# Paper_5_JI_INSuamir_2018

*by* I Nyoman Suamir

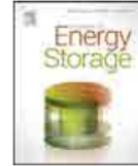# The role of vegetable oil in water based phase change materials for medium temperature refrigeration

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## ARTICLE INFO

## ABSTRACT

This research aims to investigate development of water based phase change materials (PCM) for cold thermal energy storage (CTES). Mixtures of water with small amount vegetable oil addition were chosen as candidates of the PCM that were considered to be suitable for medium temperature refrigeration application with temperature range of products between −1 °C and +5 °C. The PCM candidates were tested experimentally through DSC and T-history method. The results showed that esters of vegetable oils played very important role on the solubility of the vegetable oil in water. The esters made the investigated vegetable oil (soya oil or corn oil) mix well in water solutions and they worked as nucleating agents that could lower freezing point and reduce super-cooling of the water. It was found that addition of vegetable oils by 5% to 10% in water solution could decrease the freezing temperature from 0 °C down to respectively −3.5 °C to −6.5 °C and could also minimize degree of super-cooling of the PCM candidates.

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## 1. Introduction

Thermal energy storage is considered as one of the most perspective technologies for increasing the efficiency of energy conversion processes and effective utilization of available sources of heat [1,2]. In the last few years, much attention has been paid to the latent heat thermal energy storage systems (LHTESS) due to their various applications [3–6]. The technology is also effective to reduce gap of unbalances between energy supply and demand [7–10]. Among various applications of thermal energy storage, heat or cold accumulation of temperature ranging from −50 °C to 120 °C has a greater market potential that can be carried out using wide range of phase change materials (PCMs) [11]. Application of PCMs as latent heat thermal energy storage (known as LHTES technique) can provide larger capacity of energy storage per unit mass and transfer heat at relatively constant temperature [12–16]. The technique, therefore, can use a smaller size system with narrow temperature range during phase change process compared with sensible heat storage (SHS) [17–19].

For LHTES applications, many materials have been investigated. They included organic, inorganic and mixture of both organic and inorganic materials [20]. Materials with high thermal capacity and

constant range of phase change temperature are considered as good PCMs [21]. PCM made of organic solid-liquid has attracted much attention to various applications for their excellence properties [22–25]. Organic PCMs such as paraffin and fatty acid are reported in [26,27]. Paraffin is the most popular organic PCM, which has very small degree of super-cooling and wide range of phase change temperature. Paraffin is also chemically stable during phase change process [28]. However, paraffin waxes have disadvantages due to their low thermal conductivities, low latent heat, flammability and high change in volume [29].

Fatty acid ($CH_3$ $(CH_2)_{2n}$—COOH) has advantages compared to paraffin. Fatty acid has more accurate melting point, high latent heat, congruent melting and solidification temperatures, small degree of super-cooling, low vapor pressure, little volume change during phase change process and low cost for energy storage. Paraffin is also non-toxic, non-corrosive to metals, thermally and chemically stable, and non-flammable [30–34]. However, fatty acid has unpleasant odor compared with paraffin [35]. In order to eliminate the unpleasant odor, some researchers recommended replacing them with their derivative fatty acid esters, which could be obtained via the esterification of fatty acids with alcohols. The effect of alcohols on the thermal properties of fatty acid esters was relatively excessive [36].

The main drawback of TES technology is how to develop effective PCMs for storing energy [37]. Phase change temperature and latent heat of fusion are the two basic parameters of PCMs [38].

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Therefore, the selection of PCMs with suitable phase change temperature is critical [39]. Other parameters include thermal stability and energy storage characteristics [40]. In addition, high degree of super-cooling could make evaporation temperature of refrigerator system lower and certainly reduce cooling efficiency [41,42,43].

Water is often employed as PCMs because of its reliability, stability, low cost, high specific heat, high density, and high latent heat capacity of 335 kJ/kg. Water also has good safety issue [44–46]. Furthermore, it has been implemented in many applications especially in air conditioning systems for shifting peak loads. Water, however, has a high degree of super-cooling [47,48]. Water cannot be applied as PCM for CTES of operating temperatures below 0 °C, due to its freezing and melting point at 0 °C [49,50]. One way to make water applicable as PCMs at below 0 °C is by adding nucleation agent to trigger heterogeneous nucleation and to eliminate its super-cooling [51].

A PCM for medium temperature refrigeration application should be able to maintain product temperatures between −1 °C and +5 °C. In this application, evaporating temperature of refrigeration system is usually lower than −8 °C [52]. PCMs utilized for this application, therefore, should be melting and freezing at temperature range between −6 °C and −4 °C with assumption that the PCM would be placed in the inlet-air after evaporator. Such PCMs could be organic based materials (paraffin), salt solutions, or water based materials [53]. For salt-based PCMs, their freezing and melting temperatures can be lowered down by increasing salt concentration of the PCM solution. However, salt solutions are corrosive and have lower latent heat than water [54,55,56]. Another way to squeeze down freezing and melting temperatures of water is by adding antifreeze liquid [57].

To date, there are few studies on utilization of organic PCMs made of water with nucleating agents of vegetable oils. Vegetable oil contains various types of fatty acids. Fatty acids and their fatty acid ester or eutectic mixtures also have many superior properties as organic PCM materials [58–61]. Fatty acid esters are new material for organic PCMs. Unfortunately very limited thermal data is available in literatures [62]. Fatty acids are derivatives of materials readily found in nature and labeled as bio-based materials [63]. Another advantage of water based PCMs with vegetable oil nucleating agent is that vegetable oils offer a continuous supply [64–66].

This paper reports an investigation on very small vegetable oil (soya and corn oil ester) solution in tap water as PCM material alternatives for medium temperature refrigeration applications. The little amount of vegetable oil ester in the investigated water solution would make properties of the solution similar to the physical and thermal properties of water. This could make the solution become strong PCM candidates for below 0 °C temperature applications. Other results such as super-cooling of water and the influence of vegetable oil addition in reducing super-cooling of the PCM candidates are also discussed. This paper also explains how water and vegetable oil ester can properly be mixed to become applicable PCM solutions.

## 2. Experimental

### 2.1. Materials

Materials tested in this study were natural ester oils commonly called "vegetable oils" extracted from soya bean and corn. These vegetable oils were used without further purification. Soya and corn oil esters were chosen because they contain poly-unsaturated fatty acids (PUFA) which make its freezing and melting temperatures relatively low. Chemical composition of soya and corn oil ester was tested with Gas Chromatography Mass Spectrometry (GCMS). The test results are presented in Tables 1 and 2. The tables show that commercial soya and corn oil esters are composed mainly by methyl esters of 53.89% and 38.54%, respectively. The soya and corn oil also contains benzene (16.4%) and (17.45%), 1,3-cyclohexadiene (6.85%) and (8.29%), beta-sesquiphellandrene (11.55%) and (23.83%), and others of about 11.68% and 11.89%.

The main composition of soya and corn oil esters is methyl ester. Methyl ester is a small ester with single carbon chain. Small esters are soluble in water. Esters are derived from carboxylic acids in which one hydroxyl (—OH) group replaced by one alkyl (—O) group [61]. In water solutions, certain acid molecules of ester having —OH cluster would be ionized by releasing hydrogen (H) atom to generate ion H$^+$. Although esters can't hydrogen bond with themselves but esters can hydrogen bond with water molecules. One of the slightly positive hydrogen atoms in a water molecule can be sufficiently attracted to one of the single pairs on one of the oxygen atoms in an ester for a hydrogen bond to be formed. There is also, of course, dispersion forces and dipole–dipole attractions between the ester and the water molecules. Forming these attractions releases energy. This helps to supply the energy needed to separate water molecule from water molecule and ester molecule from ester molecule before they can mix together [67]. This explains why small esters (soya and corn oil esters) dissolve in water. In higher chain esters such as ethyl and propyl esters, as chain lengths increases, the hydrocarbon parts of the ester molecules start to get in the way. By forcing themselves between water molecules, they break the relatively strong hydrogen bonds between water molecules without replacing them. This makes the process energetically less profitable, and so solubility of esters decreases.

**Table 1**
Chemical composition of commercial soya-oil ester

| Component name | Formula | Area (%) |
|---|---|---|
| Benzene, 1-(1,5-dimethyl-4-hexenyl) | $C_{15}H_{22}$ | 16.04 |
| Zingiberene, 1,3-Cyclohexadiene, 5-(1,5-dimethyl-4-hexenyl) | $C_{15}H_{24}$ | 6.85 |
| Cyclohexene, 1-methyl-4-(5-methyl-1-methylene-4-hexenyl) | $C_{15}H_{24}$ | 11.32 |
| Dodecanoic acid, methyl ester (CAS) Methyl laurate | $C_{13}H_{26}O_2$ | 5.30 |
| 4-Octenoic acid, methyl ester | $C_9H_{16}O_2$ | 0.59 |
| Beta-sesquiphellandrene | $C_{15}H_{24}$ | 11.55 |
| 6,7-Dihydroxy 3,13,14-pentaphenetetrone | $C_{22}H_{10}O_6$ | 0.36 |
| Hexadecanoic acid, methyl ester | $C_{17}H_{34}O_2$ | 12.64 |
| decanoic acid, (2,2-dimethyl-1,3-dioxolan-4-yl) methyl ester | $C_{18}H_{34}O_4$ | |
| Hexadecanoic acid, (2,2-dimethyl-1,3-dioxolan-4-yl) methyl ester | $C_{22}H_{42}O_4$ | 24.14 |
| 2-Heptadecanone, 1- (2,2-dimethyl-1,3-dioxolan-4-yl) methoxy | $C_{23}H_{44}O_4$ | |
| 10-Octadecenoic acid, methyl ester | $C_{19}H_{36}O_2$ | 5.79 |
| Nonadecanoic acid, methyl ester | $C_{20}H_{40}O_2$ | 5.43 |

**Table 2**
Chemical composition of commercial corn-oil ester.

| Component name | Formula | Area (%) |
| --- | --- | --- |
| 3-Isopropoxy-1,1,1,7,7,7-hexamethyl-3,5,5-tris (trimethylsiloxy) | $C_{18}H_{52}O_7Si_7$ | 0.61 |
| Benzene, 1-(1,5-dimethyl-4-hexenyl) | $C_{15}H_{22}$ | 17.45 |
| 1,3-Cyclohexadiene, 5-(1,5-dimethyl-4-hexenyl) | $C_{15}H_{24}$ | 8.29 |
| Copaene | $C_{15}H_{24}$ | 0.28 |
| 8-Nonenoic acid, 5,7-Dimethylene-, methylester | $C_{12}H_{18}O_2$ | 0.50 |
| Cyclohexene, 1-methyl-4-(5-methyl-1-methylene-4-hexenyl) | $C_{15}H_{24}$ | 8.45 |
| Dodecanoic acid, methyl ester | $C_{13}H_{26}O_2$ | 10.92 |
| Beta-sesquiphellandrene | $C_{15}H_{24}$ | 23.83 |
| Hexadecanoic acid, methyl ester | $C_{17}H_{34}O_2$ | 13.28 |
| 3-Butoxy-1,1,1,7,7,7-hexamethyl-3,5,5-tris (trimethylsiloxy) | $C_{19}H_{54}O_7Si_7$ | 0.68 |
| Dodecanoic acid, (2,2-dimethyl-1,3-dioxolan-4-yl) methyl ester | $C_{18}H_{34}O_4$ | |
| Hexadecanoic acid, (2,2-dimethyl-1,3-dioxolan-4-yl) methyl ester | $C_{22}H_{42}O_4$ | 2.95 |
| 2-Heptadecanone, 1- (2,2-dimethyl-1,3-dioxolan-4-yl) methoxy | $C_{23}H_{44}O_4$ | |
| Anodendroside G, monoacetate | $C_{32}H_{42}O_{11}$ | 0.48 |
| 9-Octadecenoic acid (Z), methyl ester | $C_{19}H_{36}O_2$ | |
| 7-Hexadecenoic acid, methyl ester | $C_{17}H_{32}O_2$ | 6.21 |
| 9-Octadecenoic acid, methyl ester | $C_{19}H_{36}O_2$ | |
| Cyclopropanebutanoic acid | $C_{25}H_{42}O_2$ | 1.38 |
| Oxiraneoctanoic acid, 3-octyl, methyl ester, trans | $C_{19}H_{36}O_3$ | |
| Heptasiloxane, hexadecamethyl | $C_{16}H_{48}O_6Si_7$ | 1.63 |
| Octadecanoic acid, methyl ester | $C_{19}H_{38}O_2$ | 1.99 |
| Heptasiloxane, hexadecamethyl | $C_{16}H_{48}O_6Si_7$ | 1.06 |

## 2.2. Preparation of water based PCM with vegetable oil solution

Pure water generally freezes at 0 °C. Establishing energy conservation at temperature below 0 °C, two type substances can be mixed in their eutectic proportion to attain the lowest eutectic temperature i.e. phase change temperature. To reduce phase change temperature of pure water, vegetable oil was applied as a dispersed phase in tap water as the continuous phase. The water based PCMs were prepared at different vegetable oil solution concentrations (in% volume) which were: 5%, 7.5%, and 10% soya or corn oil ester in tap water. Volume of each tested PCM sample was prepared to be 10 ml (10 cc). Other concentrations of oil solutions such as 15%, 20%, and 25% have also been investigated for low temperature refrigeration applications but they are not included in this paper.

There was not special treatment to be conducted concerning dispersion of corn or soya oil ester homogeneously in water. A simple mixing method was applied by gently shaking the test tube less than 1 min is considered sufficient. This is because of the investigated oil esters are soluble in water as describe previously.

## 2.3. Characterization of the water based PCM

Differential Scanning Calorimetry (DSC) method was used to measure thermal properties of tap water with different percentages of nucleate agents (as PCM samples). The melting temperature ($T_m$), freezing temperature ($T_f$), latent heat of melting ($\Delta H_m$), and latent heat of freezing ($\Delta H_f$) are the main interest of CTES systems. Each PCM sample was placed in a sealed aluminum crucible pan cooled from 25 °C to −100 °C and heated back to 25 °C at a cooling and heating rate of 2 °C per minute with a constant stream of nitrogen gas at flow rate of 20 ml per minute. The largest deviation in enthalpy measurement was ±2% and the largest deviation in temperature measurements was ±0.01 °C. A semi analytical digital balance (accuracy ±0.00001 g) was also used to measure the weight of the samples (mg) for the DSC test. The melting and crystallization points were taken as onset temperatures. The latent heat of PCM sample was determined by numerical integration of the area of the peak of thermal transition. Although the phase change temperatures and latent heat of the materials could be measured by the DSC system, the specimen used in DSC was very small (about 10–30 mg) which led to high

super-cooling particularly for samples that contain water, which was not applicable for practical use. Degree of super-cooling is an important parameter for PCMs. For pure water, super-cooling indicates existence of liquid water at temperature below 0 °C, while for PCM it implies the existence of liquid phase PCM at temperatures below its freezing point. A PCM with high super-cooling would consequently require much lower evaporation temperature when applied in a refrigeration system and would accordingly reduce energy performance of the system. Moreover, before freezing, energy stored in PCMs is only in the form of sensible heat that provides a very small storage capacity. Degree of super-cooling for PCM candidates, therefore, was also tested with different method which considered to be more applicable. Schematic diagram of the super-cooling measurement system is shown in Fig. 1. It can be seen that the measurement system comprises a thermostatic bath and a data logging system.

Fig. 1 shows that PCM candidates (PCM sample) were contained in glass tubes and inserted into cooling medium of the water bath. The cooling medium was mixture of 40% (by volume) polypropylene glycol. A pump circulated the cooling medium through evaporator of a refrigeration system where it was cooled. Temperature of cooling medium can reach −25 °C. For the test, however, temperature of the cooling medium was maintained stable at −20 °C by using digital thermostat with accuracy of ±0.2 °C.

The data logging system shown in Fig. 1 was equipped with data acquisition modules and a computer for recording or display system. The data acquisition modules utilized a Datascan 7000 series from MSL (Measurement System Ltd.) which included a Datascan measurement processor 7320 and expansion modules 7020. Each Datascan module contained 16 differential input channels, individually configurable for voltage and thermocouple measurements. T-type thermocouples were applied to measure temperatures of the PCM candidates and the cooling medium. The thermocouples had temperature measurement range from −250 °C to 350 °C with specific error of ±0.5 °C. The thermocouples were calibrated using a calibration bath and precision thermometer of uncertainty ±0.04 °C. The temperature range of calibration was −25 °C to 50 °C.

For the most part, enthalpy curves of PCMs were measured either via DSC (Perkin Elmer DSC 8000) or via the T-History method, but rarely with both methods. Combined DSC and T-
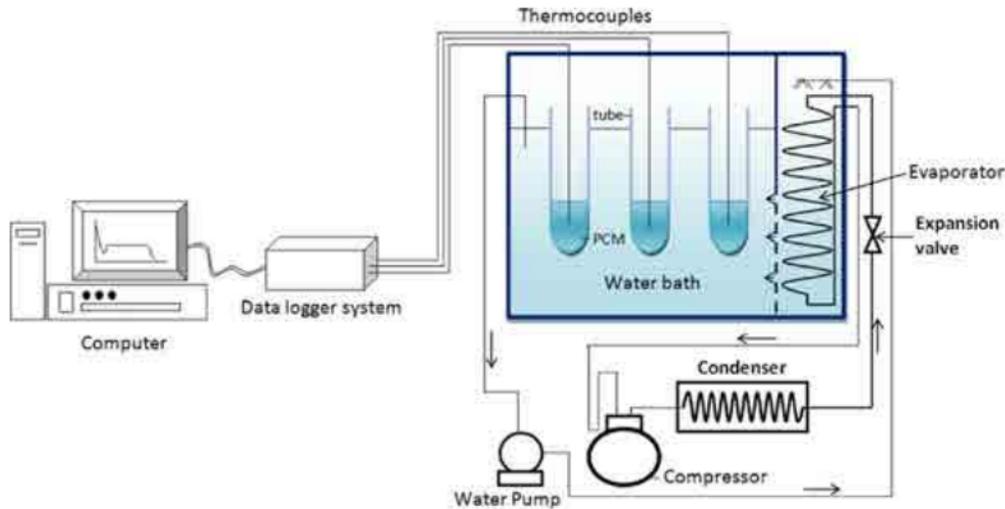
Fig. 1. Schematic diagram of experimental test equipment using T-history method.

History measurements allow an improved determination of enthalpy curves of PCM, since intrinsic material properties could be separated from properties of the investigated sample and effects of the applied methods.

## 3. Results and discussion

### 3.1. T-history test results

Fig. 2 shows temperature variation in the center of PCM samples with time as results of cooling process in water bath with constant temperature of about −20 °C. The figure illustrates super-cooling behavior of tap and mineral water during the cooling process. It can also be seen that temperatures of both samples decrease sharply to −3.5 °C at around 50 s and relatively slower when the temperatures reach −8.5 °C at around 200 s. Then ice

nucleation starts appearing and sample temperature rises to 0 °C. At this temperature level water is freezing and the temperature in the center of the PCM samples relatively constant for about 200 s. After freezing, temperature of the samples decreases toward the temperature of water bath.

Fig. 3 shows temperatures of freezing and super-cooling for tap and mineral water. From the figure, it can be seen that tap and pure water have not been frozen yet at temperature reaching −7.5 °C and −8.5 °C respectively. This is super-cooling effect of tap and pure water with degree of super-cooling of 7.5 K and 8.5 K respectively (Fig. 4). Degree of super-cooling of tap water differs from pure water. This is due to purity of water. The less pure the water the lower the degree of super-cooling. This indicates that the impurity in water acts as nucleate agents [68,69]. Therefore nucleate agents are required to reduce degree of super-cooling of pure water. The esters of corn oil and soya oil were used as nucleating agents in this paper. Nucleate agents usually used for
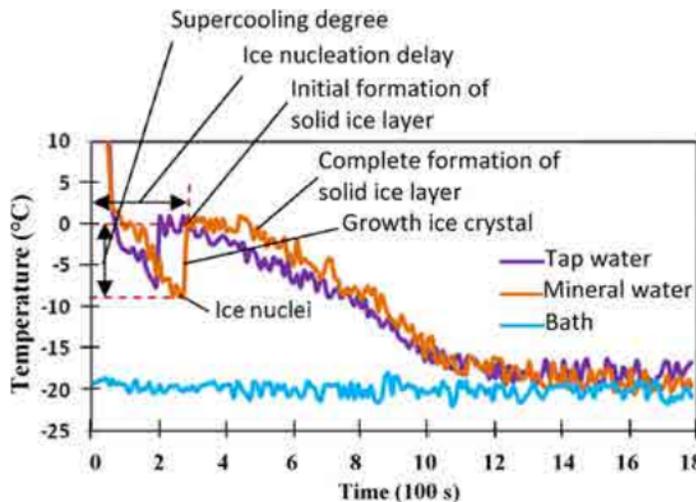


Fig. 2. Temperature in the center of samples at bath temperature of −20 °C.

1. Tap water, 2. Mineral water, 3. Salt solution, 4. Salt solution increasing, 5. 10% propylenglycol, 6. 20% propylenglycol, 7. 30% propylenglycol
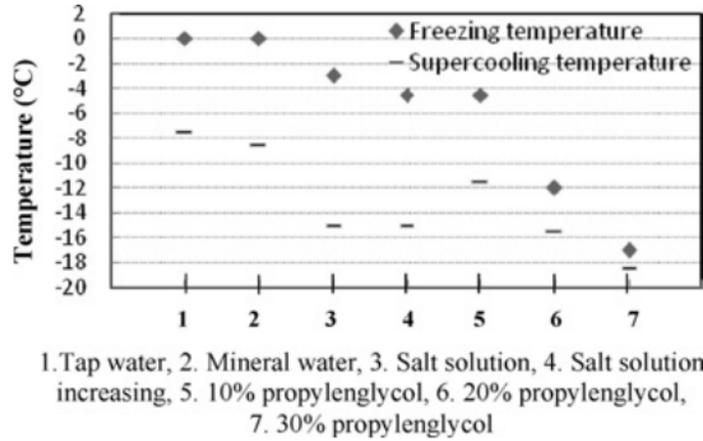
**Fig. 3.** Freezing and super-cooling temperatures of water with different nucleate agent concentrations.

water are sodium chloride (NaCl) and glycol, which were also investigated and discussed in this paper.

Fig. 3 also shows that by dissolving salt or propylene glycol can drop the freezing point of water down to below 0 °C. This may explain that freezing point of a solution is the temperature in which vapor pressure of solution and solvent are equal. The solution would not freeze at 0 °C when vapor pressure of the solution was lower than vapor pressure of the solvent (water). In order to freeze the solution, its temperature therefore should be lowered. When the solvent (water) was freezing, its vapor pressure would drop more quickly than the liquid solution. As the result at temperature level below solvent freezing point, vapor pressures of both solvent and the solution were equalized. At this condition, the solvent would freeze while the solute was still in liquid phase. This caused the solution to become concentrated and resulted in lower freezing point.

The influence of adding salt or polypropylene glycol into tap water to degree of super-cooling is shown in Fig. 4. From the figure, it can be known that addition of salt into tap water increases the super-cooling. For tap water, degree of super-cooling was found to be 7.5 K while for salt solution ranged from 10.5 K to 12 K (depending on salt concentrations). Addition of polypropylene glycol into tap water, however, could significantly reduce degree of

super-cooling. Solutions of 10%, 20% and 30% polypropylene glycol in tap water could trim down degree of super-cooling to 7 K, 3.5 K and 1.5 K respectively.

Figs. 5–7 show cooling process of PCM samples that include small vegetable oil (soya or corn oil ester) solution in tap water of different concentrations. Water bath temperature was maintained at −20 °C. Generally, temperatures of the samples change with time at three stages. Firstly, the temperatures drop sharply. Secondly, the temperatures remain constant within a certain period. These temperatures are expected to be freezing points. Thirdly, the temperatures decrease relatively smoother toward water bath temperature. At the first and second stages, the effects of soya and corn oil esters to tap water were reasonably similar. However, at the third stage the effects were slightly diverse.

From Figs. 5–7 it can also be seen that soya and corn oil esters are able to reduce the freezing point of tap water with similar effects. Soya or corn oil ester of concentration 5%, 7.5%, and 10% can drop freezing temperature of tap water from 0 °C down to −4 °C, −5 °C, and −6.5 °C respectively for soya oil ester and −3.5 °C, −5 °C, and −6.5 °C respectively for corn oil ester. Surprisingly, soya and corn oil esters could totally reduce super-cooling of the tap water.

Fig. 8 shows freezing temperatures of tap water and solutions of soya or corn oil ester with different concentrations. It can be seen
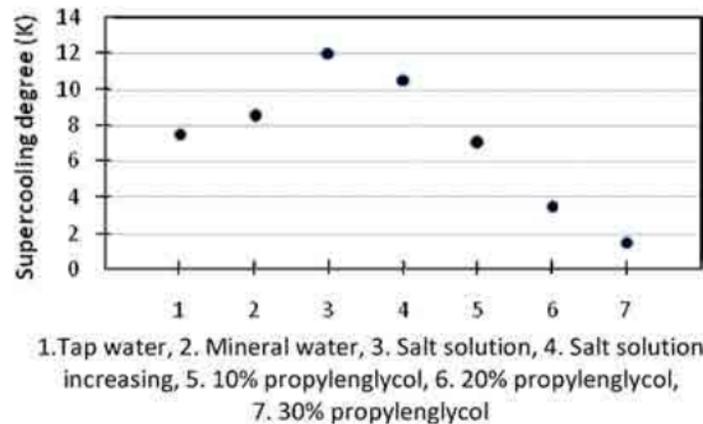


1. Tap water, 2. Mineral water, 3. Salt solution, 4. Salt solution increasing, 5. 10% propylenglycol, 6. 20% propylenglycol, 7. 30% propylenglycol

**Fig. 4.** Degree of super-cooling for water with different nucleate agent concentrations.
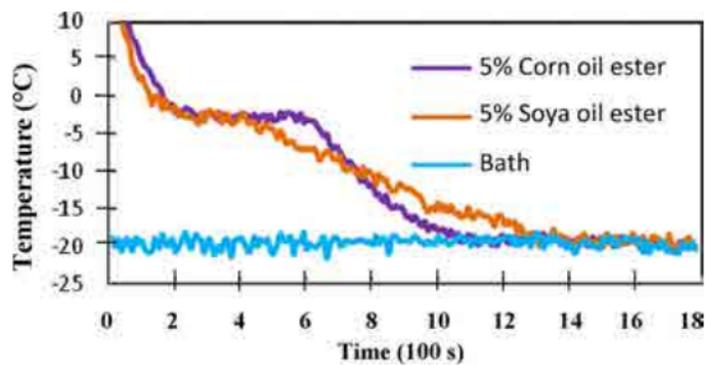
**Fig. 5.** Cooling process of 5% soya or corn oil in tap water solutions.
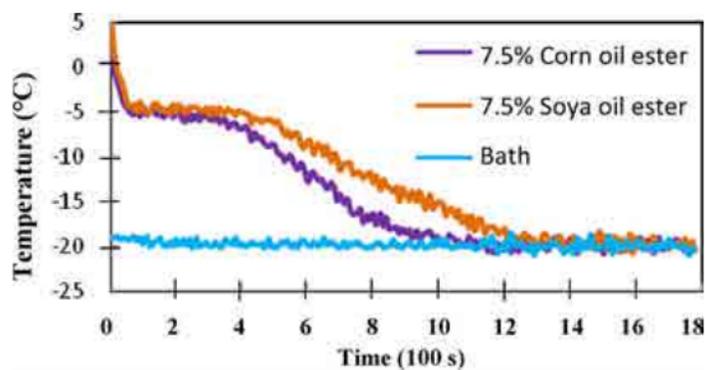


**Fig. 6.** Cooling process of 7.5% soya or corn oil in tap water solutions.

that soya and corn oil esters have almost similar effect to freezing temperature. They can significantly drop the freezing temperature of tap water almost proportionally with solution concentrations. The higher the concentration of soya or corn oil ester, the lower the freezing temperature of the solutions. This indicates that solutions of soya or corn oil ester in tap water can be considered as PCM candidates for applications at temperature level below 0 °C with thermo-physical properties are close to water.

Effects of soya and corn oil esters to degree of super-cooling are shown in Fig. 9. From the figure, it can be known that soya and corn oil esters can amazingly suppress super-cooling of tap water at any level of solution concentrations. This figure also emphasizes cooling process of the solutions without showing any super-cooling effects as discussed previously (Figs. 5–7). During freezing process, the temperature profile of PCM samples (Figs. 5–7) differed from those occurred on tap water (Fig. 2). This happened due to addition of vegetable oils into the PCM samples.

Crystallization process of PCM samples involves combination of nucleus forming and ice crystal growth inside of a crystal structure. Change of water into crystal requires nucleus in order to trigger
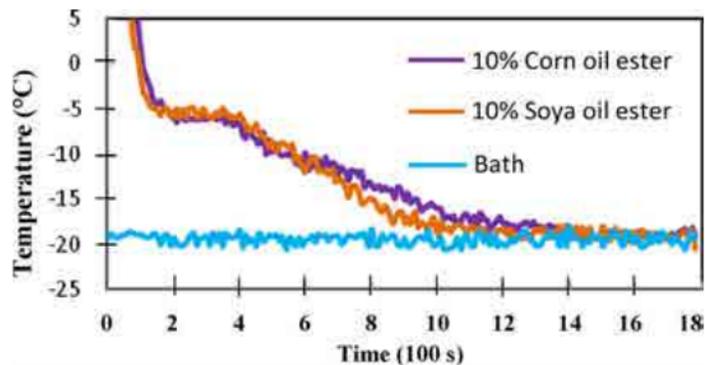


**Fig. 7.** Cooling process of 10% soya or corn oil in tap water solutions.
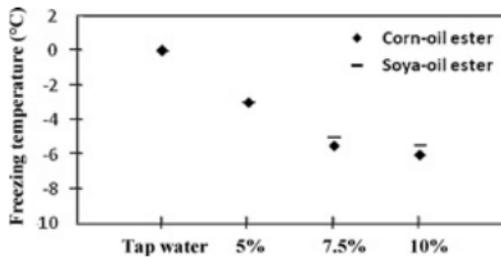
**Fig. 8.** Freezing temperatures of tap water with different percentages of soya or corn oils.
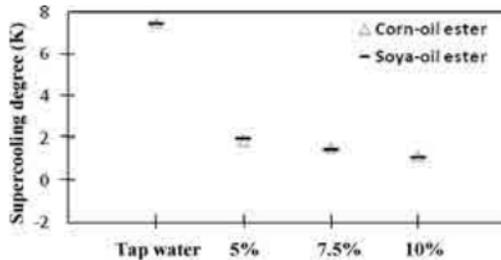


**Fig. 9.** Degree of super-cooling of tap water with different nucleate agents and concentrations.

freezing. Formation of ice crystal starts after nucleation and the water molecules join the nuclei that are being formed. Molecules compound occurred according to the appropriate size of required molecules to withstand and provide nuclei for crystal growing [70]. In the absence of nucleate particles, tap water could resist its state in super-cooled condition up to −42 °C (−43.6 °F, 231 K) before freezing homogenously [71,72]. Fig. 2 shows that the tap water is super-cooled to around −8 °C before the ice formation started.

Addition of soya and corn oil esters into the tap water could improve heterogenic nucleation. Soya and corn oil esters were able to initiate formation of nuclei at relatively warm temperature into approaching the freezing point. Water molecules merged with nuclei that were already formed in the solution. This could increase
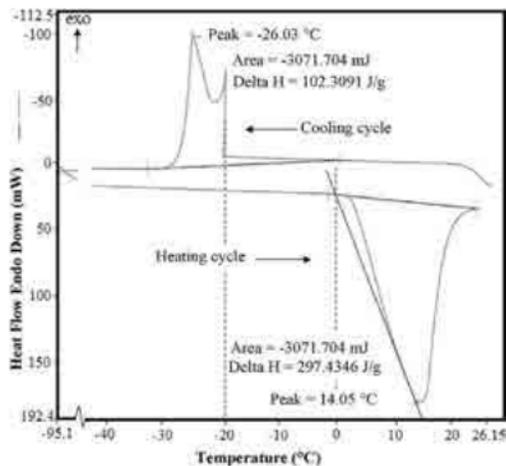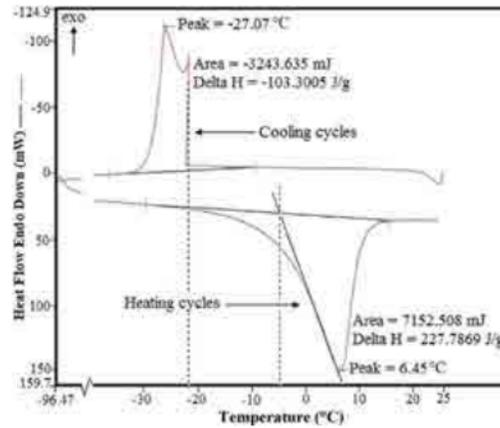


**Fig. 11.** DSC curve of corn oil in water (5/95 vol.%) binary mixture for heating-cooling cycle.

the area of crystal growth until a stable state of solution was achieved and no more crystal was formed. Due to the presence of nucleate material, the freezing point of PCM substance would be at the same point of its melting point. The exact nucleate potential of each kind of PCM substance, however, widely varied depending on the proper solution conditions. Soya and corn oil solutions were able to reduce or even eliminate super-cooling due to two reasons: (i) faster nucleation and (ii) decrease of freezing point. The decrease of freezing point was caused by the fact that the addition of soya or corn oil produced ions that strengthen intermolecular forces among solvent and solute particles. The stronger particles attracting force released much higher energy to reach the freezing point, hence the freezing point decreased.

### 3.2. DSC thermal analysis

DSC analyses have been conducted to measure the thermal properties of the PCM samples. Fig. 10 shows the DSC curve for heating and cooling processes of the tap water. The measurement results showed that melting and freezing temperatures of tap water were 0 °C and −19.5 °C, respectively. While latent heat of



**Fig. 10.** DSC curve of tap water for heating-cooling cycle.
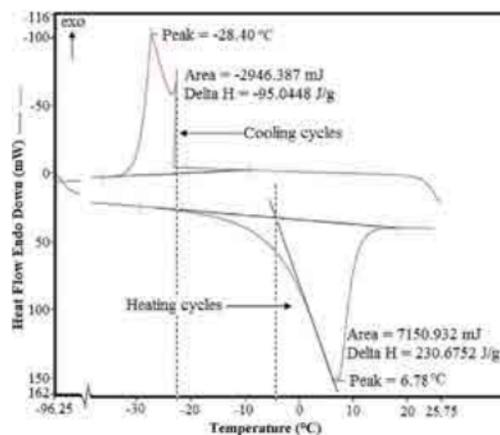


**Fig. 12.** DSC curve of soya oil in water (5/95 vol.%) binary mixture for heating-cooling cycle.
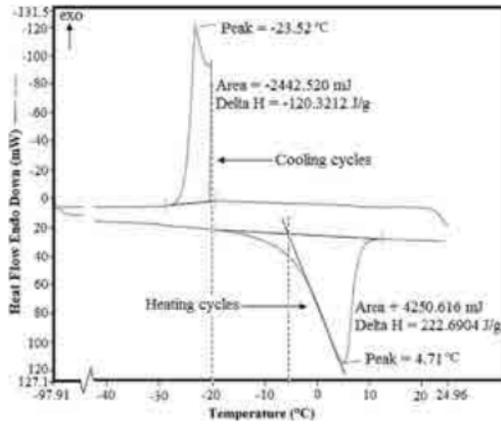
Fig. 13. DSC curve of corn oil in water (7.5/92.5 vol.%) binary mixture for heating-cooling cycle.
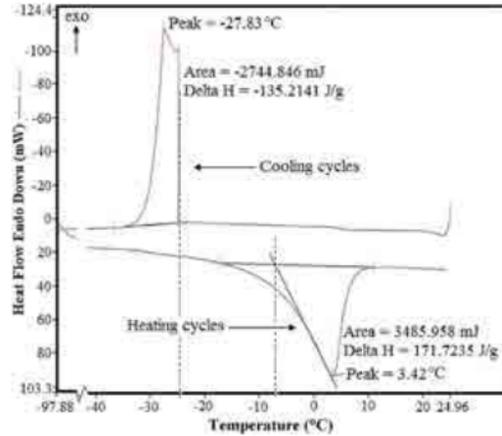


Fig. 15. DSC curve of corn oil in water (10/90 vol.%) binary mixture for heating-cooling cycle.

melting and freezing was respectively found to be 297.44 J/g and 102.39 J/g.

As there is no delay in the melting phenomenon, the melting temperature is 0 °C. This temperature is thermodynamic equilibrium temperature between ice and water. The energy released during freezing evidenced on the DSC result as an exothermic peak with imperfect bell shape. The apex temperature of the peak might be correlated to the amount of super-cooling. It is noteworthy that whatever the sample size, ice melts at 0 °C [73]. On the contrary, freezing occurs at different temperatures, depending on the water sample size [74]. From nucleation theory, it has been shown that the smaller the volume the lower the freezing temperature. For water, freezing occurred at around −14 °C at a volume of 1 cm³ and around −24 °C at a volume of 1 mm³, while for micro-sized droplets (1 μm³) freezing was found around −39 °C [75]. The energy released during the freezing process evidenced on the DSC result as an exothermic peak with imperfect bell shape when compared with melting process. The temperature difference between two intense exothermic peaks during cooling cycles may be correlated to the amount of super-cooling degree. Degree of super-cooling depend on surface conditions, cooling rate, liquid volume and nucleate agent [76].

The variations thermal properties (melting and freezing temperatures, latent heat) of 5%, 7.5% and 10% corn or soya oil ester in water mixture of the PCM samples are shown in Figs. 11–16. The addition of corn and soya oil esters could reduce the super-cooling. The super-cooling was smaller at higher corn or soya oil concentrations. It is shown by perfect bell shape exothermic peak. This is because the solution of corn or soya oil esters worked as nucleating agent that promoted faster freezing process and lower freezing temperature.

The results also show that a complete process of crystallization, which can be seen in Figs. 10–16, is very fast because with only one nucleus may initiate the solidification and simultaneously nucleation occurs. Therefore, a significant amount of energy is released in a very short time, and that is the reason why the first part of the exothermic freezing peak is so sharp. The freezing temperatures vary from one sample to another, because nucleation is a stochastic phenomenon.
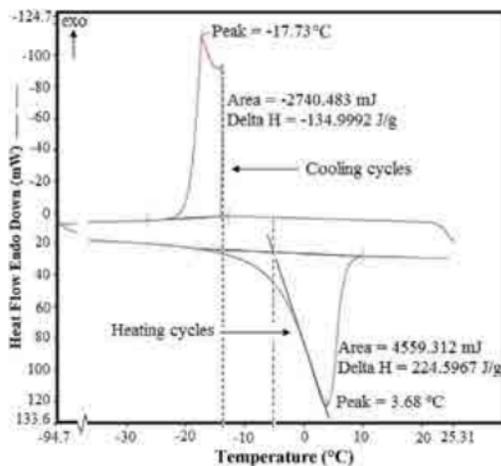


Fig. 14. DSC curve of soya oil in water (7.5/92.5 vol.%) binary mixture for heating-cooling cycle.
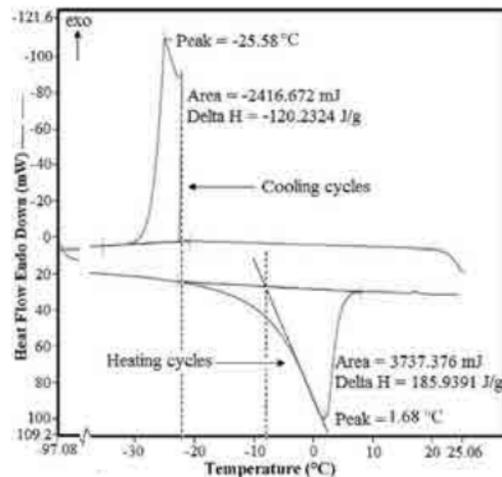


Fig. 16. DSC curve of soya oil in water (10/90 vol.%) binary mixture for heating-cooling cycle.

**Table 3**
Thermal energy storages properties of tap water and vegetable oil in water mixture.

| Samples (Vol.%) | DSC | | | | | | T-history | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| | Heating process | | | Cooling process | | | Cooling process | |
| | Melting temp. (Tm, °C) | Latent heat of melting ($\Delta H_m$, J/g) | | Freezing temp. ($T_f$, °C) | Latent heat of freezing ($\Delta H_f$, J/g) | | Freezing temp. ($T_f$, °C) | Supercooling degree (K) |
| Tap water | 0 | 297.4346 | | −19.5 | −102.3901 | | 0 | 7.5 |
| Mineral water | – | – | | – | – | | 0 | 8.5 |
| 5/95 (corn O/W) | −4.5 | 227.7869 | | −22 | −103.3005 | | −3.5 | 1.9 |
| 5/95 (soya O/W) | −4 | 230.6752 | | −19 | −95.0448 | | −4 | 2 |
| 7.5/92.5 (corn O/W) | −6 | 222.6904 | | −20 | −120.3212 | | −5 | 1.6 |
| 7.5/92.5 (soya O/W) | −5.5 | 224.5967 | | −14 | −134.9992 | | −5 | 1.5 |
| 10/90 (corn O/W) | −7.5 | 171.7235 | | −25 | −135.2141 | | −6.5 | 1.2 |
| 10/90 (soya O/W) | −8 | 185.9391 | | −22 | −120.2324 | | −6.5 | 1.1 |

O/W = Oil ester in water.

The corresponding data of thermal properties resulted from T-history and DSC tests are also summarized in Table 3. From the table it can be seen that the melting point of water with the DSC and the freezing point of water with T-history is 0 °C. The test results showed DSC melting temperature of corn oil − tap water (corn O/W) and soya oil − tap water (soya O/W) samples (5/95, 7.5/92.5 and 10/90 vol.%) were in the range from −4.5 °C to −7.5 °C for corn oil ester and from −4 °C to −8 °C for soya oil ester respectively. Melting and freezing latent heat varied in the range of 171.72 − 230.68 J/g and 95.05 − 135.21 J/g respectively. While based on the T-history, freezing points are in the range from −3.5 °C to −6.5 °C for corn oil ester and −4 °C to −6.5 °C for soya oil ester respectively.

Overall, six samples were developed with different volume fractions of vegetable oils (soya or corn oil) and tap water with proper mixing. The results acquired from DSC and T-history tests indicated that the phase transition temperatures of vegetable oil in water mixture were lower than the temperatures of the individual tap water. The melting and freezing temperatures of the mixture followed a downturn with the increase in concentration of vegetable oil in water could significantly reduce the degree of super-cooling.

This suggested that small vegetable oil solution in water mixture as PCMs has high latent heat and suitable and/or adjustable phase transition of melting and crystallization temperatures, and this would be highly desired for applications. However, there were small discrepancies between the melting temperature of DSC and freezing temperature of T-history test results. The differences between the results were most probably due to two factors: (i) the presence of a certain amount of impurities of the vegetable oils used in the mixture and (ii) the delay response due to thermal inertia of the temperature sensor at T-history analysis [77,78]. For the freezing temperature of DSC, the value is far from its melting temperature. DSC test data for water based PCM is strongly influenced by the sample size, the smaller the size of the sample the larger the difference as described previously [73].

These properties indicate that the vegetable oil in water solutions are promising PCM candidates to store and to release energy for medium temperature refrigeration applications. Moreover, they have better odor and non-corrosive properties as well as potential usage for energy storage at below zero working temperature.

## 4. Conclusions

Materials, which possess good potential for PCMs applied at medium temperature refrigeration, have been investigated in this paper. The investigated PCM candidates were mixture of vegetable oil in water solutions. The vegetable oils investigated were corn and soya oil esters. It was found that corn and soya oil esters substantially contained small ester (methyl ester) which made the vegetable oils soluble in water.

The PCM candidates were considered to be good candidates for thermal storage at temperature level below 0 °C. The PCM candidates contained 5%, 7.5%, and 10% soya or corn oil ester. The PCMs were found to be able to reduce freezing temperature of pure water from 0 °C down to respectively for soya oil water PCM candidates: −4 °C, −5 °C, and −6.5 °C while for corn oil in water PCM candidates: −3.5 °C, −5.5 °C, and −6.5 °C respectively.

Only small amount of nucleate agent (soya or corn oil ester) was required for the PCM solutions, make the soya oil or corn oil in water solution become very good PCM material candidates. The PCM candidates were found to have thermo physical properties that were close to properties of tap water. Melting latent heat of the PCM candidates varied in the range of 171.72–230.68 J/g, while tap water had melting latent heat of 297.44 J/g. The investigation also showed that the PCM candidates had better property than water due to their small degree of super-cooling especially for medium temperature refrigeration applications.

## Acknowledgement

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