

# **EXPERIMENTAL INVESTIGATION ON THE CHARGING BEHAVIOR OF WATER BASED NEPCM FOR CTES APPLICATIONS**

**A MAJOR PROJECT REPORT**

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## **BONAFIDE CERTIFICATE**

Certified that this major project report titled **“EXPERIMENTAL INVESTIGATION ON THE CHARGING BEHAVIOR OF WATER BASED NEPCM FOR CTES APPLICATIONS”** is the bonafide work of **“SHRESHTH GUPTA (RA1811002010337), SHUBHAM SAURABH (RA1811002010347)”**, who carried out the major project work under my supervision. Certified further, that to the best of my knowledge the work reported herein does not form any other project report or dissertation on the basis of which a degree or award was conferred on an earlier occasion on these or any other candidate.

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## ABSTRACT

It is vital to tackle the concerns regarding energy security and climate change in the current environment and future predictions for energy generation and consumption. From the beginning of the extraction until the end-use consumption, there are countless possibilities for energy loss to occur. Energy storage decreases necessity emergency generators, part-load plants, that rely on primary energy sources to meet short-term fluctuating energy demand. The goal of this study is to improve the K of PCM through mixing various concentrations of GNPs, as well as to decrease time to solidify and increase the cooling rate all through subcooling of the NEPCM, in order to achieve a higher overall TES efficiency for various CTES applications. DI water is taken as the base PCM along with SDS as a surfactant to reduce the surface tension forces for better interaction of the graphene nanoparticles in the concentrations of 0.2%, 0.4%, 0.6%. The thermal conductivity of the samples were measured at 4 different temperature values using the Hot Transient Wire method. A maximum improvement of 11.46% (at 25 degree C) in thermal conductivity was seen in PCM having 0.6% GNP. The freezing experiment was conducted at a steady bath temperature of -9 degrees Celsius, with ethylene glycol+water as the HTF in a 60:40 ratio. According to improved thermophysical characteristics, the process that takes place was lowered by 30.56 percent and the subcooling time was dropped by 42.3 percent. During subcooling, the cooling rate went up by 10.67 percent, allowing for a speedier beginning of solidification.

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## LIST OF SYMBOLS AND ABBREVIATIONS

<b>S.no.</b>	<b>Abbreviation</b>	<b>Full Form</b>
1	DI	De Ionized
2	GNP	Graphene Nano-platelets
3	PCM	Phase Change Material
4	NEPCM	Nano Enhanced Phase Change Material
5	TES	Thermal Energy Storage
6	CTES	Cold Thermal Energy Storage
7	HTF	Heat Transfer Fluid
8	SDS	Sodium Dodecyl Sulfonate
9	NFPCM	Nano Fluid Phase Change Material
10	NP	Nano Particles
11	K	Thermal Conductivity
12	$\mu$	Dynamic Viscosity

# CHAPTER 1

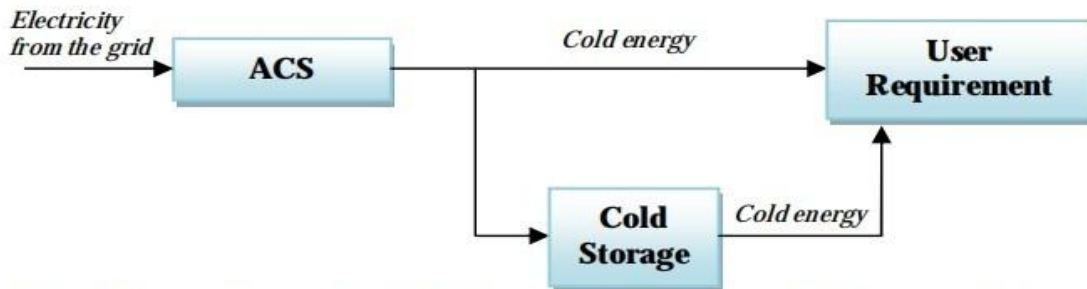
## INTRODUCTION

It is vital to tackle the concerns of energy security and climate change in the current environment and predictions for energy induction and utilization. From the beginning of the extraction until the end use consumption there are countless possibilities for energy loss to occur. Energy storage eliminates necessity backup generators or part/load power plants, that would otherwise rely on primary energy sources to meet short-term fluctuating energy demands. It allocates the energy needed during peak demand (during day) by using energy generated during the off hours (night time)

The basic principle of a thermal energy storage is that heat, cold energy can be held for some time in a medium and then recovered for later use in the same location. Heat storage is used to store and release heat, and the heat storage materials undergoes a phase shift. Isothermal transitions occur when a substance changes from solidified state to liquified state or vice versa. During the natural process, large quantities of thermal energy is reserved as heat within the material. The Cold Thermal Storage stores off-peak energy and uses it to overcome the gap between demand and supply during peak times.

Implementing energy efficient cooling for varied applications is a major challenge in businesses such as air conditioning and transportation. The construction industry accounts for almost 35% of global energy usage. Researchers and policymakers have expressed an interest in energy conservation and the use of various energy sources. In addition, for certain applications, particularly in building cooling, energy requirements are time dependent. A heat storage system (Thermal Energy Storage) is essential to store heat / cold energy in either sensible or heat of transformation using an appropriate medium due to the disparity between energy supply, demand.

Integrating the Cold TES system into the chiller makes the air-con system in large buildings in shopping malls and supermarkets so intermittent that changing unproductive hours throughout the day can significantly reduce power costs. Because of its high storage capacity and isothermal natural action, CTES systems have many advantages over sensitive storage.



**Fig. 1 Integration of a Cold Storage system in a ACS - user plant.**

Fig 1.1 - Integration of CTES System

Utilizing sensible heat, latent heat, plus thermochemical processes, alterations in a substance's internal energy can be held as thermal energy as heat. The reserved energy being influences the internal energy of storage device, elevating the temperature of the substance for Sensible Thermal Energy Storage. Mathematically it can be expressed by

$$Q = mc_p dT$$

Where Q = Energy Stored.

m = mass

C<sub>p</sub> = specific heat capacity

dT = change in Temperature

In case of **Latent Thermal Energy Storage** For storing or discharging heat energy, the heat storage material goes through a phase transformation process. The transition of a state of material takes place isothermally. Ie, the temperature remains constant, As a result, a significant quantity of heat energy can be stored in the material as latent heat throughout the phase change process. Mathematically, energy stored in case of Latent Thermal energy storage is

$$Q = mc_p \Delta T + LH$$

Where, LH is the latent heat.

In Thermochemical Energy storage, Certain materials' chemical potential is utilized as the basis for storing and releasing thermal energy with minimal thermal loss.

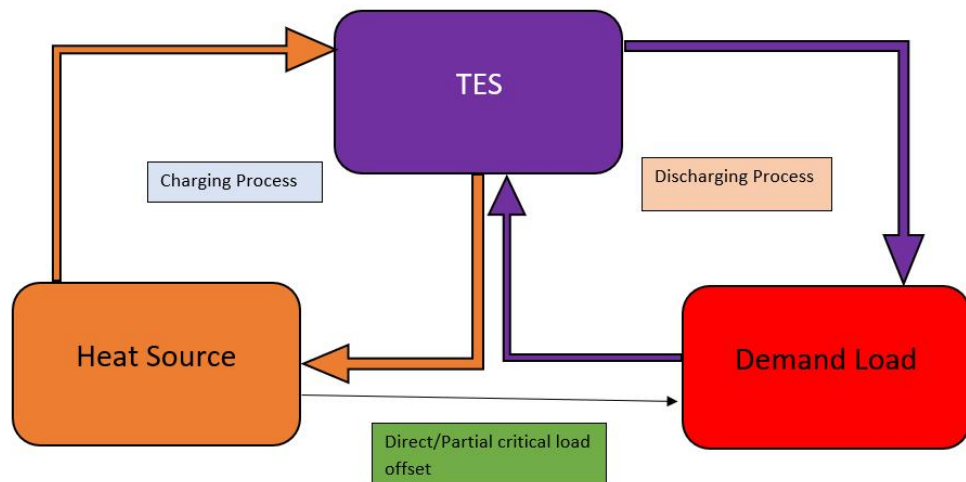


Fig 1.2 - Schematic Diagram of TES Incorporation

PCM is employed to forestall warmth within the electric cell at higher concentration ratio, to store the warmth within the type of heat of transformation and to distribute temperature uniformly throughout the photovoltaic cell. PCM is selected to support the freezing point, which should be above ambient and below PV operating temperature. It experimentally does out the thermal regulation of PV with PCM under passive cooling technique.

Due to the sheer collective oscillation of nanoscaled gaps, the temperature, melting point of the composite with the identical mass fraction were significantly lower than those of pristine octadecanoic acid. Because of the great level appropriate of graphene aerogel, one researcher constructed the PCM with 3-d graphene aerogel, saturated fatty acid, the K of the composite with 18% volume content increased by 12x compared to bottom PCM.

The thermal conductivity for biobased PCM impregnated with exfoliated graphene was reported to be three times that of the bottom PCM. Nevertheless, the Composite's heat index was just 68% of that of the bottom Phase Change Material and detection of shedded GNP significantly reduced subcooling.

The results show that the addition improves the warmth transfer capabilities of PCM in relation to the particles size, making it more suited for TES applications, according to the literature. Given GNP's excellent heat transfer properties and the need forefficient CTES systems, the above discussion disperses GNP inside of spherical capsules during HTF coagulation at various ambient temperatures. The goal is to assess the water's heat transmission properties.

## CHAPTER 2

### LITERATURE REVIEW

**V. Kumaresan et al.** in 2013 prepared NFPCM by dispersing MWCNTs with base PCM as DI Water. The solidification behavior was studied by encapsulating the NFPCM in a spherical container and the solidification time of the NFPCM was reduced with particular temperatures. The reduction in sub-cooling is due to MWCNT which acts as nucleating agent and it was also estimated that employing NFPCMs in the CTES might save roughly 6 to 9% of energy

**P. Chandrasekaran et al.** in 2015 the influence of fill volume on the solidification characteristics of DI water (Base PCM) in a spherical capsule was investigated experimentally. The spherical capsule was filled with a different volume percentage of its full capacity and it was found that the increase in fill volume led to preponing the start of solidification process and decrease in degree of supercooling which was further completely eliminated at 95% fill volume. They also observed that the heat flux was enhanced with the increase in temperature potential for the starting 50% of PCM mass. The maximum heat flux increment was observed at 95% fill volume proving its capabilities for cooling applications of large load in a short period of time.

**Dehkordi et al.** in 2017, using the Two-step approach, the nanofluid was created by the author, disseminating synthesised single walled carbon nanotubes (F-SWCNTs) with different volume fractions using an ultrasonicator in the base fluid, which is an antifreeze made up of 50% water and 50% ethylene glycol. The thermal conductivity of the specimens was tested at various temperatures using the KD2 PRO thermophysical properties analyzer. Thermal conductivity found to increase with increasing temperature and concentration, and the maximum deviation boundary value was discovered to be 2.4

percent. In the absence and presence of F-SWCNTs, the forced convective and thermal effectiveness of the antifreeze heat transfer was also tested, and it was discovered that the nanofluid was practical below 0.465 power of thermal conductivity ratio.

**A.Sathishkumar et al** in 2020 studied that the NFPCM with 0.75% mass Multi Walled Carbon Nano Tubes exhibited the highest ability to conduct heat, gains of eleven percent for liquid phase and twenty three percent for solid phase at particular temperatures. For 0.75 mass percent of Multi Walled Carbon Nano Tubes using base Phase Change Material, there were increases in latent heat of 12.8 percent and 14.13 percent for charging / discharging operations, respectively. The presence of high-conductivity Multi Walled Carbon Nano Tubes served as a nucleating substance, allowing NEPCM to begin charging ahead of DI water. Because of its magnified heat transmission behaviour, the solidification time was reduced by 28 percent and 19 percent with the NFPCM distributed with 0.75 % mass of Multi Walled Carbon Nano Tubes. Because of the greater nucleation rate, sub cooling was totally removed for NFPCM with maximal concentrations.

**V. Kumaresan et al** in 2017 studied and concluded that in the absence of GA, multiwalled carbon nanotubes distributed in solar glycol show a high inclination to aggregate and network into clusters. On rheological behaviour, the influences of particle concentration, temperature, and Ultrasonication time were investigated. Shear thinning non-Newtonian behaviour was seen in MWCNT and solar glycol derived nanofluids. The nanofluid's viscosity originally increased and dropped exponentially with increasing particle concentration and temperature. It was also discovered that the ultrasonication time has an effect on nanofluid viscosity. The viscosity of nanofluid goes up initially with a fall as sonification time increases. The increase in viscosity is linked to de-clustering of MWCNT bundle, which results in uniform dispersion, as reported. Viscosity reduction was linked to MWCNT breakage, leads to shorter MWCNT and hence weakening the MWCNT network in dispersion. Using MWCNT volume fraction of 0.6 percent, GA

weight percentage of 1.25 wt. percent, and temperature range of 30 to 50 C, maximum gains in thermal conductivity were around 28.45 percent to 30.59 percent.

**Rajendran Prabakaran et al.** In 2019, he evaluated the melting characteristic of such a fatty acid-based Phase Change Material (OM 08) comprising functionalised GNP in a spherical capsule as a heat transfer enhancer. The effects of GNP inclusion on  $K$  and  $\mu$  of PCM nano materials were examined to observe the effects of 2 parameters on discharging behaviour. The introduction of graphene nanoplatelets into liquid Phase Change Material improves both solid  $K$  as well as  $\mu$ , with the highest increases of 102.21 % and 1180.42 %, at 0.5 vol % GNP.

**Fuxian Wang et al.** in 2012 prepared graphene based ionic liquid nanofluids with no addition of any surfactant and measured an enhancement in thermal conductivity for the graphene based nanofluid at a very low mass fraction. Transmission electron microscope was used to observe the micro structure of the MWCNT. Also, it was shown that as temperature increases, the  $K$  and  $C_p$  increases, whereas, viscosity of the nanofluid decreases as compared to the base nanofluid. This novel type of nanofluid with such benefits, make it an ideal fluid for heat transfer and thermal storage.

**M. Ponrajan Vikram et al.** In 2018 investigated the agglomeration and subcooling behaviour of DI water (base PCM) dispersed in spherical encapsulation with varied mass fractions of NaCl, D-sorbitol for CTES applications. The DI water subcooled, and the subcooling was lowered. They also discovered it as the concentration of dispersants increases, the cooling rate falls, until at certain concentration where both sediments provide better cooling relative to base PCM(DI water) for a particular driving potential. Decreased subcooling and partially going to charge of water-based PCMs have been shown to improve CTES system energy efficiency.



**P. Ganesh Kumar et** in 2016 studied and concluded that the concentration of MWCNT rises, so does the divergence. This is owing to an unstructured, constrained mixing of nanotubes and solar glycol, which boosts the amount of the nanofluids for a given volume. According to the kinetics of MWCNT agglomeration and liquid layering, the thermal conductivity is increased to a maximum of 17.26% at a volume fraction of 0.4 percent MWCNT at a temperature of 70°C. At 0.2 vol. percent, 0.3 vol. percent, and 0.4 vol. percent MWCNT, the relative viscosity of MWCNT nanofluids increases significantly with temperature, although the ratio has a modest temperature dependence at 0.1 vol. percent MWCNT. The increase in the specific heat of nanofluids is greatest at the lowest particles of MWCNT (0.1 vol. percent), and this trend diminishes as the MWCNT concentration increases. The increased specific heat will be most likely owing to the MWCNT's large surface area per unit volume, which delivers a lot of surface energy.

**Rupinder Pal Singh et.** found that the specific heats of liquid and solid phases grow by 80 and 38 %, respectively, at GNP concentration of 5%, according to a study published in 2021. In the PCM, the latent heat drops by 0.35 percentage, 2.4 percent, and 5%, correspondingly, when f-GNP is added at 1, 3, and 5%. The Tmp. Lag falls by nine percent, 18 percent, and 20 percent at f-GNP concentrations of one, three, and five percent, respectively. However, nano dispersions have a modest effect on the average melting temperature. On the other hand, temperature has little effect on K of nano-composites. The overall melting time is lowered with f-GNP (5%) compared to pure PCM heat exchanger due to the high thermal conductivity of nano materials, which mitigates the negative impact of reduced convection. The f-GNP dispersions increase the molten PCM's  $\mu$ , reducing convective flow.

## **2.1 Observations from Literature Review:**

- Because of the greater nucleation rate, subcooling was eradicated for NEPCM with maximum concentration.
- The NEPCM had good cooling rate within the sub cooled region for a specified potential than that of the base PCM, allowing the CTES system to operate at more HTF temperature.
- SDS as a surfactant proved to be very effective in reducing the surface tension forces and improving the interactions between nanoparticles and base PCM.
- Deionized water as a base Phase Change Material was used because of its good thermophysical properties and easy availability.
- There is no significant increase in PCM density with graphene-based nanoparticles.. Due to the greater surface area for the production of nucleation sites, the subcooling of phase-change material is completely removed by the inclusion of GNPs.
- For CTES applications, the Solidification and Subcooling behaviour of DI water distributed with different mass percentages of NaCl and D-sorbitol in a spherical capsule showed promise.

## **2.2 OBJECTIVES**

Objectives are as follows:

- Study on the stability of NFPCM (SEM, zeta-potential distribution method).
- Study on phase change enthalpy (at 1K/min) and thermal conductivity at different temperature values.
- Study on solidification characteristics of NFPCM at -9 degree celsius HTF temperature in a spherical container.

- a. Reduction in total solidification (Charging) time.
- b. Reduction in subcooling.
- c. Cooling rate in subcooling region.

## **2.3 RESEARCH GAPS**

This study aims to investigate and comprehend the melting behaviour of water-based PCMs including Graphene-functionalized spherical shaped containers. Different load factors of GnP and two beginning temperatures of PCM were investigated for the same boundary condition.

It is essential to analyze the effect of nanomaterials on energy storage and release properties in general containers. This will give us an idea for designing CTES for various energy storage applications. Vessels of various shapes. Containers of different shapes are used to store the NFPCM for cold thermal energy storage applications.

## **CHAPTER 3**

### **EXPERIMENTAL INVESTIGATION**

#### **3.1 SELECTION OF PCM AND NANOMATERIAL**

Deionized water (DI water) is chosen as the base phase change material(PCM) because of its good thermophysical properties and easy availability. The thermophysical properties of DI water is shown below:

<b>Property of DI Water (Base PCM)</b>	<b>Value</b>
<b>Liquid Thermal Conductivity at 20°C</b>	0.598 W/mK
<b>Solid K</b>	2.2 W/mK
<b>Solid Density</b>	920 kg/m <sup>3</sup>
<b>Liquid Density</b>	1000 kg/m <sup>3</sup>
<b>Melting/Freezing Temperature</b>	0°C
<b>LH of Melting</b>	334 kJ/Kg K
<b>C<sub>p</sub> of Water</b>	4.187 kJ/Kg K
<b>C<sub>p</sub> of Ice</b>	2.108 kJ/Kg K

TABLE 3.1 - Properties of DI Water

Graphene Nano Platelets(GNPs) is selected as the nanomaterial which is procured from CHEAP TUBE,USA. GNPs show higher thermal conductivity and lower thermal contact resistance. The physical and thermal properties of GNPs are shown below:

<b>IUPAC NAME</b>	Sodium Dodecyl Sulfate
<b>Molecular Formula</b>	C <sub>12</sub> H <sub>25</sub> NaO <sub>4</sub> S
<b>Molecular Weight</b>	288.38
<b>Melting Point</b>	204-207°C
<b>Density</b>	1.03 g/mL at 20°C
<b>Flash Point</b>	>100°C

Table 3.2 - Properties of SDS Water

GNPs are chosen as the nanomaterials because of their good thermal conductivity and lower thermal contact resistance.

<b>Product Name</b>	Graphene Nano Platelets
<b>Formula</b>	C
<b>Molecular Weight</b>	12.01 g/mol

<b>Supplier</b>	CheapTubes, USA
<b>Average Particle Size</b>	8 - 15 nm
<b>Purity</b>	97%
<b>Specific Surface Area</b>	500 -700 m <sup>2</sup> /gm

Table 3.3 - Properties of GNP

### 3.2 PREPARATION OF NEPCM

A measured amount of Surfactant, Sodium Dodecyl Sulfonate (SDS) of 1% by weight is mixed with known volume of DI water (base PCM) of 200ml in all the four samples.

For the study, a total of 5 samples are prepared. For the 1st sample, only base PCM (DI water) is used for experimental analysis which is taken as reference for other samples. The second sample contains base PCM (DI water 200ml-equivalent to 200 gram) and surfactant (SDS 2 gram).

The surfactant used here (SDS) decreases the surface tension forces and improves the physical interaction between the base PCM (DI water) and nanomaterial (GNPs).

The GNPs are added in 0.2%, 0.4% and 0.6% by weight to the mixture of base PCM and SDS.

A total of 5 samples are prepared.

1. DI water
2. DI water+SDS
3. DI water+SDS+0.2% GNP
4. DI water+SDS+0.4% GNP
5. DI water+SDS+0.6% GNP

To achieve a well dispersed solution with no agglomeration of particles, The solutions are first mixed using a magnetic stirrer for 30 mins at 350 rpm. The NEPCM is gently removed from the magnetic stirrer setup after being observed and ensuring that there is no sedimentation, lump formation, or agglomeration.



Fig 3.1 - Magnetic Stirrer

Further, the solutions are ultrasonicated. Ultrasonication is basically a process where ultrasonic waves are bombarded into the sample to achieve uniform dispersion of the samples. The samples are ultrasonicated using a probe sonicator for 30 min, i.e. 10 min ultrasonication followed by a break of 10 minutes so that the temperature does not

increase and the ultrasonication process occurs at a stable temperature, and again 10 min of ultrasonication for each sample.

The specifications of the probe sonicator are given below:

<b>Frequency Probe</b>	20KHz
<b>diameter</b>	12mm
<b>Power rating</b>	500W

Table 3.4 - Specifications of Probe Sonicator





Fig 3.2 - Probe Sonicator



Fig 3.3 - Ultrasonicator Setup



Figure 3.4 - NEPCM Samples

For the study, a total of 5 samples are prepared :-

1. DI water
2. DI water+SDS
3. DI water+SDS+0.2% GNP
4. DI water+SDS+0.4% GNP
5. DI water+SDS+0.6% GNP

A measured amount of Surfactant(SDS) of 1% by weight is mixed with known volume of DI water (base PCM) of 200ml in all the four samples.

For the 1st sample, only base PCM(DI water) is used and experimental analysis done on this sample is used as a reference for other samples.

The second sample contains base PCM(DI water 200ml-equivalent to 200 gram) and surfactant(SDS 2 gram)

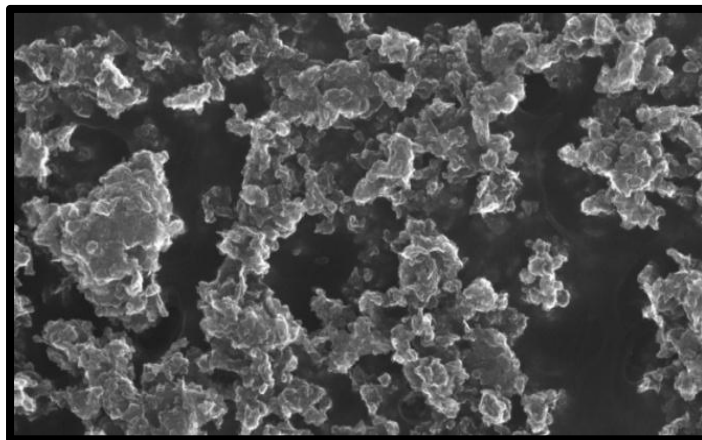
The GNPs are added in 0.2%, 0.4% and 0.6% by weight to the base PCM along with the surfactant in further 3 samples.

### 3.3 STABILITY ANALYSIS

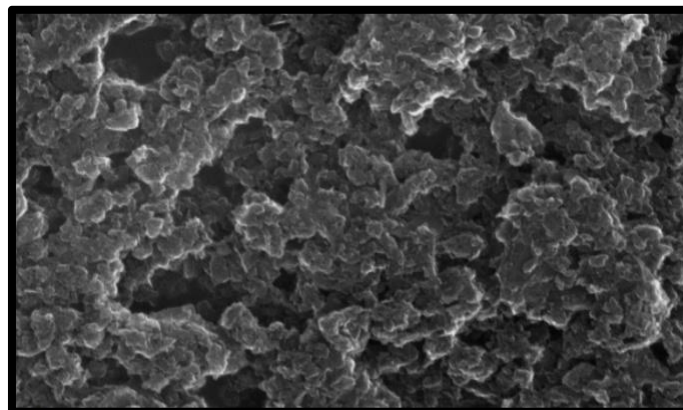
The Nanofluids(samples with GNPs) are made as a thin film using a hot surface to check the dispersion through SEM(Scanning Electron Microscope)

It is visually seen that as the GNP concentration is increased, the diffusion of GNP in the base PCM is also increased.Hence the dispersion is improved with increasing GNP concentration

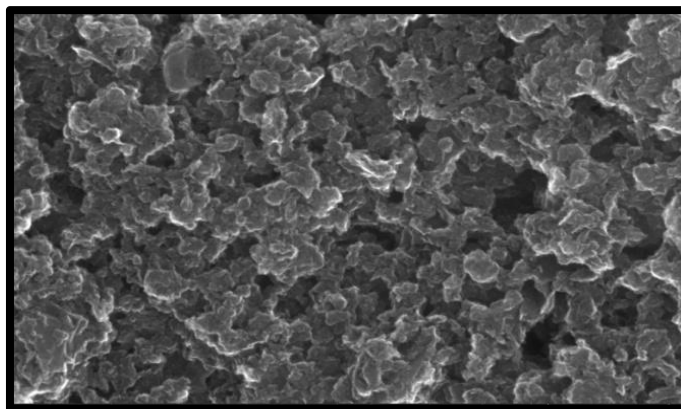
The SEM images of nanofluids(NF1-,NF2,NF3) is shown below.



NF 1- 0.2% GNP



NF2-0.4%GNP



NF3-0.6%GNP

Fig 3.5 - SEM Images of NEPCM Samples

Further, Zeta potential distribution method is used to check the stability of the prepared NEPCM samples. It is seen that for distribution values greater than 30 mV or less than -30mV, the NEPCMs show high degree of stability, while values less than 30mV or greater than -30mV can lead to agglomeration. The Zeta Potential distribution graph for maximum concentration NEPCM is shown in figure 2.

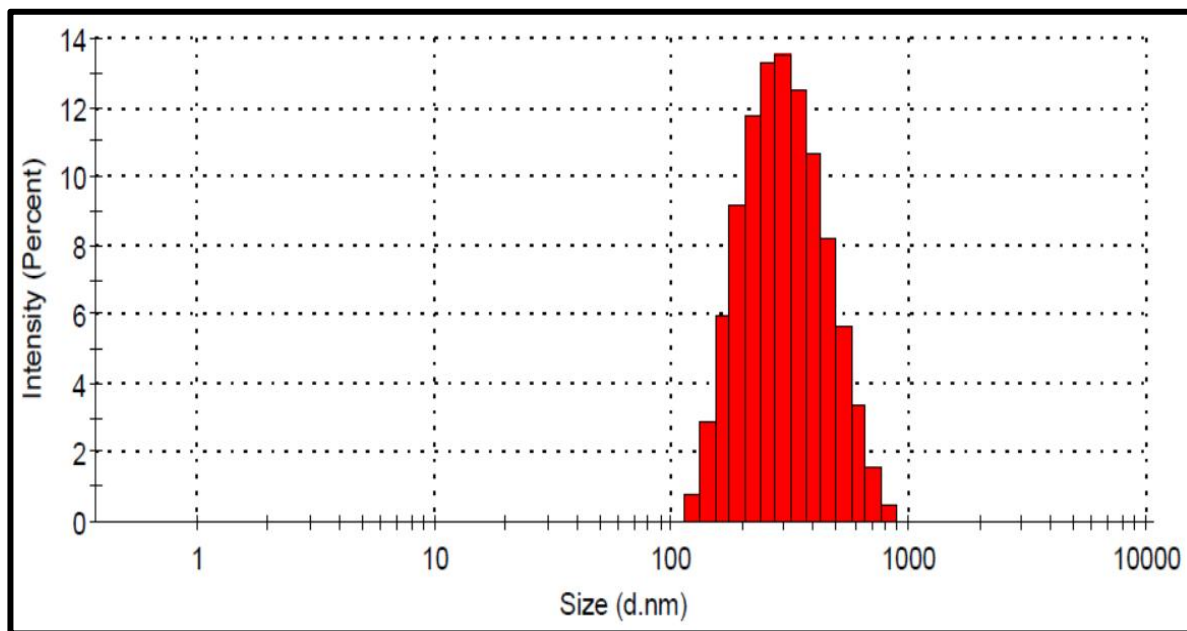


Fig 3.6 - Zeta Potential Distribution Curve for NF3

The y axis in fig 2 signifies the intensity of the NEPCM at a particular size (shown in x axis). From the figure, it is seen that every particle size is below 1000 nanometers (1 micrometer) for NFPCM having 0.6% Graphene nanoparticles by wt which further cements the argument that the solution is well dispersed and does not show any agglomeration or lumps.

### 3.4 DENSITY MEASUREMENT

The density of both the D.I. water and NEPCM are noted calculated at particular temperature (room temperature) by noting weight of a standard volumetric flask having liquid sample of Class B in it (25 ml), using a electronic weighing machine with a accuracy till 5 decimal points ( $\pm 0.00001$  g) kept within container that is airtight. The formula used to calculate density is,  $\text{Density} = \text{Mass} / \text{Volume}$ .

Sample	Mass(g)	Density(g/ml)	Percentage Deviation
DI water	24.47	0.9788	
DI+SDS	24.61	0.9844	0.57
DI+SDS+0.2%	24.7	0.988	0.93

DI+SDS+0.4%	24.801	0.992	1.34
DI+SDS+0.6%	24.852	0.994	1.55

Table 3.5 - Density Measurements

Since, The concentration of GNP is being increased in the base PCM DI water, the overall density of the NEPCM increases, which can cause agglomeration of particles leading to a non-uniform and unstable solution. But since the maximum percentage deviation of density with respect to the base PCM DI water is less than 2%, It has very less chances of agglomeration of particles. Although, the samples are still required to go through stability analysis using ZETA potential method and SEM imaging which could prove the stability and uniformity of the samples prepared.

### 3.5 Measurement of Thermal Conductivity

From inner tank to outer tank, a pipe(of stainless steel to avoid corrosion) of 10 mm diameter is welded with a ball valve to drain the inside tank. 2 plates are welded and in between Polyol + mpyol(50%+50%) solution is filled. by which the artificial foam was produced to achieve proper insulation. The setup is designed in such a way that the beaker cap does not touch the plate, and this entire environment is covered by Ethylene Glycol.

<b>Sensor Needle length</b>	60mm
<b>Sensor type</b>	KS3
<b>Name of the sensor</b>	TEMPOS

Table 3.6 - Specifications of Thermal Conductivity Setup

The measuring instrument was then connected to the sensor using a VGA cable.

### 3.6 Mechanism of Thermal Conductivity:-

The thermal conductivity of the samples is measured using TRANSIENT HOT WIRE METHOD. Heat is first supplied to the sensor from the instrument, The heat is then dissipated to the fluid in the beaker. Initially, the instrument will measure the temperature of the sample(around 25 degree celsius), then it will wait for 50 seconds to check whether the temperature of the sample is in equilibrium or not(Ethyl Glycol and Sample should be in thermal equilibrium) and if the temperature is observed to be constant(in equilibrium) by the instrument, then the instrument starts to supply heat through the wire, which further dissipates the heat into the sample for 1 MINUTE, after that, the temperature of the sample is checked, and based on its thermal conductivity, it will absorb the heat and show different raises in temperatures for different samples which is reflected in the digital measuring instrument.

Although, The measurement will have some error and its value is calculated based on one parameter i.e., if the initial temperature of the sample is varying from 25 degree celsius (eg 25.011,25.012 etc). So if any change in that 'constant' equilibrium temperature is detected, it will show a max error value of 0.002%.



Fig 3.7 - Thermal Conductivity Apparatus



Fig 3.8 - Polyol + Mpyol solution



Sr. No.	Temperature (C)	Conductivity(W/mK)	Error	% of sample	max enhancement %
1	10	0.569	0.0008	0	4.74
2		0.579	0.0007	0.2	
3		0.588	0.0006	0.4	
4		0.596	0.0005	0.6	
5	15	0.585	0.0007	0	5.81
6		0.594	0.0003	0.2	
7		0.609	0.0007	0.4	
8		0.619	0.0003	0.6	
9	20	0.615	0.0005	0	9.26
10		0.655	0.0002	0.2	
11		0.659	0.0002	0.4	
12		0.672	0.0005	0.6	
13	25	0.602	0.0008	0	11.46
14		0.652	0.0007	0.2	
15		0.667	0.0008	0.4	
16		0.671	0.0002	0.6	

Table 3.7 - Thermal conductivity for different Temperatures

Normally, as the temperature increases , the thermal conductivity value decreases in liquid solutions because of expansion of liquid molecules which causes hindrance in the movement of electrons , which are primarily the reason for heat transfer,hence the thermal conductivity value.

But in case of nano GRAPHENE enhanced PCMs, we can see that as the temperature increases, the thermal conductivity value also increases. This is because of the fact that graphene has a negative thermal expansion coefficient. This means that the material shrinks when heated, unlike conventional materials that expands upon heating. Hence it allows easier flow of electrons which improves the thermal conductivity(K) value.

The negative-to-positive transition was discovered at 900 K in the thermal expansion coefficient of single layer graphene.

### 3.7 Differential Scanning Calorimetry Test

DSC follows the principle of thermal methods analysis, which is basically used to check the stability of a compound on the basis of temperature and this technique is based on the principle of thermogravimetry where we plot a graph of temp on X axis and weight on Y axis(thermogram). DSC analysis is better than most of the other thermal methods analysis such as thermogravimetric test because of 2 primary reasons which ensure that the sample does not react with the moisture present in the atmosphere and is very well protected to get accurate results.

1. In DSC the crucibles are protected by inert gas
2. The sample is perfectly sealed and packed.

In the DSC test, we provide heat to both reference(100% stable) and sample crucible. The sample crucible starts to melt and the mass starts to reduce which is detected by the sensor. The purge gas and protective gas used is inert Nitrogen gas.

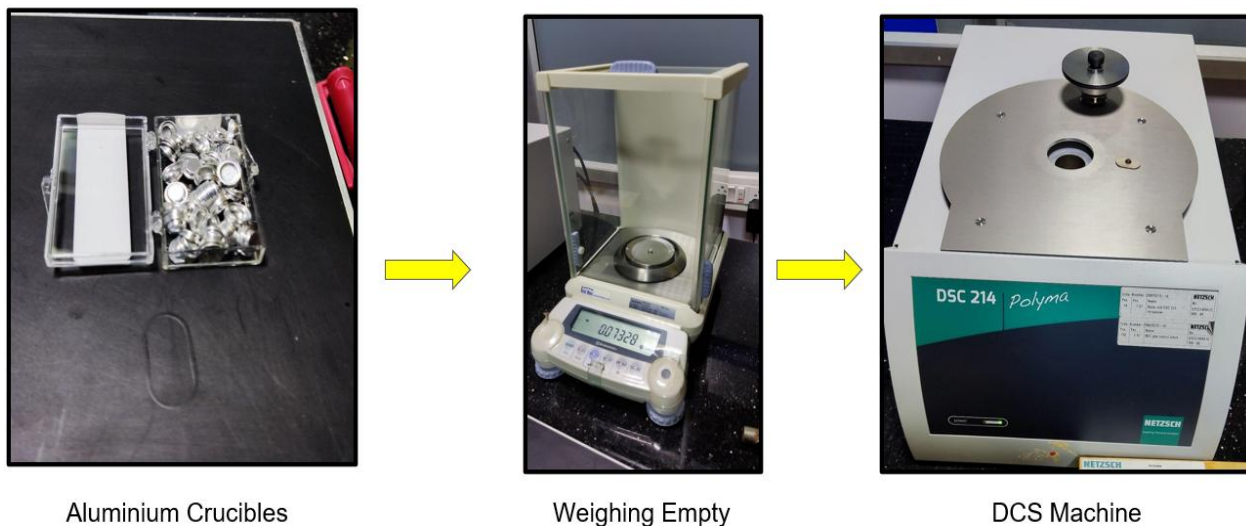


Fig 3.9 - DSC machine

### 3.8 Solidification Experiment

The schematic diagram of the solidification experiment is shown below

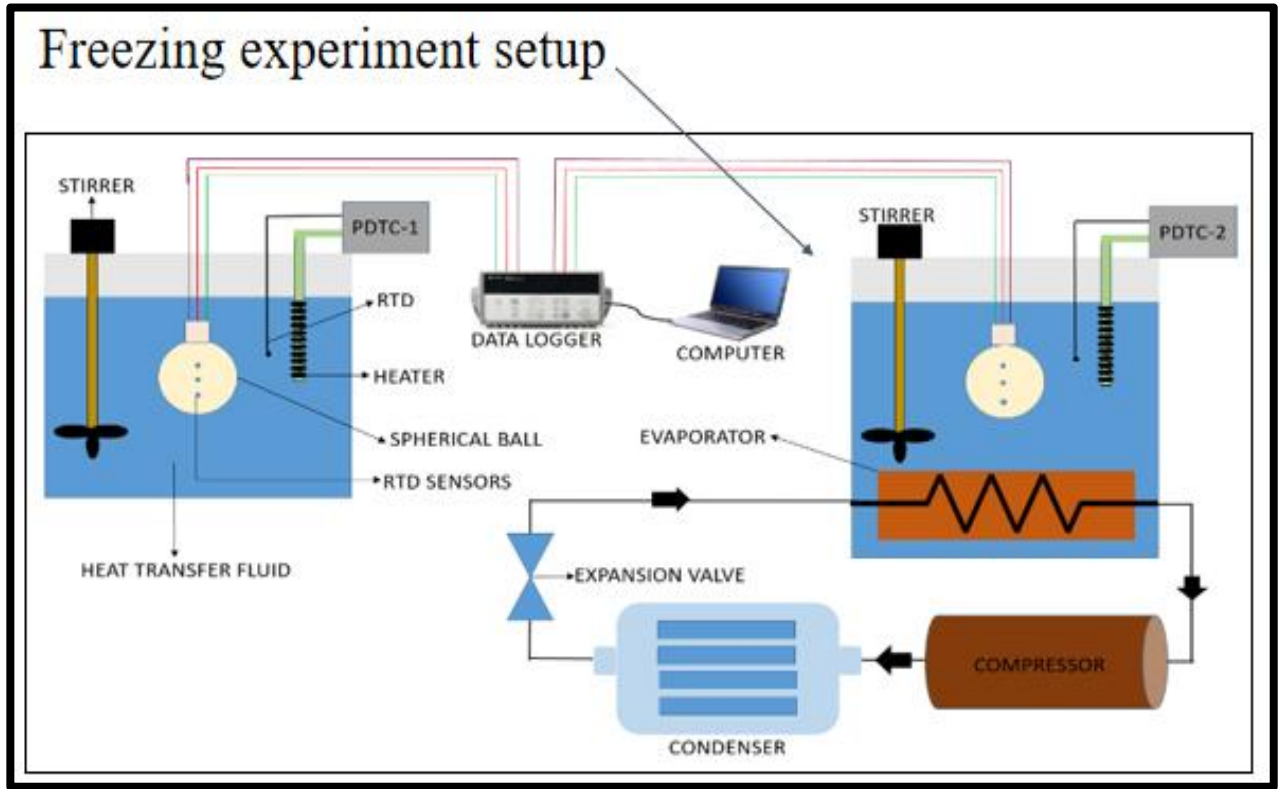


Fig 3.10 - Solidification Experiment Schematic Diagram

The freezing experiment consists of :

1. Refrigeration unit - compressor, condenser, expansion valve and evaporator. The refrigeration unit is used to keep the temperature of the bath tank at a constant -9 degree Celsius.
2. Stirrer - to keep the temperature of the bath tank near to uniform throughout the bath.
3. PDTC - Proportionate Differential Temperature Controller. This device is used to achieve the desired temperature of the HTF. It regulates the output of the cooling coil/evaporator based on the temperature of the surrounding HTF.
4. LDPE balls - Low density polyethylene balls were used as a container for NEPCM samples.

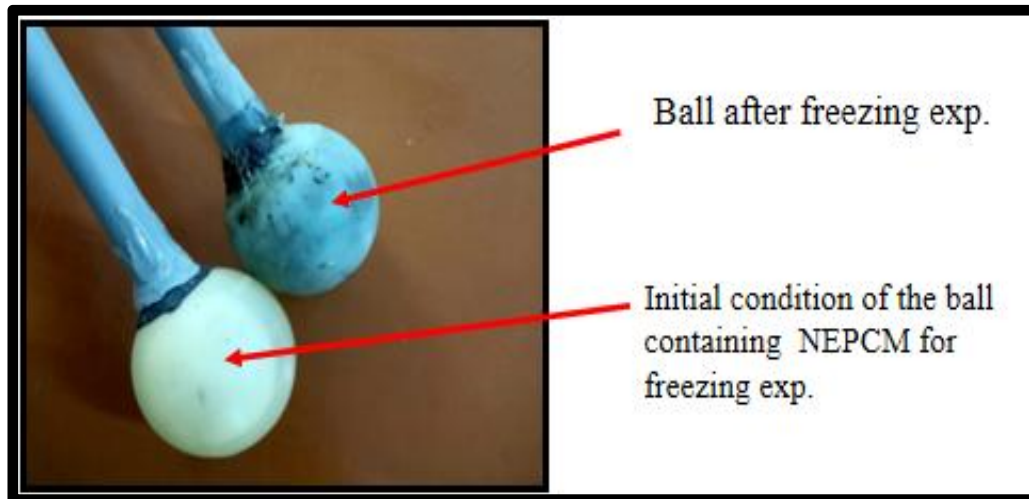


Fig 3.11 - LDPE Balls

The balls were made spherical because spheres have the minimum surface area to volume ratio which ensures that the NEPCM stored inside the balls do not freeze prior to the intended timing.

The outer diameter of the ldpe ball is 70 mm and Inner diameter is 60 mm

The total volume available is 113.09 cm cube. However, only 95 percent of the total available volume, which is 107.44 cm cube, is occupied by the NEPCM to prevent the ldpe balls from bursting due to increase in volume of water based NEPCM.



Fig 3.12 - Freezing Experiment Setup

The heat transfer fluid(HTF) is made of ethylene glycol and water in the ratio 60:40. Ethylene glycol is added to prevent the HTF from freezing at -9 degree celsius(It would freeze if the HTF was entirely made of water since water has freezing temperature of 0 degree celsius).

The RTD was placed at the center of the spherical ball to measure the variations in temperature during the solidification process.Using a data logger,Agilent 34970A, the NFPCM's transient temperature variation was continuously observed and measured every 10 seconds until thermal equilibrium was achieved for the HTF and NEPCM temperature.

<b>Refrigerant</b>	R134a
<b>HTF</b>	Aqueous ethylene glycol
<b>Temperature controller range</b>	0-20 degree C
<b>Bath tank capacity</b>	1kWh
<b>Bath section material</b>	Acrylic
<b>Design temperature</b>	-23.3 degree C
<b>Data acquisition and control system</b>	16 channel AI+ 8000 DACS

Table 3.8 - Specifications of Refrigeration Unit

### 3.8 SOLIDIFICATION CURVE FOR DIFFERENT SAMPLES

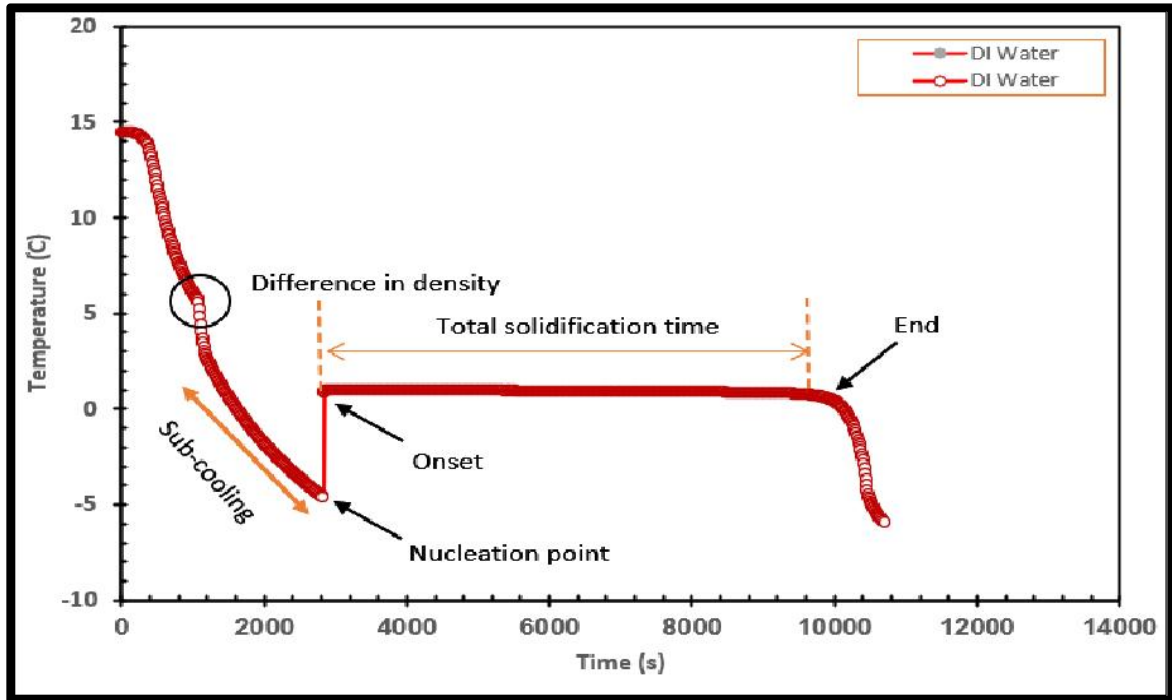


Figure 3.13 - Freezing Curve

At around 5 degree Celsius, due to difference in density of water and ice or because of the anomalous behavior of water, the cooling rate drastically increases and enters the subcooling region. Subcooling is the phenomenon of reduction in temperature below the solidification temperature to achieve the desired nucleating sites in order to kick start the solidification process. It is a major drawback in case of water based NEPCMs because it would require us to maintain even a lower temperature of the HTF surrounding the ldp ball containing the NEPCM, which in turn lowers the overall efficiency of the CTES system. After the subcooling, once the NEPCM reaches enough nucleation sites, the temperature then starts to rise to the solidification temperature.

The Onset denotes the starting of the solidification process and since the solidification

happens isothermally, the solidification process is shown by a horizontal line.

The solidification curves for different NEPCMs are shown below.

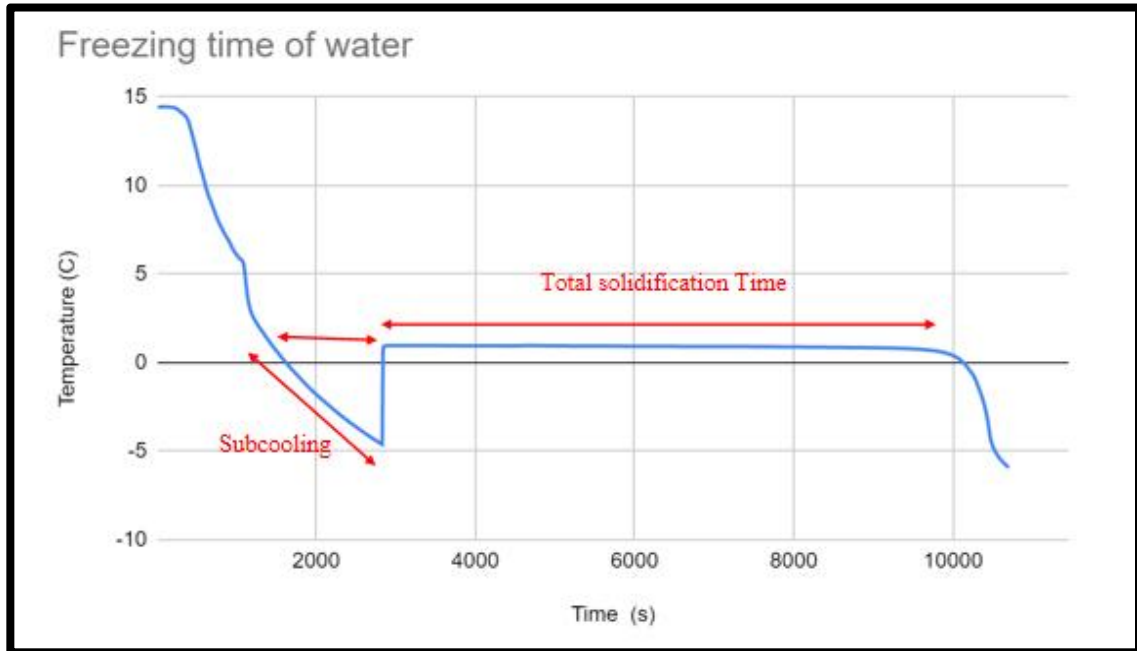


Fig 3.14 - Freezing Time of Water

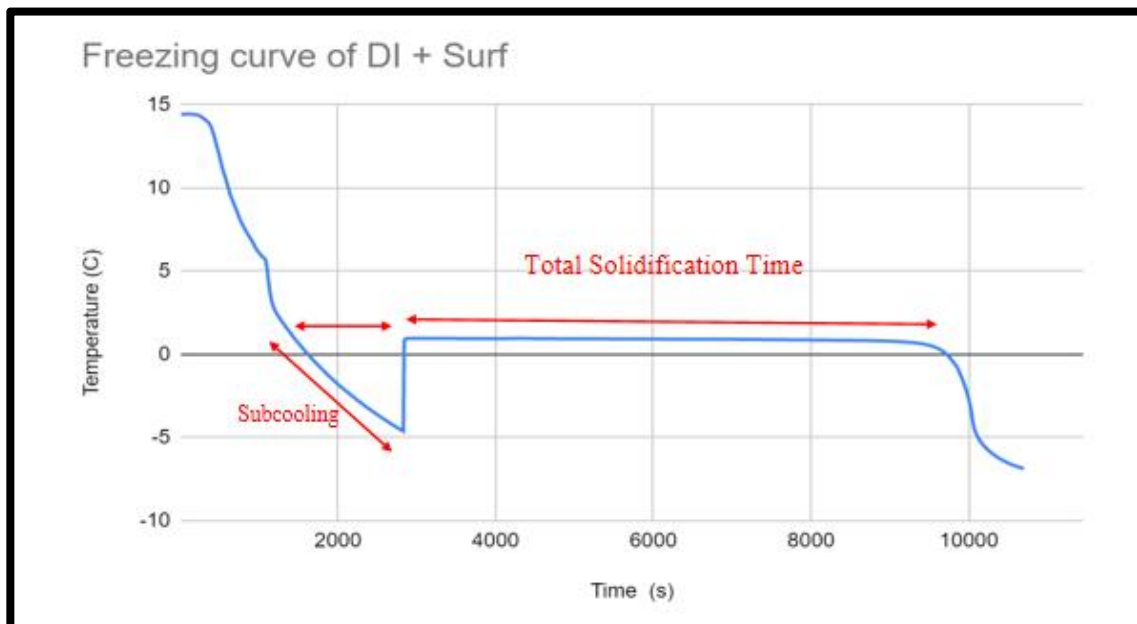


Fig 3.15 - Freezing Curve of DI + Surf



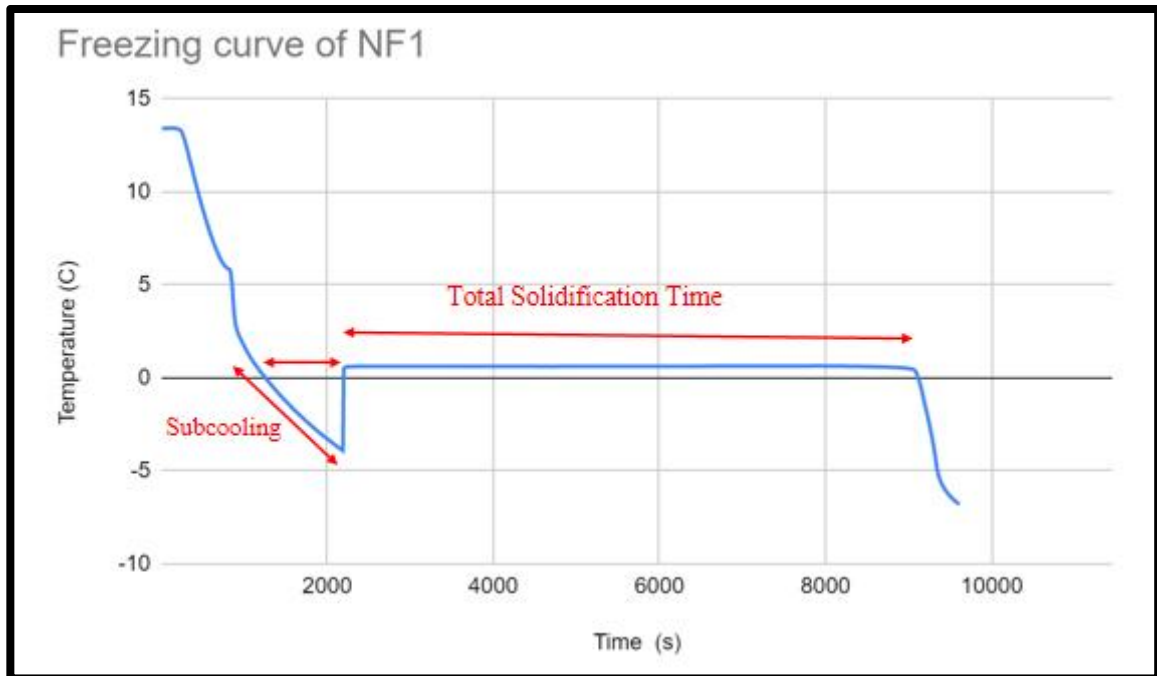


Fig 3.16 - Freezing curve of NF1

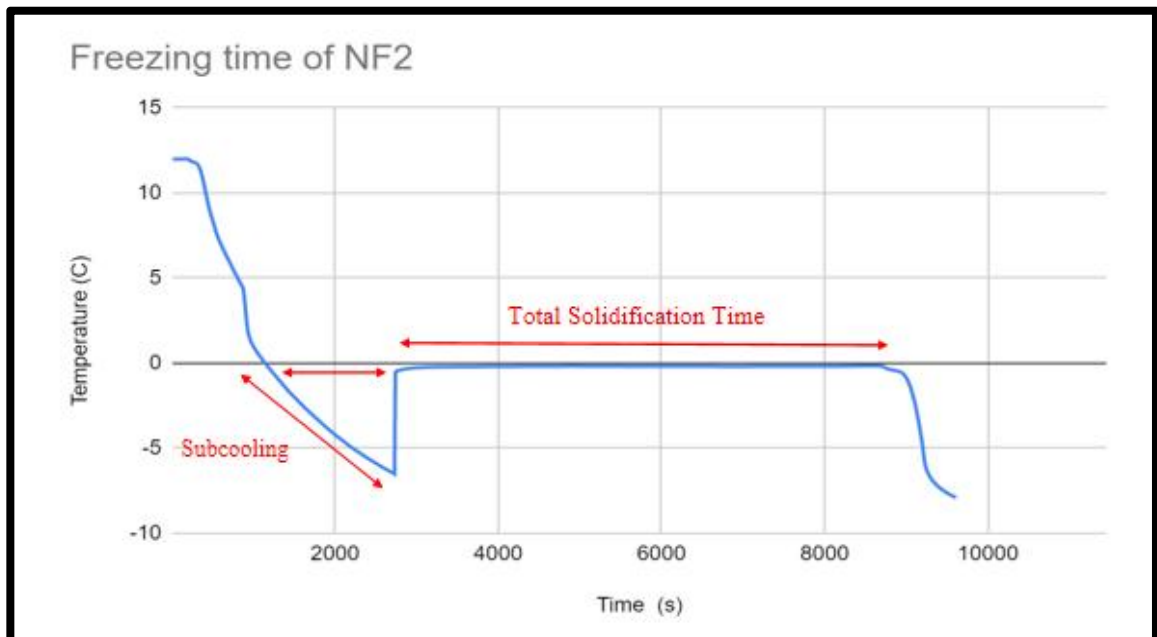


Fig 3.17 - Freezing Time of NF2



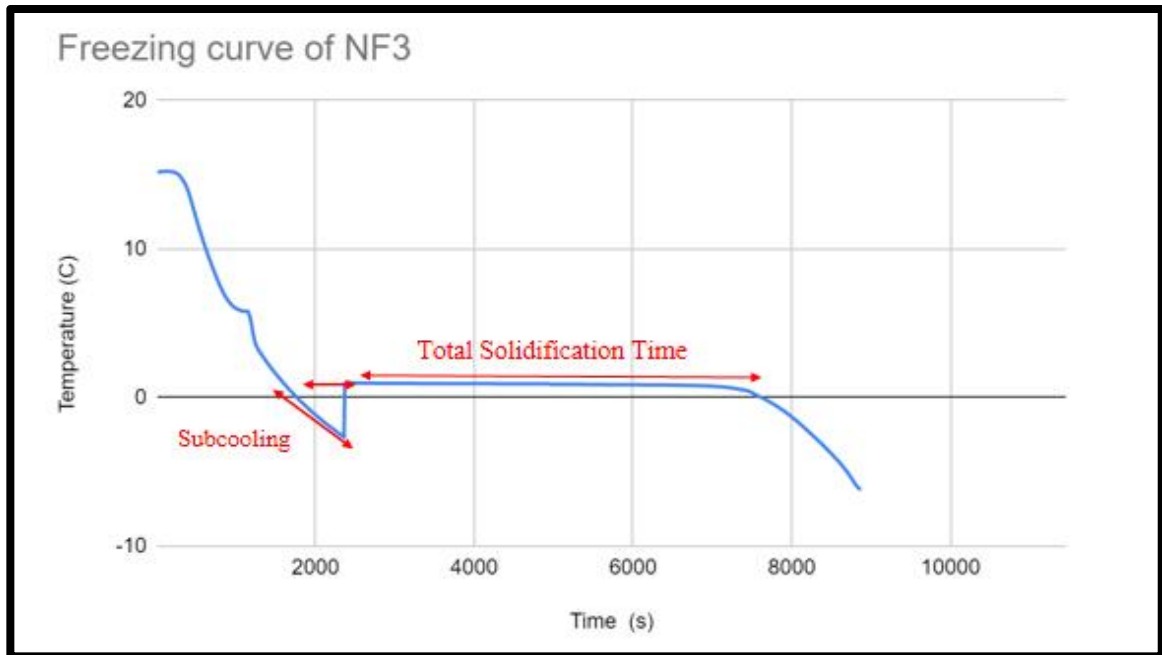


Fig 3.18 - Freezing Curve of NF3

<b>SAMPLE</b>	<b>TOTAL SOLIDIFICATION TIME (s)</b>	<b>SUBCOOLING TIME (s)</b>	<b>SUBCOOLING TEMPERATURE RANGE(°C)</b>
DI WATER	7200	1300	5.5
DI water+SDS	6900	1300	5.5
NF1 - 0.2% GNP	6800	1000	4.5
NF2 - 0.4% GNP	6000	1350	6
NF3 - 0.6% GNP	5000	750	3.5

Table 3.9 - Tabulation of Solidification Experiment

## CHAPTER 4

### RESULTS AND DISCUSSIONS

#### 4.1 Increment in Density of NEPCM samples with increasing GNP concentration

Because the concentration of GNP in the base PCM DI water is raised, the overall density of the NEPCM increases, which can lead to particle agglomeration and a non-uniform and unstable solution. However, because the greatest percentage difference of density from the base PCM DI water is less than 2%, there are extremely few chances of particle agglomeration.

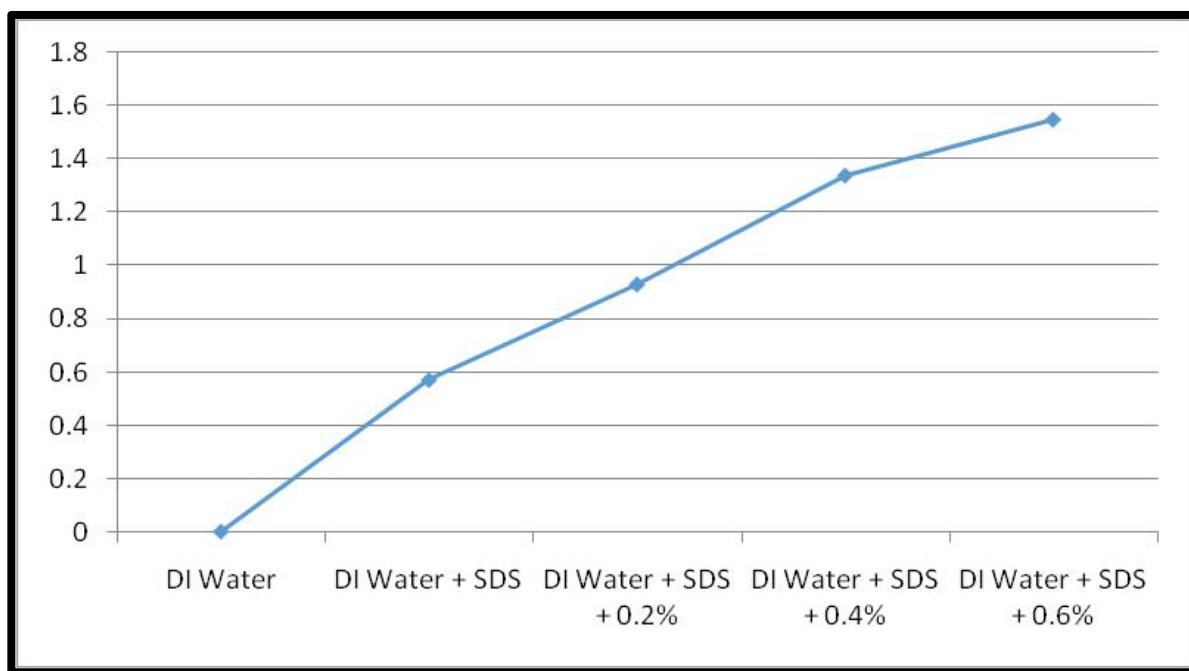


Fig 4.1: Density Deviation for different samples

## 4.2 Stability of the prepared samples

### a. ZETA POTENTIAL Analysis

According to the general-reference values (-30 mV or +30 mV), the Zeta Potential distribution curve (particles count) for the highest concentration of NEPCM(-44 mV) is in a satisfactory stability range. Low values (less than 30 mV) have been found to cause agglomeration.

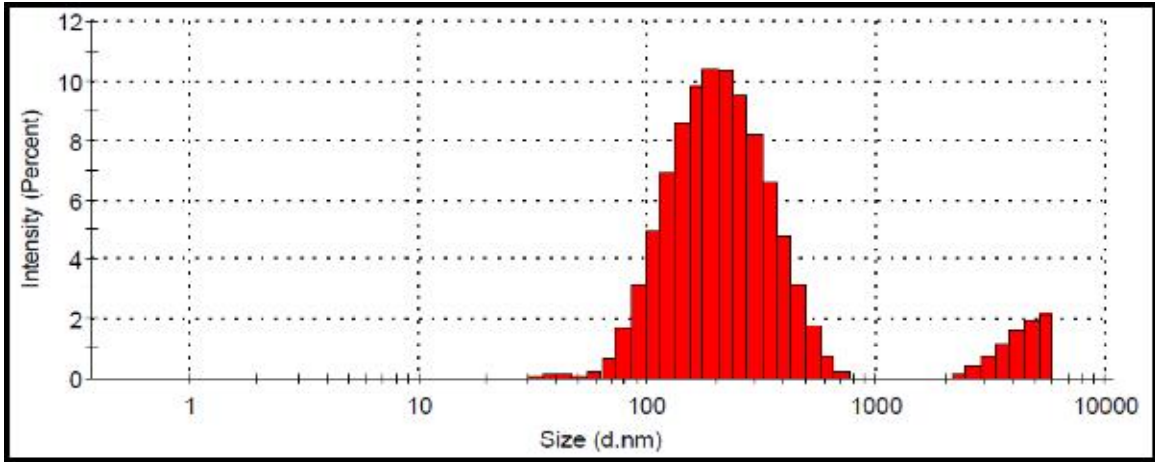


Fig 4.2: Zeta Potential curve for DI Water

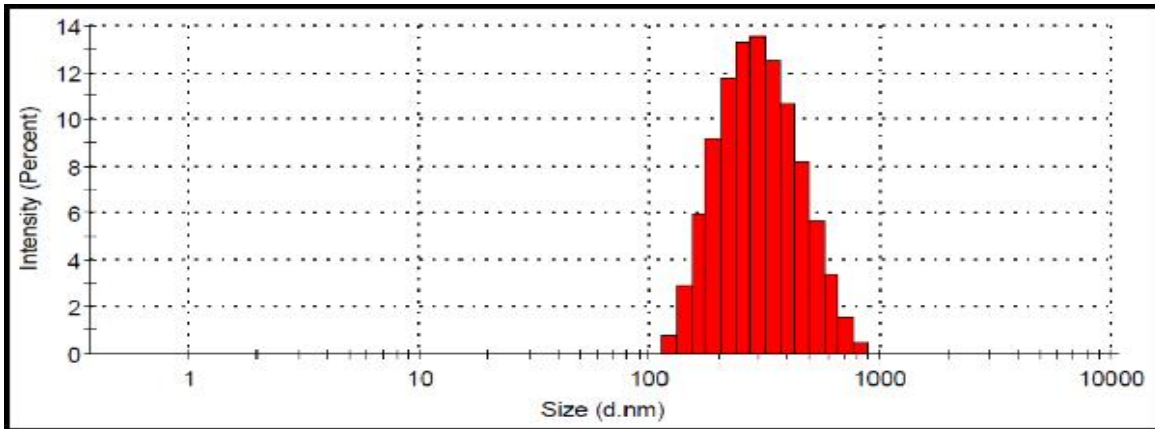


Fig 4.3: Zeta Potential curve for NF3

Nanoparticles having zeta potential values more than 30 mV or less than - 30 mV are usually very stable. Zeta potential distribution curve for DI water shows that there are some particles which forms a cluster above 1 micrometer size, which is completely eliminated in the case of NF3 having 0.6 % of GNP concentration. The size of the particles does not exceed 1 micrometer which denotes that the solution is well dispersed and shows no agglomeration of particles.

### 4.3 DSC TEST

Based on the heat flow rate, the curve gets gradual or sharp. High heat flow rate will have a sharper curve which would lead to a higher peak temperature while the phase change width would reduce. If the heat flow rate is low, the curve will be gradual and it will attain a low peak temperature resulting in larger phase change width. The heat absorption(endothermic) process is the melting process and the heat rejection(exothermic) process is the freezing process. The maximum heat flow rate is achieved at peak temperature. The onset temp of DI water is beyond 0° C because of the fact that it has some driving potential present to start the phase change process. There is more deflection(from the base PCM) in Melting point as we increase the GNP concentration because of High flow rate coupled with higher thermal conductivity value.

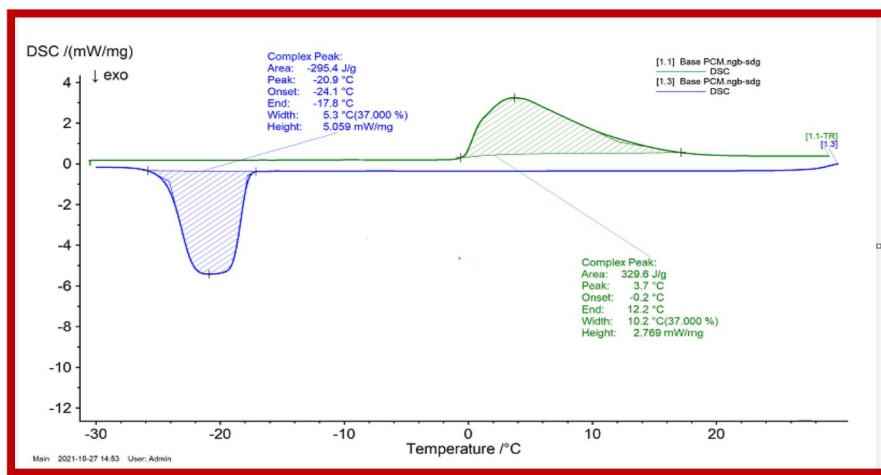


Fig 4.4: DSC Curve for DI Water

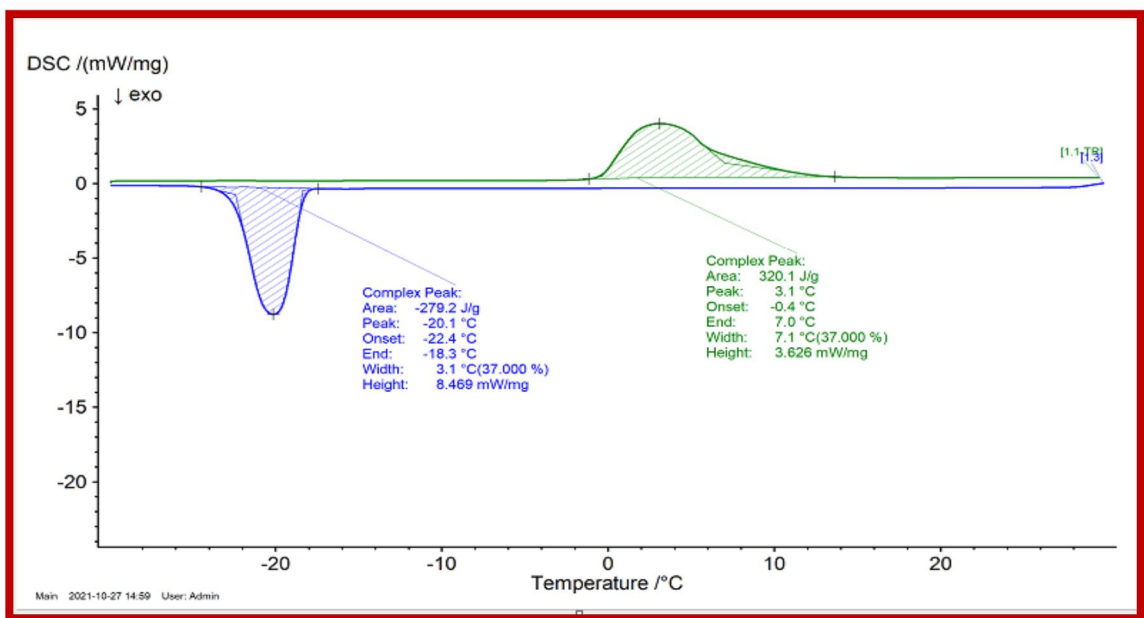


Fig 4.5: DSC Analysis of 0.2% GNP Concentration

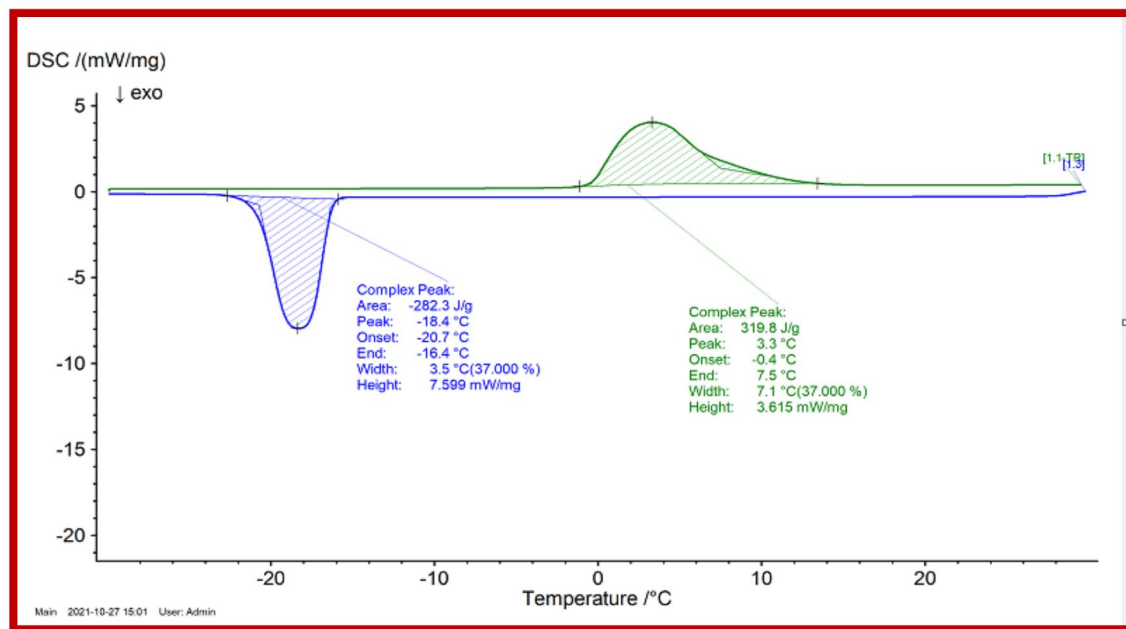


Fig 4.6: DSC Analysis of 0.4% GNP Concentration

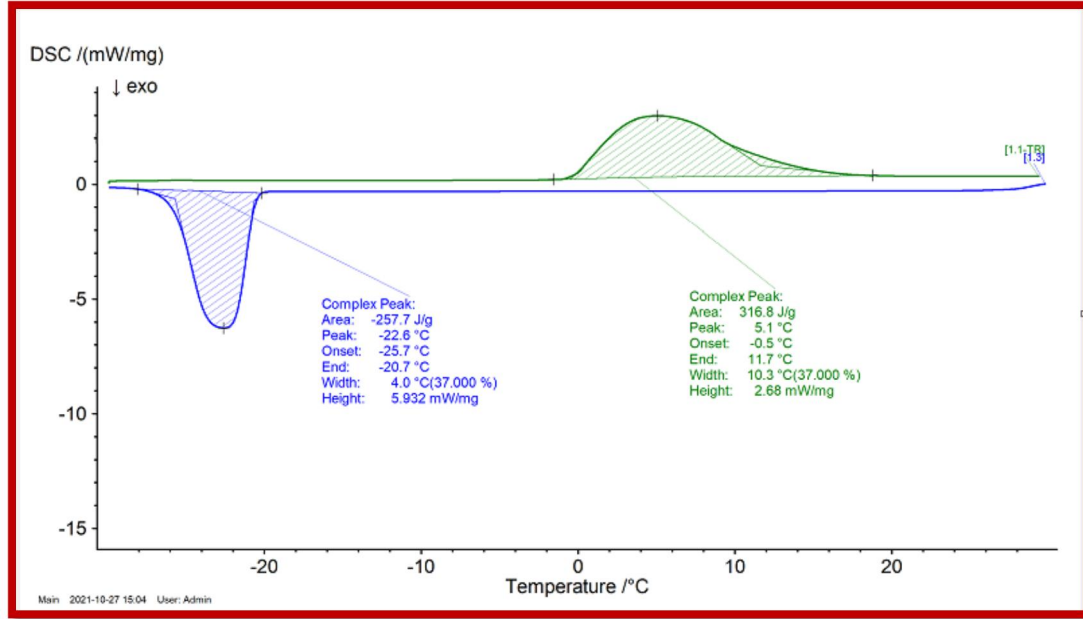


Fig 4.7: DSC Analysis of 0.6% GNP Concentration

For 0.6% concentration of NFPCM, we can see that freezing started at -20.7°C and ends at -25.7 °C with the peak occurring at -22.6 °C. Also, the rate of heat rejection and rate of heat addition increased significantly.

This demonstrates that the heat absorption area has increased, which is a result of the addition of nano-sized, highly conductive solid particles to DI water to improve its thermal transport capabilities.

#### 4.4 Reduction in solidification time

Solidification time is nothing but the charging time of the NEPCM. Greater the reduction in solidification time better the overall efficiency of the CTES system to which the NEPCM is coupled with.

$$\text{Reduction (\%)} = ((ts)_{\text{max}} - (ts)_{\text{min}}) / (ts)_{\text{min}} * 100$$

$$= (ts \text{ of DI water} - ts \text{ of NF3}) / ts \text{ of DI water} * 100$$

$$= (7200 - 5000) / 7200 = \mathbf{30.56\%}$$


Where  $t_s$  - total solidification time

Both the DI water and NFPCMs followed the same temperature drop trend with respect to time. This could be due to increase in the viscosity of the NFPCMs with addition of GNPs that suppresses the natural convection effects.

#### 4.5 Enhancement in cooling rate

The cooling rate of NFPCM increased in proportion with the concentration of GNP. This is due to enhanced thermal conductivity of NEPCM, which in turn augments the heat transfer rate during the nucleation process.

Mathematically,

$$CR = \left( \frac{\Delta T}{t} \right)_{sub} \quad CR = \left( \frac{\Delta T}{t} \right)_{sub} \quad \Delta T_{sub}$$


$$\text{Max enhancement} = (0.28 - 0.253) / 0.253 * 100 (\%) = \mathbf{10.67\%}$$

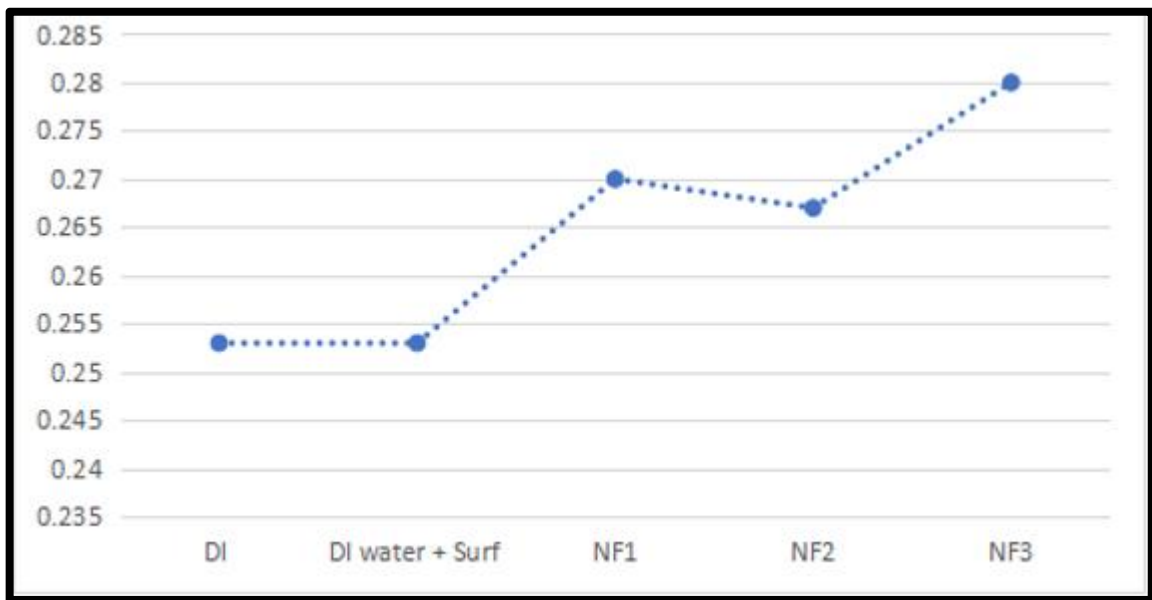


Fig 4.8 - Cooling rate for different Samples

As can be seen from the above findings, adding GNP enhances the cooling rate during subcooling for a given temperature driving potential, which is extremely beneficial for the CTES system's performance.

Further, the Subcooling time is reduced to 42.3% and subcooling range is reduced to 36.36% for NF3 with respect to the base PCM DI water due to improvement in thermophysical properties of the NEPCMs prepared.



## **CHAPTER 5**

### **CONCLUSIONS**

The thermal conductivity maximal of 11.46% was observed for 25 degree celsius surrounding temperature for NEPCM having 0.6% by wt of Graphene NP, because of its higher K value and higher specific area.

The exposure to higher conductivity GNP worked as a nucleating substance, causing the NFPCM to solidify ahead of the base PCM due to a significant drop in level of sub cooling phenomenon. The augmentation of Graphene NP also causes the total charging time to reduce by a considerable amount.

The NFPCM cooled the subcooled area more efficiently than the base PCM at the given driving potential, enabling the CTES system to run at a greater HTF temperature.

Nanotechnology breakthroughs will be critical in designing and developing an efficient Cold thermal energy storage system appropriate for a variety of industrial and architectural cooling applications.

#### **5.1 Scope for Future work**

##### **Applications:**

It is crucial in special applications like as air conditioning, refrigerated storage for space cooling, and adsorption storage for sub-zero temperatures. The thermal transport capabilities of NEPCMs contribute significantly to the improvement of the cool thermal system's specific energy storage capacity for sustainable building cooling applications.

S.No	Name	Total Solidification Time (s)	Subcooling Range (C)	Subcooling Time (s)	Cooling Rate (C/min)
1.	DI	7200	$1 + 4.5 = 5.5$	$2800 - 1500 = 1300$	0.253
2.	DI water + Surf	6900	$1 + 4.5 = 5.5$	$2800 - 1500 = 1300$	0.253
3.	NF1	6800	$0.5 + 4 = 4.5$	$2200 - 1200 = 1000$	0.27
4.	NF2	6000	$6.5 - 0.5 = 6$	$2700 - 1300 = 1350$	0.267
5.	NF3	5000	$1 + 2.5 = 3.5$	$2350 - 1600 = 750$	0.28
	Max Improvement	<b>-30.56%</b>	<b>-36.36%</b>	<b>-42.30%</b>	<b>+10.67%</b>

Table 5.1 - Tabulation of different parameters

In table 5.1 -ve sign indicates reduction as compared to the base PCM and +ve sign represents increment from the base PCM.

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