

Differential Scanning Calorimetry (DSC) Analysis Of Latent Heat Storage Materials For Low Temperature (40-80°C) Solar Heating Applications

R. Sanjay Kumar, D. Jaya Krishna

^aResearch Scholar, ^bCorresponding author and Assistant Professor,
Department of Mechanical Engineering, BITS PILANI Hyderabad campus,
Jawahar Nagar, Shameerpet, Hyderabad - 500078, Andhra Pradesh, India

Abstract

The aim of this study is to investigate properties such as melting point, latent heat and specific heat of low temperature (40 – 80°C) latent heat storage materials for solar heating applications. Materials such as sodium acetate trihydrate, paraffin wax, palmitic acid, myristic acid, stearic acid, and lauric acid have been selected for considered applications. As thermophysical properties of heat storage materials play a major role in the efficient design of the heat storage system, accurate values for properties are to be considered. But due to variations in the purity of the material discrepancies may be observed in the available values. Therefore, DSC (Differential Scanning Calorimetry) is used to determine the properties such as melting point, latent heat and specific heat of the selected materials. Before proceeding with the experiment a thorough calibration has been carried out with standard sample i.e. pure indium for different heat rates. The study attempts to provide accurate thermophysical property values which can provide an invaluable database for appropriate selection of low temperature latent heat storage materials for solar heating applications.

Keywords: Differential scanning calorimetry (DSC), latent heat storage materials, low temperature solar heating applications, thermophysical properties

1. Introduction

In recent years, energy demand has increased due to the high-energy consumption in domestic and industrial sectors. Fossil fuels have served and fulfilled all human needs of energy for long era. These fossil fuels caused significant damage to the environment as it leads to global warming, climate change, acid rain etc. In addition, the prices of fossil fuels have increased and are expected to continue in coming years because of the increase in energy demand and a decrease in reserves.

For example in India major non-alternative energy sources such as LPG, Diesel and Petrol are being rapidly depleted as they are being widely used as a source of energy particularly for cooking and transportation purpose. One of the most important alternative energy sources is solar energy and it can play a crucial role in the fulfillment of future needs. It is a clean source of energy as it does not produce any CO₂ emission. However, solar energy is intermittent and transient in nature. The incident solar radiation underlies temporal variations i.e. hourly due to weather, daily due to day and night and yearly due to the seasons. This mismatch between the availability of energy source and the demand for energy can only be alleviated by storing the solar thermal energy. Thermal energy can be stored in the form of sensible heat and latent heat.

In sensible heat storage thermal energy is stored by raising the temperature of either solid or liquid. The amount of stored heat depends on the specific heat of the medium, the temperature change and the amount of heat storage material. The amount of thermal energy stored in the form of sensible heat can be calculated by $Q = mC_p(\Delta T)$ where 'Q' is the amount of thermal energy stored or released in the form of sensible heat (kJ), ' ΔT ' is the difference between initial and final temperature (°C), ' m ' is mass of material used to store thermal energy (kg) and ' C_p ' is the specific heat of the material (kJ/kg°C). Latent heat provides much higher energy storage density and

maintains the smaller temperature difference between storing and releasing heat [1, 2]. It is the amount of heat absorbed or released during the change of material from one phase to another phase. Due to this phase change materials can be categorized as latent heat storage materials. The amount of thermal energy stored in the form of latent heat in a material is calculated by $Q = m(\Delta H)$ where ' Q ' is the amount of thermal energy stored or released in the form of latent heat (kJ), ' m ' is the mass of the material used to store thermal energy (kg) and ' ΔH ' is the latent heat of fusion or vaporization (kJ/kg).

Latent heat storage materials are classified into three types. It can be solid-liquid, solid-gas and liquid-gas. Solid-liquid, solid-gas and liquid-gas are available as phase transformations. In order to have a simple and compact system, the volume difference between the two phases should be minimum. Solid to gas and liquid to gas may not be possible to minimize the volume difference between the two phases. This means that using a gas phase would require compression, thus requiring additional energy and reducing the storage efficiency. For this reason, solid-gas and liquid-gas phase changes are not concentrated in this study. Also, in general solid – liquid phase change has high latent heat storage.

Latent heat storage materials can be referred as phase change materials. Commonly used latent heat storage materials for solar heating applications in the low temperature range are: paraffins (organic), fatty acids (organic), salt hydrates (inorganic), and eutectics [3]. Paraffins are considered to be an attractive option because it is safe, non – reactive and has negligible super cooling [3- 5]. Fatty acids namely, stearic acid, palmitic acid, myristic acid and lauric acid etc. are preferred due to their high latent heat of fusion and non- toxic nature. In inorganics, the most attractive type of materials to be used in thermal energy storage is salt hydrates. They have high thermal conductivity and large heat storage capacity compared with organics. In addition, they

have low cost and are easily available which makes them commercially attractive for solar heat storage applications.

In 1949 the exploration of phase change materials (PCMs) for storing solar heat was carried out by Telkes and Raymond [6]. Later due to its immense utility for various applications especially in solar heating, PCMs are being extensively researched. The basic principle of using a PCM for thermal energy storage in a solar heating application is that when heat is supplied to the material, the material changes its phase from solid to liquid at a constant temperature. Similarly, when heat is released, the material changes phase from liquid to solid again at a constant temperature until it solidifies completely [7]. During the melting/solidification processes large amount of heat transfer takes place with a small temperature difference which makes PCM interesting as a source of heat storage material in practical applications. As thermophysical properties of a PCM play a vital role in the optimal performance of a heat storage system hefty importance is given in determining their properties. Due to this several researchers [8-11] have focused their studies on measuring thermophysical properties of PCMs.

In order to measure thermophysical properties such as melting point, latent heat of fusion and specific heat several thermal analysis methods are available. These methods may be classified primarily into two groups, namely, i) Conventional calorimetry methods such as a) Differential Scanning Calorimetry (DSC) methods and b) Differential Thermal Analysis (DTA) technique and ii) T-history method. DSC is one of the thermo analytical technique which is used to measure the physical properties. Normally, it determines the temperature and heat flow associated with phase change material transitions as a function of temperature and time. It also determines the differences in heat transfer rates between the sample and reference products. These differences are caused by physical or chemical transformations of phase change in the sample. During physical

transformations such as phase transitions, more or less heat will transfer to the sample than the reference to maintain both at the same temperature throughout the process (either endothermic or exothermic). It allows us to measure the absorption of heat or release of heat by the sample as it undergoes phase transitions. This is the attractive feature which prompts us to use DSC to measure physical properties such as melting temperature and enthalpy. Further, DSC can be broadly classified into two categories based on the mechanism and operation. (a.1) Power compensated differential scanning calorimeter and (a.2) Heat flux differential scanning calorimeter. In power compensated differential scanning calorimeter the specimen and reference temperatures are controlled using separate identical ovens. By varying power input to the two furnaces, zero temperature difference is maintained between the sample and the reference. The power thus provided is a measure of the enthalpy or heat capacity changes in the test specimen relative to the reference. In Heat flux DSC the test specimen and reference material (which comprises of an empty aluminum pan with lid) are placed on thermoelectric disk which is enclosed in the same furnace. The furnace is heated at a linear heating rate, and the heat is transferred to the sample and reference pan through the thermoelectric disk. When the sample undergoes a phase transition a temperature difference takes place between the sample and the reference. By using calibrated experiments this temperature difference is related to find the enthalpy change of the specimen. In heat flux DSC, there is a good thermal contact between sample and reference. Due to this reason heat flux DSC is more attractive to measure thermophysical properties of the substances when compared to power compensated differential scanning calorimeter. Similar technique to DSC is differential thermal analysis (DTA). It allows determining the temperature difference between sample and reference. The difference in temperature is plotted either against time or temperature. DSC and DTA give the same kind of

information and similar results but for quantitative measurement DSC is considered to be more accurate than DTA.

T-history method was proposed by Yinping and Yi [12] for determining the thermophysical properties such as melting point, latent heat of fusion, specific heat and thermal conductivity of liquid and solid phases. Temperature-time graphs were drawn during the cooling against ambient temperature for a sample and the graphs are compared to reference material (pure water). The major drawback is that till date no commercial T-history method based instrument is available to directly measure the thermophysical properties for a given sample. Every T-history installation has to be set up individually for the evaluation of data due to which the correctness of the instrument is to be verified by quantifying measurement uncertainties.

Among the various studies relating to DSC one can cite Flaherty [13], for the characterization of hydrocarbons and natural waxes. Giavarini and Pochetti [14] reported that DSC is considered to be a more useful technique for measuring physical characteristics of petroleum products and phase change materials. They have conducted DSC analysis of petroleum products such as paraffin waxes, petrolatum, lubeoils, fueloils and bitumens. Characteristics namely, melting point and latent heat of the substances were presented. Gibbs and Hasnain [15] and Zalba et al. [16] mentioned that there is an uncertainty in the values provided by manufacturers and it is beneficial to use DSC to get more accurate results.

To study the performance of phase change materials most of the researchers [17-19] have used DSC analysis for finding out various thermo physical property values such as melting point, latent heat, and specific heat. Based on the literature it may be observed that the accurate measurement of thermophysical properties can be carried out by heat flux DSC. Therefore, in the

present study heat flux DSC is used to measure the thermophysical properties such as melting point, latent heat and specific heat of phase change materials.

In this study six heat storage materials namely, stearic acid, palmitic acid, myristic acid, sodium acetate trihydrate, paraffin wax, and lauric acid are chosen. These materials are selected based on the following criteria: (i) Desired melting point temperature (40 – 80°C) (ii) ease of availability (iii) High latent heat of fusion (iv) High specific heat (v) Nontoxic (vi) Low cost. It may be noted that the thermophysical properties for the considered PCMs can be obtained from the literature [20-33]. But discrepancies may be observed in the provided values and this may be due to the variations in purity of mass fraction of a PCM. As thermophysical properties of a PCM are very crucial for designing a long running heat storage system correct values are very much needed. Therefore in the present study an attempt has been made to provide accurate values by performing experiments on DSC. The values of melting point, latent heat and specific heats at solid and liquid states for selected materials are provided. The study is expected to help in great detail for directly selecting a suitable heat storage material to develop an efficient low cost heat storage system.

2. Experimental Methodology

2.1. Materials: In the present work the following heat storage materials namely, palmitic acid, myristic acid, stearic acid, lauric acid, sodium acetate trihydrate and paraffin wax have been selected and experiments are performed. It may be noted that the materials used are without further purification. The list of selected heat storage materials and their specifications are provided in Table 1. Myristic acid (90% purity) is procured from Sisco Research Laboratories Private Limited, Mumbai, India. Stearic acid (90% purity), palmitic acid (99% purity), lauric acid (99% purity) and sodium acetate trihydrate (99% purity) are procured from S D Fine

ChemLimited, Mumbai, India. Paraffin wax is not provided with its purity but provided with a melting point temperature range of 58-60°C isprocured from S D Fine Chem Limited, India. All the experiments are carried out at central analytical laboratory in BITSPilani Hyderabad Campus, Hyderabad, India.

2.2. Experimental setup: The experiments are performed using thermal analysis system and the setup is shown in Fig.1. The setup consists of Shimadzu DSC 60, thermal analyzer, and PC. The technical specifications of the employed DSC are given in Table 2. Shimadzu DSC 60 is a thermal analytical instrument which ismanufactured by Shimadzu Corporation, Japan. This instrument is more compatible and is essential for the evaluation of material properties. Italso enables the measurements of transitions, such as glass transition, melting and crystallization. It is a compact heat flux DSC.It consists of constantan disk, heat flux plate, chromel and alumel wires. Chromel and alumel wires are used to estimate sample temperature. Constantan disk is used to monitor the differential heat flow into the pans. Nitrogen or air is used as a purging gas to remove volatiles and is circulated in the furnace. The data collection and analysis are carried out using TA 60 WS Thermal analyzer. It is fully compatible with window XP, 32 bit application software.

2.3. Experimental Procedure:In this experiment the solid sample (5mg) is weighed in an analytical weighing balance. Weighed sample is placed in an aluminum pan which is hermetically sealed with the help of sample crimper. Reference and sample aluminum pans are placed in DSC as shown in Fig. 2. Sealed aluminum sample pan is placed at the right side of the sample holder position and sealed empty aluminum pan is placed at the left side of the sample holder position. Here the empty aluminum pan acts as a reference sample. Data collection and analysis is carried out using Thermal analyzer TA 60WS software. Experiments are conducted at

heating rates of 5°C/min, 10°C/min and 20°C/min and with a temperature range of 30 - 100°C under a constant stream of nitrogen at atmospheric pressure.

3. Results and Discussion

3.1 Calibration: In order to achieve good results on thermal analysis instrumentation, an instrument should be accurate and must have good repeatability of data. Therefore, before performing the experiment the system should be calibrated and examined under the range of parameters. In general, temperature and heat calibration factors are essential requirements for DSC analysis. Most of the researchers have used pure indium as a standard sample for the calibration of phase change materials [34, 35]. Pure indium is considered to be the most common calibration substance in DSC analysis, due to its well defined heat of fusion and melting point. Therefore, pure indium sample is used and calibration test is performed with various heating rates of 5°C/min, 10°C/min, and 20°C/min. Results are obtained in terms of heat flow as a function of temperature.

The results thus obtained for indium sample through DSC experiments for various heating rates are provided in Table 3. The table provides onset, peak and endset temperatures and latent heat for the heating rates of 5°C/min, 10°C/min and 20°C/min. Generally, the onset temperature is taken as melting point temperature and the total area obtained by integrating the curve based on onset, peak and endset temperatures give latent heat. It may be observed from Table 3 that the melting point temperature obtained for the heating rate of 10°C/min give accurate values. The plot for DSC analysis with indium sample at a heating rate of 10°C/min is presented in Fig. 3. From the figure it may be noted that the DSC output curve provides an onset temperature of 156.7°C and latent heat of fusion as 25.27 J/g. Based on this criteria the DSC analysis has been carried out for selected six materials with a heating rate of 10°C/min.

3.2. DSC results of heat storage materials: The output curves shown in Figs. 3 and 4 represents a sharp endothermic dome, corresponding to the melting process. The curve initiates with an onset temperature and then proceeds to a peak temperature. It may be noted that in the present study the onset temperature is taken as melting point temperature and latent heat is calculated based on the total area under the peaks of solid to liquid transitions. Before performing the experiments for the selected six materials the calibration has been performed using a standard indium sample. By employing standard specific heat value (0.238 J/g °C), heat flow values as a function of temperature and a heat rate of 10°C/min for the standard indium sample the value for calibration constant has been obtained based on Eq. 1.

$$Cp = \frac{E \cdot H \cdot 60}{Hr \cdot M} \quad (1)$$

Where

C_p : Specific heat (J/g°C)

(C_p of indium sample = 0.238 J/g°C)

E : Calibration constant (dimensionless)

H : Heat flow (mW)

Hr: Heating Rate (°C/min)

M : Sample Mass (mg)

It may be noted that the obtained calibration constant along with heat flow values as a function of temperature are used to determine the specific heat as a function of temperature for the considered six heat storage materials.

The output curves of DSC analyses of materials namely, (i) Palmitic acid(ii) Myristic acid (iii) Sodium acetate trihydrate(iv) Stearic acid(v) Paraffin wax and (vi) Lauric acid are presented in Fig.4. Table 4 summarizes the results of DSC, such as melting point, latent heat and specific

heat. Fig.4 (i) shows the DSC curve of palmitic acid. It may be noted from the DSC curve of palmitic acid that the melting point temperature is 62.35°C , latent heat of fusion is 212.45 J/g and specific heats at solid and liquid states are $2.15\text{ J/g }^{\circ}\text{C}$ and $2.94\text{ J/g }^{\circ}\text{C}$. Also, it may be noted from the figure that the sharp peak at 64.77°C corresponds to the solid to liquid phase change temperature for palmitic acid. The DSC curve for myristic acid is shown in fig 4(ii). It may be noted that the DSC curve of myristic acid provides a melting point temperature of 54.5°C , latent heat of fusion of 228.22 J/g , specific heats at solid and liquid states are $3.29\text{ J/g }^{\circ}\text{C}$ and $2.65\text{ J/g }^{\circ}\text{C}$ and the corresponding solid to liquid phase change temperature is 57.67°C . Similarly, for sodium acetate trihydrate based on Fig. 4(iii) the melting point temperature is 59.26°C , latent heat of fusion is 240.2 J/g , specific heats at solid and liquid states are $2.008\text{ J/g }^{\circ}\text{C}$ and $2.93\text{ J/g }^{\circ}\text{C}$ and the corresponding solid to liquid phase change temperature is 63.12°C . Similarly, for stearic acid based on Fig. 4(iv) the melting point temperature is 56.7°C , latent heat of fusion is 170.46 J/g , specific heats at solid and liquid states are $2.86\text{ J/g }^{\circ}\text{C}$ and $2.1\text{ J/g }^{\circ}\text{C}$ and the corresponding solid to liquid phase change temperature is 59.43°C . The DSC curve for paraffin wax is shown in Fig. 4(v). It can be seen from the figure that there are two phase change peaks in a DSC output curve. It may be noted that the first minor peak on the left side is due to the solid to solid transition and the second sharp peak at 63.05°C corresponds to the solid to liquid phase change [36]. Based on the DSC output curve the melting point temperature is 53.59°C , latent heat of fusion is 180.1 J/g and specific heats at solid and liquid states are $2.35\text{ J/g }^{\circ}\text{C}$ and $3.25\text{ J/g }^{\circ}\text{C}$. The DSC curve for lauric acid is shown in Fig. 4(vi). The curve provides a melting point temperature of 44.81°C , latent heat of fusion of 177.4 J/g and specific heats at solid and liquid states are $2.117\text{ J/g }^{\circ}\text{C}$ and $1.53\text{ J/g }^{\circ}\text{C}$ and the corresponding solid to liquid phase change temperature is 47.78°C . The thermophysical properties such as melting point, latent heat and specific heats at solid and liquid

states thus obtained through DSC analysis for selected six materials are consolidated and presented in Table 4. By carrying out an exhaustive literature review the available values for the considered thermophysical properties of the selected six materials are also presented in Table 4. It may be noted that discrepancies may be observed in the reported values and this may be due to the variation in purity. Based on the study it may be observed that the values for thermophysical properties if provided along with their purity values will help in overcoming the discrepancies as observed in the literature.

Conclusions

The study provides the thermophysical properties such as melting point, latent heat and specific heat of the selected six materials namely, sodium acetate trihydrate, paraffin wax, palmitic acid, myristic acid, stearic acid and lauric acid. DSC is used to perform the experiments. Results are presented qualitatively in terms of DSC output curves and quantitatively by reporting the respective property values. The experimental results prove that the selected latent heat storage materials have a desirable melting temperature, high latent heat, and high specific heat at solid and liquid states which makes them excellent heat storage materials for low temperature solar heating applications. All the mentioned heat storage materials and their physical properties have achieved good accurate results by heat flux DSC. The study is expected to provide an invaluable database which can help in selecting a suitable heat storage material for low temperature (40 – 80°C) solar heating applications.

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Table 1: Specifications of selected heat storage materials

Name	Scientific Name	Molecular formula	Type	Purity (%)	Make
Paraffin wax	Paraffina	-	Organic	-	SD Fine Chem Ltd
Sodium acetate trihydrate	-	$C_2H_3NaO_2 \cdot 3H_2O$	Inorganic	99	SD Fine Chem Ltd
Myristic acid	tetradecanoic acid	$C_{14}H_{28}O_2$	Organic	90	SISCO Research laboratories Pvt. Ltd
Palmitic acid	Hexadecanoic acid	$C_{16}H_{32}O_2$	Organic	99	SD Fine Chem Ltd
Stearic acid	n-Octadecanoic acid	$C_{18}H_{36}O_2$	Organic	90	SD Fine Chem Ltd
Lauric acid	Dodecanoic acid	$C_{12}H_{24}O_2$	Organic	99	SD Fine Chem Ltd

*- not available

Table 2: Technical specifications of Differential scanning calorimetry

Name	Specifications
Principle	Heat flux type
Heat Flow Range	$\pm 40\text{mW}$
Hold Time	0 - 999 min, hour
Temperature Range	-150 to 600° C
Program Rate	0 - 99°C/min, °C/hour
Atmosphere	Inert gas (nitrogen) or air
Temperature accuracy	$\pm 0.1^\circ\text{C}$
Calorific accuracy	$\pm 1\%$

Table 3: Results of DSC analysis of indium sample at various heating rates

Heat rate (°C/min)	Onset/melting point (°C)	Peak point (°C)	Endset point (°C)	Latent heat (J/g)	Supplied by Manufacturer	
					Melting point(°C)	Latent heat (J/g)
5	155.81	156.98	158.29	18.54	156.63	28.45
10	156.72	158.32	160.94	25.27	156.63	28.45
20	156.93	161.85	173.50	24.96	156.63	28.45

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Table 4: Comparison of present values with literature for selected heat storage materials

Materials	T_m ($^{\circ}\text{C}$)		ΔH (J/g)		C_{ps} (J/g $^{\circ}\text{C}$)		C_{pl} (J/g $^{\circ}\text{C}$)		Reference
	L	P	L	P	L	P	L	P	
Paraffinwax	47		226		2.510		-		20
	60		213		1.85		2.384		21
	58-60	53.59	214.4	180.1	0.9	2.35	-	3.25	22, 23
	53.5		266		2.5		-		24
	52		210		2.9		2.1		25
	59.9		190		2.0		2.15		26
	53		189		2.5		-		27
Palmitic acid	61	62.35	203.4	212.45	2.20	2.15	2.48	2.94	28
	64		185.4		1.9		2.8		29
Stearic acid	54-56		186.5		2.83		2.38		18
	68.8	56.7	198.8	170.46	2.83	2.86	2.38	2.1	30
	69		202.5		1.6		2.2		29
	58		169		1.59		-		20,31
Myristic acid	51.5	54.5	204.5	228.2	2.8	3.29	2.42	2.65	32
	58		186.6		1.7		2.4		29
Sodium acetate trihydrate	58	59.26	264.18	240.20	1.7	2.008	2.9	2.93	33
Lauric acid	44	44.81	177.4	160.30	1.7	2.117	2.3	1.53	29

*L-Literature, P-Present study.

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Fig.3 DSC curve of Indium sample at a heating rate of 10°C/min

Fig.4 DSC curve for i)Palmiticacidii)Myristicacidiii)Sodium acetate trihydrate
iv) Stearic acidv)Paraffin wax and vi)Lauric acid

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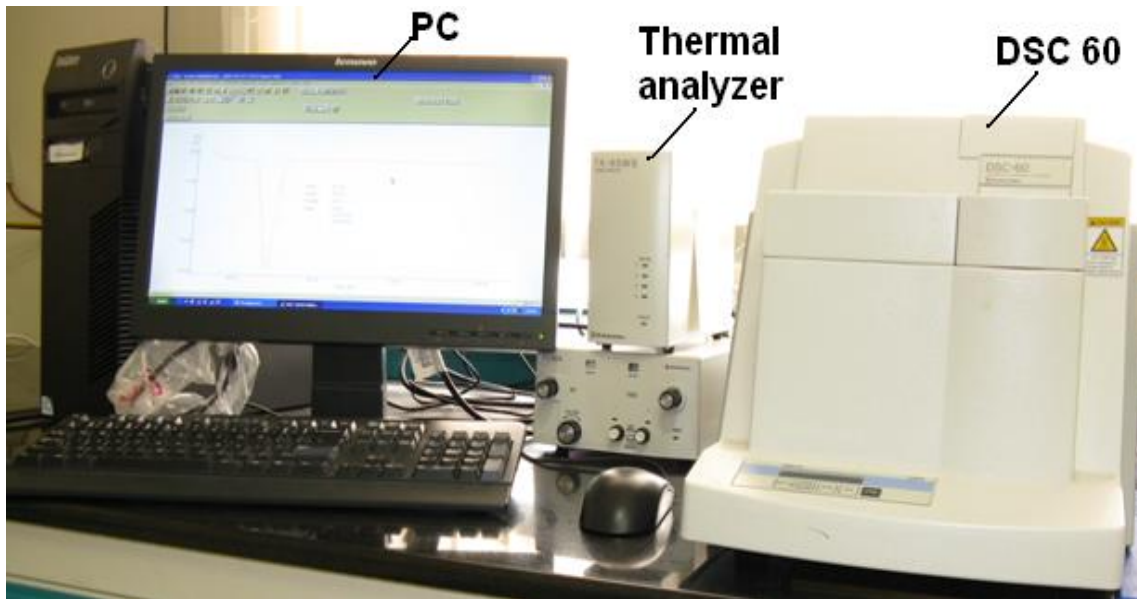


Fig.1: Experimental setup of thermal analysis system

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Fig.2: Reference and sample aluminum pans in DSC

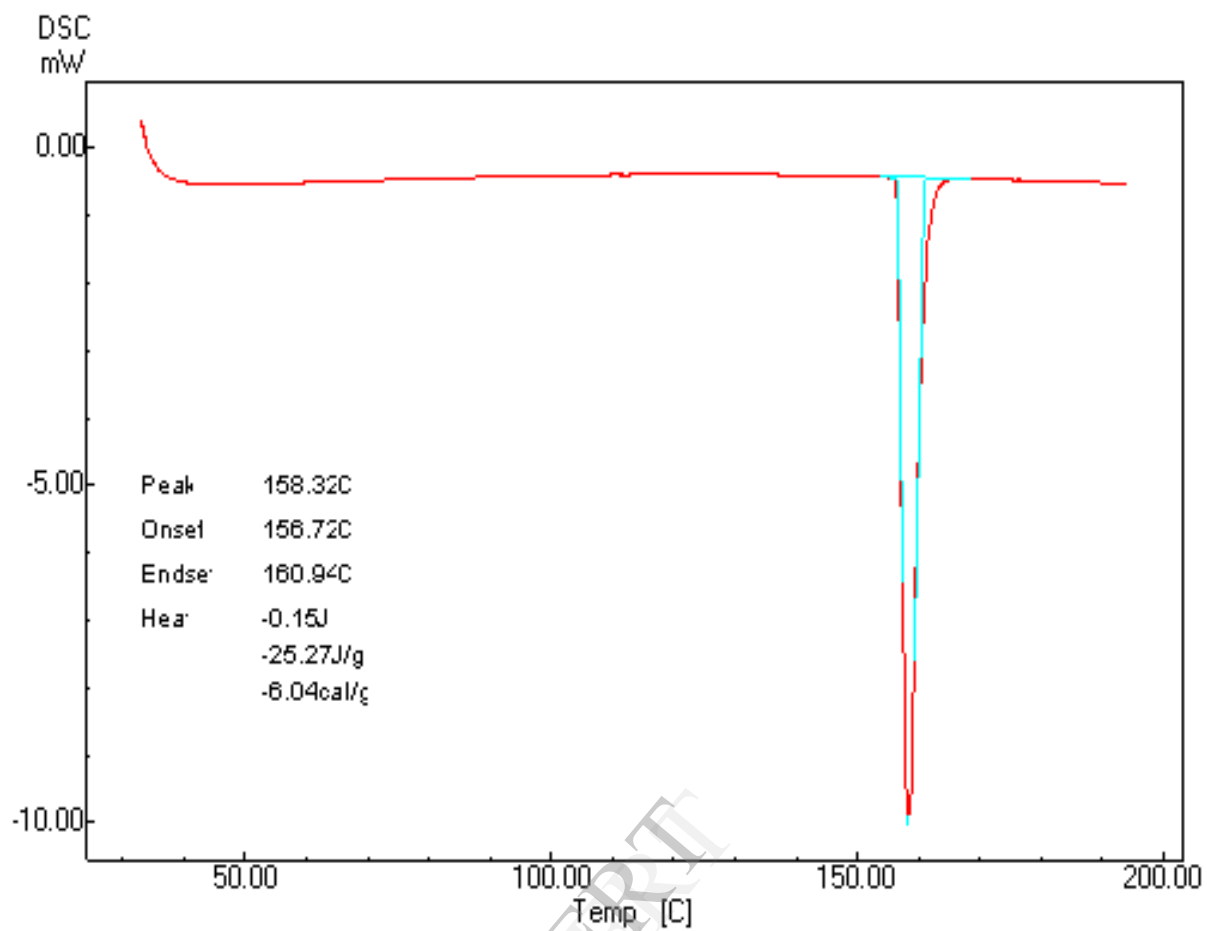


Fig.3: DSC curve of Indium sample at a heating rate of 10°C/min

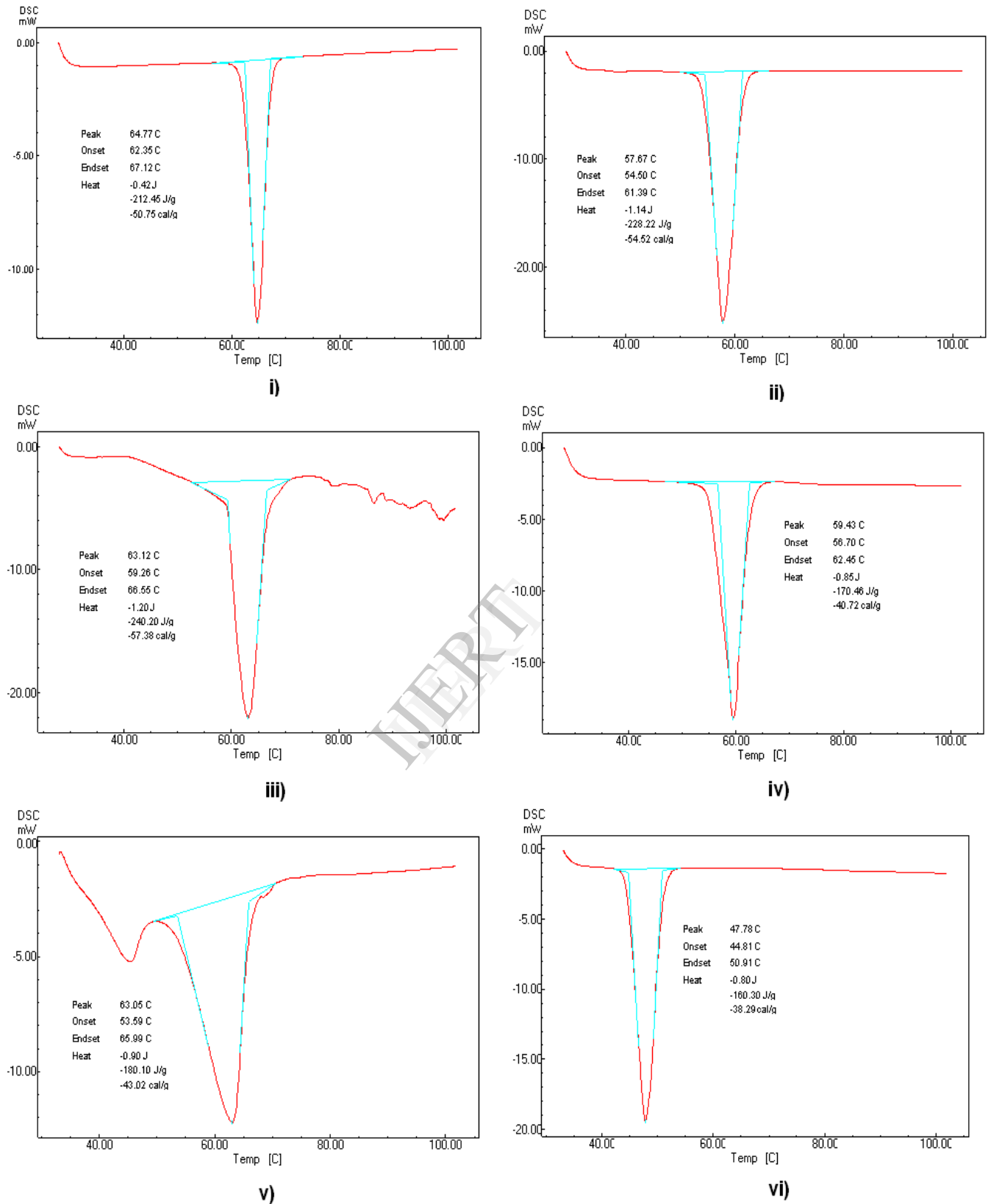


Fig.4: DSC curve for i)Palmitic acid ii)Myristic acid iii)Sodium acetate trihydrate
iv) Stearic acid v)Paraffin wax and vi)Lauric acid