Investigation of Thermal Behavior of Paraffins, Fatty Acids, Salt Hydrates and Renewable Based Oils as PCM

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Abstract— **Latent heat thermal energy storage (LHTES) using phase change materials (PCM) is a renewable energy solution that is applicable for implementation in space cooling due to its high energy storage density. A novel thermal energy storage which will encapsulate a PCM layer to absorb the rejected heat from the building during occupied hours and release it to the ambient air during night-time is going to be developed. On the grounds of this development, a selection of seven PCMs are examined. The selected materials comprise the basic classes of PCM, namely: paraffins, fatty acids, and salt hydrates. The objective of this study is to identify experimentally the thermal properties of commercial PCM, renewable based oils, PCM in water emulsions, and PCM polymer blends. The supporting materials used in the polymerization of the PCM are polyethylene glycol diacrylate (PEGDA) and polyvinylpyrrolidone (PVP). The characterization of the thermophysical properties of the PCM is achieved by using differential scanning calorimetry (DSC) in dynamic operation mode. The values of the thermophysical properties for the commercial PCM and the renewable based oils provided by the manufacturers are compared with the experimental results. The long term stability of the thermophysical properties of PCM after 50, 100, 150, and 200 thermal cycles equivalent to a 6-month duty cycle in a real-life application is presented. The cases of the PCM emulsions and the PCM polymer blends are analyzed. The obtained results demonstrate the stability of the PCM under thermal cycling and the supercooling effect during the liquid-solid phase change.**

Keywords—PCM, LHTES, DSC, Building

I. INTRODUCTION

The energy demand in the building sector has raised to satisfy the thermal comfort needs of building occupants [1]. Thermal energy storage (TES) applications are widely investigated to achieve energy reduction of buildings [2]. Phase change materials (PCM) are smart materials with the capacity to control temperature variations used for TES applications during the last decades. TES involves the storage of energy as heat in three forms: sensible, latent, and thermochemical heat storage. Latent heat thermal energy storage (LHTES) with PCM is characterized by high energy storage density in small temperature variations and is a preferred solution in TES systems. A thermal energy storage application follows three steps: charging, storing, and discharging. PCM are substances that change state from solid to liquid by absorbing heat (charging) in an endothermic process. With a decrease in the temperature, the PCM solidifies by withdrawing heat (discharging) in an exothermic process. The incorporation of PCM in TES systems accumulates the storage of a large amount of latent heat in a small volume [3]. Energy reduction in heating and cooling applications is achieved with the implementation of PCM in technical equipment. PCM can retrofit the existing cooling system in order to achieve energy saving using free cooling. In the designing process of a LHTES with PCM, the most significant requirements for the selection of a PCM are the phase change temperature and the latent heat of the material [4]. Both characteristics are indicated by thermal analysis techniques such as differential scanning calorimetry (DSC) [5], differential thermal analysis (DTA) [6], T-history method [7] and Thermogravimetric Analysis (TGA) [8]. In the ongoing project related to this study a novel heat exchanger will be designed and constructed which will encapsulate a PCM layer to absorb the rejected heat from the building during occupied hours and release it to the ambient air during the night. It is expected that the novel heat exchanger will be integrated to the already existing "two-pipe chilled beam system for simultaneous heating and cooling" [9]. The PCM layer transfers heat absorbed from the building by the chilled beam water system to the cooling tower, where it is dissipated into the atmosphere [9]. Outdoor air temperature is a preferred sustainable source in free cooling applications. For about 93% on an annual basis, the ambient temperature is below 18 °C for Copenhagen weather conditions [10]. In this respect, PCM with a phase change temperature between 15 and 20°C will be selected for the PCM LHTES since this phase change temperature range is feasible for both the charging and the discharging phase. The available in the existing literature PCM within the desirable phase change temperature limits 15 - 20°C already used in relevant studies were presented in the author's literature study [11]. In the current study, available organic and inorganic PCMs in the market i.e. organic

paraffins, organic non-paraffins and inorganic salts are compared with biobased oils, not known for their phase change properties. Organic PCM are the most commonly used PCM in the current literature [12],[13],[2] due to their phase change temperature range and their heat storage capacity. The two renewable based oils tamanu oil and coconut oil, were found as cosmetic and edible products, respectively, in the market. Recent experimental research is not focused on the long term stability of existing PCM over an extended duty cycle in a real-life application. In the current study, the long term performance and stability of five PCM were investigated.

II. MATERIALS

A. Pure PCM

In total, seven materials and their eutectic mixtures were selected and characterized by DSC. Five commercial PCM with applicability for LHTES systems, and two renewable based PCM not known for their phase change properties were selected. The five commercial PCM belong to the classes of salts, paraffins, and fatty acids. Table 1 illustrates the thermophysical properties provided by the manufacturers. All seven materials namely the two organic paraffins: RT15 and RT18, the two organic non-paraffins PT15 and PT18, the inorganic salt hydrate SP15 and the renewable based oils: tamanu oil and coconut were characterized using DSC in their first thermal cycle.

Table 1: Thermal properties of PCM candidates provided by manufacturers.

Classification	Material	Phase Change Temperature (°C)	Latent heat (J/g)
Organic non-paraffins	PT 15	15	182
	PT 18	18	192
Organic paraffins	RT 15	10-17	155
	RT 18	17-19	260
Inorganic salt hydrate	SP 15	15-17	180
Renewable based oils	Coconut oil	23-26	-
	Tamanu oil	24-26	-

Moreover, the long-term performance of five PCM samples was evaluated by DSC under 200 thermal cycles. The two organic paraffins RT15 and RT18, the two organic non-paraffins PT15 and PT18 and the renewable based tamanu oil were selected for thermal cycling.

B. PCM emulsions and PCM polymers

The organic paraffins RT15 and RT18 were dispersed in water in a ratio of 1:1 to 5:1 with the addition of emulsifiers. Water as PCM has the highest specific heat (4.186 joule/gram °C) compared to other substances, but its use is limited to fixed temperature applications (0 celsius). The emulsions of PCM with water in different ratios were formed and tested using DSC to identify whether the heat capacity of the final solution will be increased or not. Stable oil in water emulsions were formed with the use of Polyethylene glycol sorbitan (Tween 20) and Polyoxyethylensorbitan oleate (Tween 80) as emulsifiers. Tween is non-ionic detergent widely used in biochemical applications for stabilizing oil in water emulsions. No phase separation was detected in the oil in water emulsions. However, it is complex to determine an emulsion's instability.

Form stable PCM are composed by solid-liquid PCM and supporting materials. The advantage of form-stable PCM is that the material's shape can be maintained when the ambient temperature is higher than the phase change temperature. The PCM polymers of the organic paraffins RT15 and RT18 were synthesized in two different ways. In the first approach, the polymers were formed by the PCM 5% w/v with polyethylene glycol diacrylate (PEGda) 5% w/v as a synthesizer and benzoyl peroxide (BPO) 1% w/v as initiator. In the second solution, the polymers were formed by a PCM emulsion 15% w/v with polyvinylpyrrolidone (PVP) 5% w/v as a synthesizer and ammonium persulfate (APS) 10%w/v as initiator. The PCM emulsion in the second solution was a PCM-Polyvinylalcohol (PVA) emulsion. The PCM-PVA emulsions was formed by mixing 10ml of 30%w/v PCM with 8.4 mmol/L of sodium dodecyl sulfate (SDS) and 100 ml of a 9% w/v polyvinyl alcohol (PVA) solution.

III. TESTING METHODS

A process that releases energy as heat into the surroundings is classified as exothermic and one that absorbs energy as heat from the surroundings is classified as endothermic [14]. The release of heat at constant pressure signifies a decrease in the enthalpy (ΔH) of the system (exothermic process ΔH<0). The enthalpy changes at constant pressure can be measured as heat transferred from a system. Changes in internal energy are measured experimentally with calorimetry. Enthalpy can be defined by (1) and (2). At constant pressure, the equation of enthalpy is described by (3).

$$H=U+pV \quad (1)$$

$$\Delta H=Q= \int C_P dT \quad (2)$$

$$\Delta H= \Delta U+ p\Delta V \quad (3)$$

H=enthalpy, U=internal energy, p=pressure, V=volume

DSC is a versatile technique for thermal analysis introduced in the early 1960s [15], and it measures the energy transferred as heat to or from a sample at constant pressure during physical or chemical change. The dynamic range characterizes the DSC when compared to other calorimetric methods. The sample is compared to a reference material that is not involved in a physical or chemical change, and the temperatures of the sample and reference are scanned during the analysis. The scanning rate is stable in the dynamic operational mode of the DSC. Heat flow rate versus temperature is the outcome of the DSC curve due to the thermal resistance of the sample [16]. The area under the heat flux curve is proportional to the heat of fusion or enthalpy (J/g). The phase change of the PCM is occurring in a specific temperature where a peak is produced in the DSC curve. The DSC tests were performed with the use of a DSC Q2000 instrument with T-zero thermocouples. This DSC equipment offers 50 positions for samples and a temperature range of -180 to 725. A conventional empty crucible was used as a blank for the reference sample. The DSC equipment includes a furnace where a reference sample and the examined sample are heated or cooled under controlled temperature variations. A rate for the temperature fluctuation is defined, and heat is transferred to the samples. The materials response is described in equation (4). The specific heat capacity (Cp) of the examined PCM can be calculated as a function of temperature (4). Various DSC analysis were performed for the pure materials as well as for their eutectic mixtures. Table 2 depicts the technical features of the DSC equipment. The setup

temperature of the DSC was at 40°C, so the material was already melted when it was inserted in the chamber for thermal cycling. For this reason, it was attempted to cycle the materials three times in every test by heating, cooling, and heating the material again with the same scanning rate (°C/min). For industrial applications of PCM, it is essential to evaluate the thermal performance over a simulated lifetime. The samples underwent manual thermal cycling for 200 thermal cycles, which represent 6 months of lifetime in an application. DSC tests were performed for both 50, 100, 150, and 200 thermal cycles.

Table 2: Technical features of TA DSC Q2000 instruments (11)

Series	Q2000
Temperature range	-180 - 725
Calorimetric accuracy	±0.05%
Thermocouple	T-zero

$$q = C_p (dT/dt) + f (T,t) \quad (4)$$

q= heat flow, C_p= specific heat capacity, dT/dt= heating rate, f(T,t)= kinetic response at specific time and temperature

IV. RESULTS AND DISCUSSION

A. Determination of thermal properties of pure PCM

1) Short term thermal performance of PCM

Thermal properties of pure PCM measured with DSC technique are presented in Figure 1. The samples of the pure materials were examined within a temperature range of -30 to 80°C with a scanning rate of 1.5°C / min in a dynamic mode. The mass of each samples was 6 mg. The melting and freezing points are the peak temperatures at the maximum/minimum points in the melting and freezing curve. In every DSC analysis, the melting/freezing point is highly dependent on the scanning rate and the preparation of the sample. The scanning rate has a significant effect on the thermal shifts in the heating and cooling curves. A scanning rate of 1.5°C / min was selected to provide high resolution of the melting and freezing peaks. The DSC thermogram in Figure 1b illustrates a thermal shift of 2.5°C between the melting and freezing curve for the organic paraffin RT18. The same phenomenon is depicted in the DSC thermogram in Figure 1a shown that for the organic non-paraffins the thermal shift reaches 3.5°C for PT15 and 4.5°C for PT18. The DSC thermogram for coconut oil shown in Figure 1c illustrates that the thermal shift is the highest amongst the examined materials reaching 15.5°C. No thermal shift is observed for RT15 (Figure 1b) and tamanu oil (Figure 1c). A vast deviation is applied in the case of the selected salt hydrate, and it may be an outcome of the material's degradation (Figure 1d). The examined thermal properties of the pure materials (Table 3) are compared with the thermal properties provided by the manufacturers (Table 1). The expanded uncertainty for each experimental measurement is ±0.1%. The calculation of the relative error (%) was conducted with the use of equation (5) according to [17].

Relative error (%) = |(measured value - expected value)/expected value| (5)

The relative errors calculated for the melting and freezing enthalpies are 40% and 41% for PT 15, 37% and 39% for PT 18, 37% and 36% for RT15, and 45% and 44% for RT18. The relative errors for melting and freezing enthalpies for SP15 reached 99% due to the close to zero experimental melting and

freezing enthalpies. No value was provided from the manufacturer of Tamanu oil and coconut oil for the theoretical enthalpy (J/g), and thus the relative errors could not be calculated. The relative errors calculated reflect that the experimental latent heats are in all examined cases lower than the latent heats provided by the manufacturers.
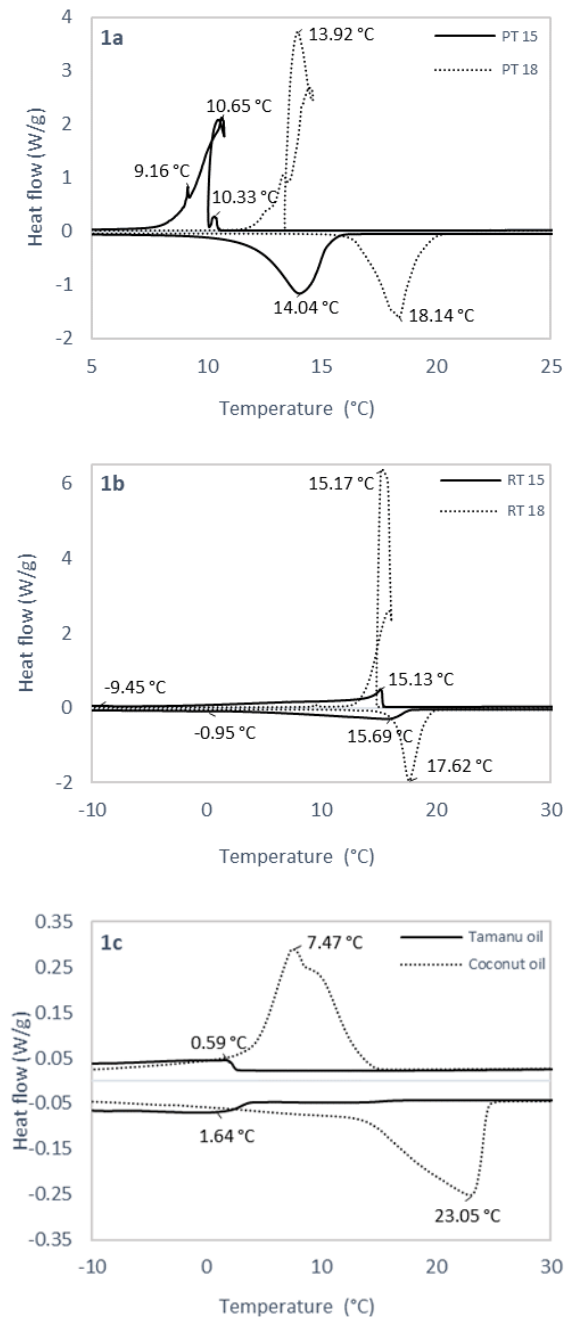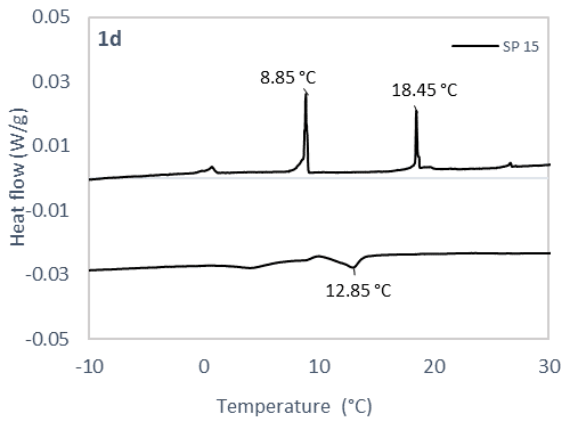
Figure 1: DSC thermogram of a)PT 15, PT 18 b) RT15, RT18, c) tamanu oil and coconut oil d) SP15 in their 1st thermal cycle.
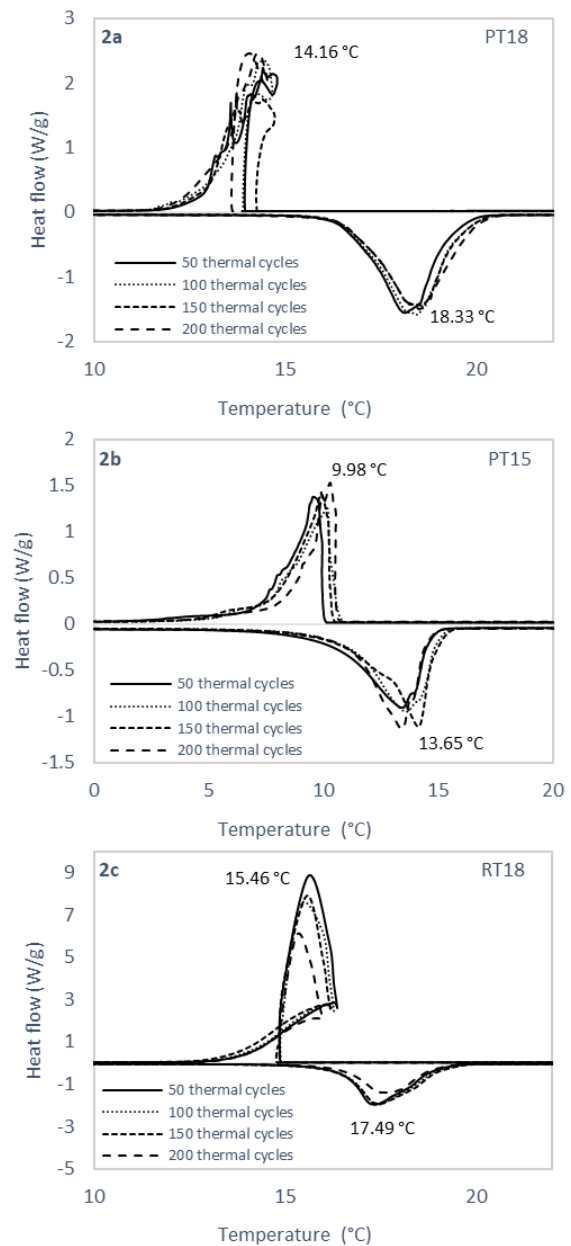
Table 3: Differential scanning calorimetry analyses for PT15, PT18, RT15, RT18, SP15, tamanu oil and coconut oil in their 1st thermal cycle.

Material	Melting Point (°C)	Latent heat (J/g)	Freezing Point (°C)	Latent heat (J/g)
PT 15	14.04	110.00	9.16	107.00
			10.65	
			10.33	
PT 18	18.14	121.50	13.92	117.20
RT15	15.69	97.38	15.13	99.12
	-0.95		-9.45	
RT18	17.62	141.80	15.17	144.00
SP 15	12.85	0.19	18.85	0.13
			8.85	0.31
Coconut oil	23.05	46.94	7.47	57.19
Tamanu oil	0.59	3.98	1.64	4.89

2) Long term thermal performance of PCM

The long-term performance of five PCM samples was evaluated under 200 thermal cycles. The samples of the five selected materials were examined within a temperature range of -30 to 80°C with a scanning rate of 1.5°C / min in a dynamic mode. Figure 2 illustrates the DSC thermogram of the five PCM, which underwent manual thermal cycling for 200 thermal cycles. The obtained data using DSC technique is shown in Table 4. The result indicates a nearly constant latent heat (J/g) throughout the heating and cooling cycles for all the examined materials. The thermal properties indicated by the long term thermal performance DSC analysis (Table 4) are in line with the first thermal cycle's DSC analysis results (Table 3). The experimental results for the melting and freezing points of the organic non-paraffins PT18 (Figure 2a), PT15 (Figure 2b) are outside the temperature range 15-20°C. According to this analysis, it is evident that RT18 is a suitable PCM candidate for a thermal energy storage application that operates in a temperature range within 15-20°C. RT18 (Figure 2c) in all data sets for 50, 100, 150, and 200 thermal

cycles performs stably in terms of variation in melting/freezing temperature (°C) and enthalpies (J/g). More specifically, the melting temperature of RT18 varies between 17.33 to 17.72 °C, and the melting enthalpy varies between 101.7 to 154.5 J/g. Correspondingly the freezing temperature for RT18 varies within 15.3-15.61 °C and the freezing enthalpy 101.7-155.1 J/g. However the melting and freezing latent heat of RT18 for the 200 thermal cycles has been decreased by 33% compared to the 50,100 and 150 thermal cycles. In the cases of PT15, PT18 and RT18, the supercooling effect is detected: the PCM solidifies at a lower temperature. In the melting and freezing curves of RT15 (Figure 2d), two peaks for the phase change temperature are observed for the 50,100, and 150 thermal cycles and three peaks for the 200 thermal cycles. The melting and freezing temperatures for tamanu oil (Figure 2e) are out of the range 15-20°C. The thermal cycling of the pure materials was conducted a period equivalent to 6 months, and this period may not be sufficient for the evaluation of the long term thermal performance.
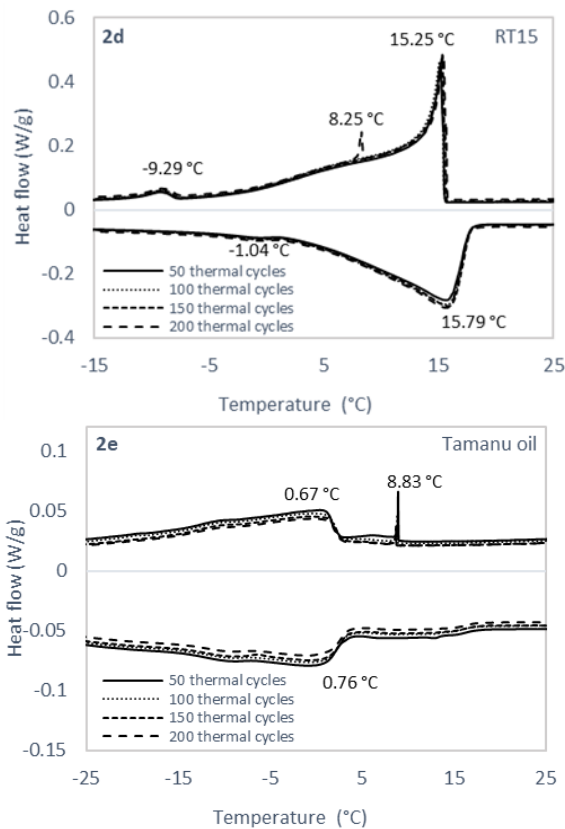
Figure 2: DSC thermogram of a) PT 15 b) PT 18 c) RT15, d) RT18, e) tamanu oil in 50, 100, 150 and 200 thermal cycles.

Table 4: Differential scanning calorimetry analyses for PT15,PT18, RT15, RT18 and tamanu oil in 50, 100, 150 and 200 thermal cycles.

Material	Melting point (°C)	Latent heat (J/g)	Freezing point(°C)	Latent heat (J/g)
50 thermal cycles				
PT 18	18.12	121.80	14.42	117.5
PT 15	13.42	105.50	9.54	105.30
RT18	17.33	144.80	15.61	144.30
RT15	15.76 / -1.00	94.27	15.15 / -9.32	93.14
T.oil	0.67	3.06	0.79 / 8.93	4.65
100 thermal cycles				
PT 18	18.40	129.90	14.41	126.60
PT 15	13.61	103.10	10.15	102.90
RT18	17.44	146.10	15.37	148.30
RT15	15.72 / -0.98	99.15	15.11 / -9.49	98.13
T.oil	0.84	3.49	0.64 / 8.86	4.64
150 thermal cycles				
PT 18	18.30	122.50	13.73	118.40
PT 15	14.15	109.90	9.93	104.9
RT18	17.46	154.50	15.57	155.10

Material	Melting point	Latent heat	Freezing point	Latent heat
RT15	15.96 / -1.00	98.44	15.32 / -9.20	96.58
T.oil	0.58	3.74	0.70 / 8.69	4.39
200 thermal cycles				
PT 18	18.50	125.00	14.08	120.70
PT 15	13.40	103.40	10.29	101.70
RT18	17.72	101.70	15.30	104.80
RT15	15.71 / -1.18	98.49	15.40 / 8.25 / -9.16	98.30
T.oil	0.96	3.35	0.54	4.71

B. PCM water emulsions and PCM polymers

The results presented in this section are focused on the two organic paraffins water emulsions in a 5:1 ratio with the addition of a 5%v/v Tween 80 in the final solution. Thermal properties of PCM water emulsions measured with DSC technique in a temperature range 5-80°C with a scanning rate of 10°C/min. The paraffins-water emulsions were prepared by mixing water and the PCM (RT15 and RT18) in a ratio of 1:5 followed by addition of 5%v/v of tween 80 in the final solution. The water, paraffins, and tween were then mixed with a T25 digital ULTRA-TURRAX disperser [18] for homogenizing the mixture. The charging and discharging characteristics of the organic paraffins - water emulsions are presented in Figure 3. From Table 5 we can observe that the water emulsion of RT18 can be used for a thermal energy storage application that operates in a temperature range within 15-20°C. The latent heat for this emulsion is around 180 KJ/kg, but the performance of the emulsion is not evaluated under repeated thermal cycles. For this reason, it can not be concluded whether this PCM-water emulsion is suitable for an industrial application. In the case of RT15 - water emulsion, it is evident that there are two peaks for both the melting curve and three peaks in the solidification curve, see Figure 3. The multiple peaks in both the exothermic and endothermic curves of RT15-water emulsion indicate the polymorphic structure of the PCM. In both RT15 and RT18 water emulsions, the solidification is observed at a lower temperature than the melting temperature of the PCM. It is suggested that the materials performance should be further evaluated since the water PCM emulsion may have undergone phase separation.

The combinations for the formation of PCM polymers with the two organic paraffins RT15 and RT18 are illustrated in Figure 4a and Figure 4b. Figure 4a shows the melting curves for the first polymer solutions with PEGda and BPO. The solid liquid crosslinked polymer RT15-PEGda-BPO stores heat at the favorable temperature range and, at the same time, maintains its solid structure. In the case of RT18-PEGda-BPO, the freezing curve contains three freezing points out of the range 15-20 °C. The latent heat for RT15-PEGda-BPO varies between 39-50 J/g and for RT18-PEGda-BPO between 76-63 J/g. Table 6 depicts the melting temperatures and heat storage capacities of the examined PCM polymers.

Figure 4b and Table 6 present as well the thermal properties of the PCM emulsions polymers with PVP as synthesizer and APS as initiator. A vast difference is indicated in the melting

and freezing point for both organic paraffins polymers in this combination. Another point underlined is that the latent heat in this case varies between 0-2 KJ/kg for both organic paraffins. The low latent heat is justified by the percentage of the PCM in the mixture, which is 0.5% w/v in the final polymer solution. The PCM polymers' performance for relevant LHTES applications should be further evaluated under repeated thermal cycles. The encapsulation of a PCM layer in an electrospun PCM fiber matrix followed the current experimental study.
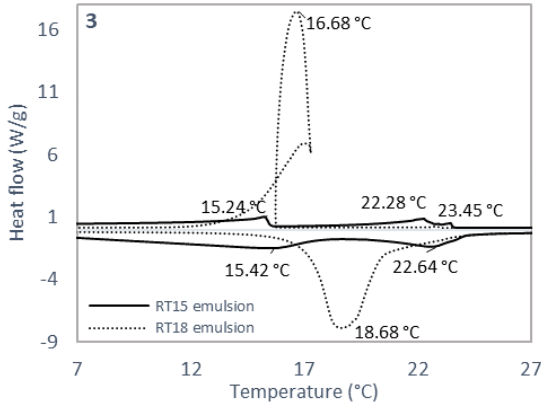

Figure 3: DSC thermogram of RT15, RT18 in water emulsions in their 1st thermal cycle.

Table 5: Differential scanning calorimetry analyses for RT15, RT18 in water emulsions in their 1st thermal cycle.

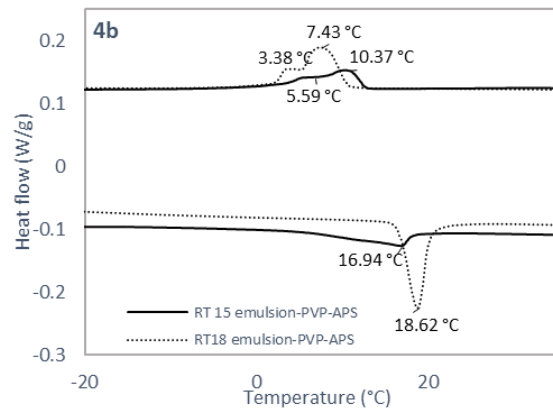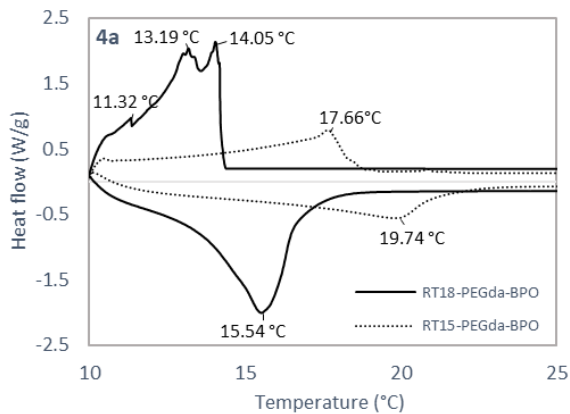| Material	Water: RT 15 (1:5)	Water: RT 18 (1:5)
Melting point (°C)	15.42	18.68
	22.64	
Latent heat (J/g)	87.70	180.70
Freezing point (°C)	15.24	16.68
	22.28	
	23.45	
Latent heat (J/g)	50.06	180.4
Mass (mg)	8.76	4.38

Figure 4: DSC thermogram of RT15-PEGda-BPO, RT18-PEGda-BPO, RT15 emulsion-PVP-APS, RT18 emulsion-PVP-APS in their 1st thermal cycle

Table 6: Differential scanning calorimetry analyses for RT15-PEGda-BPO, RT18-PEGda-BPO, RT15 emulsion-PVP-APS, RT18 emulsion-PVP-APS in their 1st thermal cycle.

Material	RT15-PEGda-BPO	RT18-PEGda-BPO
Melting point (°C)	19.74	15.54
Latent heat (J/g)	50.89	76.20
Freezing point (°C)	17.66	11.32
		13.19
		14.05
Latent heat (J/g)	39.58	63.87
Mass (mg)	7.28	4.00
Material	RT15 emulsion-PVP-APS	RT18 emulsion-PVP-APS
Melting point (°C)	16.94	18.62
Latent heat (J/g)	0.21	2.13
Freezing point (°C)	5.59	3.38
	10.37	7.43
Latent heat (J/g)	1.16	2.17
Mass (mg)	11.83	5.76

V. CONCLUSION

In a phase-change TES, energy is stored and released by a reversible change of state, such as a solid-liquid phase transition. The current study discusses the merits of seven PCM candidates and targets the selection of a suitable PCM for implementation in a LHTES system. It is essential to demonstrate the thermal properties of PCM under repeated thermal cycles, which represents the PCM life in technical implementation. Within the selected PCM, which were analysed with DSC technique in this study, the commercial available organic paraffin based RT18 is a suitable PCM candidate for a thermal energy storage application. RT18 operates in a temperature range within 15-20°C, and the latent heat varied between 101-155 J/g for the 200 thermal cycles. The water emulsion of RT18 proved to be suitable for a thermal energy storage application that operates in a temperature range within 15-20°C. The latent heat for this emulsion is around 180 J/g and the melting and freezing

points 18.68 and 16.68°C, respectively. It is suggested to evaluate the PCM-water emulsion under repeated thermal cycles. The solid liquid crosslinked polymer RT15, in combination with PEGDa and BPO, resulted in a melting point within the desirable temperature limit. Although the thermal properties of the selected PCM candidates are evaluated, it should be underlined that the sample sizes at the range of mg make the analysis insufficient for a large-scale application.

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