

Synthesis and Characteristics of Microencapsulated Decanol with TiO₂ Shell as Composites for Cold Energy Storage

Yuan LIU¹, Yang LIU², Yanghua CHEN^{3*}

¹ School of Mechanical and Automotive Engineering, Zhaoqing University, Zhaoqing 526060, China

² Department of Industrial Automation, Guangdong Polytechnic College, Zhaoqing 526114, China

³ School of Mechatronics Engineering, Nanchang University, Nanchang 330031, China

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A novel microencapsulated phase change materials for cold energy storage was synthesised through sol-gel means using decanol as phase change material and titanium dioxide (TiO₂) as encapsulated material. The micromorphology and composition of microcapsules were observed by field emission scanning electron microscope (FE-SEM), Fourier transformation infrared spectrometer (FT-IR). Using differential scanning calorimeter (DSC) and thermogravimetric analyzer (TGA) thermal properties of microcapsules were characterized. Results of FE-SEM and FT-IR indicated that micro sized decanol droplets were encapsulated with TiO₂ to form the well-developed core-shell structure, which was only physical coating between them. Furthermore, the chemical and thermal stability of the microcapsules were improved and the inflammability of the microcapsules was lowered using TiO₂ as shell material. The DSC result of the desirable ones melt at 3.87 °C with a latent melting enthalpy of 61.12 J·g⁻¹ and solidified at –1.32 °C with a latent solidification enthalpy of 59.54 J·g⁻¹. In general, the prepared microcapsules have potential for cold energy storage.

Keywords: microencapsules, decanol, titanium dioxide, thermal properties, cold energy storage.

1. INTRODUCTION

Thermal energy storage systems are mainly grouped into three types: sensible heat storage systems, latent heat storage systems and thermochemical heat storage systems [1]. Commonly used materials for sensible heat storage, such as water, molten salts, thermal oils, liquid metals [2], rocks [3] and so on, are applied in high temperature range due to their chemical stability in high temperature. However, its main drawbacks are large temperature fluctuation during the exothermic process and larger volume needed to storage the same amount of energy compared with that of latent heat storage systems [4]. Thermochemical heat storage based on reversible reactions that absorb or release heat have pros of higher thermal energy storage density than those of others, long duration and wide temperature range from 200 °C to 400 °C. But some of cons are such as high price, poor heat and mass transfer performance in the case of high density. This technology is still in progress and requires further developing before commercial applications [4]. Compared with other energy storage systems, latent heat storage systems using phase change materials (PCMs) that charge or discharge heat during phase transition possess two obvious advantages such as high energy storage density and phase change process at an isothermal state [5]. Nowadays, PCMs have been involved in diverse fields like food conserving, solar power plants, heating and cooling of buildings, greenhouse, photovoltaic electricity generation, industrial waste heat recovery, domestic refrigerators [6–12].

PCMs can be classified on the basis of phase transformation into solid-solid, solid-liquid, solid-gas and

liquid-gas. Among these kinds of PCMs, solid-gas and liquid-gas PCMs are restricted for thermal energy storage due to large volume containers during phase change process [13]. Solid-solid PCMs have pros of smaller volumetric expansion and less stringent container than those of solid-liquid PCMs, but the main con is lower latent heat compared with solid-liquid PCMs [14]. Organic solid-liquid PCMs because of their advantages such as good chemical stability, less or no subcooling, no separation, no corrosion, wide operating temperature range and high latent heat storage density, are preferred. But they can not be directly employed into practical use due to their high flammability and poor thermal conductivity (0.1 W·m⁻¹·K⁻¹ to 0.3 W·m⁻¹·K⁻¹) as well as easy leakage during the phase transition from solid to liquid [4].

The microencapsulation technique has been developed widely in the fields of chemical industry, food industry, biology industry and pharmaceutical manufacturing [15]. In recent years, microencapsulated phase change materials (MEPCMs) which use PCMs as core materials and select organic polymer or inorganic materials as shells have attracted much more attention. Because MEPCMs have the main pros like isolating and protecting the core from ambient, increasing the surface to volume ratio and preventing the liquid leakage during phase transition [16]. MEPCMs with organic polymer shell materials such as melamine-formaldehyde resins [17], polysiloxane [18], polystyrene [19] and urea-formaldehyde resins [20] are reported, but some of organic shell materials may release poisonous formaldehyde and their limited applications are attributed to their flammability and low heat conductivity.

* Corresponding author. Tel.: +86 13970944938.
E-mail address: 545422685@qq.com (Y. Chen)

Therefore, integrating the solid-liquid PCMs with inorganic shell materials to form microcapsules can avert from the above shortcomings. Zhang et al. [21] prepared the microcapsules using silica as shell material and n-octadecane as core material by means of sol-gel. Chen et al. [22] fabricated microcapsules using paraffin as core and SiO₂ as shell by sol-gel method. Pan et al. [23] microencapsulated palmitic acid with AIOOH by in situ interfacial poly-condensation method. Furthermore, studies [24, 25] indicated that titanium dioxide (TiO₂) has potential for inorganic shell materials. Because TiO₂ possesses higher heat conductivity and better mechanical performances compared with silica (SiO₂). Palmitic acid microcapsules with TiO₂ shell were synthesised through sol-gel method by Fang et al. [26].

In this paper, a novel microencapsulated decanol with TiO₂ shell as composites through sol-gel method was reported. Decanol as the organic core material is inherent with proper phase change temperature and high latent heat. The TiO₂ as shell material possesses desirable merits such as thermal and chemical stability, fire resistance and relatively high thermal conductivity, no toxicity as well as no corrosion. To the best of our knowledge, there are few literatures reported on microencapsulated alcohols with inorganic shell materials. Therefore, the main aim of this work is to prepare novel composites based on microencapsulated decanol with TiO₂ shell. And using FE-SEM, FT-IR, DSC and TGA investigates relevant characterizations of the prepared samples. It is expected that the prepared microencapsulated composites can be applied for cold energy storage.

2. EXPERIMENTAL

2.1. Chemicals

Decanol (C₁₀H₂₂O, n-decyl alcohol, percentage of purity ≥ 98 %, analytical reagent), tetrabutyl titanate (TBOT) (C₁₆H₃₆O₄Ti, analytical reagent) and sodium dodecyl sulfate (SDS) (C₁₂H₂₅SO₄Na, percentage of purity ≥ 92.5 %, analytical reagent) were obtained from Aladdin Chemical Reagent CO., Ltd. Hydrochloric acid (HCl, 36 % – 38 %, analytical reagent) which was produced in Sinopharm Chemical Reagent Co., Ltd. was selected as

catalyst. Ethyl alcohol (C₂H₅OH, analytical reagent) which was produced in Xilong Chemical Reagent Co., Ltd. and deionized water were used for solvents.

2.2. Preparation of the microencapsulated composite phase change materials

A certain amount of decanol and SDS (tabulated in Table 1) were added together in one beaker containing deionized water, and the beaker was put in the thermostatic magnetic stirring water bath at the temperature of 60 °C and stirrer kept stirring for 1 h with stirring speed of 800 rpm·min⁻¹ until the decanol oil-in-water micro emulsion was formed. Meanwhile, hydrochloric acid was dropwise added into the micro emulsion to adjust the PH value to 2.

Then TBOT and ethyl alcohol were mixed together into another one beaker whose mouth was covered with a layer of plastic wrap. Subsequently the solution was instilled into the prepared oil-in-water emulsion that continued to keep the original water bath temperature and magnetic stirring speed until the reaction was completed. During this process, the hydrolysis and condensation reaction of TBOT took place and the schematic diagrams were shown in Reaction Eq. 1 to reaction Eq. 3 [26]. The product of the reaction was white powder, which was collected with filter paper and washed several times with ethanol and deionized water, respectively and dried in the vacuum drying oven at 60 °C for 24 h. Finally, they were named MEPCM1, MEPCM2, MEPCM3, MEPCM4, MEPCM5, respectively.

2.3. Characterizations of the prepared microencapsulated composites

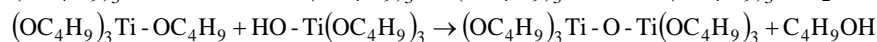
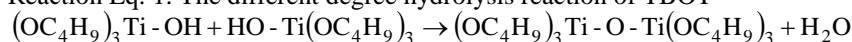
The micro morphology of the prepared four samples was observed using a field emission electron microscope (FE-SEM, JSM6701F, JEOL, Japan). The chemical composition of decanol, TiO₂ and five samples was analyzed using a Fourier transform infrared spectrometer (FT-IR, Nicolet5700), which was recorded the infrared spectra from 4000 cm⁻¹ to 400 cm⁻¹ with an accuracy of 0.09 cm⁻¹ using KBr pellets. A differential scanning calorimeter (DSC, DSC8500) measured the thermal properties of the five samples at a heating or cooling rate of 5 °C·min⁻¹ under a constant stream of nitrogen purge atmosphere of 20 ml·min⁻¹.

Table1. The composition of the decanol emulsion and solution of the tetrabutyl titanate

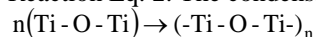
Samples	Decanol emulsion			Solution of the tetrabutyl titanate	
	Decanol, g	Deionized water, ml	SDS, g	Tetrabutyl titanate, g	Anhydrous ethanol-water, g
MEPCM1	3	60	0.5	5	16
MEPCM2	4	60	0.5	5	16
MEPCM3	5	60	0.5	5	16
MEPCM4	3	60	0.5	5	18
MEPCM5	3	60	0.5	5	20



Reaction Eq. 1. The different degree hydrolysis reaction of TBOT



Reaction Eq. 2. The condensation reaction between the products of different degree hydrolysis



Reaction Eq. 3. Network-like titanium alcohol gel (TiO₂)_n

Using a thermogravimetric analyzer (TGA4000, PE) investigated the thermal stability of decanol, TiO₂ and five samples from room temperature to 700 °C at a rate of 20 °C min⁻¹ under the nitrogen atmosphere of 20 ml·min⁻¹.

3 RESULTS AND DISCUSSION

3.1. Micro morphology of the prepared microcapsules

Fig. 1 shows the micro morphology of four samples. As seen in Fig. 1 a to d, decanol was well coated in the titanium dioxide shell to form micro-scale spherical and compact capsules. Among the previous literatures, similar photographs presents that inorganic shell materials such as silica, CaCO₃ encapsulated the PCM well [19, 24]. Compared with Fig. 1 b and c, Fig. 1 a displays relatively irregular microscopic morphology whose surface is deposited by some titanium dioxide particles, and the particle size is randomly distributed. As seen in Fig. 1 d, the microcapsules have poor shape structure with agglomeration. In short, the different micro morphology of four samples were caused by different proportion of reactants.

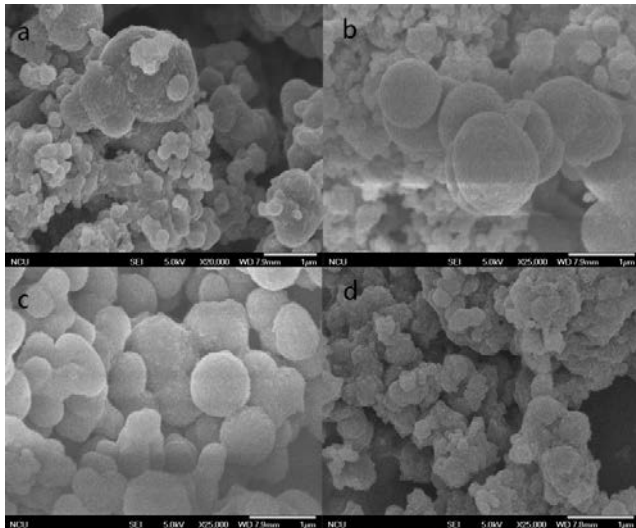


Fig. 1. The SEM images: a–MEPCM1 (20 k \times); b–MEPCM2 (25 k \times); c–MEPCM3 (25 k \times); d–MEPCM4 (25k \times)

3.2. Chemical composition of the prepared microcapsules

Fig. 2 displays the FT-IR spectra of decanol, titanium dioxide, for five samples. The FT-IR spectrum of decanol is displayed in Fig. 2 a. As shown in Fig. 2 a, the peak at 3419.30 cm⁻¹ results from the stretching vibration of the functional group –OH. The peaks at 2926.24 cm⁻¹ and at 2856.20 cm⁻¹ are attributed to anti symmetric and symmetric stretching vibration of the C–H bond in the –CH₂ group, and the scissor bending vibration and in-plane bending vibration of –CH₃ group correspond to the peak at 1462.05 cm⁻¹ as well as the peak at 1383.53 cm⁻¹, respectively. The peaks at 1348.01 cm⁻¹ and at 1262.20 cm⁻¹ are attributed to the in-plane vibration of –OH group. The out of plane bending vibration of C–H bond in the –CH₂ group corresponds to the peak at 720.69 cm⁻¹. The peak at 935.30 cm⁻¹ ascribes to the out of plane bending

vibration of the –OH group. The spectra of titanium dioxide is shown in Fig. 2 b. As seen in Fig. 2 b, the absorption peak of titanium dioxide does not appear in the diagram because it is in the far infrared region [27]. The stretching vibration of –OH group in H₂O is shown at 3750.46 cm⁻¹ and at 3418.98 cm⁻¹. The peaks at 2924.33 cm⁻¹, 2851.56 cm⁻¹ are assigned to the anti symmetric and symmetric stretching vibration of its –CH₂ functional group in absolute ethyl alcohol. The peak at 2961.88 cm⁻¹ is attributed to the asymmetric stretching vibration of –CH₃ group. And the in-plane bending vibration of –CH₃ group results in the peak at 1384.21 cm⁻¹. The peak at 1622.52 cm⁻¹ results from the deformation vibration of the –OH group in H₂O. Similar results can be obtained from relevant literatures [19, 28]. It is known that from Fig. 2 c to g, the FT-IR spectrums of five samples include all the characteristic peaks of decanol and titanium dioxide. Combined with relevant literatures for reference, it can be concluded that there is no chemical reaction between the core-shell of microcapsules [27]. This result is in agreement with the images observed by FE-SEM. Titanium dioxide formed by the hydrolysis and condensation of TBOT encapsulated the micro droplets to form the microencapsulated phase change materials.

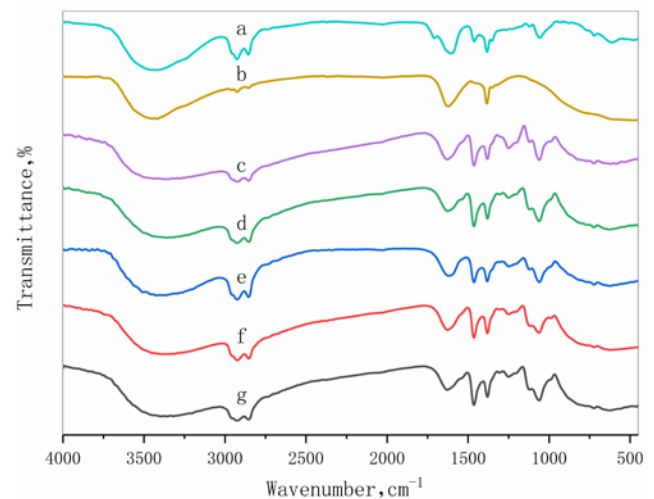


Fig. 2. The FT-IR spectrum: a–decanol; b–TiO₂; c–MEPCM1; d–MEPCM2; e–MEPCM3; f–MEPCM4; g–MEPCM5

3.3. Thermal properties of the prepared microcapsules

Fig. 3 and Fig. 4 display the DSC graphs of the melting process and solidification process of five samples, respectively. And the thermal property parameters are tabulated in Table 2. As shown in Fig. 3 and Fig. 4, five samples have similar melting and solidification process, each with only one endothermic peak and one exothermic peak. As can be seen from Table 2, the measured onset melting temperature of MEPCM1, MEPCM2, MEPCM3, MEPCM4, MEPCM5 is 0.78 °C, 3.33 °C, 3.87 °C, –2.80 °C, 0.68 °C, respectively. And the measured onset solidification temperature of MEPCM1, MEPCM2, MEPCM3, MEPCM4, MEPCM5 is 0.14 °C, –2.98 °C, –1.32 °C, –8.93 °C, –5.18 °C, respectively. The difference between the onset melting temperature and the onset solidification temperature of MEPCM1, MEPCM2,

MEPCM3, MEPCM4, MEPCM5 is calculated as 0.64 °C, 6.31 °C, 5.19 °C, 6.13 °C, 5.86 °C, respectively.

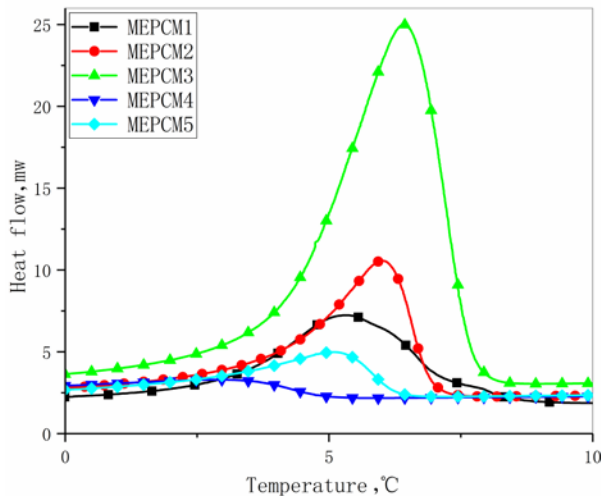


Fig. 3. The melting DSC graphs: a – MEPCM1; b – MEPCM2; c – MEPCM3; d – MEPCM4; e – MEPCM5

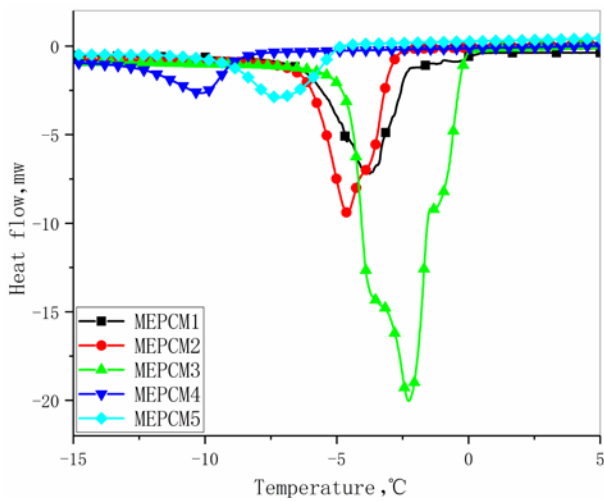


Fig. 4. The solidifying DSC graphs: a – MEPCM1; b – MEPCM2; c – MEPCM3; d – MEPCM4; e – MEPCM5

Table 2. DSC data of the decanol and MEPCM1-MEPCM5

Samples	Melting points, °C		Melting enthalpy, J/g	Solidifying points, °C		Solidification enthalpy, J·g ⁻¹
	T_{onset}	T_{peak}		T_{onset}	T_{peak}	
MEPCM1	0.78	5.62	12.94	0.14	-3.95	11.08
MEPCM2	3.33	5.78	25.11	-2.98	-4.61	25.55
MEPCM3	3.87	6.28	61.12	-1.32	-2.37	59.54
MEPCM4	-2.80	2.38	5.45	-8.93	-10.22	7.31
MEPCM5	0.68	4.57	11.27	-5.18	-7.27	13.09

Table 3. Comparison of the thermal property parameters between the microcapsules in this experiment and other microcapsules reported in the previous literatures

Materials	Types	Melting point, °C	Melting enthalpy, J/g	References
Eicosane/PMMA	MPCM	54.2	35.2	[29]
Paraffin/SiO ₂	MPCM	56.5	45.5	[30]
Zein:dodecane (70:30)	Organic	-10	34.5	[31]
Paraffin RT3	Organic	2-5	198	[32]
Paraffin RT4	Organic	2-4	281	[32]
90 % NaCl/H ₂ O	Inorganic (salt hydrate)	-5	289	[33]
Decanol/TiO ₂	MPCM	3.87	61.12	Present study

As also listed in Table 2, with the increase content of decanol in the microcapsules, the latent heat of fusion and latent heat of solidification of MEPCM1, MEPCM2, MEPCM3 all increase. In the microcapsules, the higher phase change material mass ratio represents the higher heat storage capacity, thus MEPCM3 is considered as the desirable microencapsulated phase change material in this experiment. The difference of melting enthalpy and solidification enthalpy in the MEPCM1, MEPCM4, MEPCM5 may be caused by the different amount of absolute ethanol. Absolute ethanol can influence the hydrolysis polycondensation reaction rate of butyl titanate [27].

Table 3 tabulates different thermal property parameters between the microcapsules in this experiment and other microcapsules reported in previous literatures. It can be seen that most of microencapsulated phase change materials are for thermal energy storage and some of phase change materials for cold energy storage are inorganic materials. Although inorganic cold storage materials have high energy storage capacity, obvious drawbacks of their large supercooling and phase separation limits applications of energy storage [16]. Based on the above simple analysis, the prepared microencapsulated decanol with titanium dioxide shell has large potential in cold energy storage.

3.4. Thermal stability of the prepared microcapsules

Fig. 5 displays the TGA curves of decanol, titanium dioxide and MEPCM1, MEPCM2, MEPCM3, MEPCM4, MEPCM5, and Table 4 lists some data on the onset temperature of the mass loss (T_{onset}), the temperature of maximum mass loss rate (T_{peak}) and the charred residual amount of decanol, titanium dioxide as well as five samples. As shown in Fig. 5, five samples have similar thermal decomposition curves of samples decrease with the increase content of decanol in the microcapsules. Meanwhile, the thermal decomposition process of TiO₂ is much gentler than those of others and the weight loss of TiO₂ is only 37 % at 700 °C, but the residual amount of decanol is less than 1 % at 233 °C.

As also listed in Table 4, the residual amount at 233 °C of MEPCM1, MEPCM2, MEPCM3, MEPCM4 and MEPCM5 is 64.99 %, 58.21 %, 46.61 %, 63.67 % and 52.20 %, respectively.

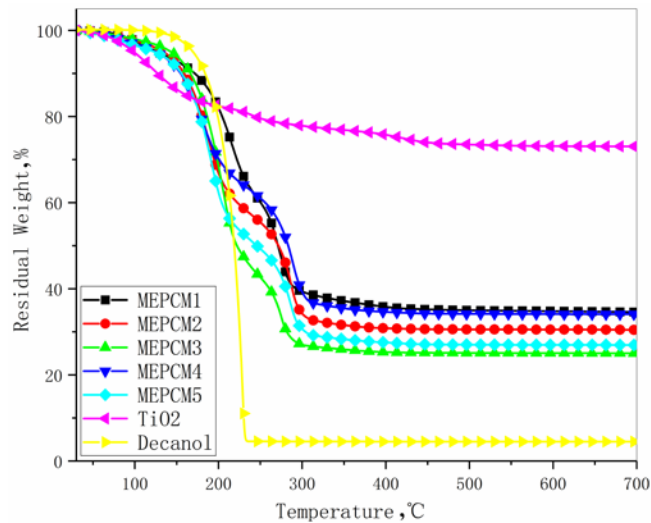


Fig. 5. TGA graphs: a –decanol; b –TiO₂; c –MEPCM1; d –MEPCM2; e –MEPCM3; f –MEPCM4; g –MEPCM5

Table 4. TGA data of the decanol, TiO₂ and MEPCM1-MEPCM5

Samples	T_{onset} , °C	T_{peak} , °C	Residual mass percentage, % (700 °C)
Decanol	167	185	0.11 (233 °C)
TiO ₂	99	163	73.06
MEPCM1	127	188	34.58
MEPCM2	134	191	30.46
MEPCM3	143	215	25.00
MEPCM4	125	198	34.09
MEPCM5	123	207	26.93

Based on the above, it is indicated that titanium dioxide as shell material can build up a layer of protective barrier on the surface of micro sized decanol droplets so that fire resistance and thermal stability of the composites can be enhanced. This result is nicely in consistent with the FT-IR analysis. Related literatures on inorganics as shell materials that can overcome the flammability of organic PCMs have been reported [21, 22, 28]. In the next work, microencapsulated eutectics with inorganic materials will be investigated.

4. CONCLUSIONS

A novel microcapsules using decanol as core material and titanium dioxide as shell was prepared via sol-gel method and a series of characterizations were employed to investigate the prepared microcapsules. The results obtained are as follows:

The micro-sized decanol droplets are well coated with TiO₂ and there is no chemical reaction occurring between decanol droplets and TiO₂. Combined with the results of TGA, it is concluded that TiO₂ enhance the thermal stability of microencapsulated composites by building up a layer of physical protective barrier on the surface of decanol droplets and lower the flammability of microcapsules. The desirable sample MEPCM3 in this experiment melts at 3.87 °C with a latent heat of fusion of 61.12J·g⁻¹ and solidifies at -1.32 C

with a latent heat of solidification of 59.54J·g⁻¹. Based on the above analysis, the prepared microcapsules can be incorporated into cold energy storage systems like domestic refrigerators, refrigerated trucks, medical products to provide thermal protection.

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