The Significant Effect of Graphene on Inorganic Salt Hydrated Phase Change Material Thermal Physical Enhancement

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ARTICLE INFO	ABSTRAK
	In this experimetal study, inorganic salt hydrated phase-change material (PCM) based on Calcium Chloride Hexahydrate ($CaCl_2 \cdot 6H_2O$) was employed to characterize phase change behavior such as the supercooling degree, phase change temperature and thermal conductivity. The $CaCl_2 \cdot 6H_2O$ nanocomposite PCM contained with different volume fraction (wt.%) of Graphene Nanoplatelets (GNP) are verified by heating-cooling curves resulted in enhancing thermal behavior. Results demonstrate the validation phase change temperature of the $CaCl_2 \cdot 6H_2O$ pure PCM are significantly referenced, while thermal conductivity and supercooling of $CaCl_2 \cdot 6H_2O$/GNP nanocomposite PCM was observed. The degree of supercooling is within the range of 6.0-15.2°C, with the maximum reductions of 68% and no phase segregation was observed. The thermal conductivity of the $CaCl_2 \cdot 6H_2O$/GNP with 1.0 wt.% 14.4% was higher than 0.5 wt.% of GNP. The $CaCl_2 \cdot 6H_2O$/GNP nanocomposite PCM presented acceptable thermal reliability and heat transfer characteristics, thus imitates its prominence for low-temperature range thermal energy utilization.

1. Introduction

Advanced energy technologies have such a great demand in improving the efficiency of renewable energy sources utilization as well as to promote the green technologies toward protecting the environment. The reliable solar photovoltaics/thermal systems, solar collectors and thermal energy storage with the present of nanotechnologies are the important components in the climate system, and plays a key role in reducing the global warming issue [1].

Thermal energy storage technique based on phase change material (PCM) has been recognized as one of advanced energy technologies. The inorganic salt hydrates PCM have a great attention of exploring due to their high-energy storage density, greater phase change enthalpy, rational price, non-flammable, multiple sources and relatively good thermal conductivity to be implemented in the

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field of energy storage technology. Commonly, $CaCl_2 \cdot 6H_2O$ [2], $MgCl_2 \cdot 6H_2O$ [3], $NaSO_4 \cdot 10H_2O$ [4,5], $CH_3COONa \cdot 3H2O$ [6], $Na_2HPO_4 \cdot 12H2O$ [5,7] and $Mg(NO_3)_2 \cdot 6H_2O$ [8] are employed as the base material of PCM storage media. Among various types of inorganic PCMs, the $CaCl_2 \cdot 6H_2O$ PCM is the most preferred inorganic salt hydrate PCM as their low cost, accessibility, and high thermal storage performance [9]. The potential inorganic PCMs substances in recent studies are shown as in Table 1 below as overview reported by Mohamad and Nor Azwadi [10] on low range temperature PCM studies [11-17]. The substances are suit in class of low and high temperature applications. Mainly, the thermophysical properties are summarized in order to review the substances performance and accuracy according to the related application.

Table 1
Thermo-physical properties of salt hydrates [10]

Salt Hydrates	Melting Temperature (°C)	Heat of Fusion (J/g)	Density (Solid) (10^3 kg/m^3)	Thermal Conductivity (Solid) (W/mK)	Specific Heat (Solid) (J/g.°C)	Reference
$LiClO_3.3H_2O$	8	253				[11]
$KF.4H_2O$	19	231	1.45		1.84	[12,13]
$Mn(NO_3)_2.6H_2O$	25.8	125.9	1.60			[11]
$CaCl_2.6H_2O$	28	174	1.80	1.088	1.42	[16,11]
$LiNO_3.3H_2O$	30	256	1.58	0.820	1.80	[11,14]
$Na_2SO_4.10H_2O$	32.4	248	1.49			[15]
$Na_2CO_3.10H_2O$	33	247			1.88	[15]
$CaBr_2.4H_2O$	34	115.5	1.52			[17]
$LiBr_2.2H_2O$	34	124				[17]
$Na_2HPO_4.12H_2O$	35-44	280		0.514	1.7	[12,13]
$Zn(NO_3)_2.6H_2O$	36	149.6	1.94		1.34	[17]
$KF.2H_2O$	42	162				[17,15]
$MgI_2.2H_2O$	42	133				[12,13]
$Ca(NO_3)_2.4H_2O$	42.4				1.46	[15]
$Fe(NO_3)_2.9H_2O$	47	155				[15]
$Na_2SiO_3.4H_2O$	48	168				[15]
$K_2HPO_4.7H_2O$	48	99				[15]
$MgSO_4.7H_2O$	48.5	202				[16]
$Na_2S_2O_3.5H_2O$	49	220	1.75	1.46		[16]

Inorganic salts containing water of crystallization is known as salt hydrates. It is formulated generally as $AB.nH_2O$, where number of water molecules and salt composition represented. The phase transformation dehydration of the salt can be expressed as

$$AB.nH_2O = AB + nH_2O - Q \tag{1}$$

$$AB.nH_2O = AB.mH_2O + (n-m)H_2O - Q \tag{2}$$

Nevertheless, $CaCl_2 \cdot 6H_2O$ is known as inorganic PCM exhibits some drawbacks such as supercooling, low heat transfer and so on which reflects to the long period cycles of thermal energy storage application. Thus, the performance of thermal energy storage systems is enhanced with the employment of nanomaterials which has recently been acknowledged. The previous studies as well recognize the effect on PCM thermal properties. Many of researchers have started to study the phase change performance and other thermal physical properties of nanocomposite PCM by adding the

nanoparticles into the PCM [18,19,20]. According to the overview by Yaxue *et al.,* [21] as shown in Table 2, many studies have been reported on graphene/graphite related nanoparticles were implemented in the base PCMs and fluids to enhance their thermal conductivity [22-29]. Recent cases overviewed by Mohamad *et al.,* [30] and Zik *et al.,* [31] also support the hypothesis that the employment of nanoparticles in which related to the size and surface area effect towards enhancement the thermal conductivity of advanced nanocomposite PCM. Surprisingly, there is no study on the combination of inorganic $CaCl_2·6H_2O$/GNP nanocomposite PCMs system reported.

Table 2
PCM-Graphene based nanoparticles combination [21]

Phase Change Material	Carbon Material Additive	Fraction of Additive	Thermal Conductivity, k of Composite PCM, (W/m.K)
Palmitic Acid [22]	Graphene nanoplatelets	5 wt%	2.75/2.54 (solid/liquid) (300 m2/g)
Beeswax [23]	Graphene nanoplatelets	0.3 wt%	2.89
Paraffin [24]	Graphene	2.0 wt%	0.46
Binary carbonate eutectic salts [25]	Graphene	1.5 wt%	-
Paraffin [26]	Nano-graphite	10 wt%	0.9362
Eutectic mixture (adipic acid and sebacic acid) [27]	Graphite nanoplates	0.5 wt%	0.131
OP10E/water emulsions [28]	Graphite nanoparticles	4 wt%	0.648
Palmitic acid/Mullite [29]	Graphite	5 wt%	0.520

In the present study, the GNP nanoparticles were embedded into the $CaCl_2·6H_2O$ as base PCM to develop advanced $CaCl_2·6H_2O$/GNP nanocomposite PCM. The study was further conducted to characterize their phase change behaviour such as the supercooling degree, phase change temperature and thermal conductivity. Furthermore, this study was comprehensively to identify the mechanism that efficiently optimized the thermal properties of $CaCl_2·6H_2O$/GNP nanocomposite PCM to be utilized in any thermal energy storage applications.

2. Experimental
2.1 Materials

Inorganic salt hydrate calcium chloride hexahydrate ($CaCl_2.6H_2O$, extra pure >98.5%, Fisher) as based PCM was purchased. Grade 4 carboxyl graphene nanoplatelets (GNP, purity >99.9%) nanoparticles were purchased from Biotek Abadi Sdn Bhd.

2.2 Preparation of Advanced Nano-Composite PCM

The two-step method is one of the more practical and effective way in preparing the $CaCl_2.6H_2O$ nanocomposite PCM [9]. The $CaCl_2·6H_2O$ material was produced by dissolved and crystallized of industrial grade $CaCl_2$. The homogeneously advanced nanocomposite PCM was prepared initially using a magnetic stirrer at a constant temperature, 50°C in 10 minutes. The graphene nanoparticle was then added to the $CaCl_2·6H_2O$-based PCM to mix into the solution and continuously stirred for extra 20 minutes. Finally, in order to improve the dispersion stability of the mixture and minimize nanoparticle aggregation, ultrasonic vibration was applied to the preparation processes for another 30 minutes. The importance of preserve liquidity is the factor to constant the vibrator temperature

above the CaCl$_2$·6H$_2$O melting point during the preparation processes. The volume fractions of the nanoparticle were 0.1, 0.5 and 1.0 wt.% of GNP. The used preparation method is schematized in Figure 1.
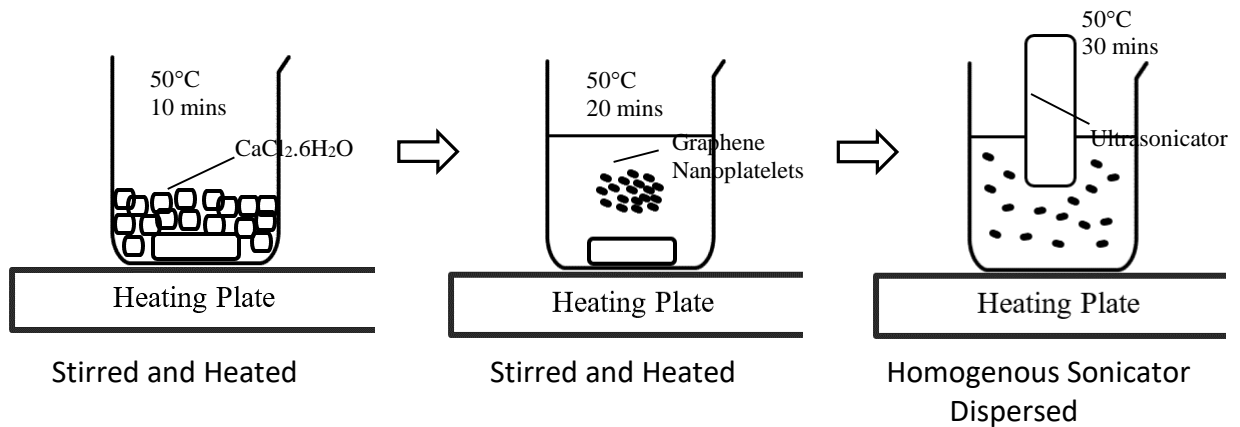

Fig. 1. Nano enhanced PCM sample preparation

2.3 Phase Change Temperature

In this study, the phase change temperature of advanced nanocomposite PCM was conducted through heat storage method. The set-up consists of a water bath and data acquisition system. The values of the CaCl$_2$.6H$_2$O/GNP nanocomposites PCM phase change temperature was determined under rapid heating rate using the DAIHAN-digital precise water bath set-up with fixed operating temperature as shown in Figure 2. The water bath had a temperature resolution of 0.1°C, with ±0.1°C temperature accuracy in range of ambient +5°C to 100°C operating temperature.
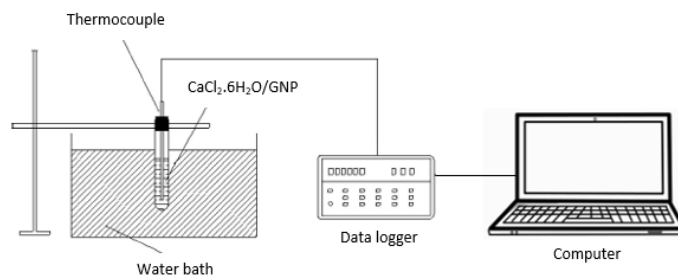

Fig. 2. Heating curve experimental set-up

2.4 Supercooling Degree

The correlation between the GNP nanoparticles concentration and the supercooling degree of the nanocomposite PCM is schematized as experimental set-up diagram in Figure 3 [32]. The set-up consists of a refrigerated circulator and a data acquisition system. The data acquisition system consists of a data collecting instrument (HIOKI-MEMORY HiLOGGER LR8431-20), a computer and a copper-constantan Omega thermocouple (T type). Temperature of the nanofluids inside the glass test tube is measured by the thermocouple which is set in the test tube. The volume of the solution was fixed at 50ml in the 30mm diameter of test tube. The average relative error was 0.75% when the temperature above 0°C. The results indicate that the supercooling degree of the CaCl$_2$·6H$_2$O/GNP nanocomposite PCM is indicative of the additional latent heat released by the nanocomposite PCM.
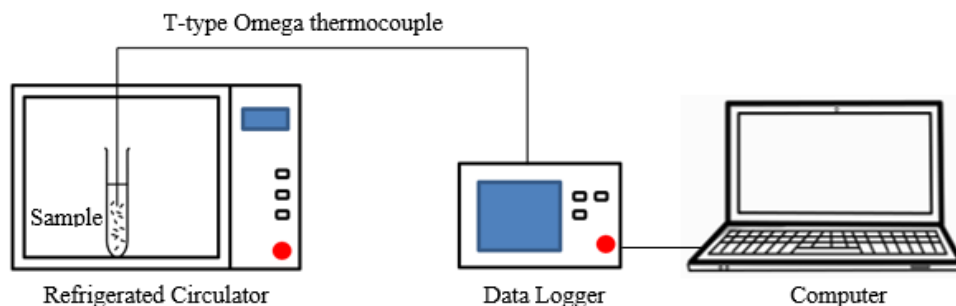
Fig. 3. Supercooling degree experimental set-up

2.5 Thermal Conductivity

The set-up consists of a thermostatic waterbath and a data acquisition system as shown in Figure 4. The data acquisition system mainly consists of KD2-Pro while data logger with a copper-constantan OMEGA thermocouple (T type) used for verification purpose. 5 constant different temperatures with 2 different volume fractions of GNP were employed in this experimental set-up in order to observe the correlation between temperature and nanoparticle concentration. The total volume of the NEPCM was fixed at 50 ml for each volume fraction of GNP. The accuracy of the measurement was ensured through the repeatability and reproducibility 10 times data measurements.

Fig. 4. Thermal conductivity experimental set-up

3. Results and Discussion
3.1 Phase Change Temperature

Figure 5 shows the heating curve of $CaCl_2.6H_2O$/GNP advanced nanocomposite PCM respected to time of phase change. The phase change temperature obtained was 28.9°C. The aim of the temperature is for the verification and validation of the melting/freezing point while completed solidification/melting was observed. The time for completed melting under rapid heating rate was

13.8 minutes which indicated equilibrium the total amount of latent heat stored. The latent heat of phase change is calculated Eq. (3) below.

$$\Delta H = \int \rho cp(T)dT \tag{3}$$

where ρ is the density, C is the specific heat capacity, T is the temperature. The correlation of latent heat of phase change was proportional with the specific heat capacity, while the other conditions remain unchanged.
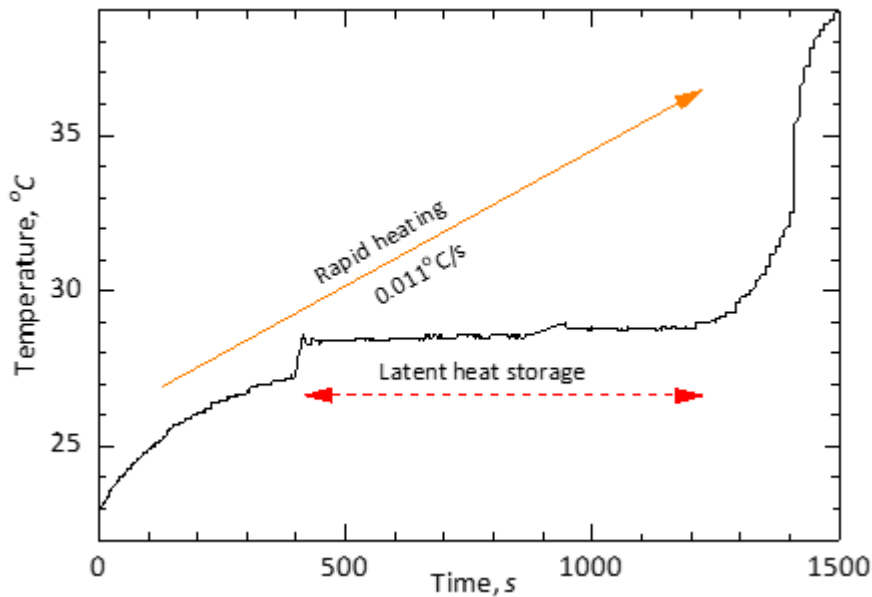

Fig. 5. Heat storage curve of CalCl$_2$.6H$_2$O/GNP

3.2 Supercooling Degree

A heating-cooling curve is a main criterion in this study to ensure the effectiveness of GNP nanoparticle in CaCl$_2$·6H$_2$O based PCM. The improved thermal physical properties of the advanced nano-composite are the key factors in this research. The GNP nanoparticle was fixed at 0.1, 0.5 and 1.0 wt.% respectively. Figure 6 shows the heat release/freezing curve of CaCl$_2$·6H$_2$O advanced nanocomposites PCM under rapid cooling condition. It was slightly displayed that the CaCl$_2$·6H$_2$O with 0.5 and 1.0 wt.% GNP nanoparticle concentration exhibited a significant supercooling degree about 5.4°C and 9.2°C compared to 0.1 wt.% of GNP nanoparticle. The degree of supercooling is within the range of 6.0-15.2°C, with the maximum reductions of 68% and no phase segregation was observed. The supercooling degree calculation of the 0.1, 0.5 and 1.0 wt.% CaCl$_2$·6H$_2$O with GNP nanoparticle applied the uncertainty formula, which was established by Moffat [33] as presented in the Eq. (4) below.
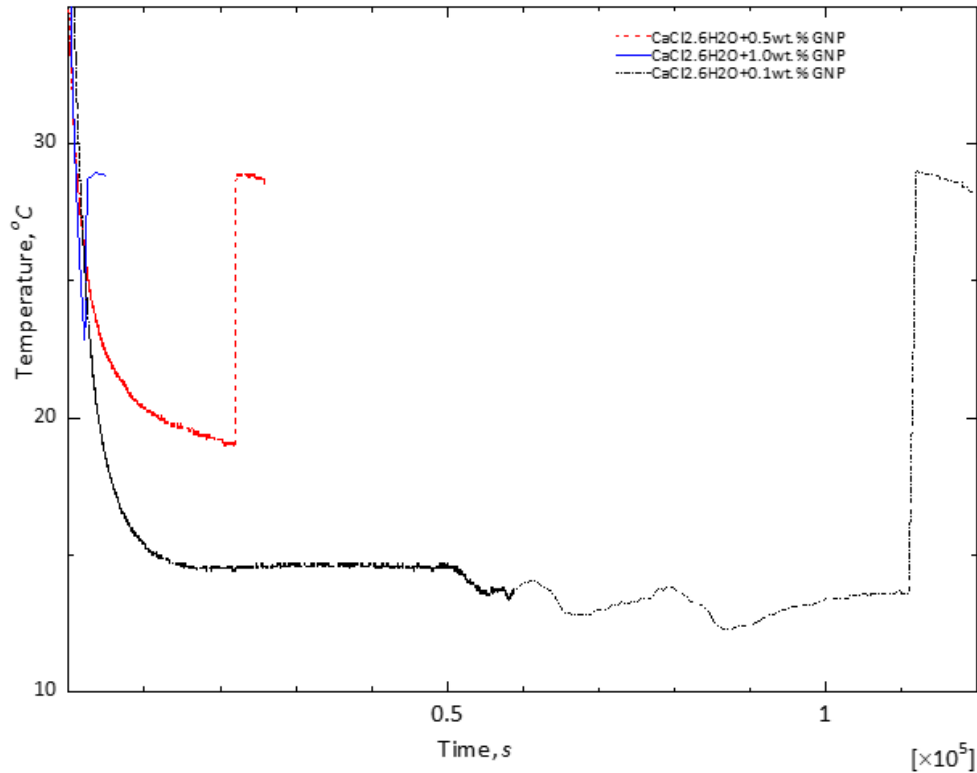
Fig. 6. Heat release curves of the pure CaCl$_2$.6H$_2$O PCM and with 0.1, 0.5 and 1.0 wt.% GNP nanoparticle

$$\frac{\delta\Delta T}{\Delta T} = \left[\left(\frac{\delta T_n}{T_n}\right)^2 + \left(\frac{\delta T_m}{T_m}\right)^2\right]^x \quad (4)$$

where,
   x = volume fraction;
   Tn = supercooling temperature;
   Tm = temperature of the phase during crystallization;
   ΔT = the supercooling degree, ΔT = Tm - Tn.

3.3 Thermal Conductivity, k

The correlation of heat transfer during melting and solidification is indicated by the thermal conductivity. A good thermal conductivity both represents a favorable in heat storage and release times, thus surge the performance of its actual application. The Eq. (5) shows the principle in obtaining the thermal conductivity [34].

$$k = \rho\alpha c_p \quad (5)$$

where
   k : thermal conductivity, $W/(m.K)$;
   ρ : density, g/cm^3 ;

α : thermal diffusion coefficient, mm^2 / s ;

c_p : specific heat at constant pressure, $J / (kg.K)$

The thermal conductivities of the $CaCl_2·6H_2O$ nanocomposite PCMs with 0.5 and 1.0 wt.% GNP nanoparticles demonstrated a marked increase following an increase in the volume of GNP nanoparticles in the composite respected to the different temperature as presented in Figure 7. The averaged thermal conductivity of the $CaCl_2·6H_2O$/GNP 0.5 wt.% NEPCM was 0.620 W/m·K while 1.0 wt.% was 0.709 W/m.k increased by 14.4%. The result demonstrated the essential contribution of graphene nanoplatelets in gaining a high value of thermal conductivity. Hence, the augmentation of $CaCl_2.6H_2O$/GNP was not only constructive in the improving the degree of supercooling, but also expedited the thermal conductivity enhancement.
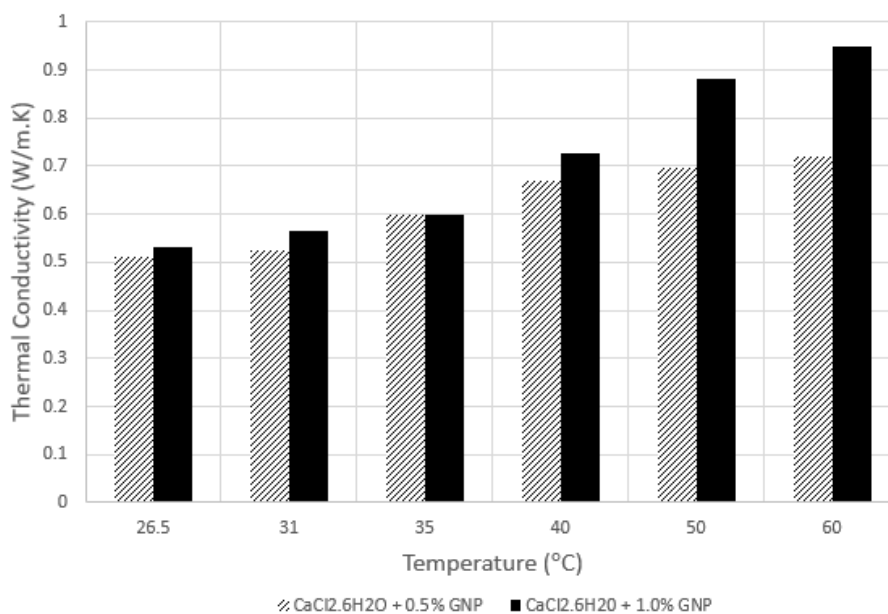

Fig. 7. Temperature-Concentration correlation of $CaCl_2.6H_2O$/GNP thermal conductivity

4. Conclusion and Recommendations

The present study was designed to determine the effect of GNP nanoparticle which can be employed to act as agent in suppressing inorganic phase change material supercooling drawback. One of the more significant findings to emerge from this study is that, the thermal conductivity of $CaCl_2.6H_2O$ PCM was slightly enhanced by employing the GNP nanoparticle. In addition, the findings of this study suggest that, the volume fraction of GNP was exhibited significantly affected the thermal conductivity of NEPCM respected to different temperature.

However, these findings are limited by the use of GNP volume fraction in $CaCl_2.6H_2O$ PCM. Further research is needed to account for the varying of nanoparticle concentration in order to obtain the correlation between volume towards thermal properties.

A greater focus on nucleating agent could produce interesting findings that account more for suppressing the PCM supercooling degree. A reasonable approach to tackle this issue could be to reduce the complete solidification time in actual application of thermal energy utilization.

Moreover, further research regarding the role of surfactant would be worthwhile and interesting. The issue of segregation and agglomeration is an intriguing one which could be usefully explored in further research. There is, therefore, a definite need for the thermal stability of the NEPCM. Furthermore, the implications of invented synthesis method will be a route to the properties study of specific thermal characterization techniques which should be considered for further studies.

Acknowledgement

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