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Preparation and Characteristics of Microencapsulated Lauric Acid as Composite Thermal Energy Storage Materials

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A novel microencapsulated phase change material was prepared by sol-gel method using lauric acid (LA) as core material and titanium dioxide (TiO_2) as shell material. The composites were characterized by field emission scanning electron microscope (FE-SEM), Fourier transformation infrared spectrometer (FT-IR), differential scanning calorimeter (DSC) and thermogravimetric analyzer (TGA). The results of the FE-SEM and FT-IR indicated that LA was well coated in the shell of TiO_2 and no chemical reaction occurred between them. The results of the DSC denoted that the composites with 43.5 % encapsulation ratio of the LA melted at 44.39 °C with the melting enthalpy of 67.54 J/g and solidified at 43.64 °C with the solidification enthalpy of 65.17 J/g. The results of the TGA signified that the thermal stability and fire resistance of the microcapsules were greatly enhanced under the protection barrier of TiO_2. The thermal decomposition of the microencapsulated LA with TiO_2 shell hardly occurred under 200 °C. That is to say, the composites possess good thermal stability in the operating temperature range. In conclusion, the prepared microencapsulated composites can be incorporated into solar energy storage like solar air heaters and into electronic devices as heat sinks.

Keyword: microencapsulated composites, lauric acid, titanium dioxide shell, sol-gel, thermal properties.

1. INTRODUCTION

Latent heat storage systems are considered as one of the most promising thermal energy storage technology, which depends on the absorption and release of thermal energy by phase change materials (PCMs) [1]. In the past decades, a wide variety of PCMs have been investigated and employed widely in different fields like solar drying systems [2], heating and cooling of buildings [3], industrial waste heat recovery [4], photovoltaic electricity generation [5], domestic refrigerators [6]. Integrating PCMs into solar energy storage system can enhance the operation efficiency by up to 26 % [7]. Meanwhile, the PCMs as heat sink incorporated with electronic devices can prolong lifespan by up to 300 % [8].

Lauric acid is one of the most desirable organic PCMs due to its obvious advantages such as proper phase transition temperature, relatively high heat of fusion, good chemical stability, no toxicity and little supercooling [9]. However, it is difficult to use it directly in thermal energy storage because of its shortcomings like flammability, low thermal conductivity, the leakage of the melted lauric acid and poor thermal stability [9, 10]. Therefore, microencapsulation technique is the good solution to avoid these drawbacks of lauric acid.

The synthesis methods have been developed to encapsulate micro-sized PCMs droplets with shell materials, for example, emulsion polymerization [11], interfacial polymerization [12], sol-gel process [13], supercritical CO_2-assisted methods [14] and complex coacervation [15]. Microencapsulated phase change materials (MPCMs) are prepared by employing the PCMs as core material and using organic or inorganic materials as shell materials. The choice of shell materials plays an important part in regulating the characteristics of the microcapsules, which might influence microstructure, thermal stability, thermal conductivity as well as mechanical strength [16]. In the previous researches reported organic polymeric shell materials such as gelatin formaldehyde resin, melamine-formaldehyde, gelatin-gum Arabic and urea-formaldehyde [17]. This kind of polymeric shell materials have been investigated widely owing to to their good leakproofness and good flexibility, but their disadvantages restrict the wide applications of microencapsulated composites. This is because polymeric shell materials have the unavoidable features of flammability and low thermal conductivity [16]. Especially Some polymeric shells like urea formaldehyde may release poisonous gas that is harmful to environment and health [10, 13].

However, using inorganic shell materials to encapsulate PCMs can become a solution to avoid the above disadvantages of organic shell materials. Several studies reported that using inorganic shell materials such as silica [18, 19], $CaCO_3$ [20], AlOOH [21] encapsulated PCMs, but using TiO_2 as shell materials is still little reported. Compared with $CaCO_3$, TiO_2 is inherent with good chemical and thermal stability. Studies [22, 23] showed that titanium dioxide (TiO_2) is higher heat conductivity and better mechanical performances than silica (SiO_2), which indicated that TiO_2 has potential for inorganic shell materials. TiO_2 as inorganic shell material can lower the flammability and enhance the thermal stability as well as mechanical strength of the microcapsules [24]. Meanwhile, TiO_2 is innocuous and un-poisonous. Fatty acids are good alternatives, which are cheaper than paraffin. For instance, lauric acid can be extracted at low cost from renewable agricultural products such as coconut oil [9]. In this research, the

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main aim is to prepare a novel composites using lauric acid as core material and using TiO_2 as shell material by means of sol-gel. And the characterization of the prepared composites will be investigated using FE-SEM, FT-IR, DSC and TGA. By analyzing the results of the above characterizations, the prepared microencapsulated composites which combine the advantages of lauric acid and TiO_2 can be utilized in solar energy storage systems such as solar hot water systems and in electronic devices for cooling.

2. EXPERIMENTAL

2.1 Materials

Lauric acid ($C_{12}H_{24}O_2$, percentage of purity ≥ 98 %, AR) as phase change material was purchased from Aladdin Chemical Reagent CO., Ltd. Tetrabutyl titanate ($C_{16}H_{36}O_4Ti$, AR, Aladdin Chemical Reagent CO., Ltd.) was selected as precursor, which can produce titanium dioxide by hydrolysis condensation reaction. Ethyl alcohol (C_2H_5OH, AR, Xilong Chemical Reagent Company) and deionized water were acted as solvents. Hydrochloric acid as catalyst and stabilizer(HCl, AR) was received from Sinopharm Chemical Reagent Co., Ltd. Sodium dodecyl sulfate (SDS) ($C_{12}H_{25}SO_4Na$, AR) as O/W emulsifier was also obtained from Aladdin Chemical Reagent CO., Ltd.

2.2. Preparation of micro sized lauric acid droplets O/W emulsion

A certain amount of lauric acid and sodium dodecyl sulfate were added to the 250 ml beaker with deionized water (tabulated in Table 1), and the beaker was placed in a constant temperature magnetic stirring water bath at 65 °C and the rotational speed of the magnetic force was adjusted to 800 rpm continuing to be 45 min until the oil/water emulsion was formed.Hydrochloric acid was added drop by drop into the prepared emulsion to adjust the PH value to 1 – 2.

2.3. Preparation of the MPCMs

A certain volume of ethyl alcohol and tetrabutyl titanate (tabulated in Table 1) were blended together in another 250 ml beaker to form solution at normal temperature, and the beaker mouth was partially covered with a plastic film. Then it was added to the prepared lauric acid microemulsion drop by drop. At the same time, the temperature and stirring speed of the constant temperature magnetic stirring water bath were controlled at 65 °C and 500 rpm lasting 1.5 h. The formation process of the titanium dioxide shell is displayed in Eq. 1, Eq. 2 [24].

The different degrees of hydrolysis reaction of tetrabutyl titanate:

$$\mathrm{Ti(OBu)_4} + n\mathrm{H_2O} \rightarrow \mathrm{Ti(OBu)_4} \bullet_n (\mathrm{OH})n + n\mathrm{C_4H_9OH} \quad (n = 1,2,3,4) \qquad (1)$$

The structural formula of titanium dioxide oligomers is presented in Fig. 1.

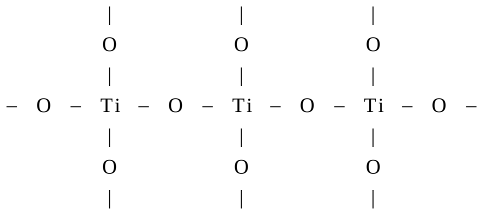

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Fig. 1. The structural formula of titanium dioxide oligomers

Under the condition of using ethanol as solvent and hydrochloric acid as catalyst, there were different degrees of hydrolysis reaction of tetrabutyl titanate occurring due to the action between Ti and -OH in H_2O molecule. Then intermolecular condensation reactions occurred in hydrolysis reaction products. And the molecules were further condensed so that the water molecules or alcohol molecules were removed to form the oligomer of titanium dioxide deposited on the surface of lauric acid microemulsion droplets. Finally, the white powder obtained from the experiment named MPCM1-MPCM4 was collected with filter paper and washed with deionized water and ethyl alcohol several times, then dried in vacuum drying oven at 60 °C for 24 h.

2.3. Characterization of the MPCMs

The microscopic morphology of titanium dioxide and microcapsules was observed and analyzed using a field emission scanning electron microscope (FE-SEM, JSM6701F, JEOL, Japan). The chemical structure of lauric acid, titanium dioxide, and microcapsules was analyzed using a Fourier Transform Infrared Spectrometry (FT-IR). A Nicolet5700 was utilized to record the infrared spectrum from $4000cm^{-1}$ to $400cm^{-1}$ with an accuracy of $0.09cm^{-1}$ using KBr tablets. The thermophysical properties were measured by a differential scanning calorimeter (DSC, NETZSCH5) at a heating or cooling rate of 5 °C/min under a nitrogen purge atmosphere of 20 ml/min [25].

Table 1. The composition of the lauric acid emulsion and solution of the tetrabutyl titanate

Samples	Lauric acid emulsion			Solution of the tetrabutyl titanate	
	Lauricacid, g	Deionized water, ml	SDS, g	Tetrabutyl titanate, g	Anhydrous ethanol, g
MPCM1	3	60	0.5	8	16
MPCM2	4	60	0.5	8	16
MPCM3	3	60	0.7	8	16
MPCM4	3	60	0.5	8	20

The intermolecular condensation reactions of hydrolysis reaction products:

$$\begin{aligned} &(\mathrm{OBu})_3\,\mathrm{Ti\text{-}OH} + \mathrm{HO\text{-}Ti}(\mathrm{OBu})_3 \rightarrow (\mathrm{OBu})_3\,\mathrm{Ti\text{-}O\text{-}Ti}(\mathrm{OBu})_3 + \mathrm{H_2O} \\ &(\mathrm{OBu})_3\,\mathrm{Ti} - \mathrm{OBu} + \mathrm{HO} - \mathrm{Ti}(\mathrm{OBu})_3 \rightarrow (\mathrm{OBu})_3\,\mathrm{Ti} - \mathrm{O} - \mathrm{Ti}(\mathrm{OBu})_3 + \mathrm{BuOH}\,. \end{aligned} \qquad (2)$$

The thermal stability was tested by a thermogravimetric analyzer (TGA4000, PE) from room temperature to 700 °C at a rate of 20 °C/min under the nitrogen atmosphere of 20 ml/min [13].

3. RESULTS AND DISCUSSION

3.1. Microscopic morphology of the microencapsulated composites

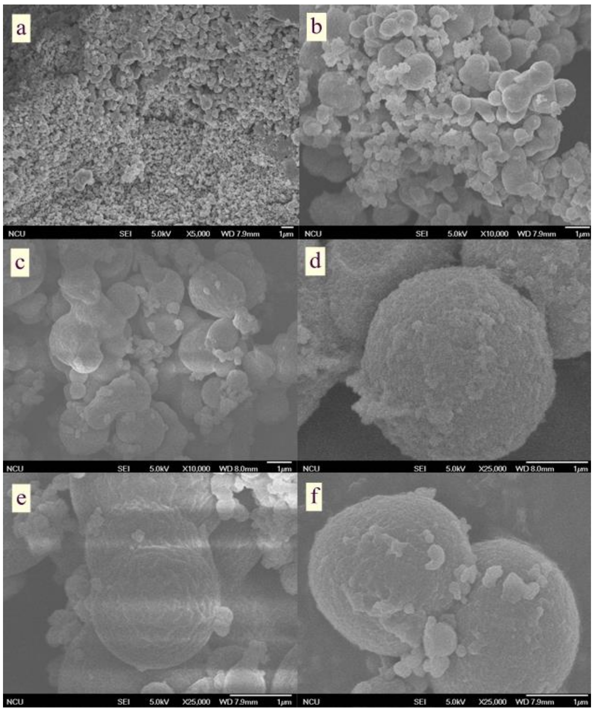

Fig. 2. SEM photographs: a – TiO_2(5k×); b – MPCM1(10k×); c – MPCM2(10k×); d – MPCM2(25k×); e – MPCM3(25k×); (f)MPCM4(25k×)

Fig. 2 presents the microstructure of titanium dioxide and four kinds of microcapsules MPCM1-MPCM4. Fig. 2 a displays a microscopic picture of blank titanium dioxide. Titanium dioxide particles have very large specific surface area at the micron level. As shown in Fig.2 b – f, it can be seen that lauric acid is well encapsulated by titanium dioxide into micron sized spherical particles or particle clusters. Similar results were observed in the previous research on microencapsulated paraffin with titanium dioxide shell [24].

3.2. FT-IR analysis of the microencapsulated composites

Fig. 3 exhibits the FT-IR spectra of the lauric acid, titanium dioxide and MPCM1-MPCM4. Fig. 3 a displays the spectrum of the lauric acid. The peak at 3449.27 cm^{-1} denotes the stretching vibration of -OH group [15]. The peaks at 2921.19 cm^{-1} and 2852.17 cm^{-1} are attributed to the asymmetric and symmetric stretching vibration of its $-CH_2$ group [13]. The peak at 1700.50 cm^{-1} is assigned to the stretching vibration of C=O. The peaks at 937.97 cm^{-1} and 723.75 cm^{-1} signify the out-of-plane bending vibration of -OH group and C-H, respectively [25]. Fig. 3 b displays the spectrum of the TiO_2. The stretching vibration of -OH group in H_2O is shown at 3418.98cm^{-1} [24]. The peaks at 2924.33cm^{-1} and 2851.56cm^{-1} are assigned to the asymmetric and symmetric stretching vibration of its $-CH_2$ group in absolute ethyl alcohol [13]. And the peak at 1384.21 cm^{-1} results from the in-plane bending vibration of $-CH_3$ group. The peak at 1622.52 cm^{-1} presents the deformation vibration of the -OH group in H_2O. The characteristic peak of titanium dioxide does not appear in the diagram because it is in the far infrared region. The result is consistent with that of this literature [24].

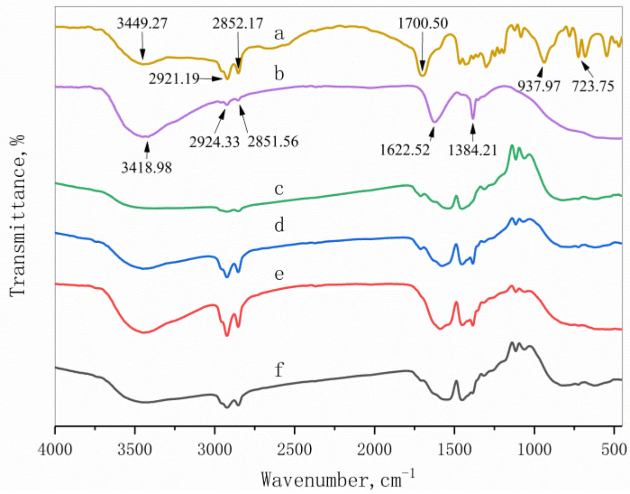

Fig. 3. FT-IR spectrum: a – lauric acid; b – TiO_2; c – MPCM1; d – MPCM2; e – MPCM3; f – MPCM4

As presented in Fig. 3 c and f, the absorption peaks of lauric acid and TiO_2 are observed in the spectrum of MPCM1-MPCM4. Based on this result that is no peak shift and occurring, it is confirmed that no chemical reaction occurring between lauric acid and TiO_2 shell.

3.3. Thermal properties of the microencapsulated composites

Fig. 4 and Fig. 5 shows the DSC graphs of the lauric acid and the microcapsules. The thermal properties parameters are listed in Table 2. It can be seen from Fig. 4 and Fig. 5 that both lauric acid and microcapsules have only one endothermic peak and one exothermic peak, and the microcapsules have similar thermal performance curves.

Table 2. DSC data of the lauric acid and MPCM1-MPCM4

Samples	Melting		Solidifying		Encapsulation ratio of the lauric acid, %
	T, °C	Latent heat, J/g	T,°C	Latent heat, J/g	
Lauric acid	44.53	155.27	42.39	152.64	100.00
MPCM1	44.77	34.85	43.58	32.57	22.44
MPCM2	44.39	67.54	43.64	65.17	43.50
MPCM3	44.20	35.79	43.78	33.64	23.05
MPCM4	44.67	38.94	43.36	37.28	25.17

As shown in Table 2, the melting temperature of lauric acid, MPCM1, MPCM2, MPCM3, MPCM4 is 44.53 ℃, 44.77 ℃, 44.39 ℃, 44.20 ℃, 44.67 ℃, respectively, and the solidification temperature of lauric acid, MPCM1, MPCM2, MPCM3, MPCM4 is 42.39 ℃, 43.58 ℃, 43.64 ℃, 43.78 ℃, 43.36 ℃, respectively.

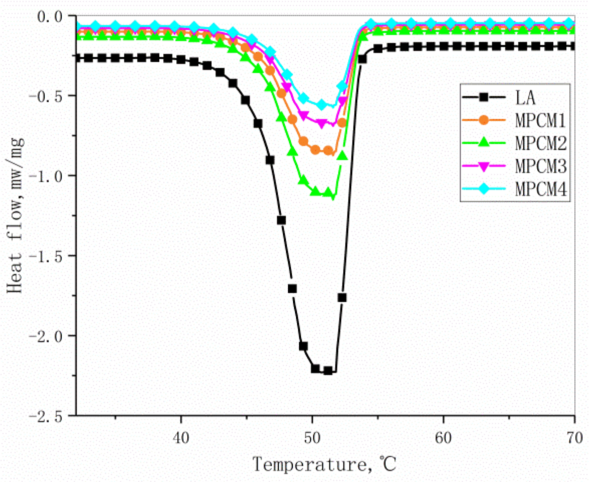

Fig. 4. The melting DSC graphs of the lauric acid, MPCM1, MPCM2, MPCM3 and MPCM4

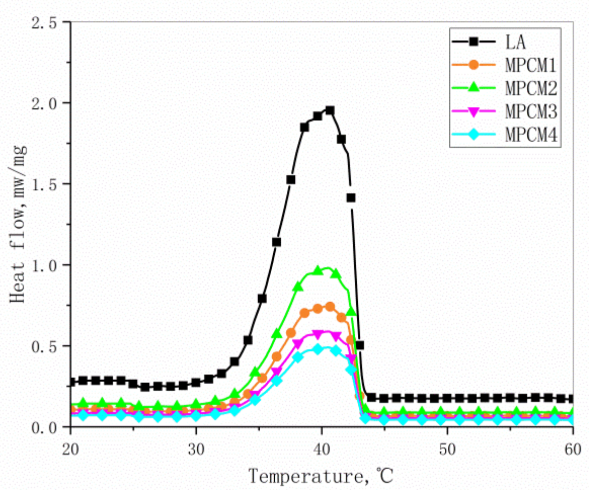

Fig. 5. The solidifying DSC graphs of the lauric acid, MPCM1, MPCM2, MPCM3 and MPCM4

The difference between the melting temperature and the solidification temperature of the lauric acid and the microcapsules are 2.14 ℃, 1.19 ℃, 0.75 ℃, 0.45 ℃ and 1.31 ℃ by calculation. The decrease of supercooling degree of microcapsules can be attributed to the good thermal conductivity of TiO_2 shell [22], which makes it act as nucleating agent during the solidification process of the microcapsules.

The encapsulation efficiency of lauric acid in microencapsuled composite phase change material can be calculated by Eq. 3 [34]:

$$\eta\% = \frac{\Delta H_{MPCM}}{\Delta H_{PCM}} \times 100; \quad (3)$$

where η is the encapsulation efficiency of the lauric acid in the microcapsules, ΔH_{PCM} represents the melting enthalpy of the lauric acid (J/g), ΔH_{MPCM} represents the melting enthalpy of the microcapsules as measured (J/g).

The encapsulation efficiency of lauric acid in the microcapsules was also listed in Table 2. It can be found that the value of lauric acid in the MPCM2 is larger than that in the MPCM1, MPCM3 and MPCM4. It can be explained that the larger mass ratio of the LA in the microcapsules is, the higher enencapsulation ratio of the microcapsules have. The difference of the encapsulation efficiency between MPCM1 and MPCM4 can be explained the amount of anhydrous ethanol, which influences the hydrolysis polycondensation reaction rate of tetrabutyl titanate [24].

Table 3 displays comparison of thermal properties between the prepared microcapsules and other microcapsules in literatures. Comparing other MPCMs in Table 3, the LA/TiO_2 microcapsules poesses a larger latent heat. It is found that these MPCMs have similar phase change temperature range within 40 – 80 ℃ for solar energy storage.

3.4. Thermal stability of the microencapsulated composites

Fig. 6 presents the TGA graphs of the lauric acid, TiO_2, MPCM1, MPCM2, MPCM3, MPCM4.The onset temperature of the mass loss (T_{onset}), the temperature of maximum weight loss rate (T_{oeak}) and the charred residue amount of the LA, TiO_2 and the MPCMs are exhibited in Table 4. As presented in Fig. 6, it is clear that the microcapsules display similar thermal stability characteristics and the TGA graphs of the microcapsules are much gentler compared to that of LA. The results are in accordance with that of relevant literature [17, 19, 23]. The charred residue amount of the empty TiO_2 is much larger than those of the microcapsules, which is indicated that TiO_2 possess good thermal stability and fire resistance. The residual amount of MPCM2 is smaller than that of MPCM1, MPCM3, MPCM4, because the content of lauric acid in MPCM2 is the largest, that is to say, the content of the titanium dioxide shell in MPCM2 is the least. It is indicated that the protective effective of TiO_2 shell is restricted due to its content in microcapsules. The residue amount of lauric acid is close to zero at 373 °C, but the residue amount of TiO_2, MPCM1, MPCM2, MPCM3, MPCM4 is 76.48%, 60.01%, 58.59%, 67.5%, 61.59%. This is the fact that TiO_2 shell builds up a physical protective layer coated on the surface of the microemulsion droplets of lauric acid,which prevents the exudation of the melted lauric acid and the escape of flammable gas produced in the process of thermal decomposition [24].

Table 3. Comparison of thermal performances between the LA/TiO_2 microcapsules and other microcapsules in literatures

MPCMs	Melting point, °C	Solidifying point, °C	Melting enthalpy, J/g	Solidifying enthalpy, J/g	Reference
Lauric acid/Activated carbon (1:2)	44.07	42.83	65.14	62.96	[25]
Docosane /PMMA	41.00	40.60	54.60	48.70	[26]
Stearic acid (25 wt.%)/TiO_2	53.84	53.31	47.82	45.60	[24]
Lauric acid (43.5 wt.%)/TiO_2	44.39	43.64	67.54	65.17	Present study

As listed in Table 4, the onset temperatures of the mass loss of the microcapsules are lower than that of the LA. This is owing to the evaporation of water and ethanol in the titanium dioxide shell [13]. In addition, the thermal decomposition temperature of the lauric acid in the microcapsules exceeds 200 °C while the operating temperature range of the composites is usually between 30 °C and 70 °C, so the prepared composites has desirable thermal stability and fire resistance in the filed of thermal energy storage.

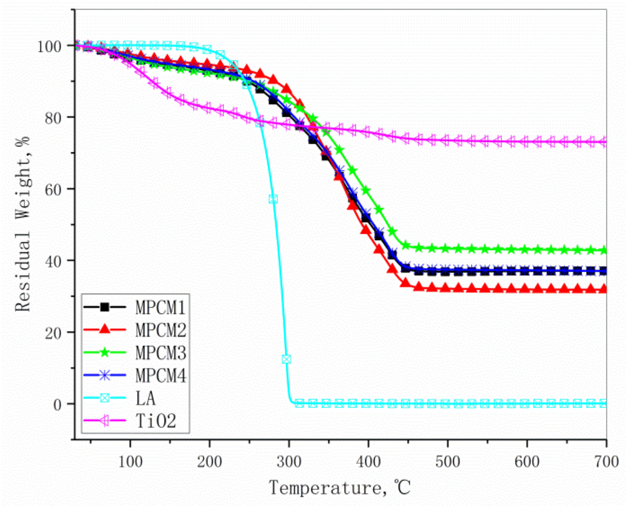

Fig. 6. TGA graphs of the lauric acid, TiO_2, MPCM1, MPCM2, MPCM3 and MPCM4

Table 4. TGA data of the lauric acid and MPCM1-MPCM4

Samples	T_{onset}, °C	T_{peak}, °C	Percent mass loss, % (700 °C)
Lauric acid	200	373	0.11
TiO_2	86	441	73.06
MPCM1	96	458	37.09
MPCM2	116	462	31.82
MPCM3	98	452	42.79
MPCM4	104	456	37.00

4 CONCLUSIONS

A novel microencapsulated composite phase change material using lauric acid as core and TiO2 as shell was prepared by sol-gel method. The prepared composites were characterized by a series of methods, and the results obtained were as follows:

1. The results of FT-IR and FE-SEM denoted lauric acid was compatible with TiO_2 each other and no chemical reaction occurred between them. Lauric acid microemulsion droplets were coated well into a spherical structure, which greatly increased the specific surface area of the composites. Meanwhile, TiO2 as the inorganic shell material is inherent with higher thermal conductivity than organic material, so that the heat transfer efficiency between the microcapsules and ambient air has been greatly improved.
2. According to the results of DSC and TGA, it is known that MPCM2 has a suitable phase transition temperature, good thermal stability and the phase enthalpy is larger than that of other three samples. MPCM2 melted at 44.39 °C with a latent heat enthalpy of 67.54 J/g and solidified at 43.64 °C with a latent solidification enthalpy of 65.17 J/g as the microencapsulation ratio is 43.50 %. Therefore, it was selected as the most satisfactory sample for this experiment. In addition, the residual amount of TiO_2 at 700 °C is still 73.06 % from the thermal stability analysis. It is concluded that TiO_2 prevents leakage of the melted lauric acid and possesses good thermal stability, which makes the thermal stability and fire resistance of the prepared microcapsules greatly enhanced. This is consistent with the results obtained from other literatures for the preparation of composite phase change materials with titanium dioxide as shell materials [24].

Based on the above results, it is concluded that the prepared microencapsulated composites have considerable thermal energy potential for solar energy storage systems and other potential applications such as electronic devices for cooling. In the next work, we will try to encapsulate the fatty acid eutectic with titanium dioxide, in order to obtain the microcapsuled composite phase change material with good thermal properties such as suitable phase transition temperature and strong heat storage capacity.

Acknowledgments

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