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Pre-Pilot Scale Microencapsulation of Phase Change Materials for Thermal Energy Storage Applications.

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ABSTRACT. Most of the researcher has attempted for the encapsulation of phase change materials in small scale for the thermal storage applications. However, energy conversion in field applications require enormous quantity of thermal energy storage materials. In this paper, a pre-pilot scale is developed for encapsulating of various binary mixtures using an in-situ polymerization technique.DSC, TGA, FTIR, XRD, and FE-SEM were used to delineate the thermal properties of obtained microcapsules in order to identify shape, mean size, functional groups, crystalline state, and weight loss of distinct microcapsules. The results show that the synthesized microcapsules were regular in shape and smooth and well encapsulated around cores. The thermal properties of different microcapsules are 34.9 ℃, 95.3 kJ/kg and 35.6 ℃, 98.5 kJ/kg resultant to microencapsulation of 75% Stearic acid (SA) + 25% Capric acid (CA) and 63% Capric acid (CA) + 37% Palmitic acid (PA),respectively. The encapsulation efficiencies were 78.2% and 73.09%. The yieldof the products are attained ~82%.

Keywords:Binary mixture; encapsulation ratio; thermal properties; energy conversation; pre-pilot scale.

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Nomenclature

CA	Capric acid
MF	Melamine-Formaldehyde
MPCM	Microencapsulated Phase Change Materials
MUF	Melamine-Urea-Formaldehyde
PA	Palmitic Acid
PCMs	Phase Change Materials
PMMA	Poly-Methyl Methacrylate
PS	Poly styrene
SA	Stearic acid
TSM	Thermal Storage Materials
TST	Thermal Cycle Test
UF	Urea-Formaldehyde

1. INTRODUCTION

Energy crisis is gradually increasing particularly in industries, residential and commercial buildings, which may lead to depletion of fossil fuels and greater impact on global warming. Advanced thermal storage materials are being integrated into various sectors such as solar energy capture, buildings, textiles, transportation shelter, etc. Thermal storage materials (TSM) are phase change materials (PCMs) which has high energy density at constant temperature. PCMsabsorbs the heat from environment during phase transition from solid to liquid and vice-versa [1, 2]. These PCMs are commercially available in the market. The researchers are choosing the PCMson the basis of low cost, superior thermal properties, availability and nontoxicity [3, 4].PCMs are integrated in heat storage systems in three ways such as immersion, macro-encapsulation, and microencapsulation. In the immersion process, the PCM was infused into the cementitious material by capillary action aided by vacuum. This process was not stable owing to liquid leakage during phase transition [5]. The macro-encapsulation of PCMs has poor thermal conductivity, which need to be protected against annihilation and more workers are required to install in storage systems. PCMs are employed as the core in microencapsulation, and that they are surrounded by a shell made of either polymer or inorganic material, which is synthesized through physical and chemical processes. It prevents the leakage during phase transition, provides a high heat transfer rate, and capable of resisting volume change during phase transition [6]. Microencapsulation of PCMs were formulated in two ways, one is physical and another is chemical. The physical method includes spray drying whereas chemical methods include in-situ polymerization, complex

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coacervation, phase separation, and suspension-like polymerization [7-9]. Microencapsulation of PCMs has wide applications, particularly in cementitious materials owing to 30–40% of total energy consumption [10]. Generally, the core materials are fatty acids, paraffin, eutectic mixture, and salts whereas shell material are Polystyrene (PS) [11-13],Poly-Methyl Methacrylate (PMMA) [14], Melamine-Urea-Formaldehyde (MUF)[15-17], Melamine-Formaldehyde (MF) [18]. The long-term viability of thermal storage systems is determined on the durability of PCMs. It has a significant impact on the PCMs' service life. As a result, researchers conducted a thermal cycle test (TST) to assess the PCMs' service life. Yang et al., [19]performed accelerating test of different fatty acids such as lauric acid, myristic acid, palmitic acid and stearic acid. They have concluded that thermal properties of fatty acids have not changed significantly after 10,000. Shilei et al., [6] conducted TST oneutectic combination of capric and lauric acid. After 360 cycles of accelerated testing, there was no substantial change in the eutectic mixture. Chinnasamy andAppukuttan [20] determined thermal properties of eutectic mixture of lauric acid / myristyl alcohol after 1000 cycles. It was determined that, there was no change observed in thermal properties. Thermal characteristics of a eutectic mixture of lauric acid/1-tetradeconal were found to be stable up to 90 thermal cycle tests by Zuo et al [21]. Zhang et al. [22] combined PCMs with lauric acid, myristic acid, and palmitic acid to create a ternary mixture. Melting point and fusion heat were found to be steady for up to 50 cycles.According to a literature review, the thermal characteristics of fatty acids remained steady for a duration of 10000 cycles, implying that fatty acids had a 27-year life span.

Researchers are preparing MPCMs in lab size using the scientific methodologies mentioned above. It is a lengthy and time-consuming procedures.Microtek-BASF preparing these MPCMs with help of Spray dryer technique (physical process) required desired phase change temperature range at large scale. It is non-economical process at lab level.However, practical applications, such as pre-pilot lab experiments on solar thermal energy systems, textiles, solar water heating systems, transportation shelters, and prototype structures, necessitate a massive amount of MPCMs.Among the physical and chemical methods, *in-situ* polymerization technique is adopted for pre-pilot scale preparation of MPCMs. This method is very feasible and economic process at lab level.

In the present work, a pre-pilot scale in-situ polymerization for encapsulation of fatty acid mixtures were prepared using existing governed model. In that, two binary mixtures such as 75% SA + 25% CA and 63% CA + 37% PA fatty acids mixtures were used as core whereas, Melamine-Formaldehyde (MF) used as polymer shell. Designed the batch agitator vessel based on density and viscosity of the materials.The description of the agitator vessel has described below Fig.1 and 2.

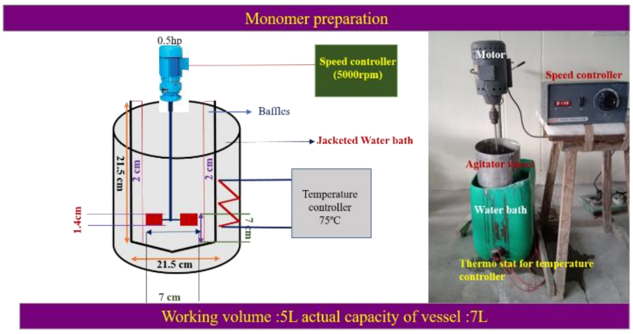

Fig. 1. Melamine –Formaldehyde Monomer preparation.

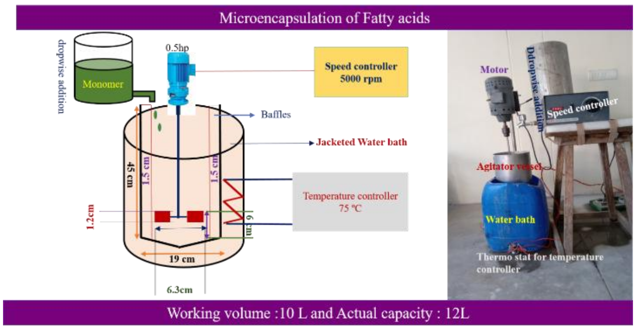

Fig. 2. Bulk scale preparation of microencapsulation of fatty acids.

Two categories of agitator vessel have been designed and developed. One was used for monomer preparation. Another was 12 L capacity of agitator vessel which was used for microencapsulation process. In situ–polymerization technique is thermal process for that the agitator vessels were placed in temperature controller water bath. Temperature was controlled with help of thermostat connected to the water bath.

The agitator vessels were designed using the following equations

$$\frac{H}{Dt} = 1 \quad (1) \qquad \frac{J}{Dt} = \frac{1}{12} \quad (2)$$

$$\frac{W}{Da} = \frac{1}{5} \quad (3) \qquad \frac{Da}{Dt} = \frac{1}{3} \quad (4)$$

$$\frac{E}{Dt} = \frac{1}{3} \quad (5) \qquad \frac{L}{Da} = \frac{1}{3} \quad (6)$$

Where H is the height of agitator vessel, Dt diameter of agitator vessel, J is width of the vessel, Da is diameter of impeller, W is the width of the impeller blade, E is distance from bottom of the tank to impeller, L is length of the impeller blade.

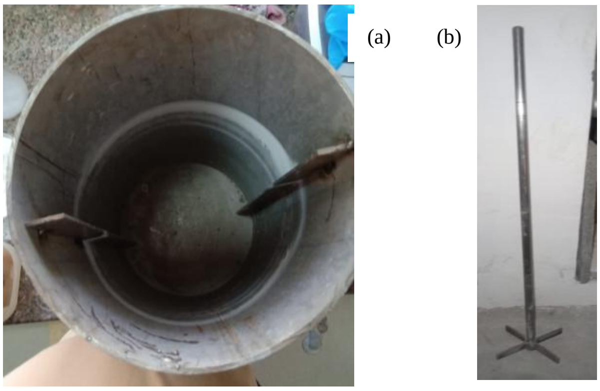

Fig. 3. Attachment of (a) Baffles (b) Impeller in Agitator vessels.

To avoid vortex in the agitator vessel, two baffles were installed Fig. 3. Open straight blade turbine is connected to shaft to the motor for agitation of viscous liquids. The power consumption of the agitator vessel has been determined using following equation

$$P = \frac{k_T n^3 D_a^5 \rho}{g_C} \qquad (7)$$

Where P is power consumption, K_T is impeller constant factor 1.27, n is rotational speed, D_a is diameter of agitator vessel, ρ is density of solution [23]. The designed and developed agitator vessel was presented in Fig.1 and 2 and the dimensions were given in table 1.

Table 1 Dimension of different agitator vessel

Dimension of monomer Agitator vessel	
Total Volume	7 L
Working volume	5 L
Height of agitator vessel	215 mm
Diameter of agitator vessel	215 mm
Width of the baffles (4Nos)	20mm
Diameter of the impeller	70mm
Width of the impeller blade	14mm
Length of the impeller blade	17.5 mm
Motor capacity	0.5 HP

Dimension of encapsulation Agitator vessel	
Total Volume	12 L
Working volume	10 L
Height of agitator vessel	450 mm
Diameter of agitator vessel	190 mm
Width of the baffles (4Nos)	15mm
Diameter of the impeller	63 mm
Width of the impeller blade	12 mm
Length of the impeller blade	15.8 mm
Motor capacity	0.5 HP

2. MATERIALS ANDMETHODS

2.1Materials:

Two kinds of eutectic mixture such as 75% Stearic acid (SA) + 25% Capric acid (CA), 63% Capric acid (CA) + 37% Palmitic acid were used as cores. The SA, CA and PA were purchased from Loba Chemie Pvt. Ltd Mumbai and Fisher Scientific Mumbai (India) Mumbai respectively. Their thermal properties were given in table 2. Melamine, and aqueous Formaldehyde procured from Loba Chemie Pvt. Ltd., Mumbai, was used as the shell. Sodium Dodecyl Sulfate (SDS) was obtained from Fisher Scientific, Mumbai (India) is an anionic surfactant used as emulsifier for the formation of the emulsion. Sodium carbonate anhydrous (Na_2CO_3) was procured from Merck, New Delhi used for maintaining basic medium, and Sulfuric acid (H_2SO_4) was purchased from Thomas baker, Mumbai (India) used for maintaining acidic medium for microencapsulation. Ethanol was purchased from Fine Chemical Co. Ltd used to remove unencapsulated PCM.For distinct solutions, two agitator vessels have been conceived and built. One was used to prepare monomers. Another was a microencapsulation agitator vessel with a capacity of 12 L. The agitator vessels were placed in a temperature-controlled water bath for the in situ–polymerization procedure, which is a thermal process. A thermostat connected to the water bath was used to regulate the temperature.

Table 2 Thermal properties of fatty acids

S. No	PCM	M.P (℃)	L.F (kJ/kg)	Ref.
1.	75% SA+25% CA	35	121.80	[24]
2.	63% CA+37% PA	36.2	134.75	[25]

2.2 Preparation of emulsion of different fatty acids

400–500 g of different eutectic mixtures was mixed with 5–7 L distilled water in a 12 L capacity of agitator vessel. The eutectic mixture heated at different melting range of 35–70 ℃ until solid becomes liquid in agitator vessel. 16–20 g of SDS was then added in the liquid solution and the solution was stirred using mechanical stirrer for 1h to make a stable emulsion. The optimized parameters for microencapsulation of PCMs such as pH 3.2, agitator speed 5000 rpm and temperature 75 ℃, core to shell ratio and composition were adopted from the previous study conducted by Srinivasaraonaik et al[24, 25]. The chemical composition of different PCMs has been provided in Table 3.

Table 3 Chemical composition of raw materials

S.No.	PCM (core) (g)	Melamine (g)	Formaldehyde (ml)	SDS (g)
1.	75% SA+25% CA (400)	160	300	16
2.	63%CA+37%PA (500)	200	375	20

2.3 Preparation of MF monomer

160–200 g of Melamine, 300–375 mL of formaldehyde and 5 L distilled water were taken in a 7L capacity of agitator vessel. The pH of the solution was adjusted to 8.8 using 10% sodium carbonate solution. The mixture was heated and stirred at 75 ℃ until it becomes transparent. The transparent colour represents the MF monomer.

2.4*Preparation of Microencapsulation of PCM*

The initial pH of the obtained emulsion was about 5. The pH of eutectic emulsion was adjusted from 5 to 3.2 by using 5% solution of sulfuric acid and the solution was heated up to 75 ℃ using thermostat (Fig. 1). The MF monomer was added dropwise into the emulsion of the eutectic mixtures and the mixture of MPCM was stirred and heated continuously for 5 hours. After 5 h, The obtained solution was cooled to room temperature. The obtained microcapsules were filtered with the help of vacuum filtration unit and laved with ethanol solvent to remove unencapsulated PCMs. The wet microcapsules were dried in the oven at 60 ℃ until completely moisture was removed on surface of the polymer (Fig. 4).

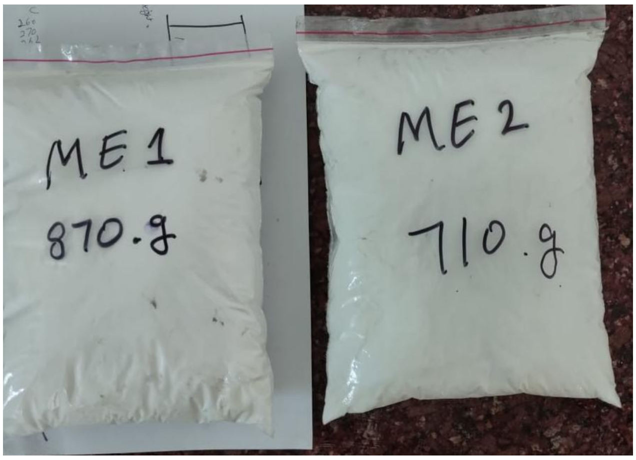

Fig. 4. Product of microencapsulation of (a) 75% SA + 25% CA (b) 63% CA + 37% PA.

2.5Charging and discharging of eutectic mixture of 63% CA + 37% PA

10 g of raw eutectic mixture and microencapsulated 63% CA + 37% PA was taken in various test tubes. These test tubes were placed in 500 mL capacity of water beaker. The calibrated K-type thermocouples were positioned in distinguish test tubes for measuring charging and discharging temperature of the samples. These thermo couples were connected to temperature data logged system. These samples were placed on a magnetic stirrer with a temperature controller. The magnetic stirrer temperature was raised to 50 °C and the charging temperature recorded to until solid completely becomes liquid. When the test tubes are engaged in ambient conditions, the discharge temperature is recorded.

3.Characterization Techniques

The obtainedmicrocapsules of different eutectic mixtures were characterized by Field Emission Scanning Microscope (FESEM), X-Ray Diffraction (XRD), Fourier Transformation Infrared Spectroscopy (FTIR), Thermogravimetric / Differential Thermal Analysis (TG / DTA), Differential Scanning Calorimeter (DSC).

3.1 Field Emission Scanning Microscope (FESEM)

The morphology of the synthesized microcapsules of different eutectic mixtures were carried out using FESEM (Make: TESCAN, Model: MIRA 3). The synthesized MPCMs adhered to carbon tape which was on the sample holder. The samples were gold-coated by using a sputter coater (Make: Electron Microscopy Sciences, Model: K550X). The samples were analysed for morphological studies and determined average particle size of microcapsule with randomly selected ten microparticles.

3.2 X-ray Diffraction (XRD)

The powdered samples were flattened on a glass slide's sample holder. The mineralogical studies of raw PCMs and different MPCMs have been analysed through X-ray diffraction (Make: Rigaku and Model: Dmax 2200) operated in two theta range of 5–80 degree at the rate of 3 theta / min.

3.3 Fourier Transformation Infrared Spectroscopy (FTIR)

The samples and KBr were grinded and made the pellet with help of pressurized device. The prepared pellets were kept in FTIR (Make: Perkin, Model: Perkin Elmer spectrum version 14) for determining the functional groups attached in SA, CA, LA, eutectic mixture and different MPCMs.

3.4 Thermogravimetric Analysis (TG / DTA)

The thermal stability and percentage of weight loss of different MPCMs have been carried out by using TG / DTA (Make: LINSEIS Model: STA PT 1600) at a constant heating rate of 10 theta / min from temperature range of 50 °C to 600 °C

3.5. Differential Scanning Calorimeter (DSC)

The thermal properties such as melting point, latent heat of fusion and specific heat of different MPCMs have been determined with help of DSC (Make: TA instruments Model: Q200) in the presence of Argon at a heating rate of 5 theta / min in the temperature range from 30 °C to 80 °C.

4 RESULT AND DISCUSSION

4.1 Morphology of eutectic mixture of microencapsulated PCMs

The morphology of encapsulation of the different eutectic mixtures was presented in Fig 5 (a) and (b). The morphology of microcapsules was observed at the same scale and 10 kV. The shapes of microcapsules are spherical and smooth. The core is well encapsulated in the shell. Randomly different 10 sizes of the samples were selected and determined the mean size of capsules. The mean particle sizesare about 3.6–6 µm and 2.5–4 µm corresponding to encapsulation of 75% SA + 25% CA and 63% CA + 37% PA, respectively.

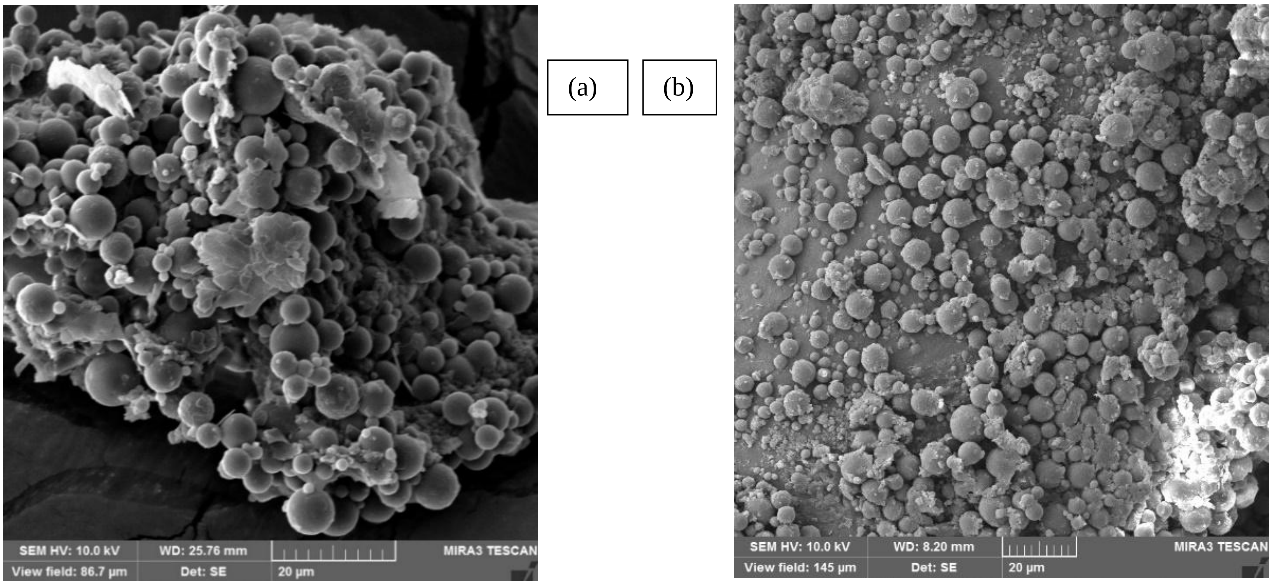

Fig. 5.Morphology of different MPCMS (a) 75% SA + 25% CA (b) 63% CA + 37% PA.

4.2 The FTIR patterns of microencapsulated eutectic mixture with MF shell

The FTIR analysis of shell, core and microcapsules were shown in Fig 6 and 7. The same shell was used for preparation of microencapsulation of eutectic mixture. The spectra of shell are represented in Fig 6 (a) and 7 (a). The stretching of N-H amine functional peak is observed approximately at 3457 cm^{-1}.The stretching vibrations of the C-H group and the C=O group are represented by the intensity peaks at 2945 cm^{-1} and 1710 cm^{-1}, respectively. 1137 cm^{-1} and 820 cm^{-1} are assigned to the C-N stretching bend and aromatic C-H vibrations, respectively. C-H stretching vibrations are represented by the intensity peaks 2918 cm^{-1} and 2852 cm^{-1}. C-H bending vibrations account for the peak at 1432 cm^{-1}. The =C-H alkene group is determined by the intensity peaks 940 cm^{-1} and 726 cm^{-1}.The spectral strong stretching vibration peak C=O is observed at 1702 cm^{-1} in Fig. 6 (b). The spectrum of 63% CA + 37% PA is shown in Fig 7 (b). The intensity peaks 2916 cm^{-1} and 2850 cm^{-1}represent to C-H stretching vibrations. The spectral strong stretching vibration peak C=O is observed at 1698 cm^{-1}. The peak at 1430 cm^{-1} is allocated to C-H bending vibrations. The intensity peaks 944 cm^{-1} and 724 cm^{-1} are the =C-H alkene group is established. The peaks are shown in Fig 6 (b) and 7 (b) almost same with little shifting because of encapsulation efficiency difference. In the distinct microcapsules depicted in Fig 6 (c) and 7 (c), the core and shell peaks are well-defined, and no further peaks were observed. The observed peaks confirm that the distinct cores are properly positioned within the shells and that no chemical interactions have occurred between them.

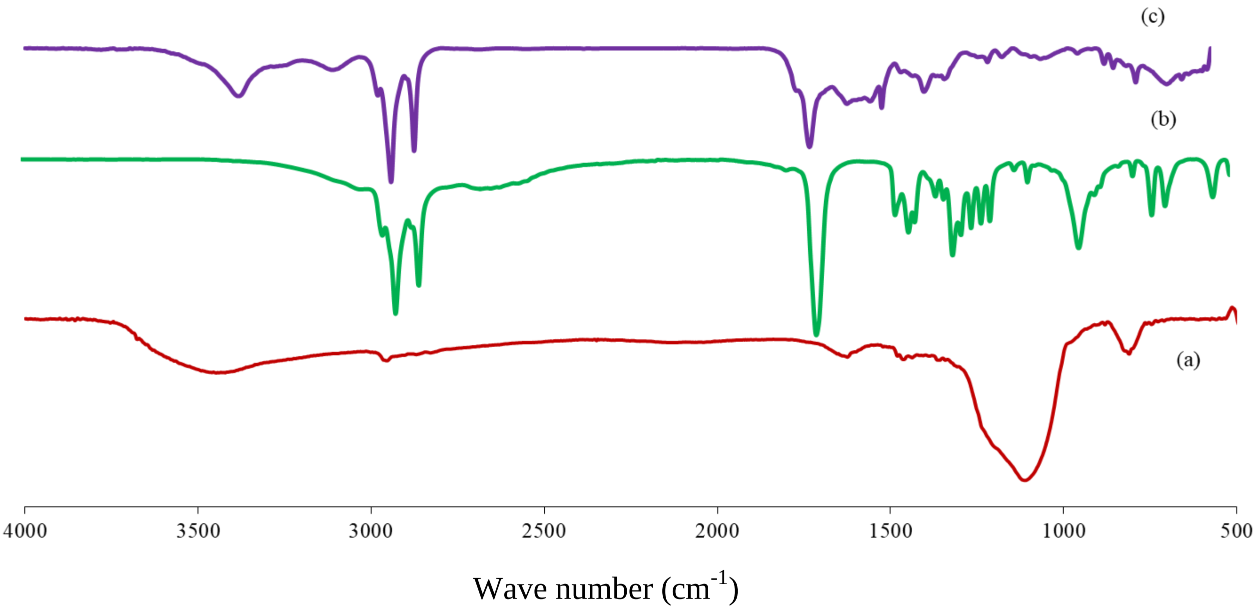

Fig. 6.FTIR analysis (a) MF (b) 75% SA + 25% CA (c) MPCM.

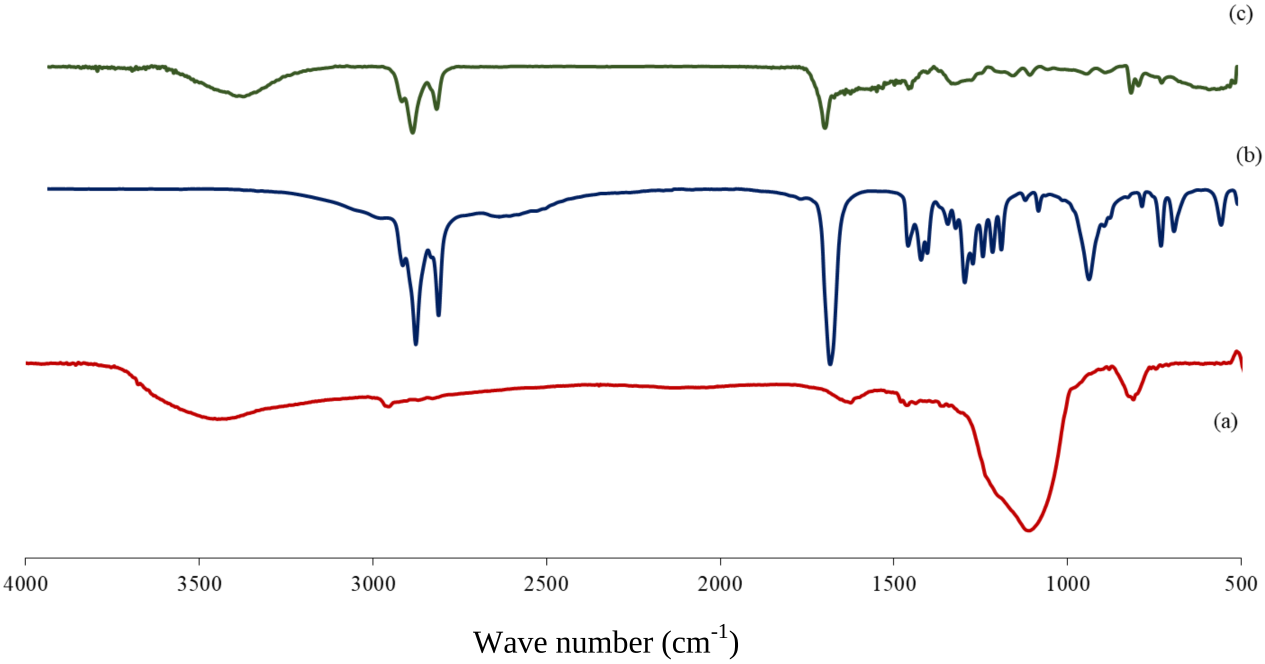

Fig. 7. FTIR analysis (a) MF (b) 63% CA+37% PA (c) MPCM

4.3 XRD patterns of microencapsulated eutectic mixtures

The XRD patterns of core, various eutectic mixtures and encapsulated eutectic mixtures were represented in Fig 8 and 9. The mineralogical study of shell is amorphous characteristic observed in Fig 8 (a) and 9 (a). The minerals peaks of different eutectic mixtures are determined at different 2 theta values. The XRD peaks at 7.2°, 9.1°, 21.72°, and 21.98° are instigated to 75% SA + 25% CA as shown in Fig 8 (b). The XRD peaks at 7.9°, 9.5°, 21.92° and 22° are engendered to 63% CA+37% PA in Fig 9 (b). From the Fig 8 (b) and 9 (b), it has been observed same peaks at slight positions happened by the same group. In Fig 8 (c), the peaks are at 6.9°, 9.3°, 21.53° and 22.01°observed with semi region of amorphous characteristic. In Fig 9 (c) 8.1°, 9.8°, 22.05° and 22.45° determined. It is clearly stated that, the shell is encapsulated around different mixtures with altered position and new peak is not revealed in Fig 8 (c) and 9 (c). The modest shift in locations is due to changes in core and shell composition

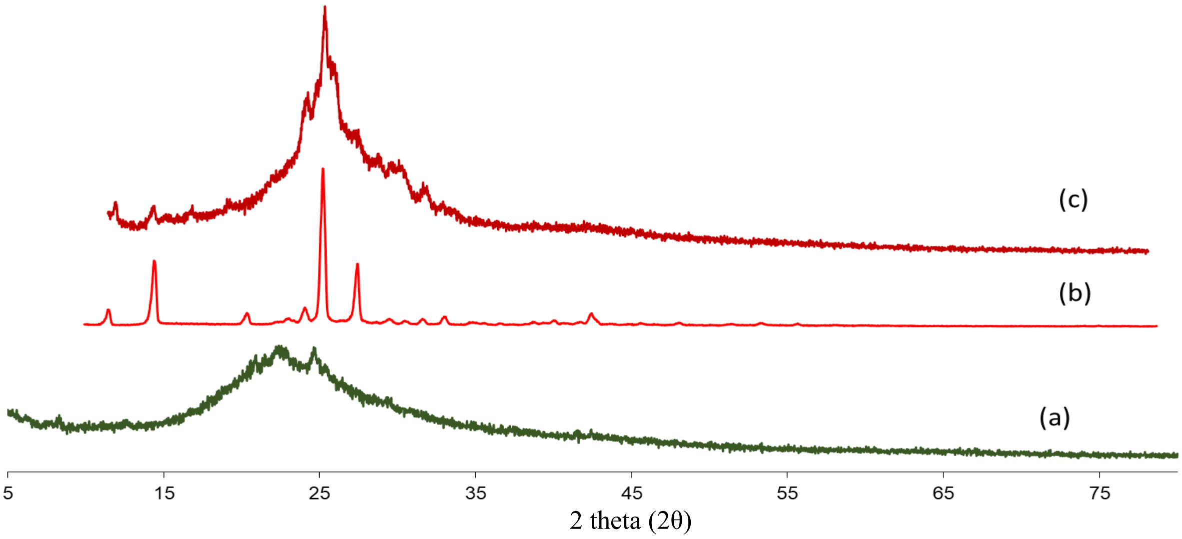

Fig. 8. XRD analysis (a) MF (b) 75% SA+25% CA (c) MPCM.

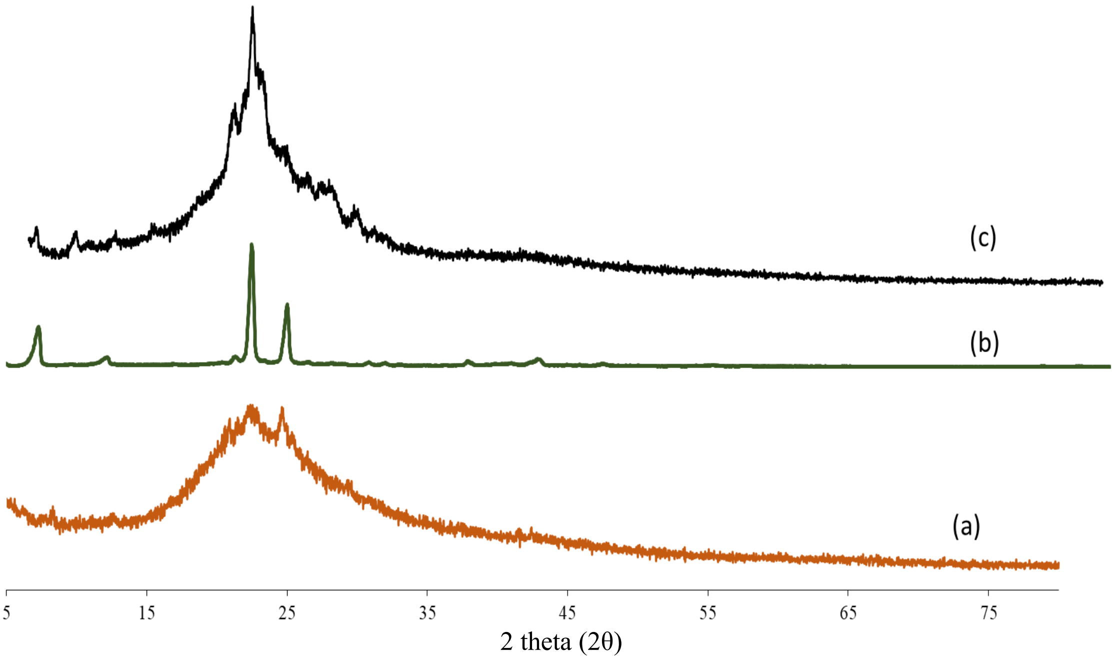

Fig. 9. XRD analysis (a) MF (b) 63% SA + 37% CA (c) MPCM.

4.4 Thermogravimetric analysis of microencapsulated eutectic mixtures

The weight loss of microencapsulated eutectic mixtures and their phase transitions were shown in Fig 10 and 11. In TGA analysis, two distinguish percentage weight losses are observed as temperature increased from 50 °C to 500 °C. From temperature 50 °C to 100 °C minor weight loss owing to moisture content over surface of the microcapsules. The primary percentage weight losses are 69% and 67% occurred in the temperature range 155 °C to 248 °C and 152 °C to 237 °C corresponding to microencapsulation of 75% SA + 25% CA and 63% CA + 37% PA due to evaporation of different composition of eutectic mixtures. The secondary weight losses are 19.4% and 23.3% due to decomposition of shell material contained in MPCMs in the temperature range of 270 °C to 450 °C Fig 10 (a) and (b). Thermal stability of phase transition of different eutectic mixtures is 202.5 °C [24] and 201 °C [25] corresponding to 75% SA + 25% CA and 63% CA+37% PA respectively. The thermal stability of microencapsulation of 75% SA + 25% CA increased from 202.5 °C to 241°C as shown Fig 11 (a) and 63% CA+37% PA from 201 °C to 228 °C Fig 11(b). Residue loss accounts for the remaining minor weight losses. When compared to microcapsules with 63 % CA+37 % PA, microcapsules with 75 % SA+25 % CA have a higher percentage weight loss. This indicates that the core content in the shell is more occupied due to encapsulation efficiency and density of eutectic mixtures. The varying thermal stability of microencapsulation of eutectic mixtures are based on their compositions between core and shell.

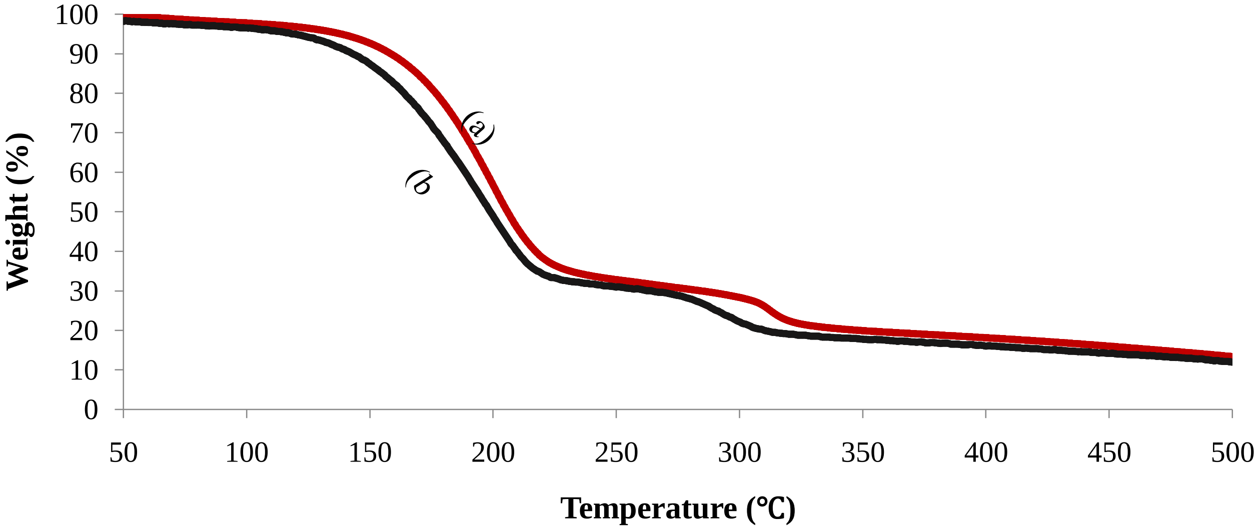

Fig. 10.TGA analysis of MPCM (a) 75% SA + 25% CA (b) 63% CA + 37% PA.

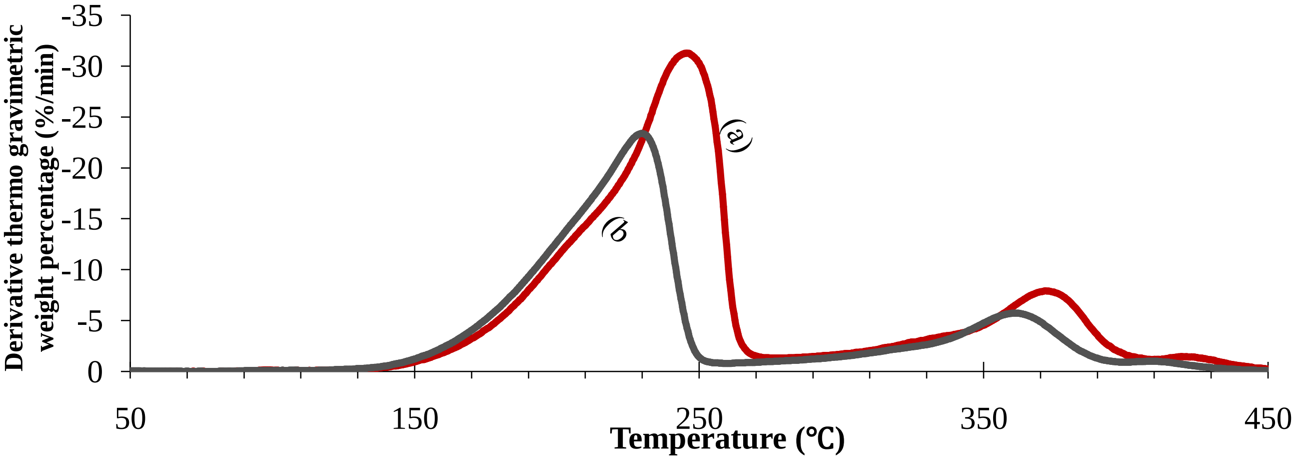

Fig. 11.DTG analysis of MPCM (a) 75% SA+25% CA (b) 63% CA+37% PA.

4.5 DSC analysis of microencapsulated eutectic mixtures

Energy can be storage in two ways such as sensible heat storage and latent heat storage. The latent heat of fusion has been carried out by using DSC instrument. The heating and cooling curve of microencapsulation of 75% SA+25% CA and 63% CA+37% PA shown in Fig 12 and 13. The melting point and latent heat of fusion of 75% SA + 25% CA and 63% CA + 37% PA are 35 °C 121.8 kJ/kg [24] and 36.2 °C and 134.75 kJ/kg [25] respectively. The tangent lines were drawn to left of the curves and determined area under curve refers to latent heat of fusion. The thermal properties of different microcapsules are 34.9 °C, 95.3 kJ/kg and 35.6 °C, 98.5 kJ/kg resultant to microencapsulation of 75% SA+ 25% CA and 63% CA + 37% PA respectively Fig 12 (a) and (b) and 13 (a) and (b). The latent heat of fusion of microcapsules of 63% CA + 37% PA is more as compared to microcapsules of 75% SA+ 25% CA. The difference in latent heat of fusion is owing to energy density of 63% CA + 37% PA is high. The core content in the shells were 78.2% and 73.09 %. The obtained results revealed that, the different microcapsules have the potential to incorporate in different applications such solar energy system and building components such as mortar, gypsum, concrete and bricks etc.

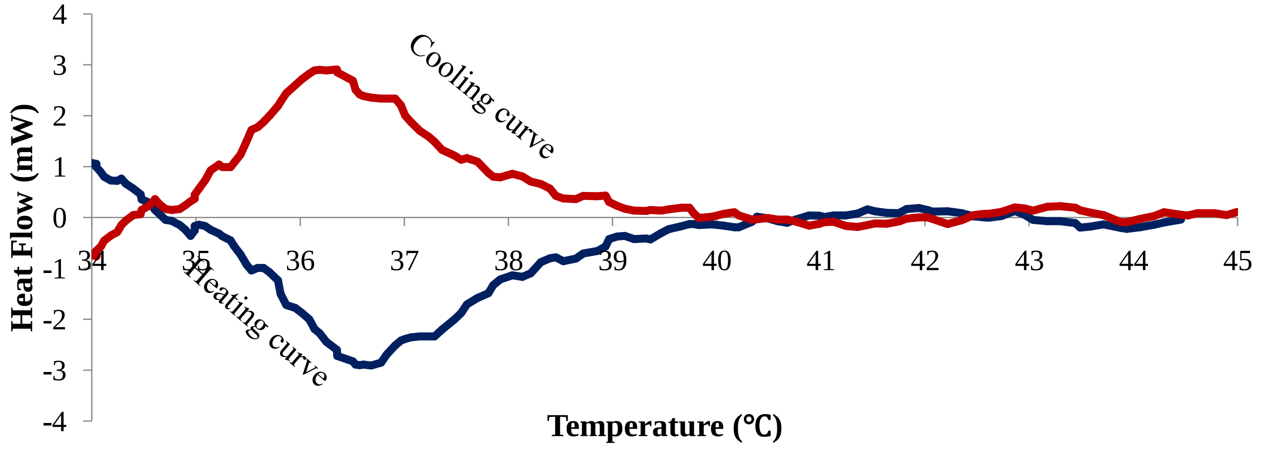

Fig. 12. DSC heating and cooling curve of MPCM of 75% SA + 25% CA.

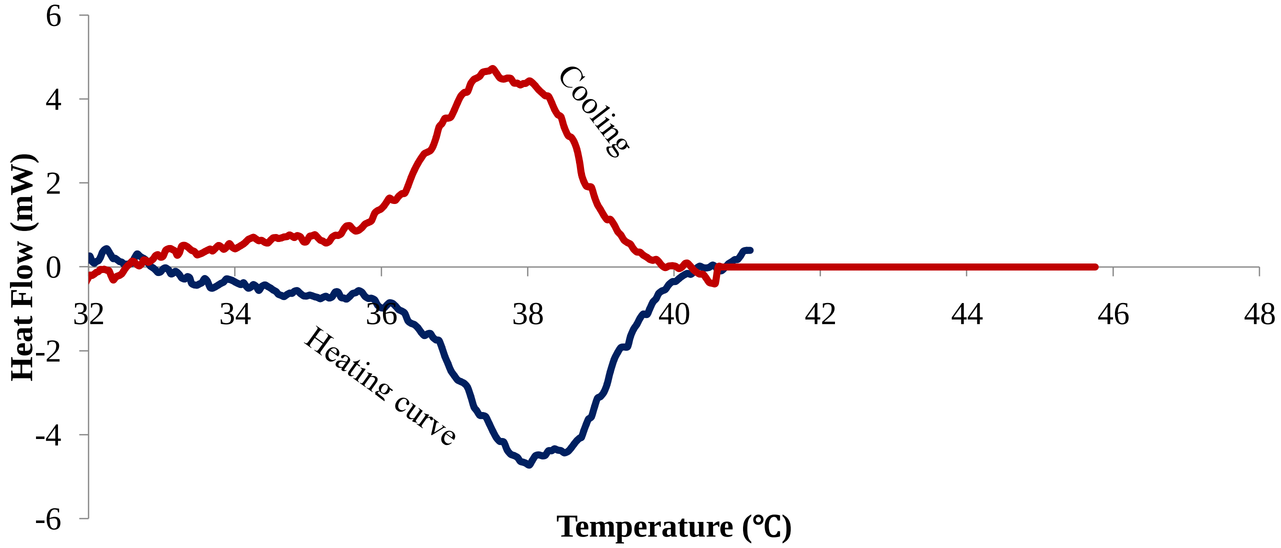

Fig. 13.DSC heating and cooling curve of MPCM of 63% CA + 37% PA.

4.6 Charging and discharging of microencapsulated eutectic mixtures

The charging and discharging of microcapsules of 75% SA+ 25% CA has been discussed in detail in our previous study[24]. The experimental setup of microcapsules of 63% CA + 37% PA was presented in Fig 14 and the obtained results are in Fig 15 (a) and(b) and Fig. 16(a) and (b). Both the eutectic mixture and the microcapsule were initiated at 33 $^{\circ}$C. The heat is fastened on account of sensible heat from 33 °C to 35.4 °C for 150 seconds. The temperature curves of the eutectic mixture and microcapsule were flat from 150 to 210 seconds at 35.4 °C. It indicates that the material melts and the heat is concentrated by latent heat of fusion for up to 210 seconds, when the material is entirely liquid Fig 15 (a) and (b). Figure 16 (a) and (b) illustrates the eutectic mixture and microcapsule discharge curves (b). The temperature drops suddenly from 41.4 °C to 35.1 °C, and the sensible heat is quickly released into the environment within 120 seconds. The heat is gently released to the atmosphere starting at 35.1 °C and continues until the substance solidifies entirely after 420 seconds. The charging and discharging of material are found to be dependent on the core content in the shell, the thermal conductivity of the shell, and the rate of heat transmission as a result of the findings.

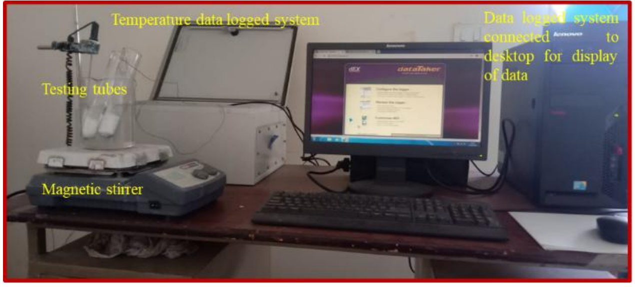

Fig. 14. Experimental set up for charging and discharging curve

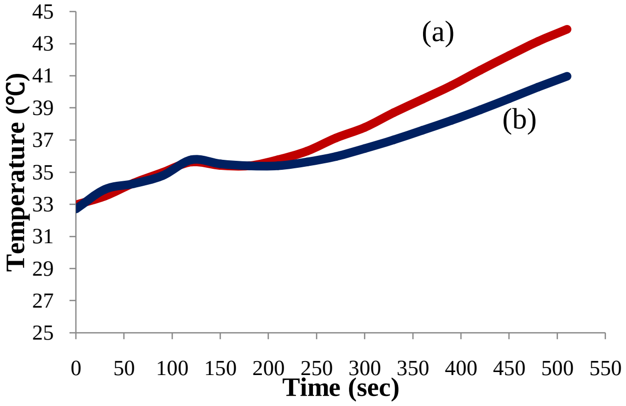

Fig. 15. Charging curve of (a) 63% CA + 37% PA (b) MPCM

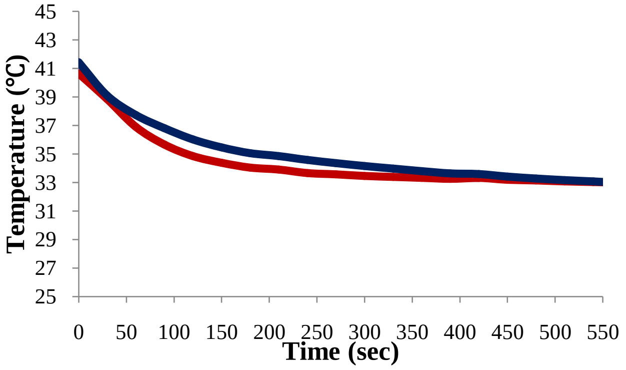

Fig. 16. Discharging curve of (a) 63% CA + 37% PA (b) MPCM

4.7 Percentage yield of the different microcapsules

The characterization of different microcapsules demonstrated that a pre-pilot scale development of in-situ polymerization process can produce around 1kg of product per day. It will be beneficial to incorporate into several domains such as building energy conversation, solar heating, paints, textiles, and food processing industries, as well as preparing various fatty acids such as LA, SA, CA, and Paraffin, among others. The resulting different products were cleaned with ethanol to remove any unformed microcapsules and dried before being weighed to determine the yield percentage. The yield of the product has been determined according to the following equation

$$\%Yield = \frac{M_1 - M_2}{M_1} \times 100 \qquad (8)$$

M_1 is amount of raw materials (g), M_2 is the amount of different microcapsules (g).

Table 4 Determination of yield of microcapsules

S. No	PCM	Amount raw materials (M1) (g)	Formation of MPCM (M2) (g)	%Yield
1	75% SA+25% CA	860	710	82.5
2	63% CA+37% PA	1059	870	81.6

The amount of individual raw materials and dosage of microcapsules were provided in table 4. The percentage yield of the products is obtained approx. 82%.According to Vogel and Furniss [26] different norms are categorized as follows quantitative yields are those that are close to 100 percent, excellent yields are those that are above 90%, very good yields are those that are above 80 %, good yields are those that are above 70%, fair yields are those that are above 50%, and poor yields are those that are below 40%. As per author, the obtained yield is falls under in very good achievement for pre-pilot scale preparation of various microcapsules. The obtained results compared with available literature as shown in table 5.

5. CONCLUSION

The microencapsulation of 75% SA + 25% CA and 63% CA + 37% PA successfully synthesized with designed and development of in-situ polymerization technique from lab scale to pre-pilot scale. The microcapsules are regular shaped, smooth and well encapsulated around eutectic mixtures. The mean particle size was about 3.6–6 µm and 2.5–4 µm corresponding to encapsulation of 75% SA + 25% CA and 63% CA + 37% PA respectively. The XRD and FTIR analysis of different microencapsulation revealed that, there is no interaction between the core and shell during preparation of microencapsulated eutectic mixtures. The thermal stability of different microencapsulation increased from 202.5 to 241 °C and 201 to 228 °C corresponding to distinguish microcapsules. The thermal properties of different microcapsules are 34.9 °C, 95.3 kJ/kg and 35.6 °C, 98.5 kJ/kg resultant to microencapsulation of 75% SA+ 25% CA and 63% CA + 37% PA respectively. The encapsulation of efficiencies was 78.2% and 73.09 %. The time taken to charging and discharging of microcapsules of 63% CA + 37% PA were 210 sec and 420 sec respectively. The generated products will be applied to evaluate thermal efficiency, energy demand reduction, and thermal characteristics such as specific heat and thermal conductivity in cementitious materials such as mortar, cement less concrete (i.e. gypsum), hollow bricks, and concrete.

Table 5 Lab scale preparation of different fatty acids.

S.No.	PCM	Amount (g)	Polymer	L.F (kJ/kg)	M.P (°C)	E.E(%)	Ref.
1.	Butyl stearate	25	MF	----	70	52.00	[27]
2.	Styrene maleic anhydride	10	MF	225	24	------	[28]
3.	n -dodecane	25	MF	125.2	26	90.00	[29]
4.	Hexadecane	120	MF	---	----	----	[10]
5.	Pentadecane	----	MUF	88.2	8.73	48.00	[30]
6.	n-Octadecane	15	MF	17.9	-----	71.20	[31]
7.	paraffin		MUF	134.3		77.10	[32]
8.	n-octadecane	7	PMMA	170	22.68	----	[33]
9.	Caprylic acid	15.31	PS	1.77–79.21	15–17	49.90	[13]
10.	Paraffin	---	PS	72.52	61	42.00	[34]
11.	LA	----	PS	167.26	43.77	91.64	[35]
12.	n-hexadecane	25	Poly (butyl acrylate	65.67	17	---	[36]
13.	LA	12	MF	84.96	45	---	[5]
14.	PA + CA	10	PS	46.3 and 77.3	13.5 and 17.1	---	[11]
15.	n-octadecane	20	MF	240	28	87.30	[37]
16.	Paraffin	----	UF	75.9	65	41.60	[38]
17.	n-tetradecane	10	polystyrene-silica	83.38	2.13	17.50	[39]
18.	$Na_2HPO_4\cdot 7H2O$	-----	SiO_2	159.8	50.1	82.41	[40]
19.	Paraffin	10-20	SiO_2	107.05	58.3	87.00	[41]
20.	n-Dodecanol	----	SiO_2	244	21	----	[42]
21.	63% CA + 37% PA	100	MF	167.26	43.77	----	[25]
22.	Paraffin	2	SiO_2	45	56.6	31.70	[43]
23.	n-octadecane	20	SiO_2	109.5	---	51.50	[44]
24.	Sodium phosphate dodecahydrate	20	SiO_2	177.0		75.3	[45]
25.	75% SA + 25% CA	20	MF	103.9	34.5	85	[24]
26.	Octadecane	15	SiO_2	227.66	28	---	[46]
27.	Na_2SO_4	1-10	SiO_2	170.60	885	----	[47]
28.	LA		SiO_2	93.80	41.5	44	[48]
29.	SA		SiO_2	46.00	71	23	[49]
30.	Paraffin		SiO_2	111.00	26.1	64	[50]
31.	75% SA + 25% CA	870	MF	95.3	34.9	78.2	Present study
32.	63% CA + 37% PA	710	MF	98.5	35.6	73.1	Present study

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Conflict of interest
The authors report no conflict of interest.

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