

# Preparation and Characterization of N-Eicosane PCM with Urea-Formaldehyde Shell

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**ABSTRACT:** Phase Change Materials (PCMs) have recently found a wide range of new application opportunities. In this study, PCMs microcapsules have been synthesized with urea-formaldehyde polymer shell. The microcapsules have been characterized by FT-IR, SEM, TEM, and DSC analysis. Then, the thermophysical characteristics of the MEPCM suspension including the thermal conductivity, and viscosity have been measured at different particle concentrations (2, 5, and 10 wt%) and different temperatures from 25 to 50 °C. New correlations are developed to predict the thermophysical characteristics of MEPCM suspensions. This work provides a practical and efficient synthetic strategy for the preparation of MEPCMs, which is expected to present a promising future in the field of energy storage.

**KEYWORDS:** Microcapsule; Energy storage; n-eicosane PCM with Urea-formaldehyde shell.

## INTRODUCTION

For several decades, the application of Phase Change Materials (PCMs) in thermal energy storage and thermal control systems has been widely investigated [1, 2]. One of the main applications of MEPCMs is for Thermal Energy Storage (TES). In this regard, many investigators studied the heat transfer and cooling performance of a MEPCM for thermal processes [3, 4]. *Ho et al.* [5] studied the performance of *n*-octadecane MEPCM particles to store thermal energy in an air-saturated enclosure. *Nomura et al.* [6] characterized a novel Al-Sialloy microspheres MEPCMs covered by  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> shells and elucidated their performance for high-temperature thermal energy storage. In another study, *Nomura et al.* [7] prepared MEPCM particles using Al-25 wt% Si core and a stable

$\alpha$ -Al<sub>2</sub>O<sub>3</sub> shell which had high cyclic durability and high heat capacity. In order to improve the thermal conductivity, *Wang et al.* [8] investigated the new MEPCM composites with a carbon network. *Song et al.* [9] reported that C-S MEPCM particles with silica shells have great potential for TES applications.

The working fluid, known as PCM slurry (PCMS), is another heat-storing concept that forms from a mixture of heat transfer fluid and PCM [10]. PCMS can be divided into one component (ice and water) and two components, including N/MPCMS, PCM emulsion (PCME), clathrate hydrate PCMS (ch-PCMS), and shape-stabilized PCMS (ss-PCMS) models [11, 12]. Each of them has been under scrutiny by various researchers worldwide to

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evaluate influential parameters and improve performance. For instance, *Alvarado et al.* [13] showed that MPCMS behave as a Newtonian fluid at mass fractions below 17.7% where the relative viscosity is independent of temperature. Also, microcapsule size has a great impact on durability and chemical stability. In another study, *Chen et al.* [14] demonstrated that the heat transfer enhancement ratio is getting larger by using the higher mass fraction of MPCMS. In a study on PCME, *Zhang et al.* [15] investigated different non-ionic emulsifiers' effects on the apparent stability, viscosity of PCME, and distribution of droplet diameter within the heat transfer fluid. *Castellani et al.* [16] classified ch-PCMS based on their melting temperature as  $T_m < 15\text{ }^\circ\text{C}$  for storing coolness in air conditioning,  $15 < T_m < 90\text{ }^\circ\text{C}$  suitable for solar heating and in heat load-leveling applications, and  $T_m$  is greater than  $90\text{ }^\circ\text{C}$  for absorption refrigeration. Each of the mentioned slurries has different advantages and drawbacks, and their potential for different applications is investigated. *Zhang et al.* [17] presented an overview of the design of two PCMS groups, namely MPCMS and semi-CHS. Comparing available materials for making PCMS based on the published data in the literature was also among their results. Later, *Zhao and Zhang* [18] carried out a comprehensive review of MPCM and MPCMS in four different aspects of fabrication, characterization, fundamental properties, and their application in the thermal energy storage systems used in textile and building industries. Furthermore, in complement to *Zhang et al.* [19] research, *Delgado et al.* [20] analyzed the information available in the literature for PCME and MPCMS, including thermophysical, rheological properties, and the heat transfer phenomenon. They presented a classification of different fabrication methods for these liquids, as well as commercially available products. *Jurkowska and Szczygiel* [21] worked on gathering data about hydrodynamic and thermal properties, such as thermal conductivity, specific heat, viscosity, pumping power, and pressure drop of MPCMS. Also, the methods and materials used for MPCMS preparation are described and compared with that of PCMS. Meanwhile, *Qiu et al.* [22] presented a review of research on MPCMS and their application in buildings.

The research was based on collecting information about the available MPCM, their coating material, thermal and structural stability as well as rheological and critical

properties of MPCMS. They suggested possible approaches for enhancing the heat transfer between MPCMS and its surroundings by considering several controversial phenomena and influential parameters. Besides available review articles about PCM slurries and emulsions, *Giro-Paloma et al.* [23] worked on the published studies and developments on MPCM in thermal energy storage systems with a focus on the different encapsulation methods and their various industrial applications.

This article aims to investigate the thermophysical characterization of such EPCM suspensions. The core material is *n*-eicosane because of its superb latent heat and desirable melting point of about  $\sim 36.4\text{ }^\circ\text{C}$ . Thus, it can be possibly employed for various applications such as building materials, thermal insulation, foams, fabrics, and thermally regulated fibers. The microcapsules have been characterized by FT-IR, TEM, and SEM analyses. The DSC is also conducted to measure the latent heat value, melting temperatures, freezing temperatures, and specific heat of MEPCMs. The thermophysical characteristics of the MEPCM suspensions including the viscosity and thermal conductivity have been determined at different temperatures and concentrations of phase change particles. Since the precise prediction of measurements is very crucial for practical and industrial applications, the predictive abilities of Maxwell's correlation [23, 24] for thermal conductivity and Vand's correlation [25] for viscosity have been compared with the experimental data.

## EXPERIMENTAL SECTION

### Working fluid preparation

The working fluid used in the experiments was the *n*-eicosane phase change capsules suspended in ultra-pure water (Millipore, USA) with a pH of about 5–6 at  $25\text{ }^\circ\text{C}$  and resistivity of  $18.2\text{ M}\Omega\cdot\text{cm}$ . The MEPCMs were produced by emulsion technique together with interfacial polycondensation [26]. *n*-Eicosane was the core of the phase change material. The water-soluble urea-formaldehyde solution (per-polymer) is used for emulsification. The required materials for the preparation process were as follows:

- *n*-Eicosane (Alfa Aesar Company, *n*-eicosane purity 99.0%, Melting point  $36.4\text{ }^\circ\text{C}$ )
- Urea monomer (Miani chemical, Inc, Urea purity 99.5%)

Table 1: Physical properties of PCM and MEPCM suspensions

$c_m$ (wt%)	0.0wt%	4.0 wt%	8.0 wt%	12 wt%	16 wt%
$\rho$ (kg m <sup>-3</sup> )	998.2	990.4	986.3	978.3	961.5
$c_{p,b0}$ (J kg <sup>-1</sup> K <sup>-1</sup> )	4180	4110(4109.2)	4040(4038.4)	3990(3967.6)	3858(3896.8)
$k_b$ (W m <sup>-1</sup> K <sup>-1</sup> )	0.601	0.581(0.591)	0.565(0.572)	0.549(0.554)	0.529(0.536)
$h_{fs}$ (J g <sup>-1</sup> ) <sup>a</sup>	-	5.74	12.8	22.59	30.54
$\mu$ (mPa.s)	0.78	1.41(0.86)	1.73(0.98)	2.18(1.12)	2.75(1.31)

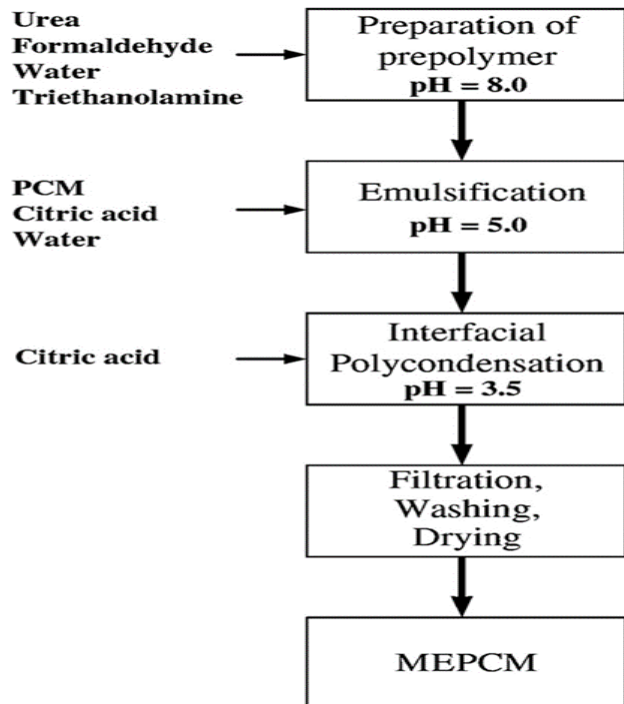


Fig. 1: Procedure for the preparation of MEPCMs

- Formaldehyde (Wako Pure Chemical Industries, Ltd., Formaldehyde purity 24%)
- Triethanolamine (Sigma-Aldrich, Triethanolamine purity 99.9%)
- Citric acid (Wako Pure Chemical Industries, Ltd., Citric Acid purity 99.0%)

The preparation procedure of MEPCMs is shown in Fig. 1. The pre-polymer was prepared from the reaction of formaldehyde and urea using an agitator in a 1-L glass reactor with a magnet stirring speed of 500 rpm for 10 min. In a typical experiment, a mixture of 60 g (0.75 M) 24% aqueous formaldehyde solution, and 30 g (0.5 M) urea monomer was heated at 80 °C for 2–3 h in the water bath. The pH was adjusted at 8.0 using triethanolamine which lowers the urea monomer and formaldehyde solution reaction rate.

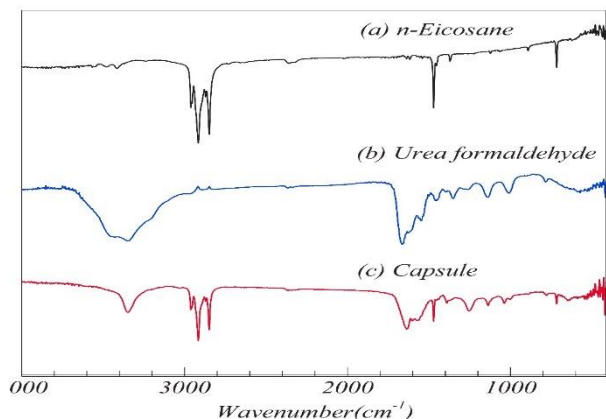
In the preparation procedure of Microcapsules containing liquid eicosane (C<sub>20</sub>H<sub>42</sub>), 10% aqueous citric acid was added into the pre-polymer solution to adjust its pH at 5.0. Then, 45 g liquid eicosane was further added while agitating. The emulsification reaction was carried out using a homogenizer at a speed of 18000 rpm and for a stirred duration of 15 min. In this step, the particle size reached the micron level. Further adjusting the pH to 3.5 with the addition of 10% citric acid to the solution at 45 °C was performed and then the agitation was done with the speed of 250 rpm for a duration of 4–5 h. Fig. 2(c) shows the microencapsulated phase change solution. As can be seen, it was floated in the upper solution because its density was lower than water. After filtration, washing for several times, and then drying, the MEPCM was yielded. In this experiment, the MEPCM particles were dispersed inside the ultra-pure water with various weight concentrations of 2%, 5%, and 10%, and the particles were distributed uniformly by a magnet stirrer.

Table 1 presents the physical properties of PCM and MEPCM suspensions.

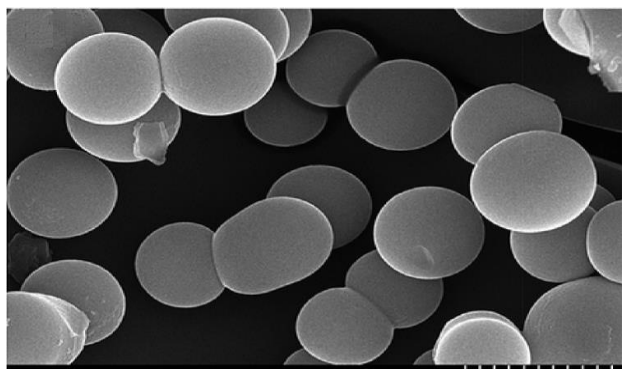
The weight concentration of MEPCM suspensions were 2%, 5%, and 10% and the corresponding concentration of phase change material (*n*-eicosane) were 1.02%, 2.35%, and 3.77% (see Table 1). Because the shell material (Urea formaldehyde resin) does not belong to the phase change material, there was no latent heat absorption and this fact led to lower concentrations of PCM rather than MEPCM.

#### Thermophysical properties measurement

Firstly, in order to measure the density of MEPCM particles, DA-505 Digital liquid density meter made by Kyoto Electronics (KRM) which had a measuring accuracy of within  $\pm 5 \times 10^{-5}$  g/cm<sup>3</sup> has been employed. The principle of the density meter was based on a vibrating U-tube method with an accuracy of up to 0.0001 g/cm<sup>3</sup>.



**Fig. 2: FTIR spectra for (a) *n*-eicosane core, (b) Urea formaldehyde shell, and (c) MEPCM**



**Fig. 3: SEM image of MEPCM under study**

The setup consisted of three main sections including the thermal conductivity/dynamic viscosity measurement device, the data acquisition system, and the constant-temperature bath. The constant-temperature bath consisted of a glass vessel (where the MEPCM suspension container was placed), a pump, and a vessel for heaters. During certain time steps, the temperature of the water inside the bath was checked and automatically adjusted by the heater to control the temperature. After reaching a steady-state condition, the temperature of the MEPCM suspension was set to the desired value which allows for measurement of thermophysical properties at various temperatures.

The transient hot-wire method by using KD2 Pro (Decagon Devices Inc., USA) was employed to determine the thermal conductivity of MEPCM suspensions. It was able to calculate this property with less than 5% error. The MEPCM suspension was held in a cylindrical container made from copper. The single needle sensor placed

in the vessel had a length and diameter of 60 and 1.3 mm, respectively.

The viscosity of water-based MEPCM suspension was determined at the temperatures of 25, 30, 35, 40, 45, and 50 °C. The Brookfield DV-II+ Pro programmable viscometer with a temperature bath was employed for measurement of the viscosity of MEPCM suspension. The Brookfield viscometer was a medium-to-high shear rate device with integrated temperature control and cone plate geometry with repeatability and accuracy of about  $\pm 0.2$  and  $\pm 1.0\%$ , respectively.

To guarantee the repeatability of experiments, they all repeated under different stirring conditions for each temperature and MEPCM concentration, and the average of the measurements was then recorded.

## RESULTS AND DISCUSSION

### MEPCM chemical characterization

Chemical characterization was done using Fourier-Transform InfraRed (FT-IR) spectroscopy. The FT-IR spectra for *n*-eicosane (PCM), urea-formaldehyde polymer (shell), and the phase change microcapsules based on *n*-eicosane (MEPCM) are shown in Fig. 2. It was seen that the characteristics absorption between 1180–1360  $\text{cm}^{-1}$  for C—N bonds, 1600–1800  $\text{cm}^{-1}$  for C=O bonds, and 3300–3500  $\text{cm}^{-1}$  for N—H bonds were obtained for *n*-eicosane MEPCM and urea formaldehyde polymer. It results indicate the successful encapsulation of alkane in the shell of the urea-formaldehyde polymer.

The SEM analyses were used to approximate the size of the MEPCM using JEOL JEM-1400 device. As shown in Fig. 3, it is clear that the shape of MEPCM was approximately spherical. Next, we analyze the median diameters of microcapsules ( $d_{50}$ ). The  $d_{50}$  value corresponds to 50% of a cumulative distribution and indicates that half the particles are smaller (or larger) than the median. The  $d_{50}$  value are 7.15  $\mu\text{m}$ .

A DSC with the Flash DSC 1 of Mettler-Toledo was used to measure the latent heat value, melting temperatures, freezing temperatures, and specific heat of MEPCMs. For DSC measurements, the heating/cooling rate was 5 K/min and the temperature ranged from 15 to 50 °C. The latent heat value of the phase change microcapsule particles is shown in Fig. 4, and its latent heat value was 136.2 J/g.

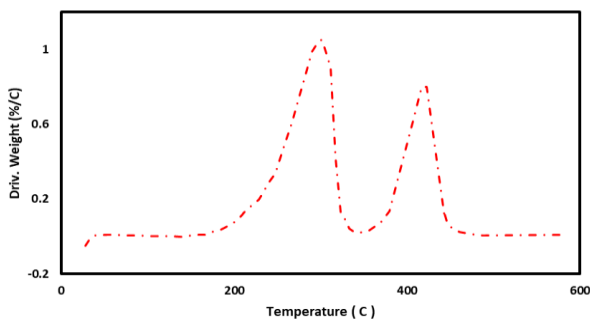


Fig. 4: DSC chart for phase change microcapsule powder

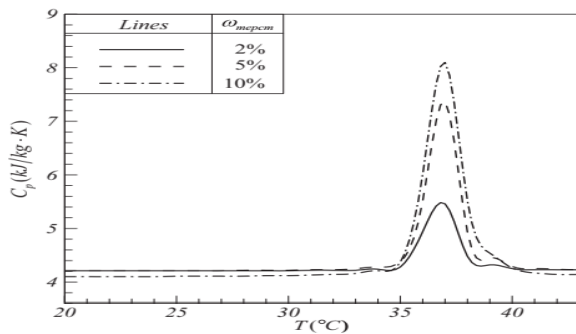


Fig. 5: The specific heat of the MEPCM suspensions with various concentrations

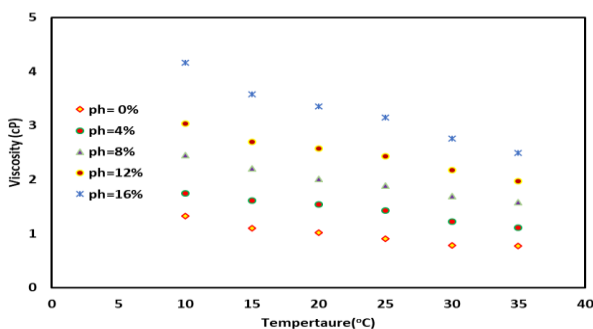


Fig. 6: Thermal conductivities of pure water and MEPCM suspension to pure water ratio.

Fig. 5 shows the specific heat of the MEPCM suspensions with different concentrations. It can be clearly seen that the higher concentration of the MEPCM suspension has a higher latent heat value, thus resulting in higher specific heat. Moreover, outside the phase change region, the increase in the concentration of the MEPCM suspension leads to a decrease in the specific heat.

#### Thermophysical characterization

The measured values of the thermal conductivity of the MEPCM suspension and pure water are given in Fig. 6.

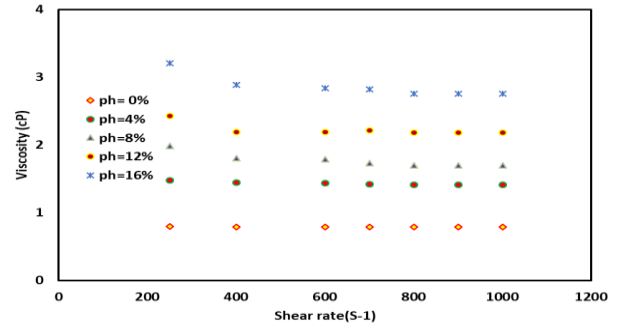


Fig. 7: Viscosities of pure water and MEPCM suspension to pure water ratio

It is seen that the thermal conductivity of pure water slightly increases with the increase in temperature. Based on the ratios of thermal conductivities, it is found that the MEPCM suspension thermal conductivity has also an increasing trend with temperature. In fact, by increasing the temperature from 25 °C (when the phase change material in the capsule is solid), the  $k_m$  enhances and by approaching the phase change periods for the suspensions, its ascending trend becomes much greater. This is because when the  $k_m$  is measured using a transient line heat-source technique, the measured value is affected by the latent heat of fusion. However, the melting process of the MEPCM particles at the temperature of  $\sim 36$  °C results in a significant reduction in the thermal conductivity at higher temperatures. When the phase change materials in the capsule are completely melted, the thermal conductivity begins to slightly increase with the temperature up to 50 °C.

Regarding the effect of MEPCM concentration, it is revealed that incrementing the MEPCM content is inversely proportional to the thermal conductivity. This relationship can be inferred from the values of thermal conductivity ratios for the concentrations of 2%, 5%, and 10% which fall below 1. This indicates the low thermal conductivity of MEPCM suspensions compared to pure water.

The viscosity of the MEPCM suspension and pure water is also concern in this research. Fig. 8 shows the relationship between temperature and viscosity coefficient at different MEPCM concentrations. The results show that the viscosity coefficient of pure water decreases with the increase of temperature. In addition, the viscosity ratios indicate that the PCM suspension has higher viscosity compared to the water since the all-viscosity ratios fall above 1.

Table 3: Comparison of empirical data with Maxwell and Vand correlations

Experimental condition		Empirical data		Correlations data	
MEPCM concentration	Temperature (°C)	Thermal conductivity ratio	Dynamic viscosity ratio	Maxwell correlation	Vand correlation
2%	25	0.9875	0.9593	0.7406	1.0685
2%	35	0.9743	0.8657	0.7307	1.0687
2%	45	0.9740	0.7705	0.7305	1.0689
5%	25	0.9692	1.2313	0.7269	1.1913
5%	35	0.9371	1.1079	0.7028	1.1918
5%	45	0.9363	1.001	0.7022	1.1926
10%	25	0.9325	1.4601	0.6993	1.5592
10%	35	0.8785	1.3281	0.6589	1.4692
10%	45	0.8770	1.2124	0.6578	1.4713

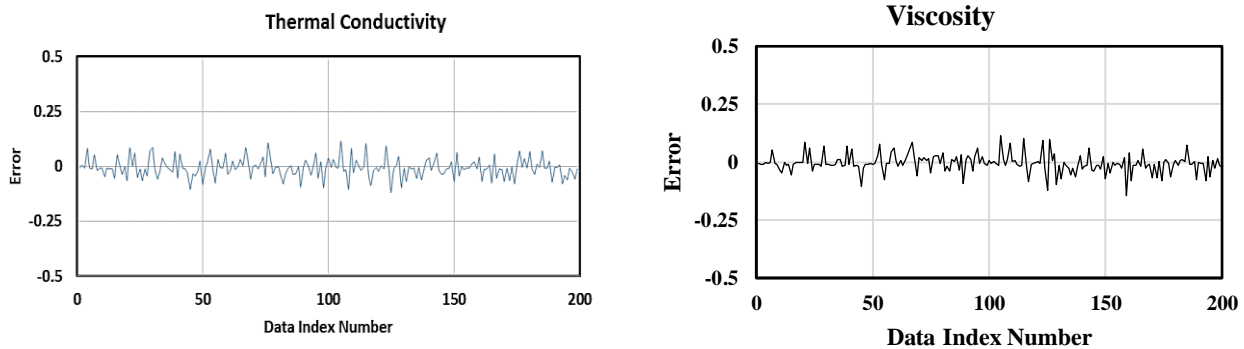


Fig. 8: Comparison between the measured and calculated thermal conductivity and viscosity

Note that the rheological behavior of suspensions of microcapsules is not affected by the melting process of PCMs due to the fact that the contacting surface with the carrying fluid is always the polymer shell.

Moreover, it is shown that for each temperature (beyond, within and over the phase change temperature) the viscosity increases with the augmentation of the particle concentration. The increase of MEPCM suspension viscosity with incrementing particles concentration is because of the strengthening of internal shear influence by MEPCM particles. These particles hinder the movements of layers of the fluid on each other which results in the increment of viscosity.

#### Proposing correlation for thermophysical characteristics

The thermal conductivity of MEPCM suspension,  $k_m$ , can be calculated according to Maxwell's correlation as suggested by other authors [23, 24]:

$$\frac{k_m}{k_{bf}} = \frac{2 + \frac{k_p}{k_{bf}} + 2c^* \left( \frac{k_p}{k_{bf}} - 1 \right)}{2 + \frac{k_p}{k_{bf}} - c^* \left( \frac{k_p}{k_{bf}} - 1 \right)} \quad (1)$$

where  $k_{bf}$  and  $k_p$  are the thermal conductivities of base fluid and that of the MEPCM particles.  $c^*$  represents the volume fraction of particles.

Furthermore, the bulk MEPCM viscosity,  $\mu_m$ , can be calculated by using Vand's correlation [25] as also recommended by several researchers [23],

$$\frac{\mu_m}{\mu_{bf}} = \left( 1 - c^* - 1.16c^{*2} \right)^{-2.5} \quad (2)$$

where  $\mu_{bf}$  is the dynamic viscosity of base fluid.

Table 3 presents the bulk thermal conductivities and viscosities calculated from the correlations (Eqs. (1), (2)) and measured by the experiments at three different temperatures. It is observed that Maxwell's correlation cannot predict the thermal conductivity of *n*-eicosane MEPCM, while Vand's correlation overpredict the measured values. This poor agreement can be attributed to the deviation of particle shape from the rigid spherical particles.

Therefore, two correlations have been proposed based on the empirical data using regression method in terms of MEPCM particle concentration and the temperature, as follows:

$$\frac{k_m}{k_{bf}} = \begin{cases} 0.0427(1 + \omega)^{-0.308} T^{0.55}, & T \leq 308 \text{ K} \\ 0.5062(1 + \omega)^{-0.12} T^{0.112}, & T > 308 \text{ K} \end{cases} \quad (3)$$

$$\frac{\mu_m}{\mu_{bf}} = 0.532(1 + \omega)^{5.589} T^{-0.109}$$

Where  $\omega$  and  $T$  represent the mass concentration of MEPCM particles and the temperature K, respectively. The comparison between the measured and predicted values of thermal conductivity and viscosity of the MEPCM suspension, as shown in Fig. 8, reveals that the proposed correlations are satisfactory and capable of accurate prediction of the thermophysical properties. The average deviations were 4.59% and 4.12% for  $k_m$  and  $\mu_m$ , respectively.

## CONCLUSIONS

Urea-formaldehyde polymer microcapsules containing *n*-eicosane PCM was successfully prepared with a particle size distribution of 7.5  $\mu\text{m}$ . FT-IR results revealed that the alkane was successfully encapsulated in the urea-formaldehyde polymer (shell). The latent heat values, freezing and melting temperatures and specific heat of MEPCMs were characterized using DSC. The thermal conductivity of the PCM suspensions was found to be lower than that of the pure water. The viscosity measurements revealed that increasing the concentration of MEPCM particles significantly increases the viscosity of the suspension for each temperature. Moreover, the rheological behavior of microcapsule suspensions was found to be not influenced by the PCM phase change process. Finally, the results of thermophysical properties were compared with the *Maxwell* and *Vand* correlation [25] and it is observed that Maxwell's correlation cannot predict the thermal conductivity of *n*-eicosane MEPCM, while Vand's correlation overpredict the measured values. Therefore, new correlations were proposed in terms of particle concentration and the temperature.

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