Effects of titanium oxide and graphene as nano-fillers on the thermal conductivity of biobased phase change materials as latent thermal heat storage.

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Title:

Effects of titanium oxide and graphene as nano-fillers on the thermal conductivity of biobased phase change materials as latent thermal heat storage

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Abstract:

Biobased phase change materials (BPCMs) have attracted much attention as they offer proper melting temperature, high heat capacity, non-corrosive, low cost, and are abundantly available. However, the BPCMs have a low thermal conductivity which limits their application. To minimise the loss of the overall heat storage enthalpy capacity, nanofillers are added to improve the thermal properties of the BPCMs. This study investigates the thermal conductivity of the BPCMs with nanofillers. The thermal conductivity was calculated using the Maxwell Garnet equation and then compared with the experimental results. The BPCMs such as palm wax, beeswax, and coconut oil were prepared with different weight concentrations of graphene and titanium oxide (0.5 wt%, 1.0 wt%, 1.5 wt%,2.0 wt%). The melting temperature for the BPCMs was between 32 and 60 °C. The thermal conductivity of BPCMs for both fillers increase between 4 and 46 % compared to pure BPCMs. Overall, the titanium oxide shows better improvement in BPCMs' thermal conductivity compared to graphene. Beeswax gives the highest thermal conductivity of 0.338 W/m.K, while coconut oil is the lowest with 0.232 W/m.K at 2 wt% titanium oxide concentration. The results from the experimental work are in agreement with the calculated value from the Maxwell Garnet model. Overall, the addition of nanofillers in BPCMs offers promising thermal performance making them more attractive to be used as latent thermal heat storage.

Keywords: Biobased; phase change materials; Nanofillers; Thermal conductivity; Graphene

Introduction

The ever-limited fossil fuels, the increase in energy usage, and the rapid growth of the greenhouse gas emissions that exacerbate the impacts of climate change have led to the effective utilisation of energy. Phase change materials (PCMs) as latent thermal heat storage have gathered widespread interest in efficient energy solutions for the buildings sector [1]. PCMs are a substance that can absorb, store and release energy equivalent to their latent heat when the temperature of the materials rises above or fall below the phase change temperature [2]. PCMs could contained more heat per unit volume compared to sensible heat storage as they able to absorb and release heat at a constant temperature [3]. They functioned as thermal energy storage (TES) systems when incorporated into the building. Latent thermal heat storage (LTHS) which employed the phase transition of a material offer extremely high energy storage density compared to other thermal energy storage system as it is able to contain heat with no change in temperature during the phase transition [4]. In buildings, PCMs are implemented to mitigate heat gain, avoid heat buildup, enhance heat gain, promote temperature moderation, and shift cooling loads to off-peak electricity periods. Studies have been conducted to investigate PCMs usage in-ceiling ventilation [5], solar roofing [6], night cooling strategy [7], free cooling [8], and in building envelopes [9].

PCMs are classified according to their chemical nature, which is organic and inorganic. Organic PCMs such as paraffin are the most versatile PCMs due to their superior properties such as high latent heat of fusion at low vapour pressure, chemical and thermal stability, which makes them a candidate for many applications. Paraffin and non-paraffin such as fatty acids, alcohols, esters, glycols, and biobased makes up the organic PCMs. Biobased phase change materials (BPCMs) are produced from renewable and environmentally friendly resources such as coconut oil, palm oil, beef tallow, soybean oil and margarine just to name a few [10]. BPCMs is favourable than paraffin as it thermally and chemically stable, constant volume change during phase transition, abundantly available and cheaper. Boussabe et. al. [11] studied the potential of vegetable fat as BPCMs. Soy oil and corn oil has been explored as BPCMs candidate by [12]. Another study carried out by [13] used natural waxes such as palm wax, soy wax and paraffin wax as PCMs. Findings showed that soy wax has better stability and favorable to be used as PCMs. However, their low thermal conductivity limits their application due to poor responsiveness of thermal changes occurring in releasing and absorbing processes, leading to a decrease in storage capacity [14]. The thermal conductivity is one of the important characteristics which defines the thermal power capacity [15].

In response to this challenge, the thermal conductivity of BPCMs could be increased by dispersion of high thermal conducive filler. Higher thermal conductivity ensures faster thermal transfer rate during charging and discharging process [16]. The dispersion of high thermal conductive nano-filler such as nanoparticles, nano-fibers, or nano-sheets can greatly enhance the PCM’s thermal and physical properties. The addition of high thermal conducive fillers improves the heat transfer rate by forming better heat transfer network leading to increase in heat transfer rate [17]. The nanoparticles modify the thermal conductivity, latent heat capacity, sub-cooling, phase change temperature, duration, density, and viscosity [5], of PCMs making them more attractive for various applications. Khodadadi and Hosseinizadeh [18] pioneered the study in enhancing the thermal properties of phase change materials by dispersing nanoparticles. The study concluded that the dispersion of nanoparticles with high surface area to volume ratio increased the thermal conductivity of the phase change materials. In previous researches, multiwalled carbon nanotubes [19], graphene [20], [21], graphite [11], copper oxide [22], [23], and titanium oxide [24] have been used as nanofiller for BPCMs. Nanoparticles exhibit distinctive properties such as macroscopic and surface effects that enable them to be steadily dispersed in a medium for better stability. Teng and Yu [25] compared the effects of metal oxide as fillers and found that the addition of 3 % of titanium oxide into paraffin has significant effects on the heat transfer performance and increases the responsiveness of thermal changes during the melting and solidifying process. Another study by Fan et. al [26] studied the enhancement of properties of PCMs using carbon fillers such as graphene, multi-walled CNTs, and carbon nano-fibers in paraffin and found 170 % enhancement of the thermal conductivity at 5 %wt concentration.

Up to date numerous studies have been conducted to evaluate the properties and potential of pure BPCMs [10], [13], [27]–[29], but only a few studies focus on improving the thermal conductivity of

BPCMs using nanofillers. Among BPCMs and nanofillers used are beeswax/graphene [20], beeswax/copper oxide [23], beeswax/carbon nanotubes [19], palm wax/diatomite [13], biobased PCM/exfoliated graphite nanoplatelets [30] coconut oil/copper oxide [22] and coconut oil/graphene [21]. Studies using beeswax by [19], [20], [23] found that the thermal conductivity improved by 10.2 % with 0.3 wt% of graphene, 132 % with 20 wt% of carbon nanotubes and 112 % with addition of 0.489 g of copper oxide. Soroush et al [22] reported that the thermal conductivity of coconut oil/copper oxide increased by 7.5 % with 1 wt% of copper oxide while another study [21] which use coconut oil/graphene saw the highest improvement of thermal conductivity by 69 % using 0.3 wt% graphene.

In the current study, work has been done to develop a cost-effective BPCMs with high thermal conductivity. Palm wax, beeswax, and coconut oil are chosen as the BPCMs. They are renewable, abundantly available, and cost-friendly. Due to their low thermal conductivity, nanofillers such as graphene and titanium oxide are introduced to increase their potential as latent thermal heat storage.

2. Methodology

2.1. Analytical model for thermal conductivity measurement

The theoretical models to investigate the thermal conductivity of the nano-fillers integrated in the phase change materials have been proposed in the literatures. The Maxwell Garnet equation is widely used to find the thermal conductivity in the liquid condition and reported to be fundamental for all other models. The thermal conductivity of the BPCMs with nanofiller was calculated based on the Maxwell-Garnett equation [31]. The effective thermal conductivity, k is given by:

$$k = [(k_p + 2k_f + 2(k_p - k_f)\phi . k_f)/(k_p + 2k_f - (k_p - k_f)\phi)] \quad (1)$$

where kp is thermal conductivity of the nanofiller (Table 1), kf is thermal conductivity of the phase change material (Table 2) and φ is the particle volume concentration. Among factors that influenced the thermal conductivity of nanofillers are the volume fraction, the surface area, the thermal conductivities of the base fluid, the temperature, and the shape of nanoparticles. The model suits well for spherical-shaped particles at small concentration at ambient surrounding. In this study, all the parameters in the model are kept constant except for the volume fraction of the nanoparticles.

Table 1. Properties of nanofillers.

Properties	Titanium Oxide	Graphene
Average size particle	21 nm	22.5–26 nm
Surface area	35–65 m^2/gm	500 m^2/gm
Purity	99.5%	95%
Thermal conductivity (k_p)	11.7 W/mK	3000 W/mK

Table 2. Thermal properties of BPCMs.

Biobased phase change materials	Thermal conductivity, k_f (W/m.K)	Latent heat (J/g)	Melting temperature (^{0}C)
Palm wax	0.249±0.001	168.3	59.8
Beeswax	0.231±0.002	240.2	51.7
Coconut oil	0.223±0.002	112.6	34.7

2.2. Materials

Beeswax, palm wax, and coconut oil in Fig. 1 were chosen as BPCMs in this study Coconut oil used is commercially available coconut cooking oil which is 100 % natural with no added chemicals, solvents, or additives. The beeswax used is KahlWax 8104 White Beeswax, purchased from EvaChem, Kuala Lumpur. It is pure, refined white beeswax obtained from honeycombs of Apis Mellifera (western honeybee), which is carefully physically bleached and refined with melting point around 61–65 °C. Palm wax MP-56 with melting point of 58–60 °C was purchased from Evachem, Kuala Lumpur.

Fig. 1. Pure biobased phase change materials (BPCMs).

Graphene and titanium oxide were purchased from Sigma Aldrich, Germany. Table 1 below shows the nano-fillers properties used in this study.

2.3. Materials preparation

The palm wax, beeswax, and coconut oil were weighed accordingly. The palm wax was melted in the oven at 58–60 °C while beeswax at 61–65 °C for 30 min. Next, coconut oil, melted palm wax, and beeswax were poured into a cylinder PVC mould and cured at ambient condition. Then, the steps were then repeated for samples with nanofillers with the addition of nanoparticles. Different concentrations (0.5 wt%, 1.0 wt%, 1.5 wt%, 2.0 wt%) of graphene and titanium oxide were added to the melted palm wax, beeswax, and coconut oil. The mixture was then stirred using a magnetic stirrer at 600 rpm on the hot plate at 80 °C for 30 min. The mixture of bio-based/nanofiller in the beaker was then placed into the ultrasonicated bath for 60 min to ensure uniform dispersion The mixture was then poured into a cylinder PVC mould and cured at room temperature. The samples prepared is illustrated in Fig. 2 below.

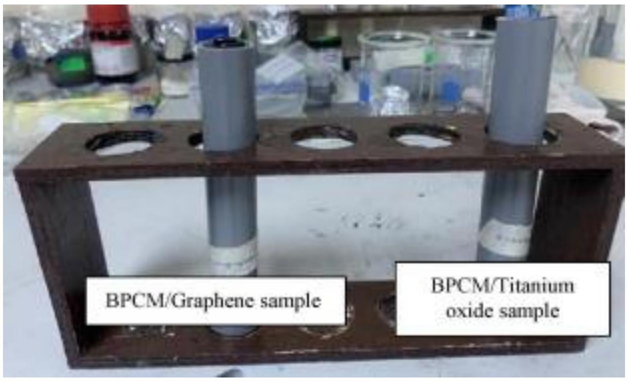

Fig. 2. Samples prepared.

2.4. Thermal conductivity measurement

The thermal conductivity of the sample was determined using the KD2 Pro as shown in Fig. 3. The KD2 Pro Thermal Analyser with KS-1 sensor (±5% accuracy) measured the thermal conductivity using a transient line heat source method. The KS-1 sensor has to be completely immersed in the sample at room temperature during the measurement and repeated three times to ensure accurate reading. The average readings were recorded. The Simultaneous Thermal Analyser (STA) was employed to determine the latent heat and melting temperature of pure samples. The sample mass was approximately 7.5 mg and the measurement was conducted at heating rate of 10 °C/min with constant flow rate of 50 mL/min of nitrogen. The accuracy for the reading is ± 0.05 °C for temperature and ± 0.01 for latent heat.

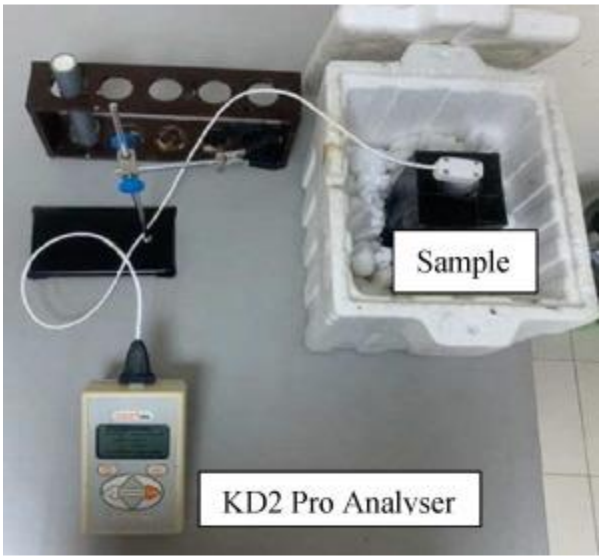

Fig. 3. Thermal conductivity measurement using KD2 Pro Analyser.

3. Results and discussion

3.1. Thermal properties of pure biobased phase change materials (BPCMs)

Thermal conductivity is a vital characteristic in BPCMs defining their responsiveness during storing and releasing energy efficiency [32]. Table 2 shows the thermal properties of pure BPCMs. The measurement for pure BPCMs was conducted as received with no further treatment. For pure BPCMs, coconut oil possessed the lowest thermal conductivity of 0. 223 ± 0.002 W/mK, followed by beeswax 0.231 ± 0.001 W/m.K and palm wax 0.249 ± 0.001 W/m.K. For latent heat, beeswax has the highest latent heat, which is 240.2 J/g compared to palm wax and coconut oil. The melting temperature for the BCPMs range between 34.7 and 59.8 °C. Results showed that for latent heat, beeswax has the highest latent heat, which is 240.2 J/g compared to palm wax and coconut oil. The findings in this study are in line with findings from literature which stated that beeswax has high latent heat of 242.8 J/g [33] with melting temperature between 52 and 66 °C [34]. However, different properties could be observed depending on the types of beeswax used in the study as suggested in [34].

3.2. Thermal conductivity of BPCMs/Graphene

Fig. 4, Fig. 5 show the theoretical and experimental thermal conductivity of biobased PCM/graphene. It can be seen that the theoretical values are in line with the experimental value. The incorporation of graphene enhanced the thermal conductivity of the BPCMs which is in line with findings from [20]. The results indicate that the thermal conductivity improves linearly with the graphene concentration. The thermal conductivity for palm wax increased by 13.6 %, beeswax 31.1 %, and coconut oil 4.9 %. The highest thermal conductivity is gained by beeswax with 2.0 wt% concentration which is 0.303 W/m.K. The thermal conductivity for the coconut oil sample shows slight improvement due to the sedimentation of graphene. It is observed that only a small portion of graphene dispersed in the coconut oil was distributed, contributing to the slight increase in thermal conductivity. It can be seen that increasing the graphene concentration doesn’t enhance the thermal conductivity due to particle sedimentation as reported by [21]. The particle sedimentation is observed in Fig. 6. This is in agreement with recent study by Khaled et al [35] which found that the increase number of particles contribute to agglomeration and aggregation which leads to sedimentation of the nanoparticles. It is suggested that surfactants such as sodium dodecylbenzene sulfonate (SDBS) [36], gum Arabic [37], or sodium dodecyl sulfate (SDS) [38] could be used to improve the dispersion of nanofillers in the sample. Other study by Asif et al [39] recommended that increased in sonication time and energy could improve the dispersion of nanoparticles.

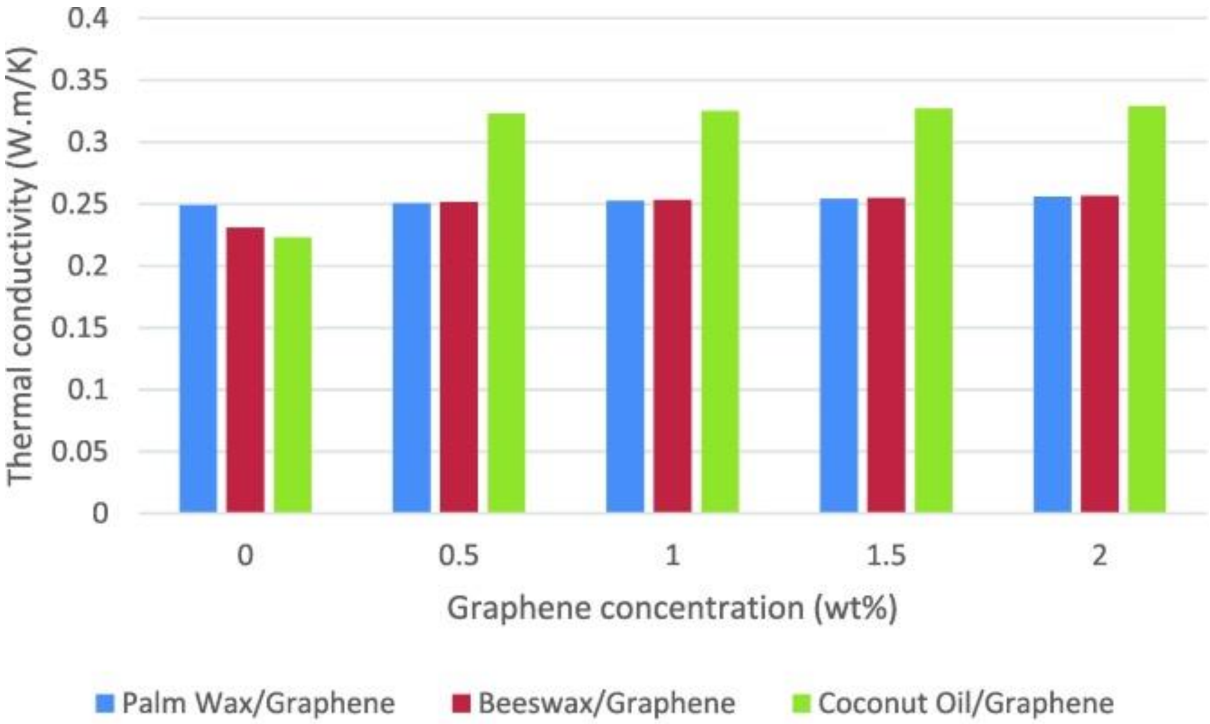

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Fig. 4. Theoretical thermal conductivity of BPCMs/Graphene as calculated from Maxwell Garnet equation.

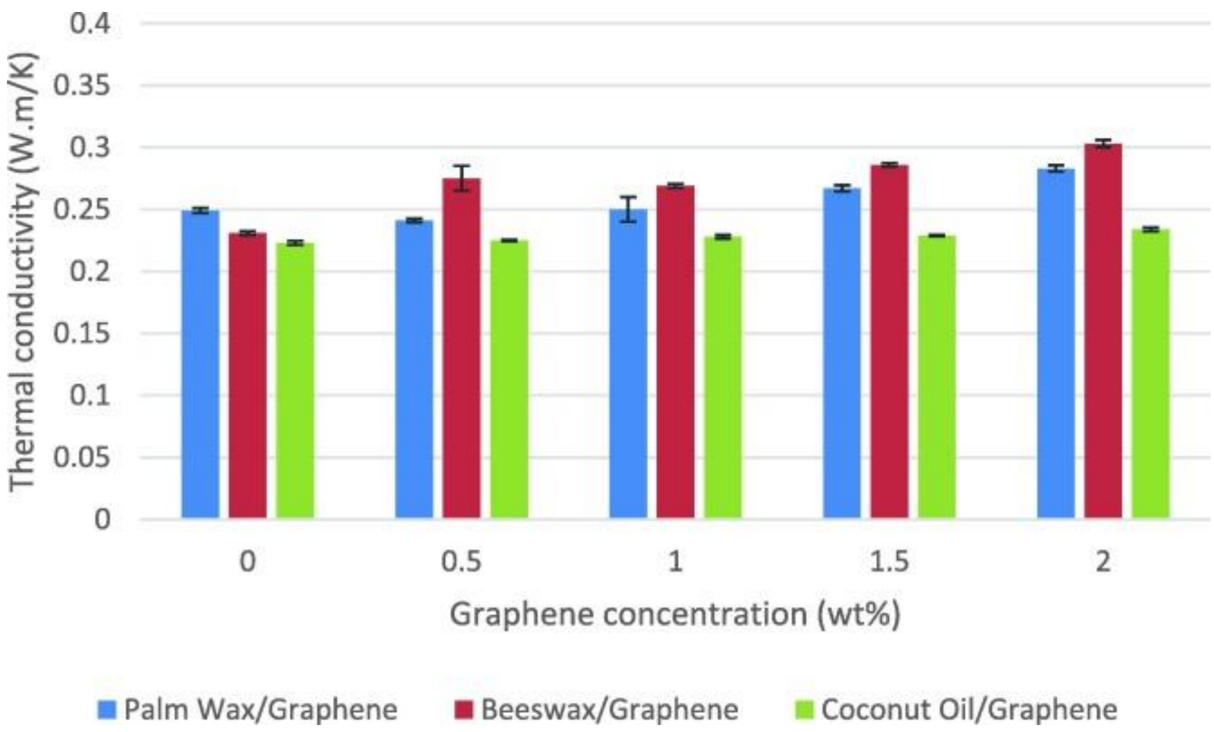

Fig. 5. Experimental thermal conductivity of BPCMs/Graphene.

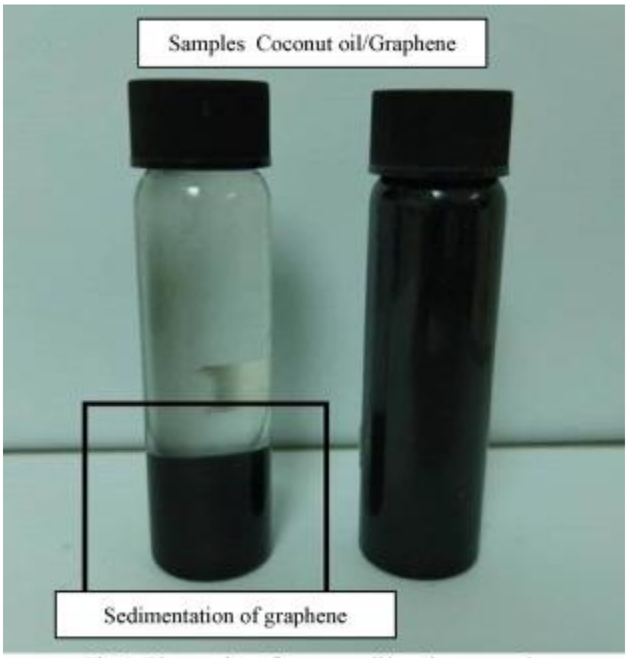

Fig 6. Observation of coconut oil/graphene sample

3.3. Thermal conductivity of BPCMs/Titanium oxide (TiO_2)

Fig. 7, Fig. 8 show the theoretical and experimental thermal conductivity of BPCMs/titanium oxide. It can be seen that the thermal conductivity of the BPCMs increased linearly with the increase in the concentration of titanium oxide. The highest thermal conductivity is obtained by beeswax with

2.0 wt% concentration which is 0.338 W/mK while coconut oil with the same concentration gained the lowest thermal conductivity, which is 0.232 W/mK. The results are consistent with findings by [24], [40]. The findings from this study are in agreement with Mahdi et al [41] where addition of titanium oxide in paraffin wax increased the thermal conductivity up to 77.9 % for 2.0 wt%. concentration. Study by Sadegh et al [42] reiterated that the addition of titanium oxide improved the conduction heat transfer. The addition of titanium oxide which acts as nucleating agent reduce the supercooling effects and contribute to congruent melting and solidification of BPCMs [43]. For coconut oil samples, it is observed that there is not much improvement in terms of the thermal conductivity. Hajar et al [44] suggested the addition of surfactant showed better titanium oxide dispersion compared to samples without surfactant. The addition of surfactants modified the titanium oxide nanoparticles surface to hydrophilic from hydrophobic resulted in better dispersion and stability [45]. Likewise, Leong et al [36] recommended that adding surfactant and increasing the sonication time could assist in enhancing the thermal conductivity of the samples. Another study stated that adding surfactants proved to be the most effective chemical method to improve the stability of the samples due the amphiphilic nature of the surfactant which extended the hydrophobic part so they act as co-polymer [46]. Both theoretical and experimental values show an increasing trend of thermal conductivity due to the addition of titanium oxide. It could be seen that the thermal conductivity of the beeswax/titanium oxide rises as the mass fraction of titanium oxide increased.

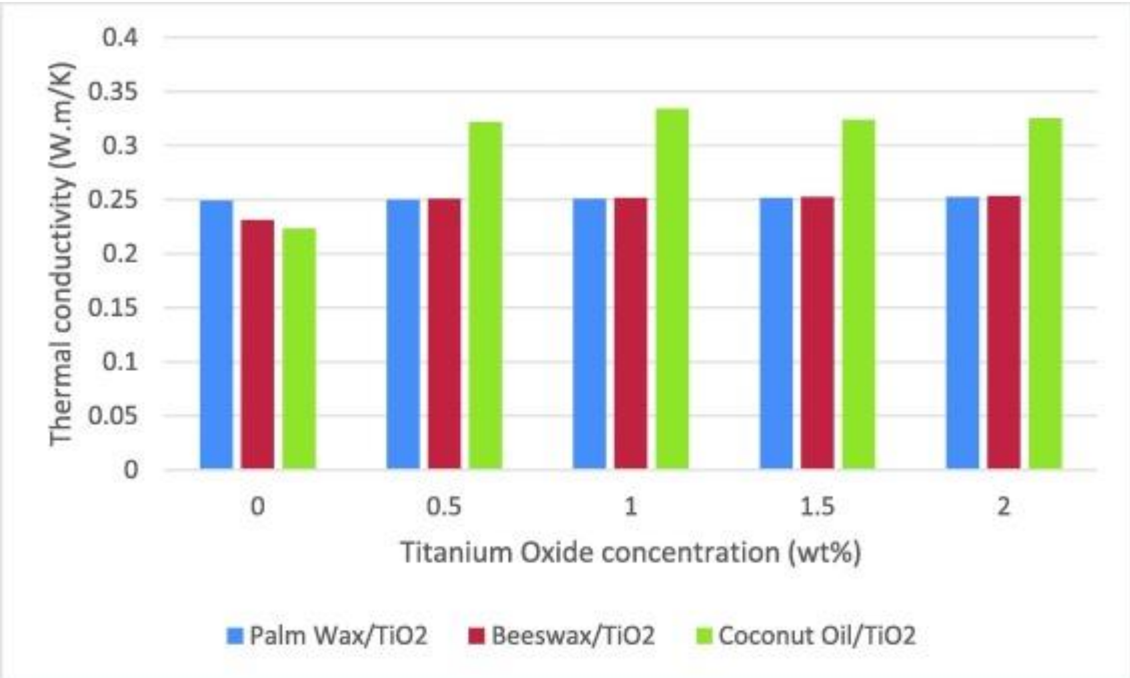

Fig. 7. Theoretical thermal conductivity of BPCMs/Titanium Oxide as calculated from Maxwell Garnet equation.

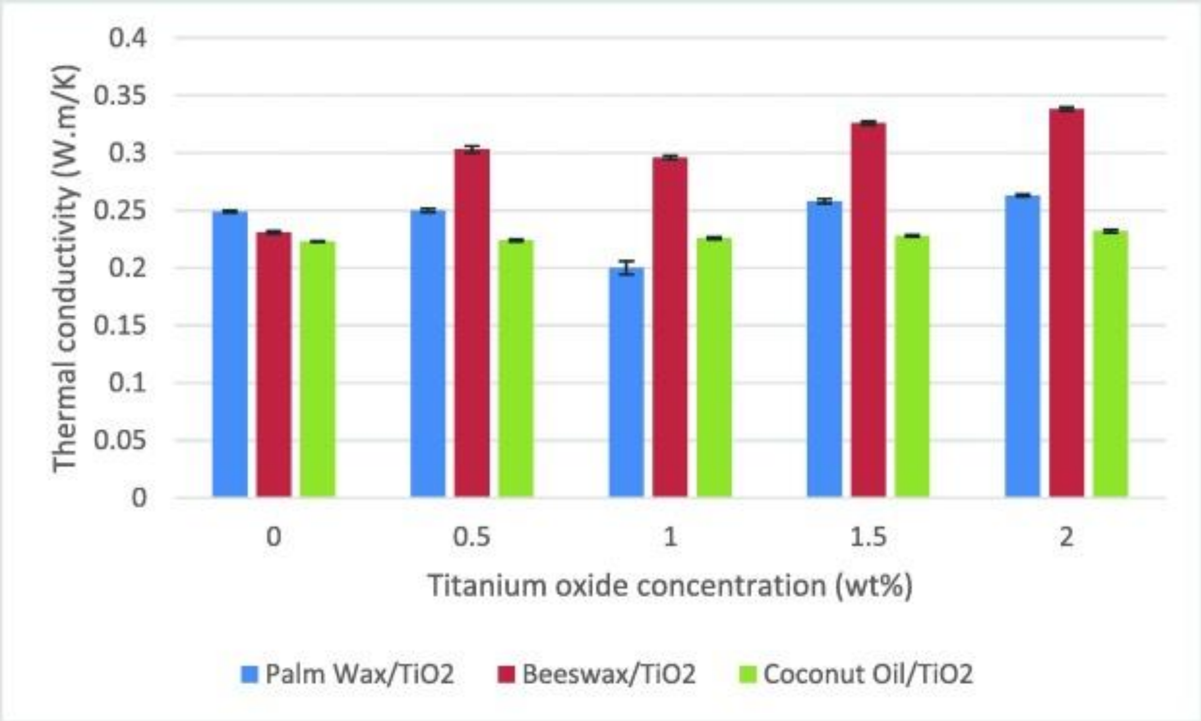

Fig. 8. Experimental thermal conductivity of BPCMs/Titanium Oxide.

4. Conclusion

BPCMs with different concentrations of nanofillers were prepared to study the thermal properties of the nano-BPCMs for LTHS in building application. It can be concluded that the addition of graphene and titanium oxide as nanofiller increased the thermal conductivity of the BPCMs. The thermal conductivity of beeswax with 2.0 % w.t concentration shows the highest increment of 31.1 % for graphene and 46.1 % for titanium oxide. Higher thermal conductivity contributes to better charging and discharging efficiency of the BPCMs. Homogenous dispersion of nanofiller in the BPCMs plays an important role in the increment of the thermal conductivity. Surfactant and sonication time have great influence in better dispersion of nanofiller in the medium, thus further studies using different surfactant and sonication time could be conducted to provide better understanding on the effects of dispersion of nanofiller in PCMs. The promising results for thermal conductivity enhancement of nano-enhanced BPCMs in this study showed that the biobased PCMs offers great potential to be use as latent thermal heat storage in building applications.

CRediT authorship contribution statement

Syafawati Hasbi: Funding acquisition, Project administration, Methodology, Writing – original draft. **Nurshahira Norazman:** Investigation. **Mohd Shahneel Saharudin:** Writing – review & editing.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Syafawati Hasbi reports financial support was provided by Malaysia Ministry of Higher Education.

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