Uncertainty of Thermal Characterization of Phase Change Material by Differential Scanning Calorimetry Analysis

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Abstract— **Phase change materials (PCMs) are used in many thermal heat storage applications because of their high heat capacity and desirable phase change temperatures. Recently many PCM products have been designed and commercialized to meet different thermal energy storage requirements. In order to correctly design commercial energy storage products, accurate knowledge of the thermal properties of the PCMs are essential. Heat flux Differential Scanning Calorimetry (DSC) is the most widely used method to characterize PCMs. However the existing standards and methods for calorimetry, which were designed for other materials, can lead to large errors if applied to PCMs. For example, the influence of non-equilibrium thermal gradients is very strong for PCMs. As a consequence, results are often systematically shifted to higher or lower values. Moreover, significant differences in the published data for the same material have been reported by many researchers. In this paper, DSC measurements were carried out parametrically with the goal of analyzing the sensitivity and resolution of the measurement technique under varying conditions. Several different types of PCMs were tested with varying sample sizes and heating rates. The phase transition temperature, onset temperature and the enthalpy of fusion were measured. The experimental results differ considerably for different measurements conditions. The results proved that using the correct heating rate and sample mass is necessary to obtain results with sufficient accuracy for PCM samples in a DSC. The optimum measurement condition was identified, and agreement between the obtained results and NIST data was demonstrated.**

Keywords— *Phase change material; Thermal properties; Differential scanning calorimetry; Thermal energy storage; Fatty Acids; PCM.*

I. INTRODUCTION

There are three common ways to store thermal energy: sensible heat, latent heat and chemical energy [1, 2]. Phase change materials (PCMs) are latent heat storage materials. Energy per unit mass is stored during melting and released during freezing at constant temperatures [1, 3]. PCMs have proven ability to improve the performance and reliability of thermal energy storage applications [4]. Recently PCMs have received great interest because of their high energy storage density and their ability to maintain a constant temperature while absorbing heat during melting and releasing heat when solidifying [3-6]. Specific interest has developed in the field of buildings, thermal insulation, solar energy systems, and greenhouses [6].

Accurate knowledge of the thermophysical properties of PCMs is very crucial for the correct design of commercial storage products. Therefore the temperature range of the phase change transition and the energy storage density must be determined accurately. Those parameters decide the system's capacity, temperature range and size. As an example, Dolado et al. [7] proved that reducing the uncertainty of the PCM melting temperature from ±1°C to ±0.25°C results in a 25% reduction in the uncertainty of the average heat exchange rate. PCMs are also widely used in buildings where the temperature change is small during the day [8], and for an effective PCM design in such applications the uncertainty should be known within ±10% for the specific enthalpy and ±0.5°C for melting temperature [9].

Differential scanning calorimetry (DSC) is a widely used method to characterize the thermal properties of PCMs [4, 6, 10-12] among other methods like the differential thermal analysis (DTA) and T-history method [13]. Ramping temperature at constant heating rate (Dynamic Method) is the most common operating mode for DSC [7, 8]. When measurements are performed in a DSC, some errors arise from measurement of the heat transferred to the calorimeter from the samples [14]. The effect of these uncertainty sources is thus of interest, particularly for PCMs where the melting enthalpy is very high and thermal conductivity is low [15, 16]. As more applications of PCMs have approached the commercial stage a precise knowledge of the thermophysical properties of PCMs has become more important. The lack of a clear standard for DSC measurements of PCMs has resulted in differing measurements being reported in the literature [9-13]. The existing standards and methods for calorimetry which were designed for other materials, can lead to a large errors if applied to PCMs [9]. Results are often systematically shifted to higher or lower values.

In the present paper several DSC measurements have been carried out to prove that these differences are not due to variability of materials, but due to problems with the measurement mode itself. More specifically, due to the strong influence of heating rate and sample mass on the DSC results for PCMs. In this study five heat storage materials, namely Palmitic Acid, Myristic acid, Lauric Acid, Capric acid, and Lauryl Alcohol are tested. These materials are selected based on their melting temperatures (24 - 62 °C), which are suitable for many low temperature heat storage applications, high latent heat, ease of availability, high specific heat, nontoxicity, low cost, and the availability of their thermophysical properties in literature [17]. The phase transition temperature, onset temperature and the enthalpy of fusion have been analyzed at different heating rates and sample weights to show that the experimental results differ considerably at different measurements conditions.

II. MATERIALS AND METHODS

A. Phase Change Materials

The PCMs used in this work have a melting temperatures ranging from 24°C - 62°C. Five heat storage materials have been selected. Myristic acid (99% purity), Capric acid (99% purity), Lauryl Alcohol (99% prutiy), Palmitic acid (99% Purity) and Lauric acid (99% Purity) were procured from Sigma Aldrich. The materials and their thermal properties are given in various references which are collected by the National Institute of Standards and Technology (NIST) [18]. Table 1 shows the chemical data and values used in this paper as a reference.

TABLE 1. CHEMICAL DATA FOR REFERENCE PCM

Name	Scientific Name	Molecular Formula	CAS Number	Melting Temp. [°C]	Melting Enthalpy [J g^{-1}]
Myristic Acid	Tetradecanoic acid	$C_{14}H_{28}O_2$	544-63-8	54.17	197.17
Capric acid	n-Decanoic acid	$C_{10}H_{20}O_2$	334-48-5	31.4	161.36
Lauryl Alcohol	1-Dodecanol	$C_{12}H_{26}O$	112-53-8	24.15	216.33
Palmitic Acid	Hexadecanoic acid	$C_{16}H_{32}O_2$	57-10-3	62.51	209.46
Lauric Acid	Dodecanoic acid	$C_{12}H_{24}O_2$	143-07-7	43.83	181.18

B. Experimental Process

DSC measurements were performed on a Seiko DSC6200 colorimeter (Seiko Instruments) and DSC2010 (TA instruments) with aluminum sample pans (TA pans #900786.901, TA Lids #900779.901). The temperature repeatability, calorimetric precision and calorimetric sensitivity for both of the calorimeters is ±0.1°C, 1μW and ±1% respectively. Temperature and enthalpy calibrations were carried out using a standard manufacturer-supplied indium sample. The experimental test samples were taken by a calibrated pipette after the PCMs were melted and stirred evenly. Sample mass was controlled in the sample pan using a precision electronic balance (Mettler MX5, METTLER TOLEDO, Switzerland) at a precision of 0.001mg. For each PCM, sample masses of 2.5, 5.0, 7.5 and 10mg were deposited in the sample pans. All samples were subjected to heating rates of 0.5, 1.0, 3.0 and 5.0 °C min^{-1}.

C. Data Analysis

The melting onset temperature, (T_o), melting temperature (T_m) and latent heat or melting enthalpy, (ΔH_m) were determined from the DSC thermograms by computer software using MUSE standard analysis (Seiko Instruments) and Universal Analysis 2000 (TA instruments). The temperature at which the PCM starts to melt, T_o, was obtained from the point of maximum slope of the leading side of the transition peak and extrapolating the heat flow base line on the same side. ΔH_m was calculated as the area under the peak by numerical integration. The experimental data was compared with NIST data to reveal the effect of different measurement conditions.

To easily compare the experimental data with the reference data from NIST, all the reference values were considered as a baseline value (P_{ref}). The variations between the experimental values (P_{exp}) and the reference values (P_{ref}) were presented as a percentage error for each parameter ($P\%$), and calculated by (1) as follows:

$$P\% = \left|\left(\frac{P_{exp}}{P_{ref}} - 1\right)\right| \times 100\% \qquad (1)$$

III. RESULTS AND DISCUSSION

In order to examine the influence of sample mass and heating rate on the accuracy of the results, measurements were carried out for five PCMs. First, measurements were performed using the Seiko DSC6200 for Myristic acid and Capric acid. Each PCM was tested at four different sample masses (2.5, 5, 7.5 and 10mg), and each sample was subjected to four different heating rates (0.5, 1, 3 and 5 °C/min). Palmitic acid and Lauric acid measurements were performed with the Seiko DSC6200 at only sample mass of 7.5mg at varying heating rates. In order to check the reproducibility of the measurements Lauryl Alcohol measurements were performed in a different laboratory using a different DSC instrument (DSC2010, TA instrument) under the same measurements conditions. A total of 56 experiments were performed. Table 2 represents the DSC experimental data for PCMs with various heating rates as function of sample mass. The melting onset temperatures, melting temperatures and melting enthalpies are presented.

Figures 1-5 display the DSC heat flow curve for five PCMs chosen to display the shapes of the peaks determined in dynamic mode at different heating rates and sample masses. The figures show that the test data and peak shapes vary significantly at different measurement conditions for all the samples. The shape of the DSC curve and the thermal behavior of PCMs in a DSC depends significantly on the heating rate and sample mass. As the onset temperature (T_o) values are not affected significantly, the melting process starts at the same temperature for all cases but continues its evolution over a wider range of temperatures for higher heating rates or sample masses. For example, as one can see in Fig. 3, the end of the phase change peak is shifted from 26.2°C (2.5mg) to about 29.3°C (10mg). It is also noted that the melting peak temperature (T_m) increases when increasing heating rate or sample mass.

The lower heating rates of 0.5°C/min and 1°C/min provide a good resolution for phase transition temperature. However, the sensitivity of the latent heat measurement is lower, especially for small sample masses of 2.5mg. The increase in latent heat sensitivity with increased heating rate or sample mass is caused by a much larger heat flow signal associated with a very small corresponding increase in signal noise. It is therefore important to choose an optimum heating rate and sample mass to maintain good sensitivity and resolution at the same time.

TABLE 2. DSC MEASUREMENTS OF ONSET TEMPERATURE, MELTING TEMPERATURE, AND HEAT OF FUSION

| Materials		T_o [°C]				T_m [°C]				ΔH_m [J g^{-1}]			
Heat Rate [°C/min] ⟶		0.5	1	3	5	0.5	1	3	5	0.5	1	3	5
Myristic Acid	2.5mg	51.07	51.42	51.40	51.90	52.57	52.97	53.66	55.37	191.1	191.3	193.1	193.5
	5 mg	51.45	51.40	51.16	51.49	52.67	52.87	53.62	54.59	193.3	194.3	196.5	197.5
	7.5mg	51.71	51.43	51.00	51.29	52.87	53.32	54.21	54.87	193.4	194.7	198.5	199.1
	10 mg	51.64	50.79	51.22	51.34	52.87	53.65	54.76	55.55	194.0	196.1	198.5	199.8
Capric Acid	2.5mg	29.5	29.27	29.15	29.59	30.16	30.27	30.64	31.28	156.3	157.9	159.0	161.2
	5 mg	29.19	29.22	29.27	29.17	30.19	30.44	31.10	31.52	158.8	159.7	161.2	162.3
	7.5mg	29.51	29.31	31.82	32.28	30.42	30.85	31.82	32.28	161.4	161.7	162.2	165.5
	10 mg	29.54	29.74	29.47	28.92	30.64	31.18	32.10	32.30	161.7	162.8	164.2	165.8
Lauryl Alcohol	2.5mg	20.55	20.89	20.98	22.01	21.87	22.34	23.05	23.57	202.2	208.2	215.1	218.1
	5 mg	20.90	20.83	20.9	21.08	21.87	22.34	23.38	23.70	204.6	212.1	215.4	218.5
	7.5mg	20.88	20.78	20.88	21.03	22.13	22.94	23.83	24.51	206.2	213.8	216.5	218.8
	10 mg	20.88	20.88	20.82	20.82	22.04	22.63	23.90	24.89	207.1	214.2	219.4	221.0
Palmitic Acid	7.5mg	60.83	60.50	60.28	63.50	61.69	62.08	62.93	63.87	205.8	207.7	209.3	214.7
Lauric Acid	7.5mg	40.64	40.78	42.72	40.79	42.56	42.78	43.98	44.35	177.3	178.0	180.8	183.4
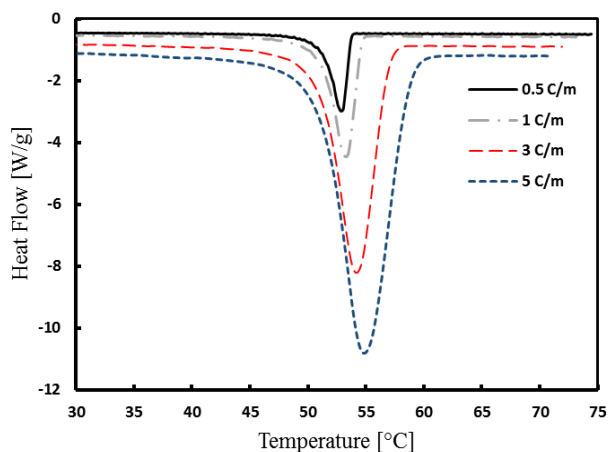
Figure 1: Results of dynamic DSC measurements of 7.5 mg of Myristic Acid at various heating rates
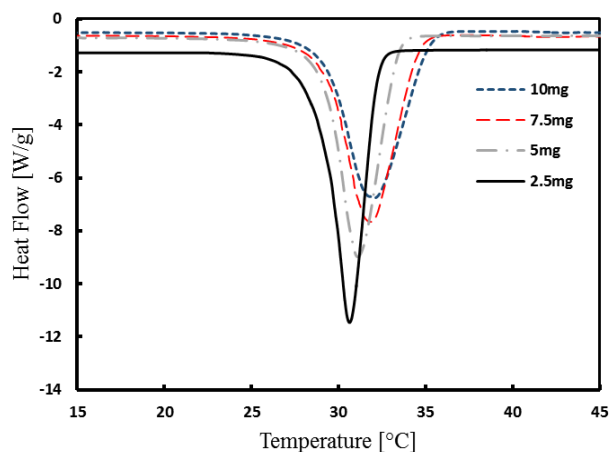

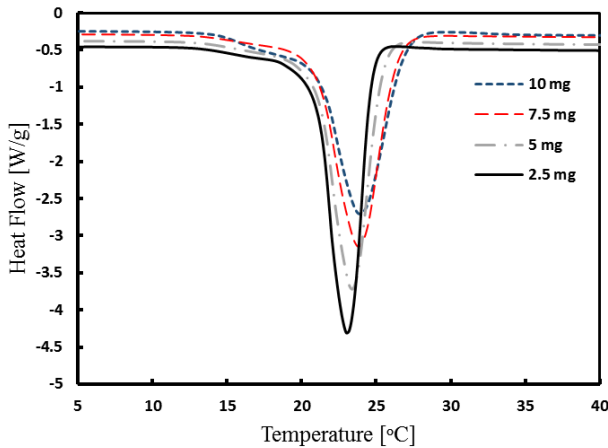Figure 2: DSC measurements of Capric Acid at 3°C/min at various samples mass

Figure 3: Results of dynamic DSC measurements of Lauryl Alcohol at 3°C/min at various samples mass
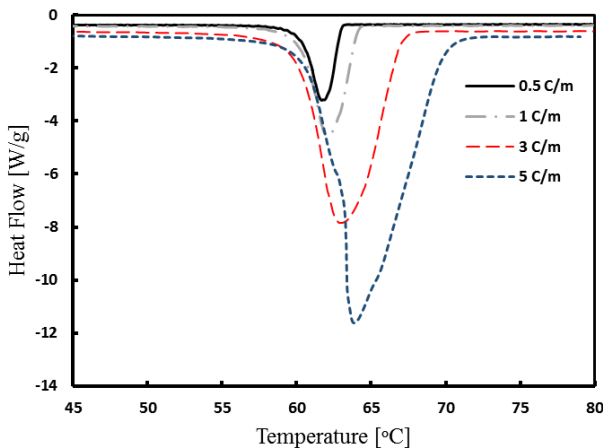

Figure 4: Results of dynamic DSC measurements of 7.5 mg of Palmitic Acid at various heating rates
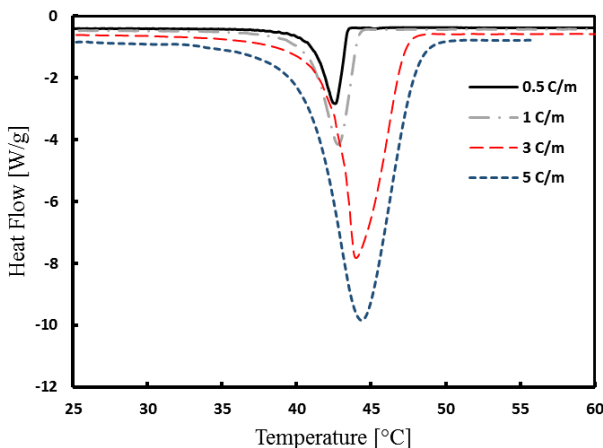

Figure 5: Results of dynamic DSC measurements of 7.5 mg of Lauric Acid at various heating rates

Figure 6 displays the melting temperatures of five PCMs (Myristic acid, Capric acid, Palmitic Acid, Lauric Acid, and Lauryl Alcohol) at varying heating rates and sample masses. The main finding is a significant difference in T_m between measurements. Figure 6-a shows the melting temperature results of PCMs of a same sample mass (7.5 mg) at varying heating rates. Figure 6-b shows the melting temperature results for PCMs at the same heating rate (3°C/min) but varying sample mass. As clearly seen in Fig. 6, the melting

temperatures significantly increase with the increase in heating rate or sample mass. For example for Capric Acid, T_m was observed at 30.64°C for a sample mass of 2.5 mg at 3°C/min heating rate, and shifted to higher temperatures with higher sample mass to 31.10, 31.82 ,and 32.10 °C at 5, 7.5, and 10 mg, respectively. It can be seen in Table 1 that similar behavior in T_m is observed when varying the heating rate for other constant sample masses (2.5, 5, and 10mg). The same trend can also be observed when varying the sample mass under other constant heating rates (0.5, 1, and 5°C/min). Samples with low heating rates (0.5°C/min) deviate strongly from the NIST reference values, especially at lower sample masses.
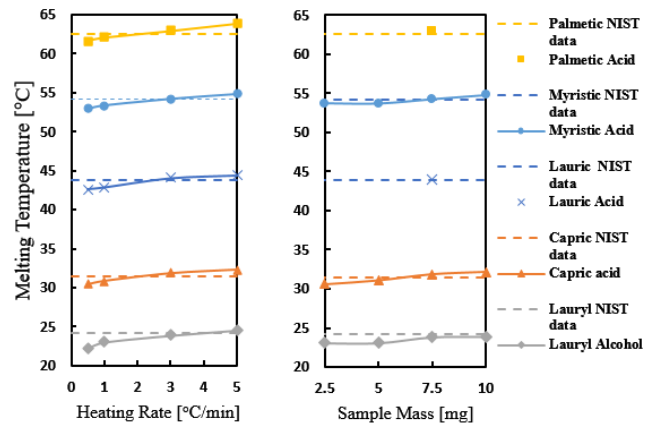

Figure 6: The effect of varying heating rate (Left) and sample mass (Right) on the measured melting temperature

The trend in ΔH_m values determined under varying heating rates and sample masses is shown in Fig. 7. In Fig. 7-a, results for PCMs of the same sample mass (7.5mg) at varying heating rates are compared. In fig. 7-b, the experimental results for PCMs at the same heating rate (3°C/min) but varying sample masses are compared. For higher heating rates and sample mass, a slight increase in ΔH_m was observed. In general, the enthalpy values do not diverge too much from the reference NIST values with varied measurement conditions, with the exception of lower masses (2.5mg). At lower masses the values deviate strongly, especially at low heating rates. When compared to T_m, The increase in ΔH_m is showing a lower dependency on heating rate and sample mass. The average variation of all the ΔH_m measurements from the NIST reference values are only within a ±1.64%, whereas the average absolute change for T_m is within ±0.95°C. This is considered to be a significant error for PCM applications, where the melting temperature must be determined with a very high accuracy due to its very important role in the system behavior. On the other hand, a ±1.64% uncertainty for the melting enthalpy is within the typical uncertainty of the DSC method [19, 20]. However, as is clearly seen in Table 1, even lower uncertainties still can be achieved if correct measurement conditions are chosen carefully.
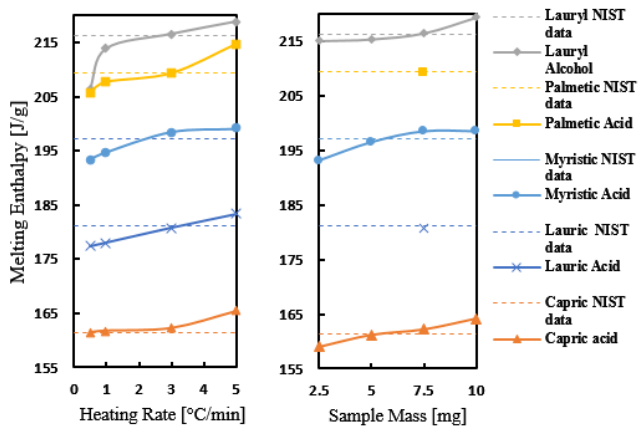
Figure 7: The effect of varying heating rate (Left) and sample mass (Right) on the measured melting enthalpy

The clear trend of peaks shifting toward higher temperatures with increased sample mass or heating rate can be explained by the internal thermal gradient inside the PCM samples. The thermal gradient in the crucible is created as the constant heating rate is supplied, and the effect increases with increasing heat rate or sample size. This thermal gradient effect is very strong for PCMs in particular, since PCMs have a high thermal energy storage combined with a low thermal conductivity and heat diffusivity [16]. As a consequence, a gradient of temperature is developed throughout the material and the temperature is not uniform throughout the sample. The measured temperature by the DSC sensor, which is in direct contact with the specimen surface, is higher than the average sample temperature - especially at higher heating rates and sample mass. In this situation, a thermal lag exists between the thermocouples and the sample, and the instrument response becomes slow. Since the temperature difference between the average sample temperature and DSC sensor is due to the time necessary for the heat flux to dissipate through the sample, lower heating rates and sample masses, up to a certain limit, can help to reduce these effects.

When measurements on PCMs are performed in a DSC some sources of error arise from the heat transferred to the calorimeter from the samples, especially for materials of high enthalpy storage [21, 22]. With materials like PCMs in particular, the effect of these uncertainty sources becomes much more significant. As mentioned above, the shift in transition temperatures is very high for substances like PCMs, in which the heat diffusivity and thermal conductivity are very low. PCMs tend to melt over wider range of temperatures, and the shift in transition event is significant under varying measurements conditions. This effect is less significant for other materials, especially for pure materials like indium samples. For measurements by Poel and Mathot [23] of indium samples studied at varying heating rates and sample masses, the maximal increase in indium melting temperatures is only within ΔT=0.5°C. These samples were studied within the same range of interest for the current study in terms of heating range (1-5°C/min) and sample mass of (1 to 8 mg), and a maximal increase of only 0.4°C for melting temperature was observed when sample mass increases from 1 mg to 8 mg at heating rage of 5°C/min. On the contrary, as seen in Table 1

for PCMs, the change in melting temperatures in some cases has reached a value of ΔT=3.8°C, as in the case of Myristic acid when heating rate increased from 0.5 to 5°C/min with sample mass of 2.5 mg.

A possible explanation for the slight increase in melting enthalpy at increased heating rate or sample mass is that at higher heating rates and samples mass, as seen in Table 1, the transition occurs over a wider range of temperature due to the internal thermal gradient. Therefore liquid PCM may exist at higher temperatures than the melting temperature, resulting in some sensible heat being included in the latent heat measurement. Hence melting is measured to be at higher temperature with a higher heat of fusion. Explained in another way as supported by Kasap's [24] observations, the increase of melting enthalpies with higher DSC heating rates or sample masses is due to the instrument sensitivity and resolution. For smaller sample mass or heating rate, the instrument's resolution is improved at the cost of its sensitivity. The DSC sensitivity can be expressed by the maximum heat flux value corresponding to the peak height [25, 26]. This effect of heating rate and sample mass on the DSC resolution and sensitivity is confirmed experimentally in Fig. 8. The sensitivity of the calorimeter is found to be directly proportional to the sample mass and heating rate.
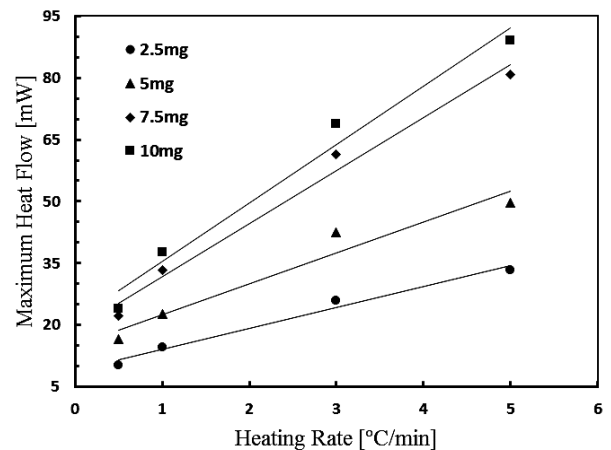

Figure 8: Maximum heat flux for varying heating rates and sample masses of Myristic acid

Figure 9 shows the experimental heat flow response to varying heating rate for the same material. At lower heating rates, a low rate of enthalpy change is obtained. In another words, the endothermic reaction of phase transition is expanded over a longer period of time. For example at heating rate of 5°C/min the phase transition from solid to liquid occurs in less than 6 minutes, compared to about 11 minutes at a lower heating rate of 0.5°C/min. Since the heat flux measurement is lower for the lower heating rate, the measurement uncertainty is higher and the instruments sensitivity is lower. Supported by these observations and according to the literature [27], a lack of accuracy results.
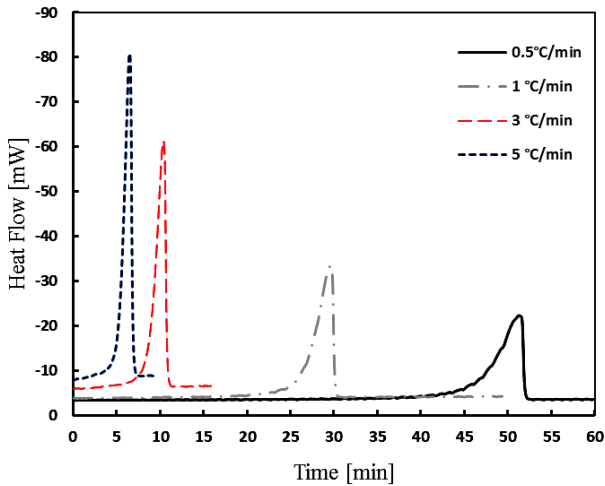
Figure 9: Effect of heating rate on the DSC resolution and sensitivity

It can be concluded that with an increase in heating rate or sample mass, transition temperatures and enthalpy values are systematically shifted to higher values. Also, it is suggested that very large samples and high heating rates are not recommended, since a temperature gradient is developed through the sample and the rate of heat transferred by conduction must be takin into account. Thereby, the thermal lag effect on transition events becomes much more significant. Lower heating rates and sample masses can help to reduce the thermal gradient effect and help to distinguish two separate transition peaks in some cases. However, use of slow heating rates or very small sample masses results in a simultaneous decrease in the DSC sensitivity due to the weaker signal. This causes a decreased accuracy in the enthalpy determination. Furthermore a very slow heating rate increases the time needed for experiments, and the results for very small samples may not be representative of the bulk material.

Table 3 shows the absolute variation for the experimental melting temperature, and the percentage variation for the melting enthalpy by applying equation (1). The variation of experimental data is compared to the NIST reference values. To provide much more representative data related to the heating rate and sample mass, the average deviation of T_m and ΔH_m of all the PCMs combined together at varying heating rates and sample's mass is presented in Table 4. The average deviation was calculated by taking the average of the deviation of the individual results for all of the measurements. It is seen that uncertainties in T_m and ΔH_m values are relatively high at very low heating rates. As previously mentioned, This can be explained by the decreased "signal to noise" ratio at very small heating rates [28] and by the decreased sensitivity with lower heating rates [24, 26]. A comparatively high heating rate is recommended for an accurate determination of the enthalpy, but only up to a certain limit where the thermal gradient effect is minimal.

TABLE 3. THE VARIATION FOR THE EXPERIMENTAL MELTING TEMPERATURE AND MELTING ENTHALPY OF ALL THE PCMS.

Heat Rate [°C/min] →	$\pm T_m$				$P_{\Delta Hm}$			
	0.5	1	3	5	0.5	1	3	5
2.5mg	±1.71	±1.38	±0.79	±0.63	4.25%	2.96%	1.37%	0.93%
5 mg	±1.66	±1.36	±0.54	±0.33	2.99%	1.48%	0.29%	0.58%
7.5mg	±1.28	±0.82	±0.27	±0.76	2.10%	1.05%	0.31%	1.68%
10mg	±1.39	±0.75	±0.51	±1.01	2.03%	0.81%	1.28%	2.08%

TABLE 4. THE AVERAGE VARIATION FOR THE EXPERIMENTAL MELTING TEMPERATURE AND ENTHALPY OF ALL THE MEASUREMENTS.

Heat Rate [°C/min] →	$P_{Tm+\Delta Hm}$			
	0.5	1	3	5
2.5mg	4.28%	2.99%	1.39%	0.95%
5 mg	3.04%	1.54%	0.34%	0.59%
7.5mg	2.14%	1.08%	0.32%	1.70%
10mg	2.08%	0.84%	1.30%	2.11%

Considering the average deviation of all the measurements of T_m and ΔH_m, it can be suggested that the most accurate results were obtained at heating rate and sample mass of 3°C/min and 7.5 mg respectively. At this measuring condition, the deviation in T_m and ΔH_m from the NIST reference values did not exceed ±0.27 and 0.31% for the melting temperature and heat of fusion, respectively. This suggests that this range of heating rate and sample mass is an appropriate choice to combine an accurate results for both of the melting enthalpy and melting temperature at the same time. For example, at this measurement condition a maximal difference of only ±0.42°C (0.14%) in T_m and ±1.33 J/g (0.67%) in ΔH_m was observed compared to the NIST reference values. Moreover, a sample size of 7.5 mg allows the bottom of most of the available standards pans to be completely covered with a thin layer of sample. At this measurements condition, a very good agreement with the NIST reference data is observed, and the shift in thermal events were minimal. Moreover, the averaged uncertainty in the determination of both T_m and ΔH_m combined together is only 0.32%.

A high heating rate mode helps to obtain a better accuracy for melting enthalpy, but at the cost of decreased resolution and an increase in thermal gradient effect and thermal lag. It is suggested that a smaller sample mass be used when DSC measurements are performed at high heating rate to reduce the drawbacks of the high heating rate and attain a high accuracy at the same time. Using smaller sample mass when an increased heating rate is applied will reduce the expected increase in the internal thermal gradient throughout the sample, avoid any extra thermal lag arising, and maintain the DSC resolution. According to Pijpers et al. [29], if the heating rate increased by a factor 'y', the sample mass has to be reduced by the same factor "y". In this way, an important aim is achieved: shorten the measuring time by using the high heating rate, attain a higher accuracy, and

avoid the drawbacks associated with the higher heating rate at the same time.

It must be mentioned that the correct choice of DSC measurements conditions is very crucial with PCMs. When incorrect results such as transition temperature range are used in the design of a PCM storage system, the phase change process may not be completed and the system will fail to store or release the total value of the heat of fusion. Accurate determination of phase transition parameters helps to design the system with an appropriate capacity, temperature range and size.

IV.   CONCLUSION

The study presents the effect of different measurement conditions on the uncertainty in the determination of the thermophysical properties of PCMs such as melting temperatures, onset temperatures, and melting enthalpy. The study demonstrates that for DSC measurements, of PCMs in particular, the sample mass and heating rate must be carefully selected due to their high latent heat combined with a low thermal conductivity. DSC results can be influenced considerably by the different measurement conditions, and sufficient accuracy can attained only if a correct heating rates and sample mass are chosen.

Samples of five PCMs each at different sample masses (2.5, 5, 7.5 and 10 mg) were examined at varying heating rates ranging from 0.5°C/min to 5°C/min. Experimental results show that:

- The onset melting temperatures are not affected by the heating rate and sample mass.

- The higher heating rates and sample masses shift the observed melting temperature to significantly higher temperatures and significantly widen the temperature range of the melting peaks. This is hypothesized to be the result of thermal gradients within the sample, resulting in measured temperatures slightly higher than the actual average sample temperature.

- A slight increase in ΔH_m was also observed for higher heating rates and sample mass. Again, it is hypothesized that this is due to thermal gradients within the sample leading to the inclusion of some sensible heat in the latent heat measurement.

- The lower heating rates of 0.5°C/min and 1°C/min provide a good resolution for phase transition temperature due to reduced thermal gradients and therefore reduced peak width. However the sensitivity of the enthalpy of fusion is lower due to the smaller heat flux signal, especially at small sample masses of 2.5mg.

- The average variation of all the ΔH_m measurements from the NIST reference values are only within a ±1.64% which is within the typical uncertainty of the DSC method. However, even lower variations still be achieved if correct measurement conditions are chosen carefully.

- It is suggested that a small sample mass be used when DSC measurements are performed at high heating rate to reduce the drawbacks of the high heating rate and attain a high accuracy.

- The best agreement between the experimental results and the NIST reference data is obtained using a heating rate of 3°C/min and sample mass of 7.5 mg. At this measurement condition, sensitivity and resolution was maintained at an acceptable level.

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