the future. The cost-benefit analysis shown in Table 7-7 clearly shows that the EC would benefit Sureclean if the unit was fully utilized.

For SAWTS, the key changes as discussed in Section 7.3.3 were to source a pump with a pressure gauge and flow meter, a bag filter prior to SAWTS was also proposed to be added. A pneumatic driven diaphragm pump and a pulsating damper could be installed. The bag filter could prevent debris and suspended solids from entering the reactor. A digital or mechanical flow

meter and a check valve could be installed at the inlet of the reactor in order to provide accurate readings of the incoming flow.

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Appendix 1- MHSR 414FTVB Decanter Centrifuge Specification

Centrifuges un-limited		MHSR 414FTVB DATA SHEET		
Contract No :		Document No :		Rev : 1
Client :		Equipment Spec :	DMNX 414FTVB	
Project :		Drawing No:	1209111	
Location :		Doc. Issuer :		

POWER REQUIREMENTS		
MAIN DRIVE MOTOR		380/460 VOLT, 3 PHASE 37 Kw
MAKE & SPECIFICATION		F&G 3000/3600 RPM . EExd IIC T4
MAIN DRIVE		VOITH TYPE TVYRC 366
BACK DRIVE MOTOR		380/460 VOLT, 3 PHASE 13/16 Kw
MAKE & SPECIFICATION		F&G 1500/1800/3000/3600 RPM . EExd IIC T4
FEED PUMP MOTOR		380/460 VOLT, 3 PHASE 7.5 Kw
MAKE & SPECIFICATION		F&G 1500/1800 RPM . EExd IIC T4
ELECTRICAL POWER OUTPUT	KW	54
MAX START UP CURRENT	Amps	97
NORMAL RUNNING CURRENT	Amps	67

TIMING DATA		
ACCELERATING TIME TO OPTIMUM SPEED		1 TO 2 MINUTES
RUNNING DOWN TIME		10 MINUTES

DIMENSIONS		
LENGTH	inches/mm	94/2400
WIDTH	inches/mm	79/2000
HEIGHT	inches/mm	59.8/1520
WEIGHT (APPROX)	lb/kg	1181/2600

Centrifuges un-limited

MHSR 414FTVB DATA SHEET

Contract No :		Document No :		Rev : 1
Client :		Equipment Spec :	DMNX414FTVB	
Project :		Drawing No:	1209111	
Location :		Doc. Issuer :		

GENERAL			
BOWL DIAMETER	mm	353	
BOWL LENGTH – CYLINDRICAL	mm	300	
BOWL LENGTH – CONICAL	mm	560	
LAMBDA (SLENDERNESS) RATIO		2.44	
HALF CONE ANGLE		8.5 degrees	
CONVEYOR TYPE		ESJBERG 110 mm PITCH	
MAX. BOWL SPEED	rpm	4000	
MAX. "G" FORCE @ MAX. RPM		3157	
AREA EQUIVALENT @ MAX. RPM		1619m³ @118mm LIQUID RADIUS	
MOMENT OF INERTIA	kgm²	9.0	
BOWL SPEED ADJUSTABLE		YES – BY CHANGING OUT PULLEYS	
MAX. DIFFERENTIAL SPEED @ MAX. RPM		104	
DIFFERENTIAL SPEED VARIABLE		YES – 5 INSTANTANEOUS DIFFERENTIAL SETTINGS	

OPERATIONAL		
VOLUME		28 LITRES MAX. DEPENDING UPON LIQUID RADIUS
DISCHARGE CAPACITY IN m³/HR		MAX. 8.0
MINIMUM D50 CUT POINT (MICRONS)		DEPENDENT UPON FLUID AND SOLIDS DENSITY AND FLUID RHEOLOGY
VARIABLE POND DEPTH	mm	46.5 to 66.5
LIQUID RADIUS (STANDARD SIZES)	mm	110 to 125
VARIABLE FEED TUBE POSITION		YES

GEAR BOX		
GEAR BOX RATIO		1:57
G/BOX TORQUE OUTPUT WORKING LIMIT	kNm	3
G/BOX TORQUE OUTPUT MAXIMUM LIMIT	kNm	3.5

PROTECTION		
OVERLOAD PROTECTION		GS COUPLING
OVERLOAD LIMITING TORQUE		65Nm
ADDITIONAL SAFETY PROTECTION		COVER OPEN/ VIBRTN SWITCH , THERMAL PROTECTION

TECHNICAL SUMMARY

MHSR 414 FTVB - VARIABLE MAIN DRIVE - 5 SPEED BACK DRIVE

1. Centrifuges Unlimited decanter type MHSR 414 multiple purpose machine suitable for high volume barite recovery and solids removal.

    All process contact parts in stainless steel, specially hardfaced conveyor flights Tm 41, bowl inlet zone hardfaced and sintered tungsten carbide solids discharge bushings. Heavy duty 3.5 kNm planetary gearbox.

Bowl diameter	353 mm
Bowl length	860 mm
Lambda (slenderness ratio)	2.44
Volume	26 litres max. Dependent upon liquid radius
Max. bowl speed	4000 rpm
Normal operating range	1588-3415 rpm
Max. g-value	3157g
Normal Operating g-value	498g – 2303g
Differential Speed	0 to 104 rpm
Moment of inertia	9.0 kgm^2

    Standard seal material is nitrile, other materials can be supplied on request

    The decanter is equipped with a hydraulic Voith Coupling and belt-pulley for main drive and a belt pulley system for the back drive, discharge funnels for solids and liquid discharge as well as a gearbox overload switch, cover open switch and vibration switch.

2. Main drive motor 380/460 volts, 50/60 Hz, 3 phase 37 kW, 3000/3600 rpm EExd 11C T4.

3. Main drive voith type TVYRC 366 turbo coupling for soft starting main drive motor complete with 3 pulleys and 3 sets of belts to give bowl speeds of 1588, 2444 & 3415 rpm at 60 Hz.

4. Back drive motor 2 direction 2 speeds with electrically operated brake, 380/460 V, 50/60 Hz, 3 phase, 13/16 kW, 1500/1800/3000/3600 rpm EExd 11C T4. This motor gives 5 different differential speeds at a given bowl speed by means of a 5 position switch mounted on the main panel.

5. Electrical control system type EExd 11B T4 including main isolator, separate short circuit and overload protection for all drives, control circuit, isolating transformers with primary and secondary mcb's, timers, relays, indicator lights etc. Main drive start/stop, feed pump start/stop and 5 position conveyor speed switch, automatic safety shut down system.

6. Oilfield type skid, sandblasted, zinc coated and enamel finish.

7. Variable speed feed pump type progressive cavity, positive displacement fitted with belt type variator complete with a 380/460 V, 50/60 Hz, 3 phase motor, 7.5 kW, 1500.1800 rpm EExd 11C T4. Totally enclosed, direct start protection, suitable for Zone 1 specifications. The complete pump unit is mounted on a skid assembly and includes couplings, guards, flanges and power cable.

Appendix 2- WAC Testing for Peaty Sludge

Waste Acceptance Criteria Testing BS EN 12457			SEVERN TRENT STL ENVIRONMENTAL LEADERSHIP
Part 2, Single Stage Process			
Interim			

Sample Details		Test Values		
Sample Number	10207295	Mass of Raw Test Portion (MW) kg		0.918
Job Number	461205	Mass of Dried Test Portion (MD) kg		0.090
Sample ID	SS73/07	Moisture Content Ratio (MC) %		926
Site	Peat Sludge	Dry Matter Content Ratio (DR) %		9.8
Job Description	Quote 44842A	Leachant Volume (L) Litre		0.067
Date Sampled		Eluate Volume (VE) Litre		0.340
Date Received	05/10/2007			
Particle Size (<4mm)	>95%			
Method of size reduction	N/A			
Non-crushable matter	N/A			

Eluate Analysis	Concentration in Eluate		Amount Leached	Landfill Waste Acceptance Criteria		
				BS EN 12457-3 Limit Values (mg/kg) at L:S 10:1		
Liquid : Waste Ratio	**10:1**		**10:1**			
Sample Number	10207296					
pH	7.19				Stable Non-Reactive	
Temperature °C	21					Hazardous
Conductivity uS/cm	150.3			Inert Waste	hazardous waste in non-hazardous	Waste
	mg/L		**mg/kg**			
Arsenic as As	<0.0050		**<0.050**	0.5	2	25
Barium as Ba	0.077		**0.77**	20	100	300
Cadmium as Cd	<0.00010		**<0.0010**	0.04	1	5
Chromium as Cr	<0.0025		**<0.025**	0.5	10	70
Copper as Cu	<0.010		**<0.10**	2	50	100
Mercury as Hg	<0.00050		**<0.0050**	0.01	0.2	2

Molybdenum as Mo	<0.0020		**<0.020**	*0.5*	*10*	*30*
Nickel as Ni	<0.020		**<0.20**	*0.4*	*10*	*40*
Lead as Pb	<0.010		**<0.10**	*0.5*	*10*	*50*
Antimony as Sb	<0.0060		**<0.060**	*0.06*	*0.7*	*5*
Selenium as Se	0.014		**0.14**	*0.1*	*0.5*	*7*
Zinc as Zn	<0.025		**<0.25**	*4*	*50*	*200*
Chloride as Cl	15		**150**	*800*	*15000*	*25000*
Fluoride as F	<0.20		**<2.0**	*10*	*150*	*500*
Sulphate as SO$_4$	82		**820**	*1000*	*20000*	*50000*
Total Dissolved Solids (TDS)	<200		**<2000**	*4000*	*60000*	*100000*
Phenol Index	<0.050		**<0.50**	*1*		
Dissolved Organic Carbon (DOC)	To Follow			*500*	*800*	*1000*
Waste Analysis						
Total Organic Carbon w/w %				*3%*	*5%*	*6%*
Loss on Ignition %						*10%*
BTEX mg/kg				*6*		
PCBs (7 congeners) mg/kg				*1*		
Mineral Oil (C10 - C40) mg/kg				*500*		
PAHs mg/kg				*100*		
pH					*>6*	
Acid Neutralisation Capacity (pH4) mol/kg					*To be evaluated*	*To be evaluated*
Acid Neutralisation Capacity (pH7) mol/kg					*To be evaluated*	*To be evaluated*

Appendix 2

Additional Eluate Analysis	Concentration in Eluate		Amount Leached
	10:1		10:1
	mg/L		mg/kg

Additional Waste Analysis	Units	Result
Moisture at 105c	%	90

Sample Comments	
10207295	
10207296	

Appendix 3- Biological Wastewater Analysis Profile: Waste stream 1 and 2 characterisations.

Waste Stream 1		
Parameters	Sampling 1	Sampling 2
pH	6.29	6.54
BOD (5 day) (mg/L O)	9850	6370
COD (total) (mg/L O)	19800	17800
TSS (mg/L)	8730	10000
As (mg/L)	<0.05	<0.05
Co (mg/L)	<0.01	0.014
Cr (mg/L)	0.086	0.17
Cu (mg/L)	2.63	0.141
Mo (mg/L)	<0.05	<0.05
Ni (mg/L)	0.032	0.098

Waste Stream 2		
Parameters	Sampling 1	Sampling 2
pH	7.04	7.12
BOD (5 day) (mg/L O)	408	402
COD (total) (mg/L O)	1050	550
TSS (mg/L)	204	750
As (mg/L)	<0.05	<0.05
Co (mg/L)	<0.01	<0.01
Cr (mg/L)	0.013	<0.01
Cu (mg/L)	0.74	0.52
Mo (mg/L)	<0.05	<0.05
Ni (mg/L)	0.011	0.12

Appendix 4- EC Prototype Trial-Heavy Metals Analysis Results

1. Shaker tank oily wastewater

Sample Name	Shaker Tank	Treatment at 45 A
As	N/A	0.111
Cr	1.1	0.03
Co	0.18	0.0289
Cu	17.8	0.002
Pb	3.77	0.012
Mo	0.19	0.002
Ni	0.7	0.11
Total heavy metals	**23.74**	**0.30**

2. Waste oil tank oily wastewater

Sample Name	Waste Oil Tank	Treatment at 30 A	Treatment at 35 A	Treatment at 45 A
As	N/A	0.018	0.022	0.018
Cr	0.24	0.028	0.23	0.11
Co	0.0525	0.004	0.0024	0.0014
Cu	3.85	0.009	0.004	0.003
Pb	2.03	0.002	0.002	0.002
Mo	1.3	0.017	0.035	0.02
Ni	0.16	0.34	0.13	0.12
Total heavy metal	**7.63**	**0.40**	**0.43**	**0.27**

3. Interceptor 5 oily wastewater

Sample Name	Interceptor 5	Treatment at 35 A
As	0.059	0.041
Cr	0.33	0.022
Co	0.029	0.0007
Cu	1.95	0.017
Pb	0.53	0.002
Mo	0.083	0.005
Ni	0.26	0.0285
Total heavy metal	**3.24**	**0.12**

4. Interceptor 6 oily wastewater

Sample Name	Interceptor 6	Treatment at 25 A	Treatment at 30 A	Treatment at 35 A
As	0.119	0.051	0.049	0.049
Cr	0.017	0.014	0.012	0.014
Co	0.0049	0.0006	0.0007	0.0008
Cu	0.23	0.015	0.016	0.006
Pb	0.075	<0.002	<0.002	0.002
Mo	0.012	0.005	0.005	0.005
Ni	0.0295	0.0217	0.0224	0.026
Total heavy metal	**0.49**	**0.11**	**0.11**	**0.10**