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Optimization of Thermal Conductivity of Phase Change Materials/ Graphene by using Taguchi Method

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Abstract

Despite the extensive research on phase change materials, studies of different phase change materials (PCM) systems are still lacking due to the complexity of the mixture properties. The objective of this study is to use the Taguchi Method to predict the optimum parameters that give the highest thermal conductivity. This study considers the effects of four factors, the PCM, weight of graphene, mixing speed, and mixing time, on the thermal conductivity, compares the thermal performance of the optimum samples by adding silicon oil and mineral oil. The Analysis of Mean (ANOM) and Analysis of Variance (ANOVA) shows that the graphene weight and type of wax are the most influential factor determining the thermal conductivity, with a contribution percentage of 60% and 31%, respectively. The thermal conductivity increases proportionally with the weight percentage of graphene (GR). This study achieves the optimum results when using paraffin wax and 5 wt% of graphene; however, mixing time and speed do not contribute significantly to increasing thermal conductivity. The confirmation experiment of thermal conductivity shows better performance than the thermal conductivity predicted results by the Taguchi Method. The PCM added with silicone oil and graphene enhanced thermal conductivity. This study contributes to the knowledge of PCM thermal conductivity optimization via various parameters and encourages a continuous effort to develop PCM as thermal energy storage.

Keywords: Phase Change Materials, paraffin, graphene, Taguchi Method, thermal conductivity

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1. Introduction

The global dependence on fossil fuels as a primary energy source is a grave concern since fossil fuels are non-renewable energy and cannot continue to fulfill the increasing demand for energy. Mofijur *et al.* (2019) contended that the rapid rise in energy usage, continuous increase in fuel cost, and greenhouse gas emissions are the primary reasons for utilizing more reliable renewable energy sources. The need to reduce the reliance on fossil fuels necessitates the exploitation of unlimited renewable energy sources, such as the sun, wind, and waves, to fulfill the demands of the growing population. Thermal energy storage (TES) is one of the breakthroughs in energy savings (Rawi *et al.*, 2018). PCM is one of the methods for managing energy storage and is considered the ideal product for thermal management solutions. The threat of climate change necessitates finding a way to store renewable energy and has resulted in utilizing phase change material (PCM) to develop new, efficient, and sustainable methods for saving energy.

Various materials, such as paraffin, beeswax, and carnauba wax, can be used as PCM. It is essential to conduct more in-depth research on the different types of PCMs to determine the suitable PCM for specific applications because of the varying thermal properties of PCMs. This study focused on optimizing thermal properties of PCM added with graphene. It also investigated the PCM's melting and solidification temperature, latent heat, and thermal conductivity, which, unlike paraffin, is derived from vegetable (carnauba wax) and animal sources (beeswax). The benefits of inorganic vegetable- and animal-based PCMs are their eco-friendliness and renewability. In contrast, petroleum-derived paraffin is fossil-based energy that causes pollution and has harmful effects on living things.

The thermal properties of pure PCM could pose a problem because of its low thermal conductivity. It is possible to improve the thermal properties of PCM composites by incorporating an additive with high thermal conductivity, such as graphene, carbon black, and carbon nanotube. This research investigated the thermal conductivity of several PCMs, namely paraffin, beeswax, and carnauba wax, incorporated with graphene using the Taguchi Method and compared the thermal conductivity of the optimum phase change materials added with silicone oil and mineral oil.

Section 2 of this article discusses the literature review, and Section 3 describes the research material and method. Section 4 discusses the research results, and Section 5 presents the conclusion.

2. Literature Review

The PCMs investigated in this research are paraffin wax, beeswax, and carnauba wax. Paraffin wax is a widely used PCM with excellent latent heat characteristics, making it suitable for a broad temperature range (Mofijur *et al.*, 2019). The paraffins in PCM applications utilize solid-liquid phase change almost exclusively as the melting and freezing cycles distribute a nearly reversible energy transformation (Pielichowska and Pielichowski, 2014). Kim and Drzal (2008) found that paraffin is a PCM with promising potential because it is non-toxic, non-corrosive, has a high latent heat, good thermal and chemical stability, and, most importantly, low cost. Beeswax is a naturally occurring wax in beehives; the wax is secreted from the glands on the underside of the abdomen of honeybees. Yellow beeswax has a honey-like scent and is brittle in the solid state, while white beeswax has less honey scent and is more flexible (Amin *et al.*, 2016). Beeswax is inert and has high plasticity. It is insoluble in water and resistant to many acids but soluble in most organic solvents. Carnauba wax is a natural vegetable wax from the leaves of *Copernicia prunifera* palm that grows primarily in Brazil. The wax is obtained by beating it off of the dried palm fronds and refining it. Carnauba wax is one of the hardest plant waxes, has the highest melting point of all vegetable waxes, and is water resistant. It is hard, lustrous, and brittle and has a clean fracture. Carnauba wax is used in various applications, including in the food industry and cosmetics, as a coating for dental floss, automobile and furniture wax polish, and molds for semiconductors devices.

Alrashdan and Alsharaeh (2015) studied the heat transfer properties of paraffin-based PCM added with varying mass concentrations of 1, 5, and 10 wt% graphene nanofiller. The results showed that adding graphene to paraffin-based PCM increased its thermal stability while preserving its latent heat storage ability, where adding 10% of graphene could increase thermal storage by 40%. Xiang *et al.* (2018) investigated the thermal properties with decrease in supercooling of graphene as additive into the paraffin emulsion (PE). The thermal conductivity continued to rise with varying graphene mass concentrations of 0.6, 0.8, 1.0, and 1.2 wt%. The thermal conductivity increased by 7.8% (0.05557 W m^{-1} K^{-1}) between the lowest and highest graphene mass concentration. Adding graphene also reduced the supercooling by up to 98%.

Ramakrishnan *et al.* (2017) conducted an investigation using paraffin/expanded perlite (EP) PCM as the base PCM and graphene nanoplatelets as an additive. They added exfoliated graphene nanoplatelets (xGNP) to produce a nano-composite by the vacuum impregnation method. Adding 1% of xGNP into the PCM material increased the thermal conductivity of the composite PCM by up to 49%. The composite reduced the heat storage/release duration by up to 33% compared to the composite without xGNP. The researchers conducted another experiment to enhance the paraffin/hydrophobic expanded perlite (EPOP) by adding 0.5% graphene nanoplatelets (GNP) and found that the additives increased the thermal conductivity by up to 49% and reduced the heat storage/release duration of the composite by up to 20%.

Previous research has compared GR and other additives. Li *et al.* (2019) found that adding 2.0% of GR to paraffin resulted in the highest increase in thermal conductivity compared to expanded graphite (EG) and graphene oxide (GO) with a ratio of 1.73 to the pure paraffin, which also increased the composite's melting point by about 4°C. Yang *et al.* (2018) discovered that GR increased the latent heat of paraffin without affecting the phase transition temperature. The authors also found that adding GO and GR to paraffin increased thermal conductivity, where GR is more effective in increasing thermal conductivity than GO.

Liu and Rao (2017) reported that GR is better than exfoliated graphite sheets in improving the thermal conductivity of paraffin. The thermal conductivity increased by 17.2% when they added 2.0 wt% GR to paraffin. The melting point of the composites is also lower than pure paraffin. Kibria *et al.* (2015) found that the thermal conductivity of graphene composite increased by about 140% relative to pure 1-octadecanol without a significant reduction in phase change enthalpy. Amin *et al.* (2017) conducted an experiment using beeswax as a base material and added GR nanoplatelets to produce a composite and measured its thermal properties. Adding GR nanoplatelets reduced the composite's melting point while increasing the solidification point by the mass fraction of graphene nanoplatelets. The composite's melting enthalpy increased by 22.32%. The thermal conductivity of the composite increased to 2.89 W/mK at the mass fraction of 0.3 wt% graphene nanoplatelets, and the heat capacity increased by 12%.

Gathering information about phase change material was done after screening information from the previous studies and experiment done by past researchers. This study used the Taguchi Method to design an experiment by implementing Orthogonal Arrays (OA). The Taguchi innovation design approach is quick and fast, making it an elementary but effective tool for many engineering scenarios. This method is chosen to reduce the research complexities and evaluate several parameters without conducting many experiments to determine the optimum parameters.

Table 1 lists previous studies on PCMs which add GR as additives. This study sought to produce the best base PCMs material (paraffin wax, beeswax, and carnauba wax) and used graphene as the additive. The researchers hope the modified composite PCMs would have enhanced thermal characteristics in terms of thermal conductivity, latent heat, and melting and solidification temperatures. The literature review reveals the following knowledge gap.

a. Despite the extensive research to improve the thermal properties of PCMs, there is a dearth of studies on combining GR with beeswax and carnauba wax.
b. Most research focused on the effects of the additives but did not consider the best parameter's process for fabricating the optimal composite PCMs.

Table 1: Summary of the fabrication methods and characterization of PCM and graphene additive

Author/Date Publication	**PCMs added with graphene and fabrication method**	**Method for characterizing the Composite PCMs**
Alrashdan and Alsharaeh (2015)	• Paraffin with graphene nanofiller	• Thermogravimetric analysis • Differential scanning calorimetry
Ramakrishnan **et al**. (2017)	• Paraffin/expanded perlite PCM with graphene nano-platelets • Fabricate composite by vacuum impregnation methods • Mix composite by vigorous stirring for two hours using a magnetic stirrer	• Scanning electron microscopy • Thermal conductivity analyzer
Ramakrishnan **et al**. (2018)	• Paraffin/hydrophobic expanded perlite (EPOP) with 0.5% graphene nanoplatelets (GNP) • Fabricate composite by vacuum impregnation methods • Mix composite by vigorous stirring for two hours using a magnetic stirrer	• Scanning electron microscopy to observe the micro-morphology • Differential scanning calorimetry
Xiang **et al**. (2018)	• Paraffin/graphene phase change material • Shear the composite at 8000 rpm for ten minutes using a high-speed shearing machine	• Differential scanning calorimetry (DSC) to measure the temperature and enthalpy emulsions • Thermal constant analyzer to measure thermal conductivity.
Amin et al. (2016)	• Beeswax/graphene phase change material • Fabricate composite by ultrasonic methods	• Differential scanning calorimetry to measure latent heat and heat capacity • Scanning electron microscopy • Thermal conductivity meter

3. Materials and Methods

3.1. Materials

This study used 100% solid pure paraffin wax without additional particles and a melting temperature of 58-60°C, beeswax with a melting temperature of 60-64°C, and carnauba wax with a melting temperature of 80-83°C. The additive for the PCMs is graphene nanoplatelets provided by Sigma Aldrich (806633-25G), the surfactant is sodium dodecyl benzenes sulfonate (SDBS), the mineral oil from Hyrax Hypertrans HR transformer oil, and the 10-oz silicone oil spray from Johnsen's.

3.2. Method

This study used the L9 orthogonal array to conduct nine experiments. Table 2 presents the actual weight of the materials, additive, mixing time, and speed. The grated paraffin wax and beeswax were heated in the oven for 2-3 hours for complete melting while the carnauba wax was melted in a water bath. The RW 20 digital model mechanical stirrer from IKA was used to stir the liquid PCM-additive mixture while heating the bottom of the container with a digital hot plate from Cimarec Digital (ATH 50053) to ensure that the mixture remains in a liquid state during the mixing. The heat transfer medium is high-temperature silicone and mineral oil with high heat transfer capabilities and thermal stability. The sample preparation for this sample consisted of weighing out the appropriate mass of both optimum sample and silicone and mineral oil in approximately a 1:1 ratio by mass to yield an overall sample mass of approximately 20 milligrams. Thermal characterisation of the composite PCMs were measured by the K2D Pro device and Simultaneous Thermal Analyzer STA 8000 from Perkin Elmer. Each characterization was run at least four times to obtain the optimum parameters and five experiments was conducted to verify the results.

Table 2: The weight of the materials, additive, mixing time, and speed

	Control Factors			
Experiment	**Phase change material**	**wt% of the graphene nanoplatelets**	**Mixing time (Min)**	**Mixing speed (rpm)**
1	Paraffin wax	0.05	15	100
2	Paraffin wax	1	30	300
3	Paraffin wax	5	60	500
4	Beeswax	0.05	30	500
5	Beeswax	1	60	100
6	Beeswax	5	15	300
7	Carnauba wax	0.05	60	300
8	Carnauba wax	1	15	500
9	Carnauba wax	5	30	100

4. Results and Discussion

Table 3 presents the measured thermal conductivity for all samples in the experiments using the L9 orthogonal array. Experiment 3, which was paraffin wax with 5wt% GNP, 60-minute mixing time, and 500 rpm mixing speed, has the highest thermal conductivity of 0.292 W/m·K, while Experiment 7, which was carnauba wax with 0.05wt% graphene, 60-minute mixing time, and 300 rpm mixing speed has the lowest thermal conductivity of 0.242 W/m·K. Figure 1 presents the Pareto ANOVA chart, which shows the control factors based on the percentage of factor effect. The figure shows that the weight percentage of GNP has the highest factor effect and contributed the most (60%) to enhance the thermal conductivity, followed by the type of PCM (31%), mixing time (5%), and mixing speed (4%).

Table 3: Thermal conductivity of the phase change materials

Experiment no.	Thermal conductivity (W/m·K)				Mean sum of squares of the reciprocals (MRR)	SN ratio
1	0.271	0.274	0.268	0.275	13.52	11.31
2	0.280	0.275	0.273	0.271	13.25	11.22
3	0.292	0.289	0.287	0.289	11.95	10.78
4	0.271	0.266	0.268	0.264	14.01	11.46
5	0.266	0.268	0.267	0.274	13.85	11.41
6	0.285	0.290	0.288	0.285	12.14	10.84
7	0.247	0.249	0.242	0.250	16.40	12.15
8	0.253	0.249	0.247	0.253	15.94	12.03
9	0.286	0.288	0.281	0.282	12.38	10.93

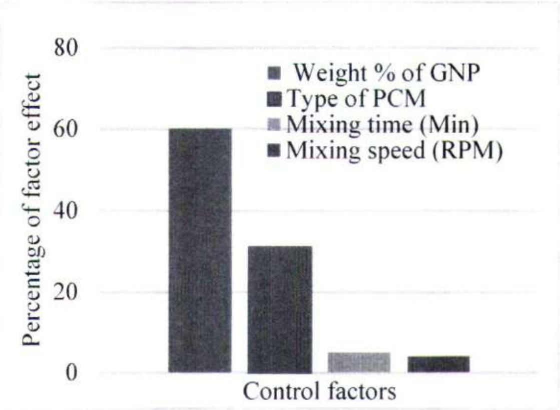

Figure 1: Pareto percentage of factor effects of the control factors.

The S/N chart in Figure 2 gives the optimum attainable level based on the control factors with the highest SN ratio. The type of PCM and the percentage weight of GNP are the dominant contributors to enhancing the thermal conductivity, while the contribution of mixing time and speed is neutral or negligible. The chosen optimum levels based on the highest SN ratio of each control factor are the paraffin wax and 5wt% of GNP. Alrashdan and Alsharaeh (2015) and Ramakrishnan et al. (2017) reported the similar result of paraffin thermal conductivity with the addition of GR. Both researchers concluded that the nanoplatelets functionally shaped and interconnected into the open paraffin pores and slightly increased the composite PCM's thermal conductivity. Praveen et al. (2019) reported anticipating higher thermal conductivity because of the high thermal conductivity of the GNP in the micro-encapsulated PCM. Table 4 summarizes the percentage contribution of the control factors and their optimum level.

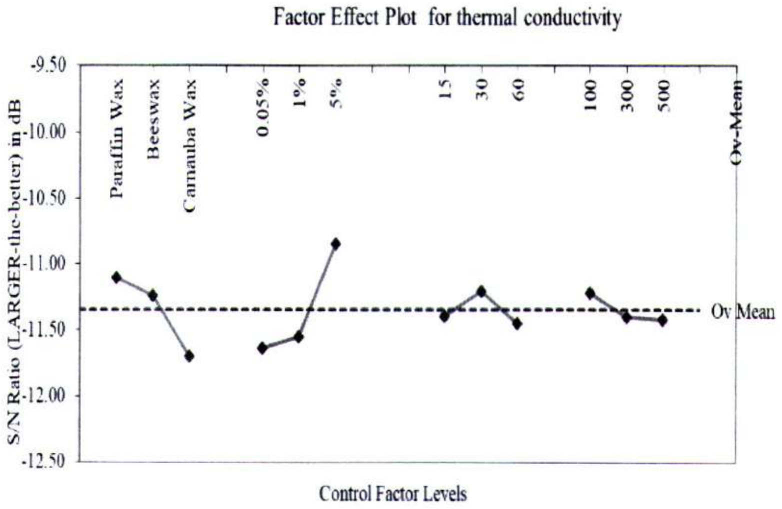

Figure 2: S/N chart for the factor effect of thermal conductivity.

Table 4: Optimum of control factors

Control Factor	**Optimum Level**	**Percentage of Factor Effect (%)**	**Rank**
Type of PCM	Paraffin Wax	31	Dominant
Weight percentage of GNP	5%	60	Dominant
Mixing time (min)	30	5	Neutral/negligible
Mixing speed (rpm)	100	4	Neutral/negligible

Table 5 presents the thermal conductivity of the optimum sample and the predicted value. The measured thermal conductivity for the optimum sample showed an average improvement of 6.6% ranging from 0.295-0.299 W/m. This result indicates that the higher thermal conductivity was related to the mass fraction of the GNP and the types of PCM. Adding GNP to PCM changed the thermal conductivity of the samples.

Table 5: Thermal conductivity of the optimum sample

Sample	Thermal conductivity (W/m·K)
Predicted	0.264-0.293
Experimental	0.295-0.299
Pure paraffin	0.271

Figure 3 shows the relationship between heat flow and temperature for the L9 orthogonal array experiments. The peaks in the graph represent the melting points in each experiment and the different transitions during the experiments. The small peaks at the low temperature at the beginning of the experiments are the minor peaks representing the solid-solid transformation change, while the peaks in the center are the major peaks for the solid-liquid phase transformation. Table 6 presents the melting point (°C) and heat of fusion (J/g).

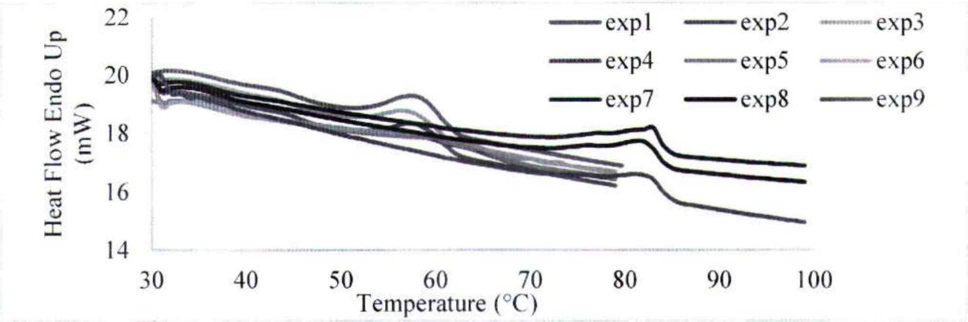

Figure 3: Graph of the heat flow endo-up versus temperature

Table 6: Thermal properties of the PCMs.

Experiment	Melting temperature (°C)	Heat of fusion or latent heat (J/g)
1	58.05	102.7974
2	57.95	79.6371
3	57.18	81.3935
4	63.58	15.6365
5	63.51	16.0274
6	62.36	16.1120
7	83.03	96.4595
8	82.12	97.0971
9	81.63	97.3562

Figure 4 presents the curve for the heat flow versus temperature of pure PCM (paraffin, beeswax, and carnauba) to compared with the L9 orthogonal array experiments in Figure 3. Experiments that used paraffin wax as one of the control factors showed higher latent heat than the experiments using beeswax and carnauba wax as the PCM. The latent heat in the experiment increased after adding the additives with a specific mixing time and speed. Table 7 shows the latent heat of pure PCMs without graphene. The experimental paraffin wax added with GNP with a specific mixing time and speed showed a 0.83-21.47% increase in the latent heat compared with the pure paraffin wax. Alrashdan and Alsharaeh (2015) obtained a similar result for the latent heat of fusion for the paraffin added with GNP . Adding 1-10 wt% of GR nanoparticles increased the thermal storage by 40%. Moreover, PCM's lower melting point temperature was achieved with higher percentages of GNP.

Adding GNP to pure beeswax with specific mixing time and speed increase the latent heat by 0.08-3.13% of the average latent heat. Amin *et al.* (2017) noted a 12% increase in latent heat capacity when adding 0.3%wt of GNP. However, adding the GNP affected the transition in the beeswax temperature. The melting temperature increased with a higher mass fraction of GNP. The melting temperature of the beeswax added with GNP increased by 0.2-2.18%, indicating that the phase change at higher temperatures of the melting process of the beeswax was due to the heat transfer rate of the sample. The experiments which used carnauba wax as the PCM also showed an increase in latent heat of 0.11-1.04% relative to the pure carnauba wax.

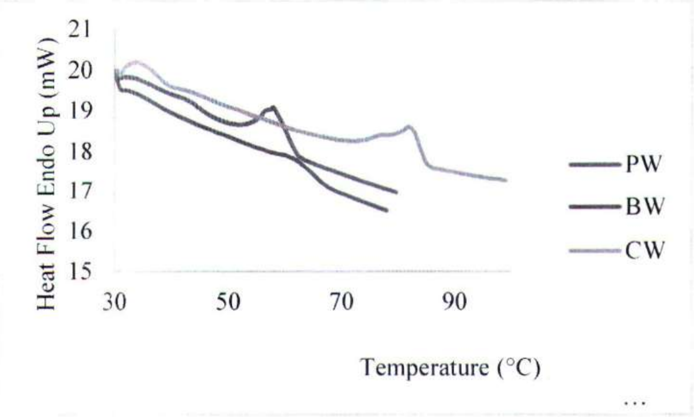

Figure 4: Graph of the heat flow endo-up versus temperature of the pure PCMs.

Table 7: Thermal properties of pure PCMs.

Phase Change Material	Melting temperature (°C)	Heat of fusion or latent heat (J/g)
Pure paraffin wax	58.01	80.7169
Pure beeswax	62.22	15.6230
Pure carnauba wax	82.25	96.3562

This study used silicon and mineral oil as the convective heat transfer medium because of their high heat transfer capabilities and good thermal stability. Figure 5 presents the curve for the latent heat and melting temperature for the pure paraffin wax, optimum PCM sample, and optimum PCM added with the convective heat transfer medium.

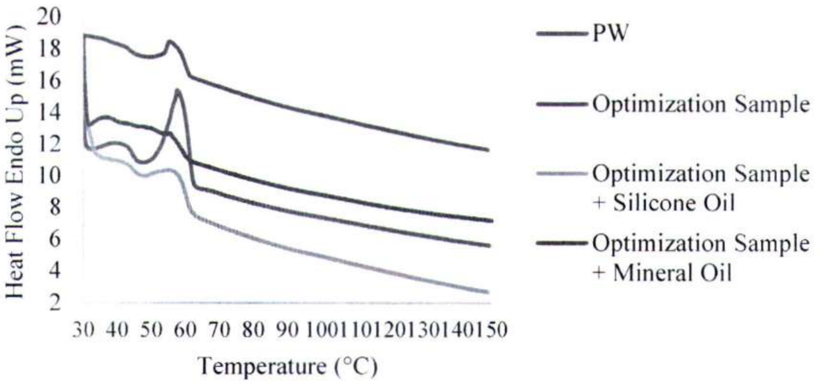

Figure 5: Graph of the heat flow endo-up versus temperature of optimum sample

Table 8 shows the melting point and latent heat of optimum samples with and without the convective heat transfer medium (silicone and mineral oil). The pure paraffin's melting point and latent heat are 57.67°C and 98.9849 J/g, respectively. However, the optimum PCM sample showed an average decrease in latent heat of 71.6% relative to the pure paraffin, which reduced the melting temperature by up to 55.42 °C. Adding thermally conductive fillers to the PCM resulted in a lower latent heat relative to the pure PCM (Haghighi *et al.*, 2020). The authors noted that, compared to pure paraffin, adding one wt.% graphene reduced the latent heat enthalpy by 18%. Adding nanoparticles reduced the latent heat of the PCM composite through molecular interactions between the nanoparticles and PCM. The optimal sample was chosen by considering the maximum thermal conductivity improvement.

Table 8: Comparison of the latent heat and melting temperature of the optimum sample with and without silicone and mineral oil

Description	PW	Optimum sample	Optimum sample + silicone oil	Optimum sample + mineral oil
Latent heat (J/g)	98.9849	28.0189	21.6666	1.2575
Melting point (°C)	57.67	55.42	57.05	55.65

Adding silicone and mineral oil to the optimum samples reduced the latent heat to 23% to 95%, respectively. However, adding silicon oil to the optimum sample resulted in a better latent heat than adding mineral oil. Lim *et al.* (2019) added silicone oil to base PCMs and noted a 50% decrease in the latent heat capacity of the mixture. The silicone oil increased the melting temperature by 1.63°C, while mineral oil increased the melting temperature by 0.23°C. The higher melting temperature indicates a significant improvement in heat transfer, which is crucial for applications in high operating temperatures. The high melting temperature and latent heat of some composite PCMs make them desirable for some applications such as waste-to-energy plants. The higher melting point of the PCMs increases their energy storage rate while retaining the high latent heat value. The optimum sample added with silicone oil has a higher latent heat and melting temperature than the optimum sample added with mineral oil.

5. Conclusion

This study used the Taguchi method to determine the optimum parameters to obtain the best thermal conductivity for the fabricated PCMs. The ANOVA and ANOM analysis showed that the weight percentage of GNP contributed 60%

to determining thermal conductivity, the base phase change materials contributed 31%, and the mixing time and mixing speed contributed 5% and 4%, respectively. The optimum result was achieved by adding 5 wt% of GNP to paraffin wax, although the mixing time and mixing speed did not have a significant effect in enhancing thermal conductivity. The experimental thermal conductivity is 9.4% better than pure paraffin and 6.6% better than the predicted values. However, the latent heat and melting point were lower. Adding silicone oil to the optimum composite sample resulted in a significantly higher latent heat than adding mineral oil, with a difference of 72% between the two types of oils. Adding silicone and mineral oil increased the melting temperature. A comparison of the results of adding silicon and mineral oil showed that silicone oil was more effective in improving the heat transfer rate of the optimum composite PCMs. The researchers recommend utilizing the optimum samples for applications such as solar collectors.

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