

Poly(methyl methacrylate) copolymer nanocapsules containing phase-change material (*n*-dodecanol) prepared via miniemulsion polymerization

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ABSTRACT: Poly(methyl methacrylate) copolymer nanocapsules containing the phase-change material *n*-dodecanol, which could be used for energy storage, were prepared with different comonomers via miniemulsion polymerization. The thermal properties, morphology, and composition of nanocapsules were characterized with differential scanning calorimetry, thermogravimetric analysis, transmission electron microscopy, and Fourier transform infrared spectroscopy. The results show that the thermal properties and morphology of the nanocapsules were influenced greatly by the type and amount of comonomers. Under the same dosage of 4 wt %, the nanocapsules prepared with the comonomer acrylamine and which had a moderate hydrophilicity showed the highest phase-change latent heat of 109.3 J/g; the acrylamine that had a moderate hydrophilicity and the highest encapsulation efficiency of 91.3%. The size of the nanocapsules ranged from 50 to 100 nm with a uniform spherical shape and apparent core-shell structure. We also found that when the amount of the soft comonomer butyl acrylate was increased, the phase-change latent heat of the nanocapsules first decreased slightly, then increased to the maximum value with deformed spherical and conglomerated morphology, and finally decreased continuously. The thermal stability of the nanocapsules became weaker with higher contents of core material. © 2015 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2015**, *132*, 42334.

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INTRODUCTION

Because energy resources are intermittent in nature and the energy crisis is becoming more and more serious today, the storage of available energy has become important and necessary. Among the methods of conserving energy, the use of new functional materials plays an important role.¹ Recently, phase-change materials (PCMs) have attracted more and more attention for thermal energy storage and utilization because PCMs can absorb or release a high amount of thermal energy while undergoing phase changes with small temperature variations.^{2–5} PCMs have been widely applied in many fields, including energy efficient buildings,^{6,7} waste heat recovery,⁸ solar energy storage,^{9,10} electronics cooling,^{11,12} smart textiles,^{13,14} and preservation of food.¹⁵ Nevertheless, the practical use of PCMs suffers from many problems, such as dimensional instability, corrosiveness, decomposition, incompatibility, and low thermal conductivity. To solve these problems, the microencapsulation and nanoencapsulation of PCMs in physically and chemically stable polymeric capsules was examined in this study; this can be an effective method.

In recent years, a lot of studies have been done to prepare microcapsules containing PCMs with different polymeric shells. Fortuniak *et al.*¹⁶ used coemulsification method to microencapsulate *n*-eicosane with a siloxane polymer as shell. Lone *et al.*¹⁷ prepared highly monodisperse polyurea microcapsules containing *n*-octadecane with tubular microfluidics. Onder *et al.*¹⁸ fabricated microcapsules containing three types of PCMs, including *n*-hexadecane (HD), *n*-octadecane, and *n*-nonadecane, through complex coacervation of natural and biodegradable polymers, a gum Arabic–gelatin mixture. Platte *et al.*¹⁹ used surface Michael-type addition polymerization of thiols and acrylates to synthesize a microcapsule containing alkaline salt hydrates. In our previous work,²⁰ microcapsules containing *n*-dodecanol were prepared with *in situ* polymerization with melamine–formaldehyde resin as the shell. However, there are many shortcomings for the practical application of microcapsules containing PCMs in several fields, including latent thermal fluids, smart textiles, and phase-change thermal storage building materials because of the large size of the capsules. In thermal fluids, the large particles of microcapsules

containing PCMs cause an increase in a fluid's viscosity and crush during pumping.^{21,22} The use of microcapsules containing PCMs in the building materials matrix causes a decrease in the strength of the composite building materials.²³

As a result, the size of capsules containing PCMs plays an important role in the effect of the use of capsules in those fields. The decrease in the size of the capsule containing PCMs can not only improve those problems but also increase the heat-transfer rate to the surroundings. This is attributed to its larger surface-to-volume ratio. Thus, the nanoencapsulation of PCMs provides many merits for the application of capsules containing PCMs to improve the energy efficiency and enlarge the application fields. A lot of works have shown that miniemulsion polymerization is a convenient encapsulation method for synthesizing nanocapsules.^{24–31} The nanocapsules can be easily obtained and stable in the miniemulsion system. The aim of this study was to encapsulate PCM *n*-dodecanol into copolymer nanocapsules. Different types and amounts of comonomers were used to prepare the nanocapsules through miniemulsion polymerization. The effects of the comonomer on the morphology and properties of nanocapsules containing *n*-dodecanol were studied.

EXPERIMENTAL

Chemicals

Methyl methacrylate [MMA; analytical reagent (AR), Tianjing Fuchen Chemical Reagent Co.] was used as monomer of shell material. Butyl acrylate (BA; AR, Tianjing Yaohua Chemical Reagent Co.), acrylamine (AM; AR, Tianjing Daomao Chemical Reagent Co.), and acrylic acid (AA; AR, Shanghai Runjie Chemical Reagent Co.) were used as comonomers of the shell material. *N*-Dodecanol (Chemically Pure, Shanghai Chemical Reagent Factory) was used as the core material. Ammonium allyloxymethylate nonylphenol ethoxylate sulfate (DNS-86, industrial grade, Qingxin Hanerchem Chemical Technology Limited Co.) was used as a polymerizable emulsifier. HD (AR, Tianjing Daomao Chemical Reagent Co.) was used as the coemulsifier. Azobisisobutyronitrile (AIBN; Chemically Pure, Shanghai No.4 Reagent & H.V. Chemical Limited Co.) was used as the initiator.

Preparation of the Copolymer Nanocapsules Containing PCM *n*-Dodecanol

Emulsifier DNS-86, the coemulsifier HD, and deionized water were mixed together according to the ratio in Table I. The mixture was dispersed for 10 min under an ultrasonic amplitude of 90% with an ultrasonic cell disruptor (S-450D, Branson Ultrasonics Corp.) in an ice–water bath to obtain a stable water phase. The main monomer, MMA; the comonomers; PCM *n*-dodecanol; and the initiator, AIBN, were mixed together; this constituted the oil phase. The oil phase was added to the water phase, and then, the mixture was pre-emulsified mechanically at a stirring rate of 1500 rpm for 30 min to obtain the pre-emulsion. The pre-emulsion was miniemulsified for 10 min with the ultrasonic cell disruptor (S-450D) at an amplitude of 90% in ice–water bath to get the miniemulsion. The miniemulsion was placed into a 250-mL, four-necked flask with a mechanical stirrer, nitrogen inlet, and reflux condenser and was then heated to 80°C to start a miniemulsion polymerization

Table I. Basic Formulas of the Nanocapsules Containing *n*-Dodecanol Prepared by Miniemulsion Polymerization

Component	Mass ratio (%)
Monomer (including MMA and comonomers)	10
<i>n</i> -Dodecanol	10
AIBN	0.2
DNS-86	0.6
HD	0.4
Deionized water	78.8

under a stirring rate of 300 rpm. After 4 h of reaction, the nanocapsules latex containing *n*-dodecanol was slowly cooled to room temperature. Figure 1 shows the schematic view of the formation of the core–shell structured polymeric nanocapsules containing PCM *n*-dodecanol via miniemulsion polymerization.

The resulting nanocapsules latex was demulsified by the addition of a mixture of ethanol and 50 wt % aqueous sodium chloride solution, and then, the demulsified latex was filtered with double filter paper under reduced pressure to obtain a solid sample of nanocapsules containing *n*-dodecanol. The solid nanocapsule was washed with a 50 wt % ethanol–water solution at 50°C once and with distilled water twice to remove the unencapsulated *n*-dodecanol or *n*-dodecanol that emerged from the cracked nanocapsule. The washed solid nanocapsule was left to dry at 30°C for 24 h under negative pressure. The dried nanocapsule was stored at room temperature.

Characterization

The chemical composition of the copolymer nanocapsules containing *n*-dodecanol prepared with different comonomers were analyzed with a Bruker Vector 33 Fourier transform infrared (FTIR) spectrometer. The samples were mixed with KBr, and the FTIR spectra were recorded at 450–4000 cm⁻¹.

The thermal storage properties of the nanocapsules containing *n*-dodecanol prepared with different comonomers were measured with a Netzsch 204F differential scanning calorimeter at a heating rate of 10°C/min among the range 10–60°C under a nitrogen atmosphere. The phase-change latent heat and phase-change temperature of nanocapsule containing *n*-dodecanol were obtained through analysis of the results of differential scanning calorimetry curves with a Netzsch TA4 instrument.

The content of PCM *n*-dodecanol in the nanocapsules was estimated according to the measured phase-change latent heat:

$$\text{PCM content (\%)} = H_w / H_p \times 100\%$$

where H_w is the phase-change latent heat of the nanocapsules (J/g) and H_p is the phase-change latent heat of PCM (*n*-dodecanol of 248 J/g²⁰). The encapsulation efficiency of PCM *n*-dodecanol in the nanocapsules was calculated as the ratio of the measured PCM *n*-dodecanol content in the nanocapsules to the theoretical value. This depended on the amounts of PCM *n*-dodecanol and monomers added to the system of fabrication.

The dried nanocapsules containing *n*-dodecanol prepared with different comonomers were analyzed with a Netzsch TG 209F

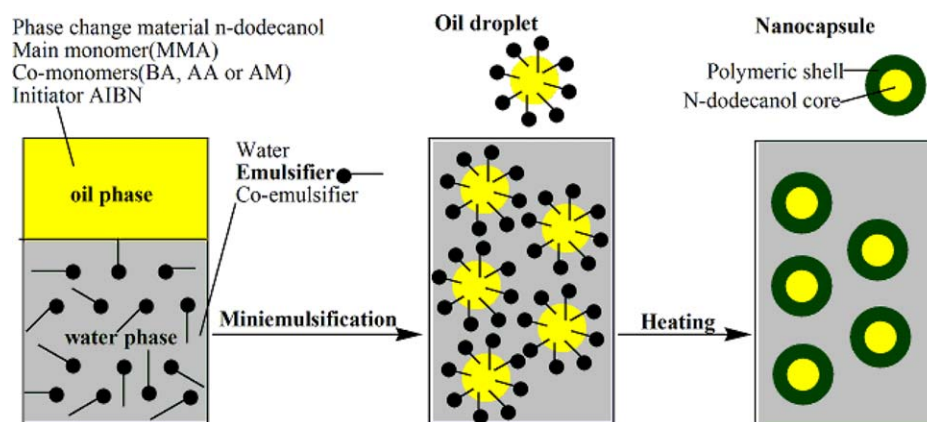


Figure 1. Schematic view of the formation of the core-shell structured copolymer nanocapsules containing PCM *n*-dodecanol via miniemulsion polymerization. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

thermogravimeter at a heating rate of 20°C/min among the range 30–600°C under an air atmosphere.

The morphology and microstructure of the nanocapsules containing *n*-dodecanol was observed with a JEOL JEM-100CXII transmission electron microscope. A drop of nanocapsules emulsion was diluted 20 times with distilled water. The diluted sample was placed onto a 300-mesh copper grid and left to dry before transmission electron microscopy imaging.

RESULTS AND DISCUSSION

Effects of the Comonomer Type on the Thermal Properties of the Nanocapsules Containing PCM *n*-Dodecanol

The differential scanning calorimetry curves of the copolymer nanocapsules containing *n*-dodecanol prepared without comonomer and with three different types of comonomers, BA, AA, and AM, under the same dosage of 4 wt % are presented in Figure 2. The phase-change temperature, phase-change latent heat, and encapsulation efficiency of *n*-dodecanol of the nanocapsules are reported in Table II.

As shown in Table II, the phase-change temperature of the nanocapsules prepared with different comonomers was near 20°C; this was consistent with that of *n*-dodecanol. The type of comonomer had no effect on the phase-change temperature of the copolymer nanocapsules containing PCM *n*-dodecanol. However, the phase-change latent heat and encapsulation efficiency of *n*-dodecanol were different from those of the nanocapsules of four samples prepared with different comonomers. As compared with nanocapsules prepared without any comonomer, the addition of 4 wt % of the BA comonomer caused a slight decrease in the phase-change latent heat and encapsulation efficiency of *n*-dodecanol, whereas the addition of 4 wt % AA or 4 wt % AM did the opposite. Under the same little dosage of comonomer, the hydrophilicity of the comonomers played a decisive role. The hydrophilicity of the BA comonomer was lower than that of the main monomer, MMA. This reduced the hydrophilicity of the polymer chain synthesized with BA; this was harmful for the phase separation of the polymer to encapsulate the oil-soluble PCMs. On the other hand, the hydrophilicities of the comonomer AA and AM were higher than that of the main monomer MMA, and the hydrophilicities of the polymer chain synthesized

with AA and AM increased. More oil-soluble PCMs were encapsulated in the polymeric nanocapsules synthesized with AA and AM, and the phase-change latent heat and encapsulation efficiency of *n*-dodecanol increased. Nevertheless, the hydrophilicity of the AA comonomer was too strong to easily cause the self-polymerization of AA in water and the formation of empty polymeric nanocapsules;^{32,33} this decreased the encapsulation efficiency. Therefore, the nanocapsules prepared by the AM comonomer with moderate hydrophilicity got the highest phase-change latent heat and encapsulation efficiency under the same small dosage of 4 wt %.

Effects of the BA Comonomer Amount on the Thermal Storage Properties of the Nanocapsules Containing PCM *n*-Dodecanol

The glass-transition temperature of the polymeric shell of the copolymer nanocapsules should have been too high; this would be unfavorable for its practical application. So, the soft

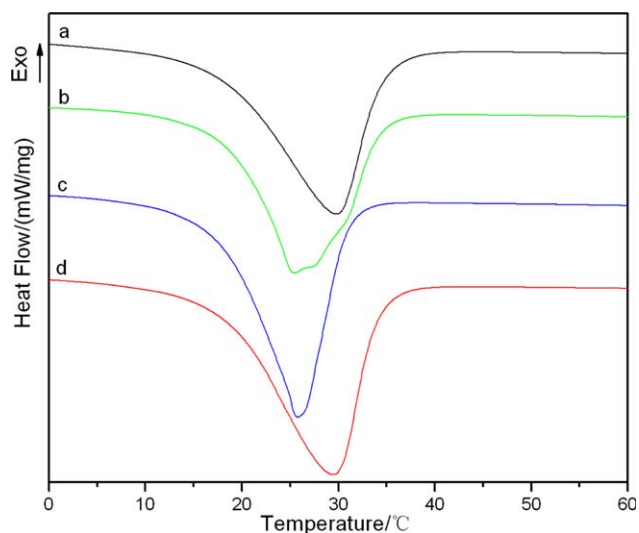


Figure 2. Differential scanning calorimetry curves of the copolymer nanocapsules containing *n*-dodecanol prepared (a) without any comonomer or (b–d) with different comonomers: (b) 4 wt % BA, (c) 4 wt % AA, and (d) 4 wt % AM. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table II. Thermal Storage Properties of the Copolymer Nanocapsules Containing *n*-Dodecanol Prepared with Different Comonomers

Comonomer	Phase-change temperature (°C)	Phase-change latent heat (J/g)	Encapsulation efficiency of <i>n</i> -dodecanol (%)
No comonomer	18.2	98.8	82.9
BA	19.5	94.9	79.6
AA	18.8	99.4	83.3
AM	18.4	109.3	91.6

monomer BA was chosen as the comonomer to be copolymerized with the hard main monomer MMA. The effect of the BA comonomer amount on the phase-change latent heat and encapsulation efficiency of the nanocapsules containing *n*-dodecanol is shown in Figure 3.

The phase-change latent heat and encapsulation efficiency of the nanocapsules decreased slightly first and then increased to the maximum value when the dosage of BA reached 10 wt % with increasing BA comonomer amount. However, when the dosage of BA exceeded 10 wt %, the phase-change latent heat and encapsulation efficiency of the nanocapsules decreased dramatically as the BA comonomer amount continuously increased. The phase-change latent heat and encapsulation efficiency of the nanocapsules prepared with 4 wt % BA decreased slightly because of the poor hydrophilicity of BA. As more soft comonomers were introduced, the polymer chain became more mobilizable, and more PCM was encapsulated in the polymeric nanocapsules.^{32,33} When the amount of soft comonomer BA increased to 10 wt %, the mobility of the polymer chain played a more important role than the hydrophilicity of the comonomers. So, the phase-change latent heat and encapsulation efficiency of the nanocapsules increased as the dosage of BA reached 10 wt %. However, when the dosage of the soft comonomer BA increased too high to 20–30 wt %, the mechanical strength of the nanocapsule shell became much weaker, and the core material of the *n*-dodecanol leaked out greatly in the washing and drying processes. Therefore, the phase-change

latent heat and encapsulation efficiency of the nanocapsules decreased greatly, as the dosage of BA increased continuously after 10 wt %.

Morphology of the Nanocapsules Containing PCM *n*-Dodecanol

Figure 4 shows the transmission electron micrographs of the copolymer nanocapsules containing *n*-dodecanol prepared with different types and amounts of comonomers.

As shown in Figure 4(a), the size of the nanocapsules containing *n*-dodecanol prepared with 4 wt % BA comonomer ranged from 50 to 100 nm with uniform spherical shape. However, the nanocapsules with deformed spherical shape conglutinated with each other, as shown in Figure 4(b); this was different from those shown in Figure 4(a). It was attributed to the decrease in the glass-transition temperature of the polymeric shell prepared with a high level of 10 wt % BA soft comonomer. The structure of the nanocapsules, as shown in Figure 4(c,d), with a uniform spherical shape was more apparent than that of those shown in Figure 4(a,b); this was due to the hydrophilicity of the comonomers AA and AM. The hydrophilic comonomers AA and AM were enriched in the interfacial area between the oil phase and the aqueous phase after miniemulsification; this was helpful for the polymer chain to encapsulate the oil-soluble PCM *n*-dodecanol and form nanocapsules with a core-shell structure. The size of nanocapsules with moderate hydrophilicity, as shown in Figure 4(d), prepared by the comonomer AM was bigger than that of the nanocapsules shown in Figure 4(c) because more PCM was encapsulated in the nanocapsules. This was consistent with the differential scanning calorimetry results of nanocapsules with 4 wt % comonomer AA or AM.

FTIR Spectra of the Nanocapsules Containing PCM *n*-Dodecanol

The FTIR spectra of the copolymer nanocapsules containing *n*-dodecanol prepared without comonomer or with different comonomers are presented in Figure 5.

As shown in Figure 5, the FTIR spectra curves of the four samples of the nanocapsules prepared with different comonomers were nearly the same. The strong and wide absorption peaks at 3346 cm⁻¹ were associated with O—H stretching vibrations of the core material *n*-dodecanol.³⁴ The two strong absorption peaks at 2997 and 2952 cm⁻¹ and the weak absorption peaks at 2855 cm⁻¹ were assigned to the asymmetric and symmetric C—H stretching vibrations of the shell material poly(methyl methacrylate) (PMMA), respectively.^{35–37} The multiple strong peaks at 2923 and 2854 cm⁻¹ were attributed to the aliphatic

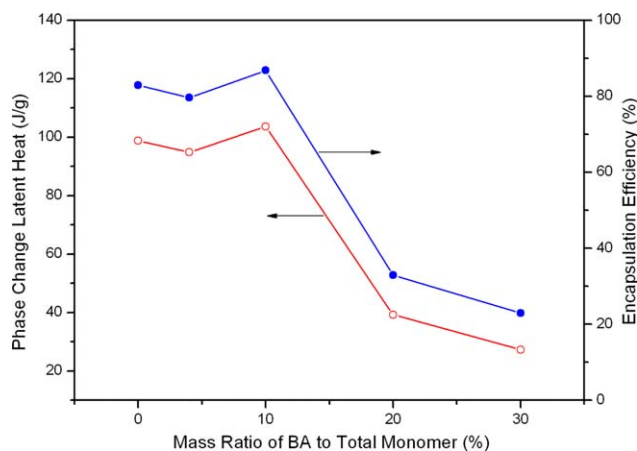


Figure 3. Effect of the amounts of the comonomer BA on the phase-change latent heat and encapsulation efficiency of the nanocapsules containing *n*-dodecanol. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

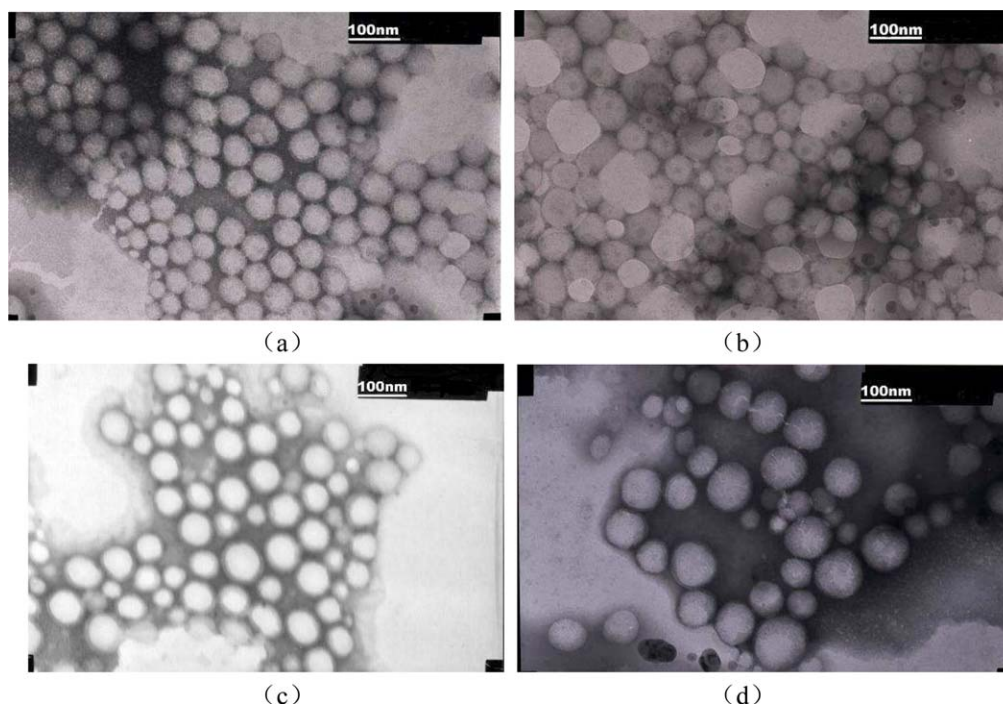


Figure 4. Transmission electron micrographs of the copolymer nanocapsules containing *n*-dodecanol prepared with different comonomers: (a) 4 wt % BA, (b) 10 wt % BA, (c) 4 wt % AA, and (d) 4 wt % AM. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

C—H stretching vibrations of the methyl and methylene groups in *n*-dodecanol. The strong and sharp absorption peaks at 1730 cm^{-1} were related to the C=O stretching vibrations of PMMA. The characteristic absorption peak of *n*-dodecanol at 1057 cm^{-1} with a moderate strength was also found in the FTIR spectra curves of the four samples; this was assigned to the C—OH stretching vibrations of primary alcohol. We concluded that *n*-dodecanol encapsulated in the nanocapsules

without any chemical reaction between *n*-dodecanol and PMMA resin. However, the characteristic absorption peaks of the BA, AA, and AM comonomers could not be found in the FTIR spectra curves of the corresponding nanocapsules; this might have been due to the low dosage of the comonomers.

Thermogravimetric Analysis of the Nanocapsules Containing PCM *n*-Dodecanol

Figure 6 shows the thermogravimetry curves of the copolymer nanocapsules containing *n*-dodecanol prepared without

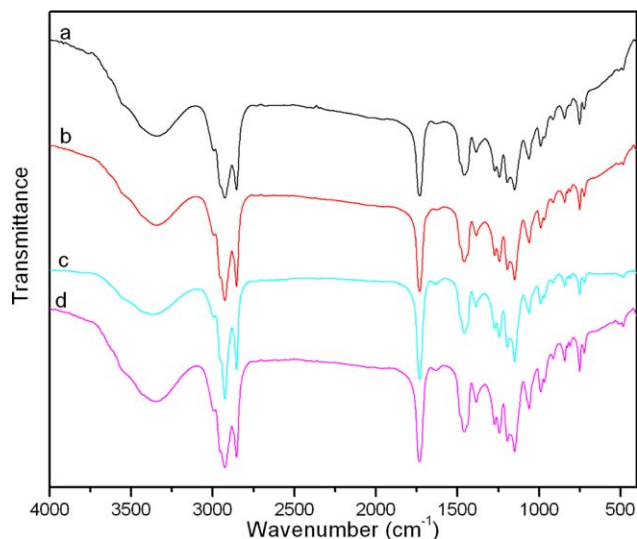


Figure 5. FTIR spectra of the copolymer nanocapsules containing *n*-dodecanol prepared (a) without any comonomer or (b–d) with different comonomers: (b) 4 wt % AM, (c) 4 wt % AA, and (d) 4 wt % BA. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

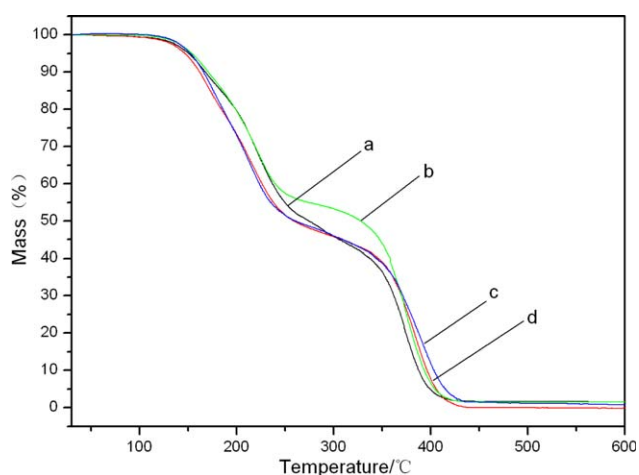


Figure 6. Thermogravimetry curves of the copolymer nanocapsules containing *n*-dodecanol prepared with (a) no comonomer or with different comonomers: (b) 4 wt % AM, (c) 4 wt % AA, and (d) 4 wt % BA. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table III. Thermogravimetric Analysis of the Copolymer Nanocapsules Containing *n*-Dodecanol Prepared with Different Comonomers

Sample	T_{onset} of step 1 (°C)	Mass loss of step 1 (%)	T_{onset} of step 2 (°C)	Mass loss of step 2 (%)
Nanocapsules prepared without a comonomer	161.3	50.9	357.1	47.5
Nanocapsules prepared with 4 wt % BA as a comonomer	163.0	45.3	354.8	53.1
Nanocapsules prepared with 4 wt % AA as a comonomer	149.2	51.8	356.2	47.3
Nanocapsules prepared with 4 wt % AM as a comonomer	146.3	52.4	357.1	47.2

comonomer or with the different comonomers, BA, AA, and AM. The thermogravimetric analysis results are listed in Table III.

As shown in Figure 6, there were two steps of mass loss for all of the nanocapsules prepared without comonomer or with different comonomers. The first step of mass loss corresponded to the volatilization of the core material *n*-dodecanol; this was due to the cracking of the shell of nanocapsules. The order of mass lost in the first step was AM > AA > No comonomer > BA; this was consistent with that of the core material content in the corresponding nanocapsules. However, the starting temperatures of mass lost in the first step for the nanocapsules prepared with 4 wt % BA or without comonomer with a value of more than 160°C were higher than that of the nanocapsules prepared with 4 wt % AA or 4 wt % AM with a value of less than 150°C; this might have been due to the thinner shell of the nanocapsules prepared with 4 wt % AA or 4 wt % AM possessing a higher content of core material. The second step of mass loss was attributed to the decomposition of the shell material of the nanocapsules. The order of mass lost in the second step was BA > No comonomer > AA > AM; this was the opposite to that of the first step. The starting temperatures of mass lost in the second step for all of the nanocapsules were nearly the same with a value of about 355°C. We concluded that the thermal stability of the nanocapsules was mainly decided by the content of core material. The thermal stability of the nanocapsules became weaker with a higher content of core material.

CONCLUSIONS

Copolymer nanocapsules containing PCM *n*-dodecanol were successfully synthesized with MMA as the main monomer and different comonomers via miniemulsion polymerization. The type and amount of comonomers had a great influence on the thermal properties and morphology of the nanocapsules. The hydrophilicity of the comonomers increased the hydrophilicity of the polymer chain, which played an important role in the encapsulation of oil-soluble PCM *n*-dodecanol at the same small dosage of 4 wt %. The nanocapsules prepared with 4 wt % comonomer AM with moderate hydrophilicity showed the highest phase-change latent heat of 109.3 J/g with the phase-change temperature of 18.4°C and the highest encapsulation efficiency of 91.3%. The nanocapsules with uniform spherical shape and core-shell structure range from 50 to 100 nm in size.

The amount of the comonomer affected the mobility of the polymer chain greatly; it also showed a great effect on the formation of the copolymer nanocapsules when the amount increased to 10 wt %. As more soft comonomers were introduced, the polymer chain became more mobilizable; this was attributed to the encapsulation of more PCM in the copolymer nanocapsules. However, because of the poor hydrophilicity of the soft comonomer BA, the thermal storage properties of the copolymer nanocapsules slightly decreased first and then increased to the maximum value with a deformed spherical and conglomerated morphology. When the amount of BA increased too high, the thermal storage properties of the nanocapsules decreased continuously. The thermal stability of the nanocapsules became weaker with a higher content of core material.

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