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## Novel organometallic catalyst for efficient valorization of lipids extracted from *Prunus domestica* kernel shell in sustainable fuel production

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### ABSTRACT

This study focuses on converting Plum Kernel Shell (PKS) waste biomass into biodiesel using a novel synthesized heterogeneous catalyst, contributing to the pursuit of renewable fuel from sustainable resources. Plum Kernel Shell (PKS) is waste biomass generated from plum fruit and available abundantly; utilizing it can help in many ways, such as overcoming environmental issues and promoting a circular economy. The precursor for the heterogeneous catalyst is derived from post-oil extraction waste biomass and further modified with metallic oxides (CuO and Mo) due to its acidic nature to enhance its efficacy for biodiesel production. Thorough characterization of the synthesized catalyst was conducted using analytical techniques such as XRD (X-ray diffraction), SEM (Scanning Electron Microscopy), EDS (Energy-Dispersive X-ray Spectroscopy), BET (Brunauer-Emmett-Teller), and XPS (X-ray Photoelectron Spectroscopy) to elucidate its nature and performance. The transesterification process was systematically optimized by varying parameters such as temperature, time, methanol-to-oil ratio, and catalyst loading. The optimized yield of 92.61 % of biodiesel resulted under ideal conditions, specifically at 65 °C, 150 min, 5 wt% catalyst loading, and an 18:1 M ratio. The biodiesel derived from PKS oil exhibited promising fuel properties encompassing cold flow properties, density, viscosity, cetane number, and flash point, validating its potential as a viable alternative fuel source. Furthermore, the synthesized novel catalyst demonstrated exceptional efficiency, retaining stability over five cycles without significant reduction in biodiesel yield. These findings underscore the viability of PKS biomass as a renewable and sustainable source for both catalyst synthesis and biodiesel production.

### Introduction

The consideration of alternative energy resources is imperative due to the declining reserves of fossil-derived fuels [1,2]. The combustion of

these conventional fuels emits greenhouse gases, contributing to environmental degradation [3]. With industrialization and a burgeoning global population, the demand for such fuels is on the rise, underscoring the need for viable alternatives [4–6]. Among various options like solar,

**Abbreviations:** PKS, Plum Kernel Shell; CuO, Copper oxide; Mo, Molybdenum; XRD, X-Ray diffraction analysis; SEM, Scanning Electron Microscopy; EDS, Energy Dispersive X-Ray Spectroscopy; BET, Brunauer-Emmett-Teller; XPS, X-ray photoelectron spectroscopy; FFA, Free Fatty Acid; GC–MS, Gas Chromatography–Mass Spectrometry; ASTM, American Society for Testing and Materials; EN, European Standards; JCPDS, Joint Committee on Powder Diffraction Standards.

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wind, and geothermal energies, biomass stands out as a versatile solution, especially in liquid fuel production, offering convenient storage and transportation possibilities. Biomass-derived oil, processed into biodiesel, presents a viable substitute for traditional diesel, eliminating the need for extensive engine modifications [7,8].

During the early 20th century, methyl esters derived from triglycerides were identified as potential fuel sources [9,10]. Initially sourced from edible oils, their use raised concerns regarding food scarcity versus fuel production [11]. To avoid this debate, researchers turned their focus to nonedible resources, aiming to mitigate these controversies and lower the final product's cost [12–15]. Nonedible biomass, often considered waste and cultivable on infertile or marginal lands, emerged as an economically viable alternative to edible resources [16,17]. Addressing the commercialization challenges of biodiesel, markedly cost, nonedible feedstock became pivotal in reducing production expenses [18,19]. Various nonedible sources like rubber seed oil, jatropha oil, and date seed oil have been utilized for biodiesel production. *Prunus domestica* (Plum), a widely available fruit year-round, comprises both an edible component and a hard-shelled seed [20,21]. This study focuses on utilizing Plum kernel shell (PKS), a nonedible part, as a potential source of oil. The transesterification process converts this oil into methyl esters (biodiesel) using a suitable catalyst [14,18,22,23].

The catalysts employed in the production of methyl esters (biodiesel) are categorized as either homogeneous or heterogeneous. Presently, the focus on commercializing biodiesel production emphasizes the necessity of using heterogeneous catalysts [12,24]. Heterogeneous catalysts are prized for their recyclability and reusability, key aspects enhancing process feasibility and scalability for commercial applications [25,26]. The selection of a heterogeneous catalyst for biodiesel production depends on the composition of the source oil intended for conversion. Oils with minimal free fatty acid (FFA) content, notably edible oils, are typically compatible with basic catalysts [17,27,28]. Conversely, oils with higher FFA content (Acid value > 3) pose challenges if directly subjected to a basic catalyst, potentially resulting in saponification [29–31]. Consequently, oils with elevated acid values often necessitate a two-step process: first, reducing the acid value by treating the oil with methanol in the presence of an acidic catalyst, followed by transesterification employing a basic catalyst [3,14,32]. However, certain bi-functional heterogeneous catalysts have been developed to circumvent this two-step procedure, enabling simultaneous esterification and transesterification. In this study, a synthesized bi-functional catalyst will be employed for biodiesel production, leveraging these advancements [33–36].

To enhance the market competitiveness of biodiesel, catalysts sourced from waste biomass have emerged as a viable option. The utilization of metallic oxides as catalyst precursors for biodiesel production has been widely reported [33,37,38]. Leveraging waste biomass for catalyst synthesis offers a promising avenue to economize biodiesel production [39–43].

Herein, PKS serves as the feedstock for oil extraction, with the remaining biomass after oil extraction earmarked for the synthesis of the catalyst precursor. The carbon precursor derived from this waste biomass is modified using active metallic oxides (CuO & Mo) to boost its efficiency towards biodiesel production. Subsequently, the synthesized catalyst is employed in the transformation of PKS oil into biodiesel—a pioneering approach, to the best of the authors' knowledge, unreported in existing literature, and it is a novel of its kind. The optimization of the biodiesel production process entails considering various process parameters, followed by an intricate characterization of the resultant biodiesel to assess its fuel properties.

## Materials and methodology

### Materials

Plum kernels were sourced from the local market, then meticulously

deshelled, ground, and thoroughly washed with water. The cleansed Plum Kernel Shell (PKS) powder underwent oven-drying at 110 °C for an entire day. This dried PKS powder was subsequently subjected to a Soxhlet extraction assembly [44], resulting in an oil-hexane mixture that was later separated using a rotary evaporator to isolate the oil. Hexane, methanol, potassium hydroxide, and sulfuric acid were procured from Merck, while copper and molybdenum salts were obtained from Sigma-Aldrich. All chemicals utilized in this study were of reagent grade.

### Catalyst synthesis and characterization

The residual biomass left after PKS oil extraction underwent a thorough wash with warm water to eliminate water-soluble impurities and was subsequently oven-dried at 110 °C overnight. The resulting dried powder underwent carbonization in an inert atmosphere furnace at 400 °C for 4 h, employing a heating rate of 3.5 °C/min. To enhance the physicochemical properties of the carbon material, it was treated with a 0.5 N H<sub>2</sub>SO<sub>4</sub> acidic solution, followed by rigorous washing with water until the filtrate reached a neutral pH level. The resultant material (carbon) was impregnated with varying weights per cent of copper nitrate solution (5, 8, 11, and 14 wt%), leading to the formation of distinct samples labeled as follows: 5 wt% CuO-Carbon (Cu-C1), 8 wt% CuO-Carbon (Cu-C2), 11 wt% CuO-Carbon (Cu-C3), and 14 wt% CuO-Carbon (Cu-C4). All samples were calcined in a muffle furnace at 500 °C for 4 h using a heating rate of 3.5 °C/min. The resulting materials were carefully preserved in airtight containers to prevent moisture contamination. Subsequently, the Cu-C2 sample underwent further modification by impregnating it with Mo active material, followed by a similar calcination process in a muffle furnace at 500 °C for 4 h using a linear heating rate of 3.5 °C/min. The resultant catalyst (Mo-Cu-C2) was then securely stored in an airtight container to prevent moisture contamination before its application in a chemical reaction.

Synthesized novel catalysts were analyzed through the following characterization techniques XRD by using a machine with the following details PANalytical, Xpert PRO instrument, USA, equipped with rotating anode and Cu K $\alpha$  radiation, SEM and EDXS analysis were conducted by using a machine with following details such as Jeol, JSM 7800F, Japan equipped with EDXS detector (Oxford instrument, UK). Physicochemical analysis was conducted by using the BET analysis technique using a machine with the following details ASAP 2020, Micromeritics Instruments Inc., Norcross, GA, USA.

### Catalyst application

The synthesized catalysts were used for the transesterification of PKS oil in a three-neck flat-bottom flask. Initially reaction vessel with oil with a magnetic stirrer was placed on a hot plate, which has provision for temperature control and stirring. Oil was heated up to the desired temperature with continuous stirring followed by the addition of a mixture of methanol and catalyst. All the reactions for biodiesel production were conducted based on reaction parameters mentioned in Table 1. After the reaction stopped, it was allowed to cool down. Once the mixture comes to room temperature it is centrifuged to separate the catalyst to liquid. After the separation of the catalyst, the mixture is

**Table 1**

Independent process parameters with the defined ranges for the experimental plan to produce biodiesel from PKS in the presence of a synthesised novel catalyst.

Parameter	Experimental ranges	
	Low	High
Temperature (°C)	55	75
Time (min)	60	180
Methanol to oil molar ratio	9	21
Catalyst loading (wt.%)	2	8

poured into a separating funnel to separate the biodiesel product from the glycerol byproduct. The mixture is allowed to stay in a separating funnel for 24 h, and after that, two distinguished liquids can be observed: which upper layer of biodiesel and the bottom of glycerol. After the separation of biodiesel, it is washed with warm water to remove any water-soluble impurities as a standard procedure reported earlier as well [45]. In the end, sodium sulphate beads were added to biodiesel to remove moisture from it completely. The product biodiesel is further analyzed by using the GC-MS technique by machine with the following details such as Perkin Elmer Clarus 600 fixed with DB-Wax column. Fuel properties, including acid value, density, viscosity, Cetane number, flash point and low-temperature properties of product biodiesel, were determined by standard methods defined by ASTM and EN.

## Results and discussions

### 3.1 Catalysts analysis

#### XRD analysis

The XRD analysis was conducted to determine the phase and structure of the novel synthesized catalyst. Fig. 1 illustrates the results obtained. The pristine carbon revealed a porous nature, as indicated by a

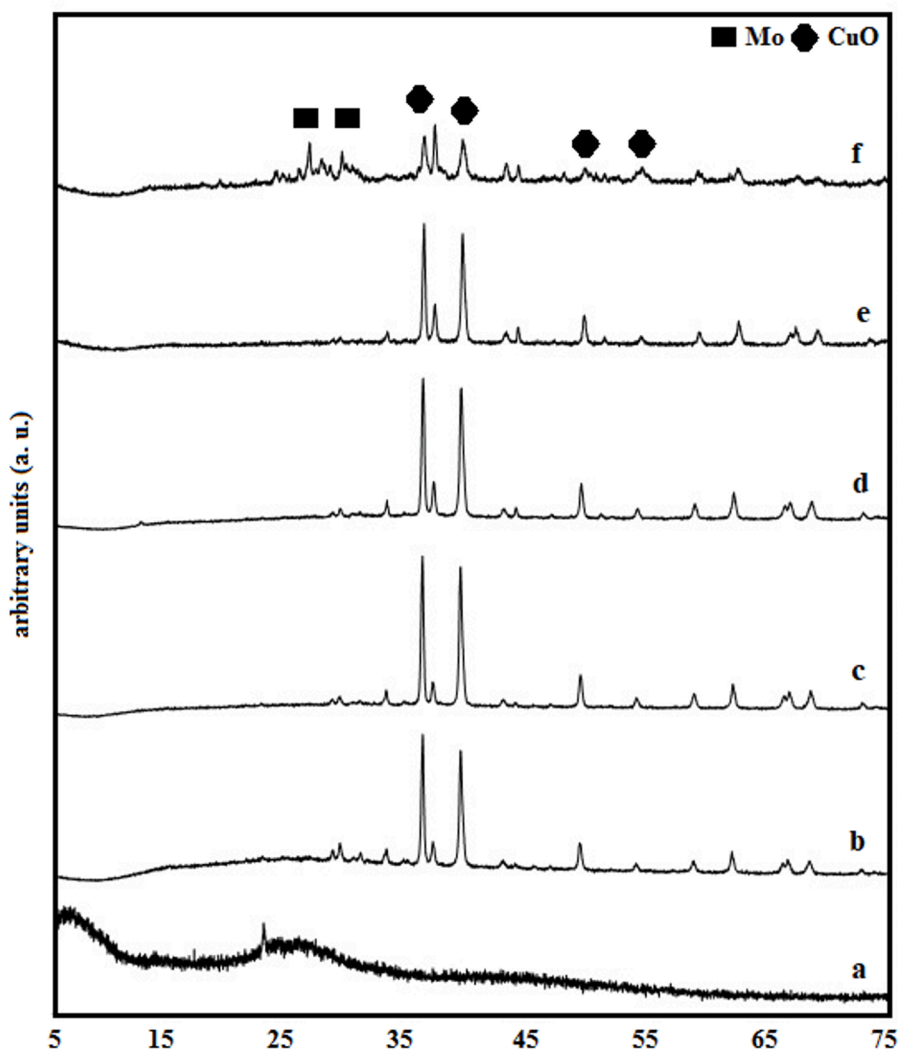
broad peak ranging from  $15^\circ$  to  $30^\circ$ . This porous characteristic is typical of carbon derived from biomass, aligning with previous reports [46].

Upon impregnation with metallic oxide (CuO), changes in the phase of the resultant material were observed, displaying additional peaks in each catalyst sample containing carbon as a precursor and copper oxide as an active material. Diffraction peaks at  $35.07^\circ$ ,  $38.17^\circ$ ,  $46.92^\circ$ , and  $52.81^\circ$  signify the presence of copper oxide on the pristine carbon, in accordance with the JCPDS library (89-5895) [47]. Notably, all catalyst samples modified with copper oxide exhibited sharp peaks, indicating a transformation into a crystalline phase.

The subsequent modification of one catalyst sample through the inclusion of Mo as an active material revealed additional diffraction peaks at  $27.14^\circ$  and  $30.95^\circ$ , affirming the presence of Mo. The observed sharpness of the Mo diffraction peaks suggests that its addition did not alter the catalyst phase. Thus, it can be inferred that the synthesized catalyst possesses a crystalline phase with a porous precursor, indicating its potential suitability for reactions involving long-chain oxygenated hydrocarbons.

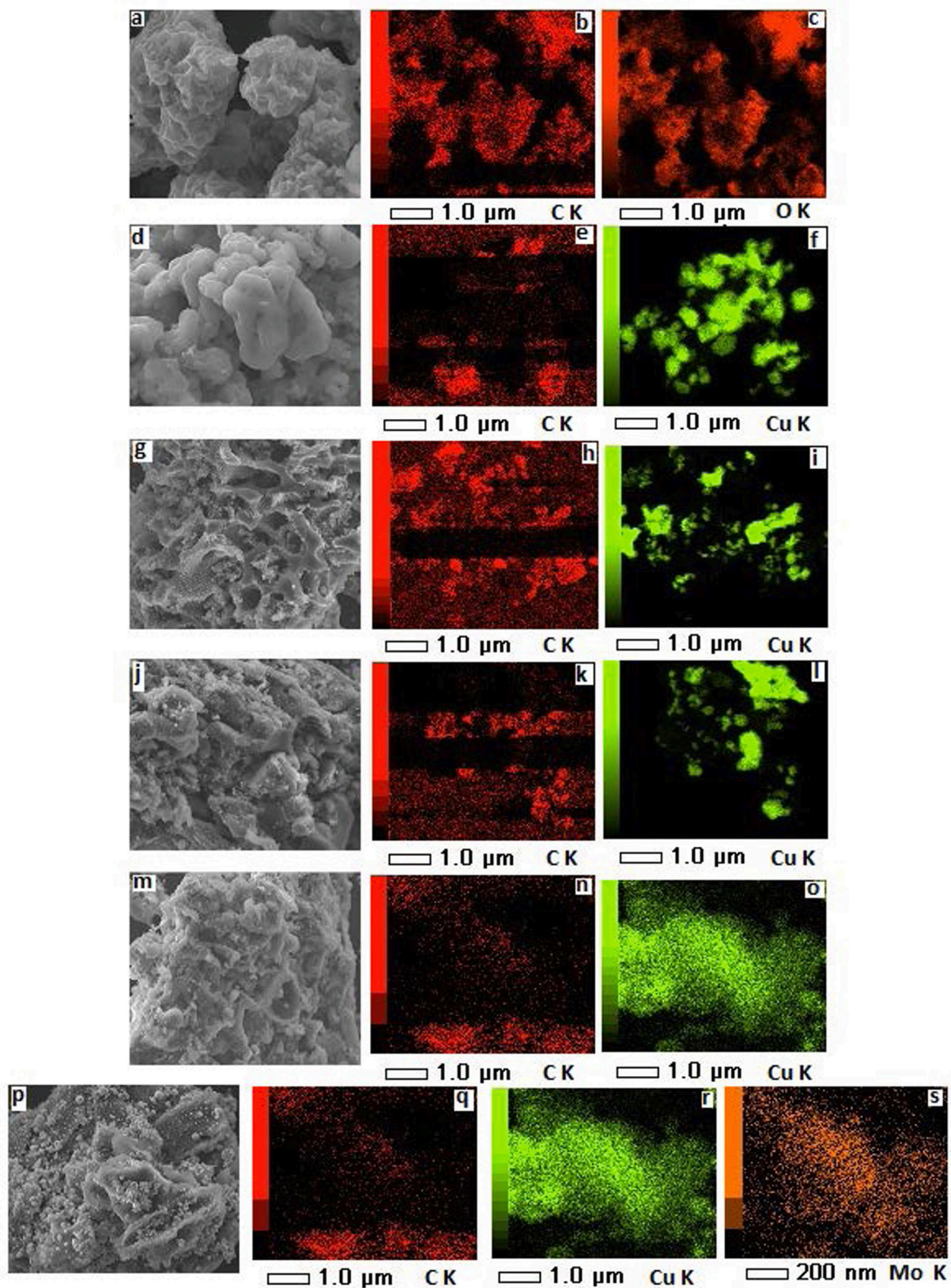
#### SEM and EDXS analysis

SEM analysis was employed to assess the surface morphology of the synthesized heterogeneous catalyst, along with elemental mapping for all samples, where Fig. 2 depicts the results obtained. Pristine carbon



**Fig. 1.** XRD analysis patterns for synthesized catalysts; (a) pristine carbon, (b) 5 wt% CuO-Carbon (Cu-C1), (c) 8 wt% CuO-Carbon (Cu-C2), (d) 11 wt% CuO-Carbon (Cu-C3), (e) 14 wt% CuO-Carbon (Cu-C4) and (f) 8 wt% CuO-Carbon (Cu-C2) with Mo.





**Fig. 2.** SEM and EDS images for synthesized catalysts; (a-c) pristine carbon, (d-e) 5 wt% CuO-Carbon (Cu-C1), (g-i) 8 wt% CuO-Carbon (Cu-C2), (j-l) 11 wt% CuO-Carbon (Cu-C3), (m-o) 14 wt% CuO-Carbon (Cu-C4) and (p-s) 8 wt% CuO-Carbon (Cu-C2) with Mo.

exhibited irregularly shaped porous particles, indicative of its porous nature. This porous structure suggests suitability for reactions involving long-chain oxygenated hydrocarbons.

Images representing the modified pristine carbon with metallic oxide illustrated particles dispersed on the porous precursor, with even

distribution apparent in the elemental mapping images. However, an increase in metallic oxide content resulted in lump or cluster formation. While metallic oxides may enhance catalyst activity, excessive loading could potentially block active sites and disrupt pore channels, leading to variations in product yield.

The SEM and elemental mapping observations corresponded well with the XRD findings. The presence of broad diffraction peaks in pristine carbon aligns with its porous structure, while the modified catalyst samples exhibited sharp diffraction peaks indicative of crystalline material. These observations collectively suggest that pristine carbon retains its porous nature even after modification with metallic oxide, rendering it suitable for catalyzing reactions involving complex or long-chain oxygenated hydrocarbons.

#### BET analysis

Physiochemical properties of the synthesized catalysts, including surface area, pore size, and diameter, were determined and are detailed in Table 2. For reactions involving organic hydrocarbons with large chains, porous catalysts are advantageous, facilitating complete interaction between reactants and active sites. Pristine carbon exhibits a substantial surface area, aligned with SEM images, and possesses suitable pore diameter and volume, indicating its capability to handle reactants like oil.

When pristine carbon is modified with metallic oxide (CuO), the surface area increases, signifying effective dispersion and bonding of the metallic oxide to the precursor and thereby exposing more surface area for reactants. However, beyond an 8 wt% impregnation of metallic oxide, the surface area decreases, along with pore volume and diameter. This reduction suggests that additional material is blocking the precursor surface and clogging the pore channels.

Modification of Cu-C2 with another active material (Mo) did not significantly alter the surface properties of the parent material. Given that the diameter of a triglyceride molecule (the main component in organic oil) is approximately 5.8 nm, the determined properties indicate that the synthesized catalyst is well-suited for the desired reaction.

#### XPS analysis

Fig. 3 presents the comprehensive XPS survey spectrum of various catalyst surfaces. The base material (CAR), comprising carbon, exhibits the presence of carbon, oxygen, phosphorus, as well as traces of nitrogen and chloride. Successful Cu impregnation on the base substrate is evident from the observed core level Cu 2p peaks on the surfaces of Cu-C1, Cu-C2, Cu-C3, and Cu-C4 catalysts.

Following Cu impregnation, a reduction in the intensity of the core level C 1s peak suggests that the surface of the synthesized catalysts was predominantly covered with Cu. This inference is further supported by the noticeable increment in the core level O 1s peak for Cu-impregnated catalysts. Notable amounts of nitrogen were also detected on the surfaces of CAR and Mo-Cu-C2 samples. Fig. 2b demonstrates the core level O 1s peaks of the base, Cu-modified, and Mo-modified catalysts. A series of peak shifts to lower binding energy were observed from the base carbon material to the Mo-Cu-C2 catalyst. Previously, we have reported a similar phenomenon where the chemical interaction between CaO-CeO<sub>2</sub> was witnessed [48]. These shifts affirm the chemical interaction between the base carbon material and the impregnated materials, Cu and Mo. Such shifts indicate an increase in basic sites on the catalysts' surface, potentially enhancing biodiesel production yield, a topic to be discussed in a subsequent section [49].

Regarding the core level Cu 2p peaks (see Fig. 1c), these exhibited lower binding energy shifts at low Cu<sup>2+</sup> loadings (Cu-C1, Cu-C2, and Cu-

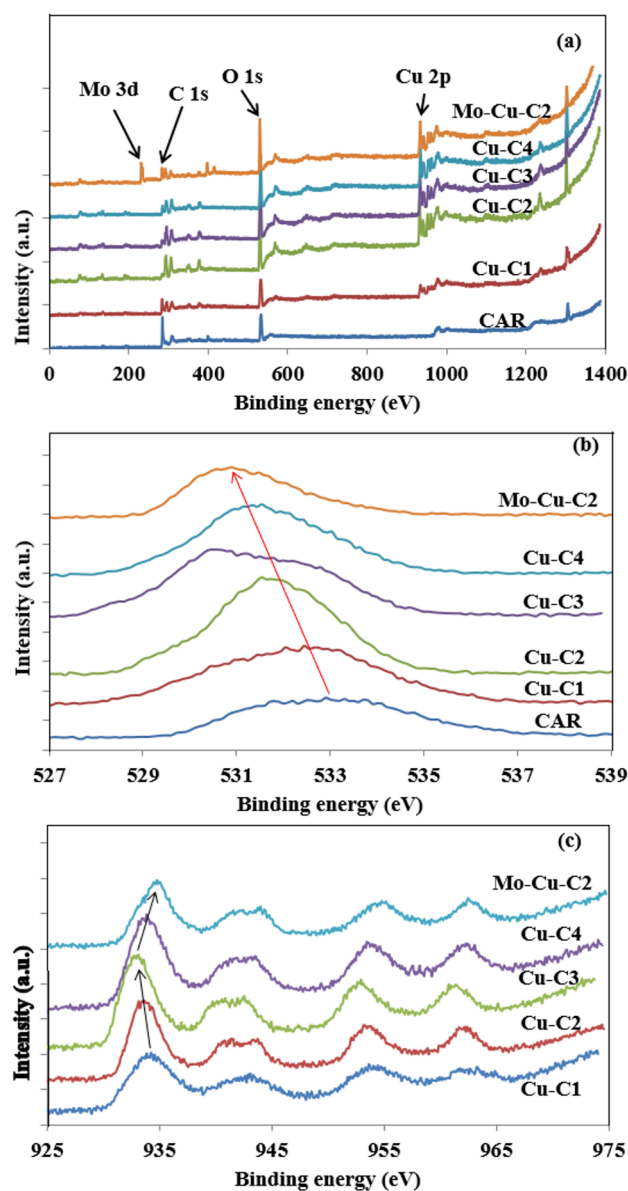


Fig. 3. XPS analysis of (a) Survey spectrum (b) O 1s peaks (c) Cu 2p peaks of catalyst base (CAR) and Cu impregnated catalysts (Cu-C1, Cu-C2, Cu-C3 and Cu-C4) and Mo modified catalyst (Mo-Cu-C2).

C3), potentially due to electron information originating from Cu-O and C-O (base material) [50]. However, at higher Cu<sup>2+</sup> loadings and Mo-impregnated samples (Cu-C4 and Mo-Cu-C2), the binding energy returned to its initial value. This shift could be associated with a complete surface chemistry change attributed to the high concentration of Cu impregnation, along with Mo impregnation.

#### Catalysts Evaluation

The fatty acid composition of PKS oil, as detailed in Table 3, suggests its potential suitability for biodiesel (methyl ester) production. The synthesized catalysts were assessed for their efficacy in generating methyl esters from PKS oil, depicted in Fig. 4.

Initially, when the reaction was conducted using pristine precursor carbon, a lower yield of biodiesel was observed [51]. This lower yield can be attributed to the scarcity of active sites within the carbon structure, resulting in decreased methyl ester production. However, upon employing the modified form of carbon in the reaction, the yield

Table 2

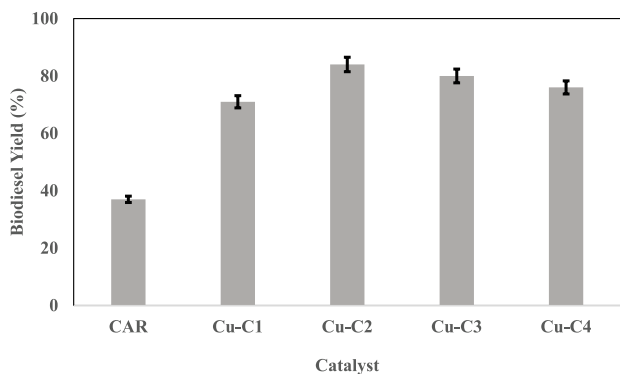
BET analysis for physiochemical properties, which includes surface area, pore volume and diameter of a synthesised catalyst from PKS post-oil extraction.

Catalysts	Surface area (m <sup>2</sup> /g)	Pore volume (cm <sup>3</sup> /g)	Pore diameter (nm)
CAR	212.57	0.328	36.24
Cu-C1	228.74	0.257	34.29
Cu-C2	236.29	0.238	29.47
Cu-C3	215.37	0.217	18.97
Cu-C4	198.37	0.206	15.27
Mo-Cu-C2	230.68	0.231	27.32

**Table 3**

Detailed composition analysis of PKS oil, which is further to be transformed into biodiesel.

Composition	Quantity (wt.%)
Lauric (C 12:0)	1.08
Myristic (C 14:0)	1.45
Palmitic (C 16:0)	9.38
Stearic (C 18:0)	2.95
Oleic (C 18:1)	65.79
Linoleic (C 18:2)	19.35



**Fig. 4.** Evaluation of synthesised catalysts (pristine carbon (CAR), 5 wt% CuO-Carbon (Cu-C1), 8 wt% CuO-Carbon (Cu-C2), 11 wt% CuO-Carbon (Cu-C3) and 14 wt% CuO-Carbon (Cu-C4) for biodiesel production from PKS oil.

significantly improved. This improvement indicates that the modification of pristine carbon with copper oxide provides suitable active sites for the reaction.

Moreover, an increase in metallic oxide quantity further increased

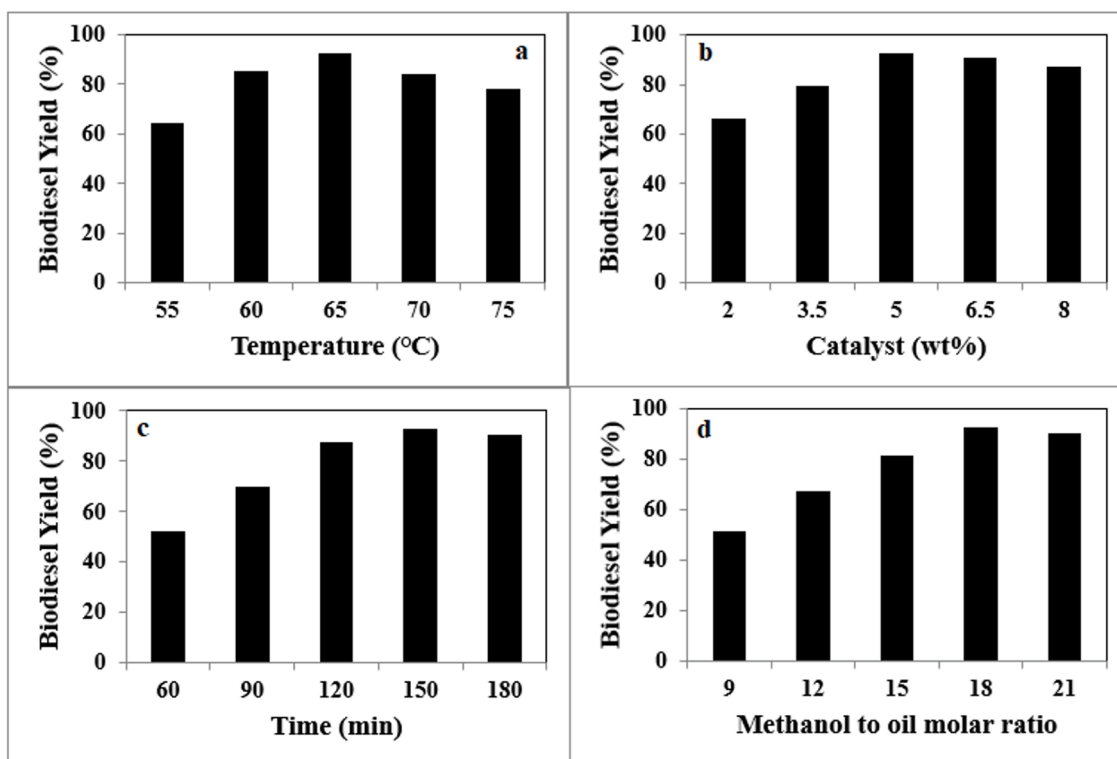
the yield. The boosted methyl ester yield with increased metallic oxide content can be linked to the addition of extra active sites to the pristine carbon, thereby providing more pore channels for reactants to interact with active sites. However, this ascending trend in biodiesel yield showed a declining pattern beyond a specific limit of metallic oxide addition. This reduction in yield could be attributed to the limited availability of active sites for reactants, potentially linked to changes in physiochemical properties. Surface area, pore diameter, and volume decreased upon the addition of copper oxide, potentially leading to channel blockages.

Consequently, it can be inferred that while the inclusion of metallic oxide is advantageous for providing active sites, an excessive amount of copper oxide may decrease surface area and block pore channels. Thus, the most suitable catalyst identified was labeled Cu-C2 (8 wt% CuO-Carbon), which, when further enhanced by molybdenum, exhibited more improved performance. This resultant catalyst was used for the process optimization based on the parametric study.

#### Parametric studies and optimization

The transformation of PKS oil into biodiesel utilizing the synthesized catalyst (Mo-Cu-C2) underwent an optimization process involving independent variables such as temperature, methanol-to-oil molar ratio, time, and catalyst loading. The conducted experiments encompassed the ranges defined in Table 1. These parametric studies offer valuable insights crucial for economic analysis and product commercialization [45,52].

Fig. 5a illustrates the impact of process temperature on biodiesel yield, which emerges as a pivotal parameter directly tied to energy consumption. Initially, at lower temperatures, the observed biodiesel yield remained low, indicating insufficient energy input to excite reactant molecules for transformation into products. However, as the temperature increased, the yield increased due to enhanced entropy. The higher energy input at increased temperatures rendered the immiscible



**Fig. 5.** Parametric studies for biodiesel production (a) effect of process temperature on biodiesel yield, (b) effect of catalyst loading on biodiesel yield, (c) effect of process time on biodiesel yield and (d) effect of methanol to oil molar ratio on biodiesel yield.



reactants more active, leading to higher product yields. This increasing trend peaked at 65 °C, beyond which the yield started to decline. This decrease in yield at higher temperatures was attributed to reduced methanol availability in the reaction media, despite continuous refluxing. Hence, 65 °C was identified as the optimal temperature for biodiesel production within the specified range.

Catalyst loading, detailed in Fig. 5b, showcased a parabolic trend concerning biodiesel yield. Minimal loading resulted in low yields due to inadequate active sites for the reaction. Increasing the catalyst quantity initially augmented yields, yet beyond 5 wt%, yields declined due to potential mass transfer limitations and separation challenges.

Fig. 5c illustrates the biodiesel yield concerning process time. Longer process times typically enhance the interaction between reactants, thereby improving yield. However, beyond 150 min, a decrease in yield was noted, likely due to downstream separation issues, making 150 min the optimal process duration.

Fig. 5d displays the biodiesel yield concerning the molar ratio of reactants. Lower ratios resulted in lower yields due to methanol scarcity for the reaction. However, increasing the ratio boosted yields up to a ratio of 18. Beyond this, yields decreased, likely due to excessive methanol in the reaction media, causing downstream issues [53].

Based on the parametric study, the optimized set of independent variables includes a process temperature of 65 °C, a process time of 150 min, a catalyst loading of 5 wt%, and a molar ratio of reactants at 18. These settings ensure the maximum yield of biodiesel from PKS oil.

#### Quality analysis of biodiesel

The quality assessment of the produced biodiesel involved examining various properties vis-à-vis standards defined by ASTM and EN, as detailed in Table 4.

The acid value reflects the acidic content in the fuel. A low acid value in biodiesel ensures minimal unconverted fatty acids, allowing for safe storage and transportation [54–56]. Thus, in the current study, the produced biodiesel has an acid value less than the maximum value defined by standards ASTM and EN, ensuring its transportability and storage without container damage [57].

The density of produced biodiesel is 889 kg.m<sup>-3</sup>, which satisfies the standard range defined by EN. The density of fuel can affect fuel atomization as well, and dense fuel can cause an imbalance in the quantity to be used in an engine, so it's essential to make sure the density of fuel is within the defined range to avoid any issues during combustion. The viscosity of produced biodiesel is 4.57 mm<sup>2</sup>.s<sup>-1</sup>, and this is within the range defined by ASTM and EN standards. Ideal viscosity contributes to proper fuel atomization and performance in colder regions. Meanwhile, biodiesel, which has considerable viscosity, contains unsaturated in higher amounts, and fuel has better cold flow properties moreover, it helps in the proper atomization of fuel when injected into the engine.

**Table 4**

Detailed analysis of biodiesel to determine the fuel properties and comparison with the international standards.

Characteristics	Biodiesel	EN 14,214	ASTM 6751
Acid Value (mg KOH/g)	0.31	0.50 max	0.80 max
Density (kg.m <sup>-3</sup> ) at 25 °C	889	860–900	–
Viscosity (mm <sup>2</sup> .s <sup>-1</sup> ) at 40 °C	4.57	3.5–5.0	1.9–6.0
Flashpoint (°C)	176	120 min	93 min
Cetane number	58.34	51 min	47 min
Cloud point (°C)	0.91	-a	–
Pour point (°C)	–3.03	-a	-b
Cold Filter Plugging Point (°C)	–1.97	-a	-b
Free Glycerin (%)	0.09	0.020 max	0.020 max
Total Glycerin (%)	0.207	0.250 max	0.240 max

<sup>a</sup>Not specified. EN 14214 uses time- and location-dependent values for the cold filter plugging point (CFPP) instead.

<sup>b</sup>Not specified.

The Flashpoint of biodiesel is determined, and it is found to be 176 °C which is higher than the minimum value defined by EN and ASTM standards. Flashpoint refers to the safety aspects of biodiesel as it is the maximum temperature which a fuel can withstand without ignition. So, the higher the flash point, the safer it is to transport and store the biodiesel. The cetane number of product biodiesel is 58.34, which is higher than the minimum value defined by the EN and ASTM standards. The cetane number is considered to be an anti-knocking property of biodiesel/diesel, which is higher enough to combust fuel properly in the engine, so product biodiesel based on its Cetane value has a better anti-knocking property.

The analysis of the biodiesel's properties reveals its viability as a fuel, meeting ASTM and EN standards. However, its performance in colder climates remains a concern due to its susceptibility to solidification at low temperatures. Biodiesel's composition of methyl esters, particularly the higher proportion of saturated components, can result in poor low-temperature properties. The cloud point, indicating the temperature at which crystals appear on the fuel's surface, is 0.91 °C. Although this temperature is relatively low, it could still lead to fuel solidification in colder environments. The cold filter plugging point, at –1.97 °C, signifies the minimum temperature where fuel flows through a standard filter. Below this point, clogging may occur, affecting engine performance.

Moreover, the pour point, at –3.03 °C, indicates the temperature below which the fluid might lose its flow properties and solidify. Despite being relatively low in very cold conditions, it could still lead to fuel solidification and potential engine issues. However, the free and total glycerin content is within acceptable limits, indicating effective filtration to remove impurities, enhancing the biodiesel's overall quality.

While the produced biodiesel meets international fuel standards, its performance in extremely cold temperatures might still present challenges due to its composition and identified low-temperature properties. Further research or additive modifications might be necessary to enhance its cold weather performance for broader geographical usage.

#### Reusability check of synthesized catalyst

The reusability study of the synthesized catalyst for biodiesel production reveals crucial insights for its potential commercial application. The results exhibit a substantial difference in performance based on whether the catalyst undergoes treatment before reuse, as shown in Fig. 6. When the catalyst is washed with ethanol and calcined before being reused, the biodiesel yield remains relatively consistent across multiple runs, demonstrating a notable level of reusability. This treatment process appears to effectively remove alcohol-soluble impurities and reactivate the catalyst's active sites by eliminating unnecessary components. Consequently, the biodiesel yield maintains consistency, indicating the sustained efficacy of the catalyst in facilitating the conversion process.

However, when the catalyst is reused without prior treatment, there is a considerable decline in biodiesel yield in each successive experimental run. This decrement is attributed to the blocked active sites, hindering efficient interactions between the catalyst and reactants. The accumulation of residues from reactants and products negatively affects the catalyst's performance, leading to reduced yields over subsequent uses.

The findings affirm the importance of catalyst activation before reuse, emphasizing the need to wash the catalyst with ethanol and perform calcination to restore its active sites and ensure optimal performance. The study indicates that the synthesized catalyst exhibits reusability for up to the fifth run without a significant decrease in biodiesel yield when appropriately treated before each reuse. This information underscores the catalyst's potential for practical application in biodiesel production at a commercial scale.

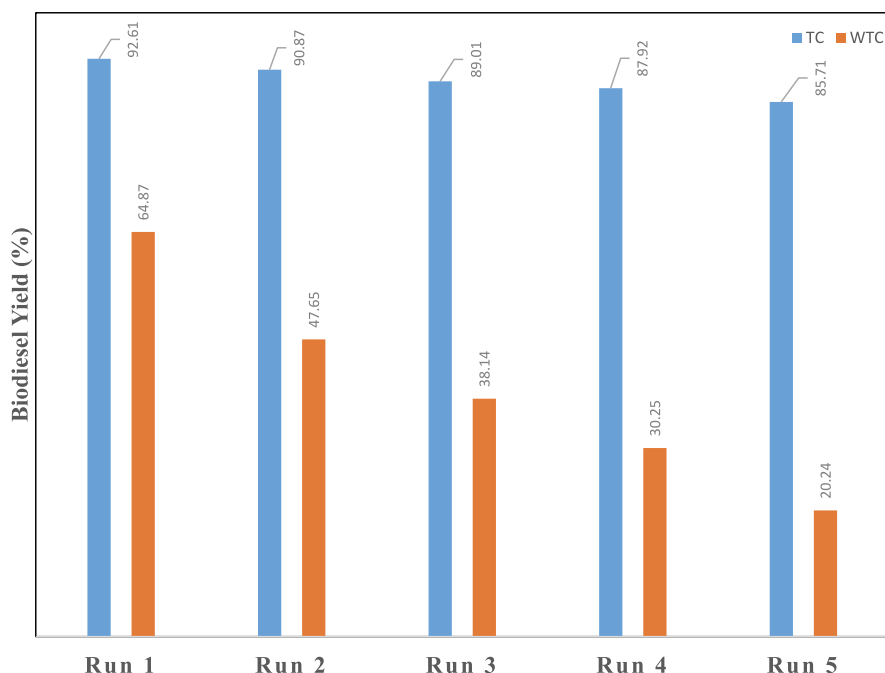


Fig. 6. Reusability studies of synthesized catalyst for biodiesel production from PKS oil where TC represents treated catalyst, and WTC represents catalyst without treatment.

## Conclusions

With the concerns of depleting fossil fuel reserves and escalating greenhouse gas emissions, this research presents a stride towards sustainable energy solutions. The focus on producing biodiesel, a green and renewable alternative derived from PKS oil, aligns seamlessly with sustainability imperatives and SDGs. Notably, this study employs waste PKS both as an oil source and for catalyst synthesis, representing resourcefulness in enhancing eco-friendly and cost-effective production methods. Optimization efforts within the biodiesel production process have yielded significant results, achieving a yield of 92.61 % under specific parameters, which include a process temperature of 65 °C, a processing time of 150 min, a catalyst loading of 5 wt%, and a molar ratio of reactants at 18. This notable efficiency not only underscores the viability but also the scalability of this biodiesel production method. Importantly, the resulting biodiesel conforms to the rigorous standards outlined by ASTM and EN, affirming its credibility as a feasible fuel option for conventional diesel engines.

Moreover, this research unveils the untapped potential of PKS as a versatile biomass resource, serving not only as an oil source for biodiesel but also as a catalyst precursor. Such a holistic approach not only contributes substantially to sustainable fuel production but also addresses the urgent need for alternative energy sources.

In summary, this study not only emphasizes the viability of harnessing PKS as an invaluable biomass resource for biodiesel production but also underscores the significance of repurposing waste biomass for catalyst synthesis. Such multifaceted endeavors pave the way for a more sustainable and environmentally conscious approach in the production of biodiesel.

## CRedit authorship contribution statement

**Farrukh Jamil:** Investigation, Methodology, Writing – original draft. **Ala'a H. Al-Muhtaseb:** Conceptualization, Supervision, Writing – review & editing. **Ahmed I. Osman:** Methodology, Investigation, Formal analysis, Writing – review & editing. **Lamya Al-Haj:** Writing – original draft. **Myo Tay Zar Myint:** Methodology, Investigation, Formal analysis. **Abrar Inayat:** Visualization, Validation. **Murid Hussain:**

Validation, Visualization. **Abdallah Shanableh:** Resources, Validation, Visualization.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

No data was used for the research described in the article.

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