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Development of Corn-Oil Ester and Water Mixture Phase Change Materials for Food Refrigeration Applications

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Abstract

This research aims to investigate development of corn-oil ester and water mixtures as novel solid-liquid phase change material candidates for chilled and frozen food refrigeration applications. Thermal properties of both water and its mixture with corn-oil ester were tested by DSC and T-history methods. The results showed that corn oil could mix well in water solutions. Phase transition temperatures of the mixtures were lower than those of individual water. Corn-oil ester in the mixtures was acted as a nucleate agent and it was able to lower freezing point and to trigger ice nucleation in water which could diminish super-cooling. Addition of corn oil ester by 5% to 35% in water solutions could decrease freezing temperature from 0°C down to respectively -3.5°C to -28°C. The PCM candidates were also found to have excellent thermal properties that could fulfill requirements of thermal energy storage systems for food refrigeration applications.

Keywords: corn-oil ester; phase change materials; thermal energy storage; food refrigeration system;

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1. Introduction

Phase change material (PCM) is one of thermal energy storage (TES) technology which can improve the performance and reliability of energy systems. The technology could also potentially provide energy savings, which in turn could reduce environmental impact related to energy use [1]. Phase change materials (PCMs) store heat by using latent heat, commonly from solid to liquid, as they can exhibit latent heat of phase change and have attracted interest as possible heat thermal storage [2]. Phase change storage with PCMs is one of the most efficient ways to store thermal energy [3]. One advantage of the PCMs is it has high energy storage density with small temperature variation during the process of phase change [4]. They have been used to improve the TES capacity of different systems [5]. PCMs with large latent heat of fusion are also increasingly being used for thermal management of air conditioning in buildings in order to achieve a better balance between cooling supply and demand [6]. In the last 15 years PCMs have also been gradually used for food storage and transportation system [7].

Nomenclature

COP	Coefficient of Performance
DSC	Differential Scanning Calorimeter
GCMS	Gas Chromatography Mass Spectrometry
PCMs	Phase Change Materials
TES	Thermal Energy Storage

Many researchers have also carried out investigations on the feasibility of application of PCMs in improving the performance of refrigerated cabinets, chest freezer and domestic refrigerators. Applications of integrated thermal energy storage with PCMs have the potential to increase the energy efficiency of the refrigeration systems. The improvement can be achieved by reducing compressor cycling frequency and cycling losses. Moreover, the use of PCMs can also maintain product temperature within a safe temperature range in the event of electrical power failure [8,9]. Experimental investigations on the performance of household refrigerators using PCM were carried out by Azzouz *et al*. [10,11]. The PCM was placed in a container at the back of the evaporator plate between. The results showed that the response of the refrigerator to the addition of PCM and its efficiency were strongly dependent on the thermal load. The integration of PCM allowed 5 to 9 hours of continuous operation without electrical supply. This could increase the coefficient of performance (COP) of the system by 10% - 30% depending on the cooling load. While studies on the use of PCMs in freezers were reported by [9,12,13]. The PCM was introduced close to the evaporator wall. The results indicated that, during electrical power failure the use of PCM could maintain product temperature for longer compared to the freezer without PCM. Lu *et al.* [14] and Jouhara *et al.* [15] investigated the use of PCM and heat pipes to provide product temperature uniformity on the shelves of vertical multi-deck food display cabinets. The PCMs were placed within the structure of the shelves of the display cabinet. The results showed that the use of heat pipes could homogenize the temperature profile of the products and improve the heat transfer between the cabinet, the shelves and the products.

PCMs are generally grouped into organic and inorganic compounds [16,17]. Organic PCMs are very important class materials because of their unique thermal properties such as congruent melting process and narrow melting-freezing temperature ranges [18,19]. Paraffin, the most commonly used organic PCM, have been widely used for energy storage due to its wide range of phase change temperatures, negligible super-cooling, no corrosive behavior and chemical stability [6,20]. However, paraffin relatively has higher cost, high volume change, lower latent heat and lower thermal conductivity. Another serious issue of the paraffin is its high flammability [20]. The low thermal conductivity of paraffin requires heat transfer enhancement methods such as the incorporation of materials with high thermal conductivity [21-23], increasing heat transfer surface area [24-28] or application of compact heat exchanger [6,29]. While salt water solutions are very common inorganic PCMs. The solutions have advantages of higher thermal conductivity, fusion heat and density, and lower flammability. However, salt water solutions possess serious issues of corrosion and super-cooling.

The best-known PCM is water. It has very good thermal properties such as reliability, low cost, high specific heat, high density, high latent capacity of 335 kJ/kg and safe [30]. Unfortunately, water cannot be used on its own as a PCM in food refrigeration of temperature range below 0 °C [31]. Water also has a big degree of super-cooling during solidification process [32]. In some applications, degree of super-cooling can have major effect on a system performance [33]. In order to make water applicable as PCMs at temperatures below 0 °C, nucleation agent could be added to trigger heterogeneous nucleation. This could also eliminate the super-cooling of water [34]. A food grade antifreeze or nucleation agent in the water would be required [35]. This will maintain the high percentage of water in the solution and the high latent heat of the PCM making it a good candidate for applications just below 0 °C.

The main objective of this paper is to develop phase change material candidates for medium and low temperature food refrigeration applications. The PCM candidates were made by mixing corn-oil ester which worked as nucleation agent. Corn-oil ester and water solutions to be investigated are applicable for medium and low temperature food refrigeration of evaporating temperature of the system between -35 °C and -8 °C. The solutions contain only small portion of corn oil ester. Larger part of the solutions is water which makes them become strong PCM candidates for food refrigeration applications. Moreover, corn-oil ester also contains various types of fatty acids which have many superior properties as organic PCM materials [36-38]. Fatty acids are also derivatives of materials that are readily found in nature such as vegetable oils and labeled as bio-based materials [39]. However, fatty acid ester is more expensive compared with corn-oil ester. Another advantage is that corn-oil ester offers a continuous supply [40,41], no corrosive behavior, non-flammable, and non-toxic, therefore it is suitable for food refrigeration.

2. Materials and Characterization

2.1. Materials

Materials used in this study were tap water and corn oil ester as nucleating agent resulted from esterification of corn oil. Corn oil ester was chosen because it contains a lot of unsaturated fatty acids so it has low freezing and melting points. The corn oil ester is composed mainly by methyl esters of 38.54%. The oil ester also contains benzene (17.45%), 1,3-cyclohexadiene (8.29%), beta-sesquiphellandrene (23.83%) and others of about 11.89%. Esters are polar molecules that have a very important role on the solubility of corn oil in water. These chemical compositions of corn-oil ester were obtained from Gas Chromatography Mass Spectrometry (GCMS) [42]. The test method comprised analysis of corn oil ester which was performed on a GC-MS Shimadzu type QP 2010 with a split/split less injector. The GCMS test results are presented in Table 1.

Table 1. Chemical composition of investigated 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-, methyl ester	$C_{12}H_{18}O_2$	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 ester	$C_{16}H_{48}O_6Si_7$	1.63
Octadecanoic acid, methyl ester	$C_{19}H_{38}O_2$	1.99
Heptasiloxane, hexadecamethyl ester	$C_{16}H_{48}O_6Si_7$	1.06

Methyl ester, which mainly contained in corn oil ester, is a small ester with single carbon chain. Small esters are soluble in water. This plays a key role in solubility of corn oil ester in water. The solubility of corn oil ester can also be explained as certain acid molecules of ester in water solution having -OH cluster which are ionized by releasing hydrogen atom to make ion H+. Even though esters cannot hydrogen bond with themselves but esters can hydrogen bond with water molecules. Individual positive hydrogen atom in a water molecule can be attracted to one of the single pairs on one of the oxygen atoms in an ester for a hydrogen bond to be formed. Moreover, there are also dispersion forces and dipole-dipole attractions between the ester and the water molecules which release energy. This helps to supply energy required to separate water molecule and ester molecule from others before they can mix together [43]. This explains why corn oil ester dissolves in water. The corn-oil esters were chosen as nucleating agents for the purpose of obtaining a food grade PCM which was considered as one important factor for food refrigeration applications. PCMs made from the mixture of tap water and corn oil esters are also economically competitive compared to paraffin based PCM. At present, the market price of fatty acid esters is relatively high. This is because of the cost of producing the fatty acid esters is higher than that of corn oil esters because the production line of fatty acid ester includes purification process. While the corn-oil esters can be used without further purification.

2.2. Characterization of PCM

Thermal properties of the PCM candidates (of corn-oil ester in water mixtures) were measured by differential scanning calorimeter (Perkin Elmer Jade DSC). The properties included melting and freezing temperatures and latent heat of melting and freezing. The analyses were performed at temperatures between 25 °C and -100 °C for cooling and from -100 °C to 25 °C for heating at 2 °C per minute of cooling and heating rate. The analyses were also performed under a constant stream of nitrogen gas at flow rate of 20 mL per minute. The temperature accuracy was ± 0.01 °C and heat flow repeatability was 0.2 µW. A 30 mg sample of PCM candidate was sealed in an aluminum pan. The melting and crystallization points were taken as onset temperatures.

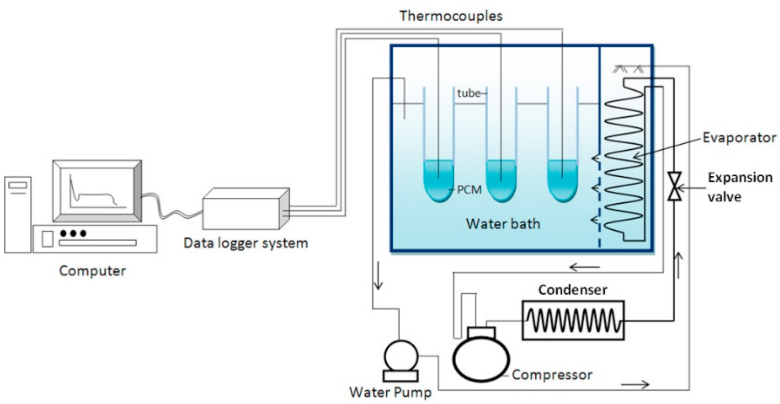

Fig. 1. Schematic diagram of T-history method

The latent heat of PCM candidates was determined by numerical integration of the area of peak thermal transition. Even though phase change temperatures of the solutions can be measured by the DSC system, the specimen used in DSC is very small (of about 10-30 mg) which is not applicable for practical use especially for samples that contain water with high degree of super-cooling [44,45]. Whereas degree of super-cooling is an important parameter for PCMs. In this research, phase change temperatures and degree of super-cooling of the PCM candidates were tested by using T-history method which is considered more suitable for this application. Schematic diagram of the T-history method is shown in Fig. 1. The PCM candidates tested include the mixtures of 5%, 7.5%, 10%, 12.5%, 15%, 20%, 25%, 30% and 35% corn oil ester in water.

3. Results and Discussion

3.1. Super-cooling analysis

Super-cooling occurs when the temperature of a liquid is lowered below its freezing point without becoming a solid [46]. Fig. 2a shows that tap and mineral water was super-cooled to reach -7.5 °C and -8.5 °C respectively before the ice formation process started. The ice crystallization process involves combination of nucleation and growth of ice crystals within a crystalline structure. Ice crystal formation occurs after nucleation, at which the water molecules join the already formed nuclei. For comparison, in Fig. 2b the super-cooling of propylene glycol solutions is illustrated. Super-cooling occurs at lower concentration of propylene glycol solution. At higher concentration, the super-cooling disappears.

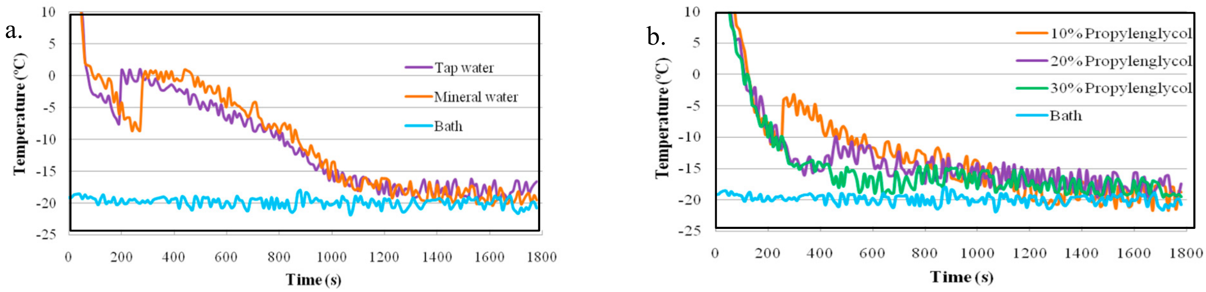

Fig. 2. Super-cooling: tested at bath temperature of -20 °C; a. pure water, b. propylene glycol in water solutions

Fig. 3a and 3b shows that PCM candidates with different concentration of corn-oil ester are able to initiate formation of ice nuclei quickly at somewhat higher temperature than its approaching freezing-point of the solution. For the history-T method, the test can be done up to 25% corn oil ester in water solution due to limitation of the minimum bath temperature. It can be seen the additions of 5%, 7.5%, 10%, 15%, 20% and 25% of corn-oil ester in the PCM candidates can decrease tap water freezing point to -3.5 °C, -6 °C, -7.5 °C, -10 °C, -15 °C and -19.5 °C respectively. They can also reduce super-cooling of the pure water. The addition of corn-oil ester as solute particle into the tap water as solvent produce some ions that contribute to intermolecular force between solvent and solute particles.

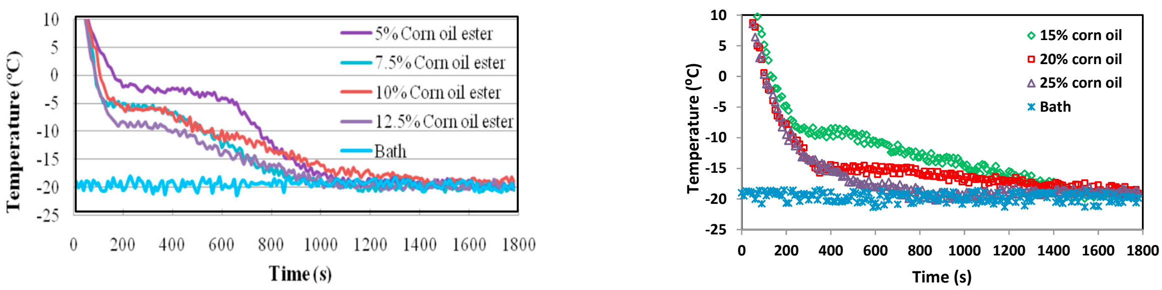

Fig. 3. Cooling process of corn-oil ester in tap water solutions at bath temperature of -20 °C

During cooling process, the pulling force between solvent and solute particles release more heat hence the freezing point of the solution is lowered. Therefore, corn-oil ester solution is able to reduce or even eliminate super-cooling due to: (i) faster nucleation and (ii) lower freezing point.

3.2. Thermal properties of the PCMs

In order to compare thermal properties and phenomena in melting and freezing processes of the PCM candidates, the results of DSC for melting and freezing processes of tap water is also presented in Table 2. The melting and freezing temperatures of tap water resulted from DSC were 0 °C and -19.5 °C respectively, and the latent heat of melting and freezing were 297.4 J/g and 102.4 J/g respectively. It is noteworthy that, whatever the sample size, ice melts at 0 °C. On the contrary, freezing occurs at different temperatures, depending on the water sample size [45]. From nucleation theory, it has been shown that the smaller the volume, the lower the freezing temperature. For bulk water, freezing occurs at -14 °C for a volume of 1 cm3 and at around -24 °C for a volume of 1 mm^3, while for micro-sized droplets (1 μm^3) freezing is found around -39 °C [44]. The energy released during the freezing process is evidenced on the DSC result as an exothermic peak with imperfect bell shape when compared with melting process.

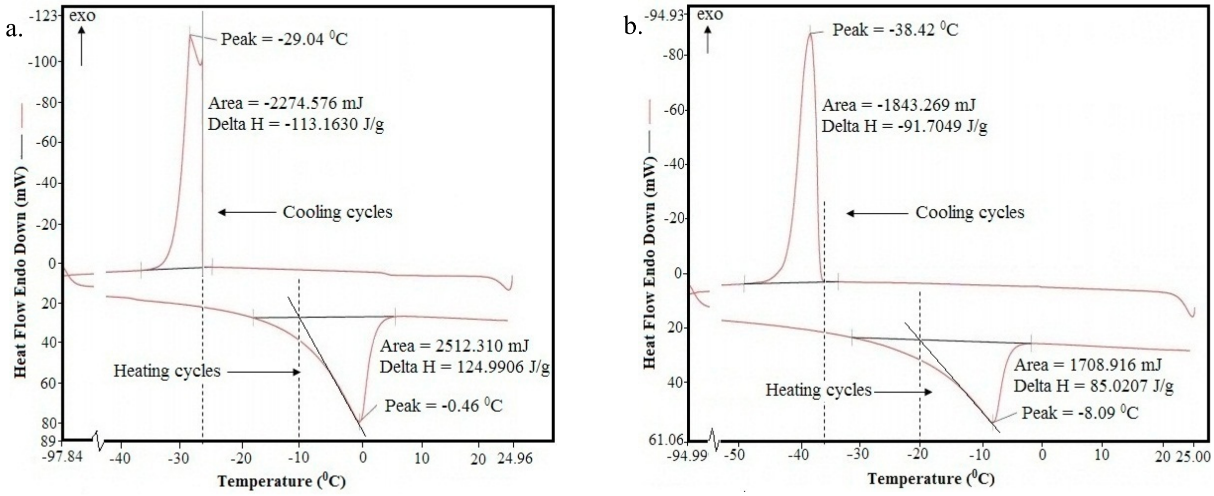

Fig. 4. DSC curves of heating and cooling processes: a. 15% corn-oil ester, b. 25% corn oil ester

Thermal properties of PCM candidates which contain tap water and various compositions of corn-oil ester can be seen in Table 2. While Fig. 4 shows only two DSC curves in order to show super-cooling. Other curves of the corn oil ester solutions are not presented. Fig. 4 shows thermal properties of PCM candidate with 15% and 25% corn-oil ester. From the figure it can be seen that the addition of 15% corn-oil ester into tap water still demonstrates the occurrence of super-cooling. Increasing the concentration of the corn-oil ester to 20% (it is not shown in the figure) causes degree of super-cooling of the PCM solution to decrease. Degree of super-cooling is totally disappeared as the concentration of corn-oil ester reaches 25% (Fig. 4b). This is indicated by a perfect bell shape shown in Fig. 4b.

Table 2. Thermal properties of the PCM candidates, DSC Test Results

Samples (Vol. %)	DSC			
	Heating process		Cooling process	
	Melting temp. (Tm, °C)	Latent heat of melting (ΔH_m, J/g)	Freezing temp. (T_f, °C)	Latent heat of freezing (ΔH_f, J/g)
Tap water	0	297.4	-19.5	-102.4
5/95 (E/W)	-3.5	227.8	-22	-103.3
7.5/92.5 (E/W)	-6.0	222.7	-20	-120.3
10/90 (E/W)	-7.5	171.7	-25	-135.2
15/85 (E/W)	-10.5	125.0	-27	-113.2
20/80 (E/W)	-15	107.3	-33	-109.8
25/75 (E/W)	-19.5	85.0	-36	-91.7
30/70 (E/W)	-23	78.6	-38	-84.7
35/65 (E/W)	-27	68.7	-43	-67.2

E/W = Corn-oil ester in water

The freezing temperatures vary from one sample to another, because nucleation is a stochastic phenomenon. Results of DSC test method are also summarized in Table 2. The table clearly shows that melting temperatures of the PCM candidates are lower than those of the tap. The melting and freezing temperatures of corn-oil ester in water solutions of concentration between 5% and 35% (by volume) range from -10 °C to -27 °C. While melting latent heat varies from 68.7 J/g to 227 J/g respectively. The results indicated that by increasing concentration of corn-oil ester in water solution can reduce melting temperature and minimize or even negate the super-cooling. These properties make the solutions potential to be PCMs with large latent heat and suitable phase change temperatures for medium and low temperature food refrigeration applications. For comparison, Fig. 5 shows melting temperature of PCM candidate corn oil ester (COE) in water solutions, propylene glycol solutions and NaCL solutions at different concentrations. The PCM candidates (Corn oil ester solutions) require lower concentration to achieve the same melting temperature below 0 ºC. This indicates less material is needed for the corn oil ester PCM.

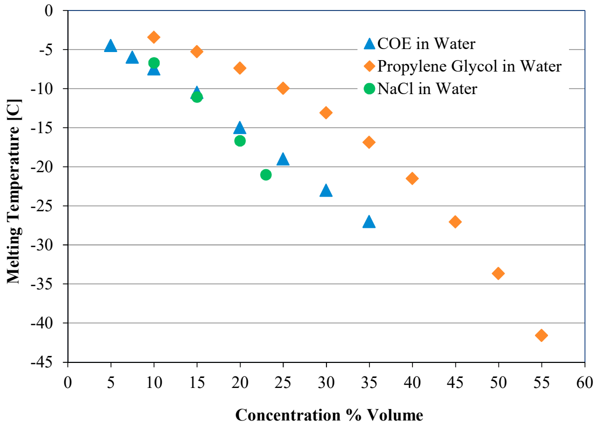

Fig. 5. Melting temperatures of Corn oil ester solutions in comparison to NaCl and Propylene glycol solutions

4. Conclusions

Corn oil ester in tap water mixtures have been investigated for development of phase change materials (PCMs) as thermal energy storages that can be applied for food temperature refrigeration systems. DSC and T-history thermal analyses were applied in the investigation and it has been found that the water-based mixtures contain 5% up to 35% corn-oil ester have freezing temperatures -3.5 ºC to -27 ºC respectively. The investigation also found that the PCM candidates at test conditions have minimum or even no or negligible degree of super-cooling. Additionally, corn-oil ester and water solutions offer a continuous supply and cheaper option compared with fatty acid esters that has been frequently used for below 0 °C applications. Corn oil ester showed no corrosive behaviour and is non-toxic as well. These make the mixture of corn oil ester and water become applicable as low cost novel PCMs for food refrigeration applications.

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