Experimental Study on GNP Based Nanofluid Phase Change Material for Cooling Application

Thode Ranjith and A Sathishkumar
Department of Mechanical Engineering, SRM Institute of Science and Technology, Kattankulathur, Chennai 603203, Tamilnadu, India.

Email: ranjiththode1820@gmail.com

Abstract. The current study aims to analyze the solidification and melting characteristics of nanofluid phase change material (NFPCM) for building cooling applications. The NFPCM were prepared by using graphene nanoplatelets (GNP) in different concentrations (0.2, 0.4 & 0.6 Wt. %) and DI water as base PCM. The NFPCM was prepared using two step methods and stability of the NFPCM was analyzed using visual sedimentation method. The experimentation was conducted in -12 ºC and -9 ºC surrounding bath temperatures. The Sub-cooling was eliminated completely for the addition of SDBS and GNP in maximum concentration. The experimental results showed that the 10 % and 8.5 % reduction in solidification time for 0.6 wt. % GNP in base PCM for the surrounding bath temperature of -12 ºC and -9 ºC respectively. Also, the DSC analysis was conducted for the heating rate of 5 K/min and the reduction of enthalpy during heating and cooling was noticed 14 % and 11 % respectively. It is observed that the reduction of overall solidification time of the NFPCM will have the predominant effect in the chiller operation time.

1. Introduction
In the modern world, with the advancement in technology energy is required in every field for operational purposes, along with which the need for its storage has also increased. This need has driven scientist’s to harvest energy by new sources including both natural and artificial means to reduce the energy consumption requirement worldwide. But along with harvesting, storage of this energy is equally necessary as it serves various applications in different fields. The initial extensive use of Fossil fuels, Natural Gas and Petroleum have caused a decrease in its availability due to which the mankind has been forced to use alternate sources for energy production, sensible utilization of the available resources and higher efficient Energy Storage Systems. Energy can be stored in various forms by different methods, some providing short term energy storage while others are capable of bulk energy storage [1,2]. One major objective of storage involves the conversion of energy from different forms which are more difficult to the ones more economical and convenient. Thermal Energy Storage system serves the purposes of energy storage for the air conditioning systems of buildings. They make use of an energy storage tank and standard cooling equipment that would shift the cooling of the air conditioning of the buildings to an Ice storage unit during peak off hours. The formed ice in the storage is used for the cooling of the building during the night hours or power shortage thus maintains the building temperature. In this way, during the on peak hours the cool energy stored on the TES due to the formation of ice would be sent to the building cooling coils that would result in the air conditioning of the building [3]. Phase Change Materials (PCM) as the name states are materials that are capable of changing their phase at different temperatures with a unique characteristic of High Heat of Fusion, which make them capable of storing and releasing energy at a certain defined temperature. As we know matter exists in three phases Solid, Liquid and Gases, substances like water are capable of existing in all three states at different temperatures [4]. Water has
been researched as the prospective PCM for the CTES applications because of its inordinate latent heat, ample accessibility and effortlessness of taking care of, yet the presence of subcooling in water postpones the beginning of hardening and needs to diminish the evaporator operational temperature inside the chiller unit that effects with development in explicit energy absorption of around 3 - 4 %. [5, 6], has been endeavoured by the analysts on advancement of utilitarian latent heat storage materials with an objective of eradicating the sub-cooling of the PCMs. The incorporation of nucleating specialist with the PCM is a significant strategy to lessen the sub-cooling and those might be either being steady particles or precious stones that bring about the crystallization of the PCMs. The stage change qualities of different PCMs like the paraffin with unique stage change temperature. [7, 8], barium-chloride [9], oleic corrosive [10] and stearic corrosive [11] have just been scrutinized and proclaimed. By the benchmarks, their results uncovered that the expansion of nanoparticle prompts improvement inside the thermal-conductivity and decrease in freezing and melting time in contrast with that of base PCM. An investigation on freezing characteristic of nanofluid PCM for building applications was carried out [12, 13]. The experiment made use of different concentrations of graphene nanoparticles added to the PCM. It was seen that the base PCM indicated a 9.5% expansion in warm conductivity with expansion of 0.6% of graphene Nano-platelets because of the increment in surface zone and high warm conductivity. The paper also talks about the effect of density of the solution and shows that increase of density is not recommended.

It is ascertained from the writing that the incorporation of graphene improved the thermal-properties of the Phase change material with respect to the molecule focus which drives them to turn out to be extra appropriate for Thermal energy storage solicitations. Considering the top notch thermal-transport-properties of GNP, to build an energy productive cool thermal energy storage system, the overarching contemplates work objectives to explore heat-transfer attributes of water scattered with the GNP in a circular case all through solidification at a few encompassing temperature states of the HTF.

2. Materials and methods

2.1. Selection and Preparation of Nano fluid phase change material

The selection of the PCM depends on its various thermal properties and its application for thermal energy storage for cooling purpose. It has a high latent heat of combination per unit with moderately high specific-heat, high density and high thermal-conductivity. It has complete reversible charging and discharging cycle in order to store and release the energy during peak hours. Taking this into consideration DI Water is selected with a Thermal Conductivity of 0.608 W/mK for the research.

The addition of nanoparticle to the PCM solution has enhanced the thermal transport properties of the solution and has resulted in the reduction in the solidification time hence, increasing the efficiency of the energy storage system. Taking the various thermal properties and factors into consideration for the cooling energy system application, Graphene Nanoplatelets (GNP) have been selected for the research.

2.1.1. Specifications of GNP

| PRODUCT       | GNP                        |
|---------------|----------------------------|
| supplier      | Cheap tubes (USA)          |
| Purity        | 97%                        |
| Dimensions    | >2um                       |
| thickness     | 8-15nm                     |
| Specific Surface Area | 500 – 700 m2/g    |

2.1.2. Preparation of Nano fluid phase change material. NFPCM's arrangement is the fundamental advance for strengthening the heat transfer execution of Phase change material in thermal storage
prerequisites. The appropriate blending and compensation nanoparticles inside the base phase change material are required in order to sort out a consistent NFPCM.

In the current research, deionized water (DI water) as the base Phase Change Material, surfactant as sodium-dodecyl-benzene-sulfonate (S-D-B-S) and graphene Nano-particles (GNP) are used for preparation of Nano fluid phase change materials by two step method. TEM image of (GNP) as shown ‘Figure 1’

![Figure 1. TEM image of GNP.](image1)

The focus taken in (0.2wt%,0.4wt%,0.6wt%) was first blended in with DI water by using an attractive stirrer for ten minutes and this blend was ultra-sonication for ninety minutes in a ultrasonication vibrator at a recurrence of 40 kilohertz. Prior to the including of GNP with the water arrangement, the GNP were disturbed utilizing planetary ball plant utilizing ten mille meter balls for forty-five minute, trailed by ultra-sonicate process for sixty minute under thirsty conditions to untangle the Nano-tubes. The (S-D-B-S) (0.2, 0.4, 0.6 wt. %) was included distinctive fixation to the blend of DI water and nucleate specialist. This blend was mixed consistently utilizing an attractive stirrer for 10 min. The GNP of various convergence of (0.2, 0.4, 0.6 wt. %) was included with the above blend along these mixing was proceeded for an extra twenty minutes. The sample arrangement was then moved into the ultra-sonication vibrator and blended for sixty minutes at a recurrence of 40 kilohertz. In the wake of guaranteeing no recognizable sedimentation was seen inside the arrangement considerably following a time of three days, the solidification investigation were led with the readied NFPCM at different encompassing bath temperatures.

![Figure 2. SEM image of GNP.](image2)

The size of the scattered GNP in water was watched utilizing a scanning electron microscope (SEM; NRC SRMIST). The SEM images of the NFPCMs as shown in ‘Figure 2’. It can be observed within the figure that the dimensions of the dispersed GNP was in 20nm. No visual sedimentation was watched considerably following a time of a quarter of a year of preparation, subsequently guaranteeing the unfaltering quality of the prepared NFPCMs. In the wake of guaranteeing the consistent quality and size of the scattered GNP in water, the thermo physical properties enthalpy, specific heat (Cp), density (ρ), was estimated in differential scanning calorimetric (DSC) analysis.

2.2 Experimental setup

The experimentation setup as shown in ‘Figure 3’. It consist of cool thermal energy storage with chiller unit with refrigerant setup (R134a), design temperature -23.3°C. Temperature controller range is (0 to 20°C), compressor (KCH 431 LAGC 310DSK 689, kirloskar make), air cooled condenser, cool storage tank capacity is (1kWh), cryo pump is (0.025 kW), flow meter (100 to 1000LPH), the low temperature thermostat bath’s temperature was set at -23.3 °C for freezing and 30 °C for melting. Agilent data logger (34972A) with 16 channel AI 8000+ data acquisition and control system, computer. To start the solidification bath and melting, NFPCM was filled in spherical capsule ball in 70mm diameter up to 90% of volume of the full capacity of the ball. 3 RTD sensors are fixed in specific positions of the poly ethylene spherical ball. The first sensor is placed at 30 mm from the
centre sensor, second sensor was positioned at 15mm from the centre sensor and the third sensor being the centre sensor as shown in ‘Figure 4’.

![Experimental setup of solidification and melting bath.](image1)

**Figure 3.** Experimental setup of solidification and melting bath.

All the Resistance Temperature Detector sensors are associated with a data-logger to collect the consistent information created throughout the analyses. The round ball was loaded up with NFPCM and submerged in the solidification reservoir at a predetermined distance and the test was started. The experiment is conducted for total freezing time, for every 10sec the data logger will monitor continuously thereby continuing the experiment until the PCM within the centre of the spherical ball reaches the freezing temperature. After the solidification process gets completed, remove the ball from the solidification tank and immerse the spherical ball into the melting bath with different temperature range. The experiment conducted in different temperature for freezing is (-12 °C, -9 °C, -6 °C) and for melting is done for (17 °C). After the experiment, a graph will be initiated by the computer setup for the result.

![RTD sensor fixed in spherical ball.](image2)

**Figure 4.** RTD sensor fixed in spherical ball.

Volume calculation of Nano-fluid in spherical capsule

\[
\text{diameter of the spherical capsule} = 75\text{mm}
\]

\[
\text{thickness of the spherical capsule} = 2.5\text{mm}
\]

\[
\text{radius of the spherical capsule} = 36.25\text{mm}
\]
3. Results and Discussions
The measured amount of surfactant and GNP added for the preparation of 5 samples are given in the ‘Table 2’.

| Sample | Amount of DI water used (ml) | Amount of surfactant added (gm) | Amount of GNP added (gm) |
|--------|-----------------------------|-------------------------------|--------------------------|
| S.1    | 200                         | 0.00                          | 0.00                     |
| S.2    | 200                         | 1                             | 0.00                     |
| S.3    | 200                         | 1                             | 0.4                      |
| S.4    | 200                         | 1                             | 0.8                      |
| S.5    | 200                         | 1                             | 1.2                      |

The ‘Table 3’ shows 5 samples that have been considered for the experiment with their respective concentration of GNP particles and surfactant that has been added to DI water.
Once the samples are prepared, density was measured for each concentration using the following formulae:-

\[ M_1 = \text{mass of shell, DI water, surfactant (if present), GNP (if present)} \]

\[ M_2 = \text{mass of empty shell} \]

\[ V_1 = \text{volume of GNP and surfactant in shell} \]

\[ \text{Mass of the NFPCM} = (M_1 - M_2) \]

\[ \text{density}\ \rho = \frac{(M_1 - M_2)}{V_1} \]

3.1. Measurement of percentage increase in density of the sample
The measurement of density variation between the samples can be calculated from the given relation

\[ \text{percentage increase of density} = \frac{(\rho_{\text{NFPCM}} - \rho_{\text{PCM}})}{(\rho_{\text{PCM}})} \]

\[ \text{volume of spherical capsule} = \frac{4}{3} \pi r^3 \]

\[ = \frac{4}{3} \pi \times 0.03625^3 \]

\[ = 1.995 \times 10^{-4} m^3 \]

90% volume of nano fluid filled in spherical capsule = 1.810 \times 10^{-4} m^3 = 180ml approx
Table 3. Measurement of percentage increase in density for different samples.

| Sample No. | Composition | Mass of NFPCM | Volume of the beaker | Density (Kg/m³) | % Increase of Density |
|------------|-------------|---------------|----------------------|----------------|----------------------|
| S1         | DI Water (100 ml) | 99.10         | 100                  | 982            |                      |
| S2         | DI Water + 1 wt.% Surfactant | 99.18         | 100                  | 983.6          | 0.16                |
| S3         | DI Water + 1 wt.% Surfactant and 0.2 wt. % of GNP | 99.55         | 100                  | 991            | 0.916               |
| S4         | DI Water + 1 wt.% Surfactant and 0.4 wt. % of GNP | 99.89         | 100                  | 997.8          | 1.6                 |
| S5         | DI Water + 1 wt.% Surfactant and 0.6 wt.% of GNP | 99.98         | 100                  | 999.6          | 1.79                |

It is observed from the ‘Table 4’ the percentage of increasing the density is maximum for 0.6 wt. % of GNP. Also, the variation of density (minimum) will not affect the stability of NFPCM.

3.2. Solidification curves

3.2.1. Transient temperature variation of unadulterated PCM of DI water in Tsurr = -9° C. The ‘Figure 5’ shows the charging conduct of Phase Change Material (PCM) comprising of DI water. It tends to be seen that the overall freezing time is 5800 seconds (at centre). Additionally the RTDs fixed in 10 mm and 20 mm from centre is demonstrating that 4400 and 2400 seconds respectively. The sub-cooling of the PCM is estimated as - 6.01 °C for - 9 ° C surrounding bath temperature.

![Figure 5. Solidification of Di water in Tsurr = -9° C.](image)

3.2.2. Transient temperature variation of unadulterated PCM of DI water in Tsurr = -12°C. The ‘Figure 6’ shows the charging conduct of Phase Change Material (PCM) comprising of DI water. It tends to be seen that the general freezing time is 4900 seconds (at centre). Additionally the RTDs fixed in 10 mm and 20 mm from centre is demonstrating that 4400 and 2400 seconds respectively. The sub-cooling of the PCM is estimated as - 7.1 °C for - 12 ° C surrounding bath temperature.
3.2.3. Transient temperature variation of unadulterated NFPCM of DI water in $T_{\text{sur}} = -9^\circ C$ (centre RTD alone) for different concentration.

![Figure 7](image)

Figure 7. Solidification curve for $T_{\text{sur}} = -9^\circ C$ in different concentrations (centre RTD sensor alone).

The ‘Figure 7’ shows the charging conduct of Nanofluid Phase Change Material (NFPCM) comprising of DI water and graphene nanoplatelets (GNP) in various fixation (0.2, 0.4, 0.6Wt %). It is observed that the overall freezing time for unadulterated PCM, PCM + 1 Wt. % of SDBS + 0.2 Wt. % of GNP, PCM + 1 Wt. % of SDBS + 0.4 Wt. % of GNP, PCM + 1 Wt. % of SDBS + 0.6Wt. % of GNP as 5800, 5400, 5000, 4800 seconds respectively (at centre). It is observed that the most extreme concentration of 0.6 Wt.% of GNP with base PCM can decrease the overall solidification time in 8.5 % and furthermore, the sub-cooling got totally disposed of while including SDBS and GNP in greatest concentration.

3.2.4. Transient temperature variation of unadulterated NFPCM of DI water in $T_{\text{sur}} = -12^\circ C$ (centre RTD alone) for different concentration.
The ‘Figure 8’ shows the charging conduct of Nanofluid Phase Change Material (NFPCM) comprising of DI water and graphene nanoplatelets (GNP) in various fixation (0.2, 0.4, 0.6Wt %). It is observed that the overall freezing time for unadulterated PCM, PCM + 1 Wt. % of SDBS + 0.2 Wt. % of GNP, PCM + 1 Wt. % of SDBS + 0.4 Wt. % of GNP,PCM + 1 Wt. % of SDBS + 0.6 Wt. % of GNP as 4900, 4200, 3800, 2900 seconds respectively (at centre). It is observed that the most extreme concentration of 0.6 Wt.% of GNP with base PCM can reduce the overall solidification time in 10 % and furthermore, the sub-cooling got totally disposed of while including SDBS and GNP in greatest concentration.

3.3. Variation of Enthalpy during Freezing and Melting

3.3.1. Variation of enthalpy for (DI) water in 5K/min heating rate:

‘Figure 9’ shows the freezing and melting of base PCM was led in Differential Scanning Calorimeter to discover the Enthalpy of heating and cooling for PCM. The investigation was led between the temperature ranges from -30 to 30°C. It is observed that the enthalpy of heating and cooling is 325.5kJ/kg and -277.8kJ/kg for 5K/min heating rate. Additionally saw that the peak temperature during melting and freezing is 21.6°C and 4.9°C respectively.
3.3.2. Variation of enthalpy for NFPCM of (0.2, 0.4, and 0.6 Wt. %) in 5K/min heating rate:

Figure 10. (a)

Figure 10. (b)

Figure 10. (c)

Figure 10. Enthalpy of freezing and melting (a) NFPCM with 0.2 Wt. % of GNP (b) NFPCM with 0.4 Wt. % of GNP (c) NFPCM with 0.6 Wt. % of GNP.

‘Figure 10 (a-c)’ shows that the variation of enthalpy for both freezing and melting of NFPCM in different concentrations (0.2, 0.4, 0.6 Wt. %) of GNP. From Figure 10, (a) is observed that the
enthalpy of heating and cooling is 310.9kJ/kg and 261.2kJ/kg for 5K/min heating range in temperature range of -30 to 30°C and the peak temperature during melting and freezing is 21.2°C and 4.8°C respectively, from ‘Figure 10. (b)’ the enthalpy of melting and freezing is 310.9kJ/kg and 253.7kJ/kg and the peak temperature for melting is 21.5°C freezing is 4.7°C in the temperature range of -30 to 30°C in 5K/min heating range. ‘Figure 10. (c)’ the enthalpy of heating and cooling is 281.4kJ/kg and 246.2kJ/kg and the peak temperature for melting is 22.2°C freezing is 4.5°C in the temperature range of -30°C to 30°C in 5K/min heating range. From the DSC analysis the variation of enthalpy for both freezing and melting of NFPCM in different concentration of GNP. By the expansion of greatest Nano-material the enthalpy of PCM is getting decreased because of high surface zone. The decrease of enthalpy during heating and cooling was observed 14 % and 11 % respectively.

4. Conclusion
The accompanying ends were shown up from the exploratory work

- There is no appreciable increase in density of the PCM for the addition of graphene Nano-platelets in different concentrations
- The subcooling of phase change material is completely eliminated for the addition of (0.6 Wt. %) GNPs due to the higher surface area for the formation of nucleation cites.
- The scattering of the Nano-material likewise diminished the freezing time of the NFPCM by 10 % and 8.5 % than that of DI-water at the bath temperature of - 12 °C and - 9 °C, separately.
- The reduction in enthalpies of NFPCM with maximum concentration were observed 14 % and 11 % for melting and solidification processes respectively. It is noticed that the addition of surfactant and nanomaterial to the base PCM will increase the thermal conductivity as well as reduce the enthalpies.

The recent developments in the nano-synthesization field will leads to improve an energy efficient cool thermal energy storage system applicable for various building cooling applications.

Acknowledgment
We acknowledge the support received from the DSC laboratory, department of mechanical engineering SRMIST kattankalathur, Chennai, Tamilnadu, India.

5. Reference
[1] Al-shannaq R, Kurdi J, Al-muhtaseb S, Dickinson M and Farid M 2015 Supercooling elimination of phase change materials (PCMs) microcapsules Energy 87 654–662
[2] Altohamy A A, AbdRabbo M F and Sakr R 2015 Effect of water based Al2O3 nanoparticle PCM on cool storage performance Appl. Therm. Engineering 84 331-338
[3] Cheralathan M, Velraj R and Renganarayanan S 2007 Performance analysis on industrial refrigeration system integrated with encapsulated PCM based cool thermal energy storage system Int. J. Energy. Res. 31 1398e1413
[4] Sharma R K, Ganesan P, Tyagi V V 2016 Long-term thermal and chemical reliability study of different organic phase change materials for thermal energy storage applications J. Therm. Anal. Calorim. 124 1357–1366
[5] Okawa S, Saito A, Kadoma Y and Kumano H 2010 Study on a method to induce freezing of supercooled solution using a membrane Int. J. Refrig. 33 1459–1464
[6] Hossain R, Mahmud S, Dutta A and Pop I 2015 Energy storage system based on nano particle enhanced phase change material inside porous medium Int. J. Therm. Sci. 91 49–58
[7] Kumaresan V, Velraj R and Das S K 2012 The effect of carbon nanotubes in enhancing the thermal transport properties of PCM during solidification Heat Mass Trans. 48 1345–55
[8] Sari A and Karaeplek A 2007 Thermal conductivity and latent heat thermal energy storage characteristics of paraffin/expanded graphite composite as phase change material Appl. Therm. Eng. 27 1271–7
[9] He Q, Wang S, Tong M and Liu Y 2012 Experimental study on thermophysical properties of nanofluids as phase-change material in low temperature cool storage Energ.Convers. Manage. 64 199–205
[10] Harikrishnan S and Kalaiselvam S 2012 Preparation and thermal characteristics of CuO–oleic acid nanofluids as a phase change material *Thermochim Acta* **533** 46–55

[11] Li H, Jiang M, li Q, li D et al 2013 Aqueous preparation of polyethylene glycol/sulfonated graphene phase change composite with enhanced thermal performance *Convers. Manage.* **75** 482–7

[12] Pachappan Murugan, Anbalagan Sathishkumar, Vellaisamy Kumaresan, Ramalingam Velraj, 2017 melting / solidification characteristics of paraffin based nan composite for thermal energy storage applications *Thermal Science* **21** 2517-2524

[13] Sathishkumar A, Kumaresan V and Velraj R 2016 Solidification characteristics of water-based graphene nanofluid PCM in a spherical capsule for cool thermal energy storage applications *Int. J. Refrig.* **66** 73–83