The Effect of Nucleation on DI-Water Based Phase Change Material for Cool Thermal Energy Storage

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Abstract. This report addresses the study on nucleating effects of DI–water based phase change material by adding salt hydrate like a Sodium chloride, Magnesium chloride and Calcium chloride. DI-water is selected as base PCM in which nucleating agents are added in concentration of 0.3, 0.6 and 0.9 Wt.% to study the solidification characteristics of the PCM. The samples are prepared with the help of magnetic stirring and ultra-sonification process which ensures that the nucleating agents are completely dissolved in the base PCM. An experimental trial is carried out with surrounding heat transfer fluid temperature of -7°C and the solidification history is generated for all PCM samples. From the observation it can be concluded that NaCl 0.9 Wt.%, MgCl₂: 0.6 Wt.% and CaCl₂: 0.6 Wt.% shows a significant reduction of supercooling degree. Only NaCl of 0.9 Wt.% shows a reduction of 91.32% in supercooling degree with least increase of 6.73% in solidification time. Thus, Phase change materials proves to be effective in eliminating the discrepancy between demand and supply during peak hour by storing energy during non-peak hours and retrieving when demanded. Thus, it can be used to reduce power consumption during peak hour’s demand and reduce the effects on environment.

1. Introduction

The energy demand has been increased during the last few years. Due to the limited sources of non-renewable energy, it is in great demand to find out other sources of energy using renewable energy sources. Global warming is one of the major issues which can only be resolved by switching to renewable mode of energy. But there exists a complication in integrating it with electrical grid and more energy consumption during peak hours which can’t be covered by renewable mode of energy. This leads to temporal mismatch between energy consumption and renewable energy availability. This discrepancy between energy demand and supply can only eliminated by storing energy during non-peak hours and retrieving it during peak hours demand [1]. The PCM can be used to eliminate this discrepancy between demand and supply by storing energy in the latent heat region of the PCM during non-peak hours and retrieving it during peak hours. It has large energy storage density due to its high latent heat of fusion. But it has leakage problem, low thermal conductivity and subcooling problem[2]. Inorganic PCM shows high latent heat storage, good thermal conductivity and low cost over organic PCM.[3]. Use of phase change materials for cooling applications provide many diverse applications such as protection of temperature sensitive products like food, beverages, pharmaceutical products, blood derivatives, electronic devices, bio medical products, air-conditioning, and many more. Eutectic salt-water solutions are PCM which generally melt below 0 ºC, usually have good storage density, and are available at low cost. All these factors attract the interest of their use for the cooling applications and for cold storage
systems. On the other hand, this PCM could produce phase separation and subcooling, problems that should be addressed [4]. Viscosity has influence over enthalpy, storage capacity, supercooling and phase stability. Nucleating agents proved to be effective in reduction and elimination of degree of supercooling [5]. Salt hydrates shows good results in reduction in supercooling phenomenon and thickening agents are added to avoid phase separation during freezing cycle [4]. By adding calcium chloride, heat transfer property of the base fluid can be increased and it doesn’t form any particles during cooling process [6]. Solidification can be triggered by adding nucleating agents to the base fluid and over 90% reduction in degree of supercooling can be achieved by conserving the high latent heat property of the material. The nucleation time can be controlled by using ultra-sonification or electrical and mechanical nucleation techniques [1]. Ultra-sonification should be carried out in order to disperse the nanoparticles completely in base fluid to get better results or-else it might affect the degree of supercooling [3]. From the literature survey, nucleating agents shows better reduction in supercooling degree when added to the base PCM. This paper studies the effect of nucleating agents like NaCl, MgCl₂ and CaCl₂ when its added to the DI-water and study their solidification behavior.

2. Methods and Materials

2.1 Materials

DI water is used as base fluid during charging and discharging cycle. The Nucleating agent is purchased from Sisco chemicals private limited. The properties of DI water are listed in the Table 1 and the concentration of nucleating agents are 0.3, 0.6 and 0.9 Wt.% of individual components of NaCl, MgCl₂ and CaCl₂ respectively.

| Properties                              | DI water        |
|-----------------------------------------|-----------------|
| Liquid thermal conductivity at 20°C     | 0.598 W m⁻¹K⁻¹ |
| Solid density                           | 920 kg m⁻³      |
| Liquid density at 4°C                   | 1,000 kg m⁻³    |
| Melting/freezing temperature            | 0°C             |
| Latent heat of melting                  | 334 kJ kg⁻¹     |
| Specific heat water                     | 4.187 kJ kg⁻¹K⁻¹|
| Specific heat ice                       | 2.108 kJ kg⁻¹K⁻¹|

2.2 PCM Solution Preparation

The DI water is added with nucleating agents like NaCl, MgCl₂ and CaCl₂ to study its solidification behavior. It undergoes Magnetic stirring for 20 minutes and Ultra-sonification for 20 minutes using bath sonificator. PCM solution preparation method is shown in Figure 1.
DI-water is taken in 200 ml beaker and nucleating agents are added in the corresponding concentration. Then magnetic stirring and ultra-sonification is carried out for 20 minutes individually in-order to completely dissolve the nucleating agents added to base PCM. If not it will affect the degree of supercooling and the properties [3].

2.3 Experimental setup

The main components of experimental setup are explained below. This setup is suitable for altering the various bath temperatures to give a better analysis of the solidification and its properties. The experimental setup consists of Vapour compression and refrigeration system, Heat Transfer fluid, Stirrer, Compressor, Evaporator, Condenser, Expansion Valve, RTD (Two wire), Spherical capsule, PTDC and Agilent 34970A Data Logger. The schematic representation of the experimental setup is shown in Figure 2. The data logger is used measure the temperature change for every 10 seconds interval and spherically encapsulated capsules are placed in the VCR evaporation tank after attaining the steady state temperature. The tank contains 60:40 volume ratio of water and ethylene glycol. The constant temperature is maintained with the help of VCR unit and PDTC switches the heater on and off if the temperature deviates from the specified bath temperature. The spherical encapsulation is made of low density polyethylene material with 73 mm diameter and contains PCM till 90% volume capacity [7]. The temperature change is measured with the help of 2 wire RTDs which are placed radially.

![Figure 2. Schematic diagram of Experimental Setup](image)

At first the testing of the experimental setup is carried for calibration purpose in-order to place the encapsulated PCM in the setup. The desired surrounding bath temperature is set at -7°C for all samples. Then temperature indicator is constantly checked because at the starting of the experiment the temperature oscillates from the specified bath temperature and after attaining steady state temperature the spherically encapsulated PCM is kept in the VCR evaporation tank. Each sample is repeated three times in-order to get accurate results.
Inside the spherical encapsulation there are three thermocouples such as RTD 1 (At Centre), RTD 2 (At 22.0 mm from the Centre) and RTD 3 (At 27.8 mm from Centre) are placed firmly as shown in Figure 3. The neck of the spherical ball is sealed using Mseal to avoid the leakage of the phase change material. Then it is connected to the data logger which in-turn connected to the computer to measure temperature change and to generate solidification curves. Low density polyethylene is used for spherical encapsulation of phase change material because of its good chemical resistance, electrical resistance and good insulation property.

![Figure 3. Spherical Encapsulation of PCM](image)

3. Results and Discussion

From the figure 4, the curve initially has room temperature 31°C. As the surrounding bath temperature is kept at -7°C, the PCM starts to solidify as temperature drops following sensible region. At 4°C, the liquid density gets higher and follows constant temperature over a period of time and density decreases as temperature decreases. It’s a unique property of the water. Then the water is cooled below 0°C that leads to the formation of subcooling region up to -4.771°C due to lack of nucleation sites. After attaining maximum supercooling temperature, the temperature rises and follows a constant path called latent heat energy storage region where thermal arrest takes place. The point at which it follows a constant path is called onset of solidification or latent heat region where the subcooling region ends. It follows a constant path for a period of time and drops down gradually to sensible region. The point at which the curve drops is called offset of solidification or latent heat region. Solidification time is the time between onset and offset of the latent heat region or phase change region. From the figure 4, it takes 104 minutes to solidify completely at 100% volume of DI-water. DI-water at surface or circumference of the spherical ball (At 50% volume) freezes faster than at Centre (At 100% volume) and radial location (At 75% volume). Thus, at Centre solidification time is more when compared to other radial locations. The formed subcooling region should be reduced in-order to reduce power consumption. Therefore, it stores more energy in latent heat zone (Phase liquid – solid transition zone).
3.1 Study on DI water at -7°C bath temperature

![Figure 4. T-t history of DI-water at -7°C](image_url)

3.2 Comparison of DI-water with nucleating agents at -7°C

3.2.1 T-t history of NaCl at -7°C

Adding NaCl in DI-water, 0.3 Wt.% shows a reduction of 31.02% in supercooling degree with 9.62% increase in solidification time. 0.6 Wt.% shows an increase of 14.46% in supercooling degree with 8.65% increase in solidification time. Whereas 0.9 Wt.% shows significant reduction of 91.32% in supercooling degree with least increase of 6.73% in solidification time.

\[
\text{NaCl} + \text{H}_2\text{O} \rightarrow \text{NaOH} + \text{HCl}
\]  

(1)

Comparing all three concentration of NaCl at -7°C, 0.9 Wt.% shows significant reduction of 91.32% in supercooling degree with maximum supercooling temperature of -0.414°C because it triggers the nucleation cites that tends to reduce the subcooling region.

3.2.2 T-t history of MgCl₂ at -7°C

Adding MgCl₂ in DI-water, 0.3 Wt.% shows an increase of 27.06% in supercooling degree with 17.31% increase in solidification time. 0.9 Wt.% shows reduction of 21% in supercooling degree with 12.5% increase in solidification time. Whereas 0.6 Wt.% shows significant reduction of 97% in supercooling degree with 20.19% increase in solidification time.

\[
\text{MgCl}_2 + 2\text{H}_2\text{O} \rightarrow \text{Mg(OH)}_2 + 2\text{HCl}
\]  

(2)

Comparing all three concentration of MgCl₂ at -7°C, 0.6 Wt.% shows significant reduction of 97.09% in supercooling degree with maximum supercooling degree of -0.139°C.

3.2.3 T-t history of CaCl₂ at -7°C

Adding CaCl₂ in DI-water, 0.3 and 0.9 Wt.% shows 12.5% increase in solidification time. But, in-case of supercooling degree former shows a reduction of 43.58% whereas the latter shows an increase of
0.92%. Among these 0.6 Wt.%, shows a reduction of 93.21% in supercooling degree and 14.42% increase in solidification time.

\[
\text{CaCl}_2 + 2\text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2 + 2\text{HCl}
\]  \( (3) \)

**Figure 5.** T-t history of NaCl at -7°C

**Figure 6.** T-t history of MgCl₂ at -7°C

Comparing all three concentration of CaCl₂ at -7°C, 0.6 Wt.% shows maximum supercooling degree of -0.324°C and significant reduction of 93.21% in supercooling degree.
3.3 Comparison on reduction of supercooling degree

On comparing the reduction effect on supercooling degree by all concentrations of nucleating agents, NaCl 0.9 Wt.%, MgCl₂ 0.6 Wt.% and CaCl₂ 0.6 Wt.% shows significant effect. All the above-mentioned concentration shows an average reduction of around 93.87% in supercooling degree. But there is an increase in solidification time, in-order to provide this reduction in supercooling degree. MgCl₂ 0.6
Wt.% shows highest reduction of 97.09% in supercooling degree with 20.19% increase in solidification time when compared to other two concentrations. Only NaCl 0.9 Wt.% shows 91.32% reduction in supercooling degree with least increase of 6.73% in solidification time. Only NaCl 0.9 Wt.% shows highest reduction of 97.09% in supercooling degree with 20.19% increase in solidification time when compared to other two concentrations. Only NaCl 0.9 Wt.% shows 91.32% reduction in supercooling degree with least increase of 6.73% in solidification time.

4. Conclusion

From the above experimentation on solidification characteristics of DI-water added with nucleating agents the following conclusions were made. NaCl 0.9 Wt.% shows significant reduction of 91.32% in supercooling degree with least increase of 6.73% in solidification time. Thus, NaCl triggers nucleation sites owing to reduce supercooling degree. MgCl₂ 0.6 Wt.% shows highest reduction of 97.09% in supercooling degree with 20.19% increase in solidification time. CaCl₂ 0.6 Wt.% shows a reduction of 93.21% in supercooling degree with 14.42% increase in solidification time.

NaCl 0.9 Wt.% shows significant effect in reduction of supercooling degree with least increase in solidification time when compared to MgCl₂ 0.6 Wt.% and CaCl₂ 0.6 Wt.. The addition of nucleating agents can reduce and even eliminate the major subcooling problem that exists in water. But, negligible presence of the subcooling region are observed. This promotes that phase change materials can be used as an energy storage medium during non-peak hours. This reduces power consumption during peak hour’s demand and the effects on environment.

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