



Effects of type and contents of microencapsulated *n*-alkanes on properties of soft polyurethane foams

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ABSTRACT

Soft polyurethane (PU) composite foams containing microencapsulated *n*-alkanes were fabricated with general polyether and a combination of polyether polyols. The elongations at breakage and the permanent deformations of foams are decreased with an increased content of microcapsules. It has no obvious effect on tensile strength of the foams when the content of microcapsules was less than a certain value. When the general polyether was used, the foams with different densities could be fabricated by adjusting the content of foaming agent. The enthalpies of the foams rise steadily as the content of microcapsules increased from 6.4 to 25.2 wt%. The enthalpy of the foam containing 25.2% microencapsulated *n*-octadecane (MicroC18) is 28 J/g.

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1. Introduction

Microcapsules are tiny particles composed of shells and cores, and the shells protect the core materials [1]. The protective shells are usually natural or synthetic macromolecular. The core materials of the microcapsules can be drugs, enzymes, dyes, fragrant oils, water, or salts, for example.

Microcapsules have been applied to a wide range of products such as pharmaceuticals [2–5], microencapsulated electrophoretic display systems [6,7], and liquid crystals [8].

Phase-changed materials (PCMs) can absorb or release latent heat when the temperature of the material surpasses or undergoes the temperature of phase change [9]. However, the PCMs are not easy to handle in practical applications due to their inherent characteristics. Microencapsulated phase-changed materials (MicroPCMs) are powder-like materials; and have been widely investigated since the late 1970s because they are regarded as renewable and clean energy storage materials [9,10]. Microencapsulation turns PCMs into permanent solids for various applications. MicroPCMs have been widely studied as active or pumped coolants [11–14], solar and nuclear heat storage systems [15], and heat exchangers [16]. The application fields of MicroC18 also relate to the manufacture of thermo-regulated fiber, fabrics and coating [17–20].

The foam containing MicroPCMs is very useful in the heat-insulating application, such as footwear, clothing, automotive interiors and medical products [21,2]. The application of MicroPCMs in PU foams to improve their thermal performance was developed in the 1990s [22]. However, there is little information available about the fabrication of this kind of foam. MicroC18 with melamine-formaldehyde co-polymer (MF) shells have been applied in the fabrication of heat-insulated PU foams in our previous study [23]. The process of fabrication was complex because of the defoaming action of MicroC18 with MF shells. Since then, the novel microencapsulated alkanes with different shell materials have also been reported [24–26]. MicroC18 with a shell of styrene (St)-divinylbenzene (DVB) co-polymer was tested to be used in a foaming system. It was found that MicroPCMs with a shell of St-DVB co-polymer were able to be added in the foaming system directly without heat-treatment, compared to MicroC18 with a melamine-formaldehyde co-polymer shell [23,27]. In addition, the PU foams containing MicroC18 were fabricated with a combination of polyether polyols in our previous research. Though the PU foams exhibited thermo-regulated performance, their mechanical properties were not satisfactory.

In this study, the kind of general purpose polyether applied in the slabstock foaming process (general polyether) was used for fabricating the composite PU foams, and the influences of MicroPCMs on the morphology, heat properties, and mechanical properties of the foams were investigated. In addition, the mechanical properties of the foams synthesized with these two different materials are compared.

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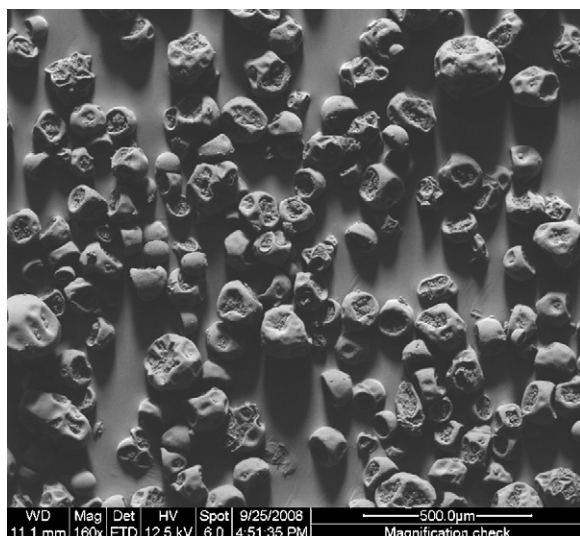


Fig. 1. SEM micrograph of MicroC18.

2. Experimental

2.1. Materials

General polyether (TMN-3050) was purchased from the Third Petrochemical Factory, Tianjin Petrochemical Inc., China. The hydroxyl value of TMN-3050 is 54.5–57.5 mg KOH/g and the viscosity (25 °C) is 400–600 mPa s.

Toluene 2,4-diisocyanate (TDI, purity 95 wt%, Tianjin Yuanli Chemical Co., Ltd.) was used as monomer. PU foam stabilizer (AK-6618) was provided by Dymatic Shichuang Chemical Co., Ltd., Nanjing, China. Methylene chloride (MC, purity 99.5 wt%, Tianjin Jiangtian Chemical Co., Ltd.) was used as physical foaming agent and distilled water was used as chemical foaming agent. Stannous octoate (C.R., Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) and triethylenediamine (TEDA, purity 98 wt%, Tianjin Jinke Chemical Co., Ltd.) were used as catalyst. In this study, 33 g triethylenediamine were dissolved in 67 g diglycol, and the solution was used in the experiment.

2.2. Preparation of MicroPCMs

MicroPCMs were fabricated by suspension-like polymerization using St-DVB co-polymers as shell. MicroC18, microencapsulated *n*-nonadecane (MicroC19) and microencapsulated *n*-eicosane (MicroC20) were fabricated, respectively and the preparation process was reported previously [27]. An SEM micrograph of synthesized MicroC18 is shown in Fig. 1. The microcapsules are spherical particles and many concaves are observed on the surfaces

Table 1
Phase change properties of MicroPCMs.

PCM/MicroPCMs	T_m^a (°C)	ΔH_m^b (J/g)	T_c^c (°C)	ΔH_c^d (J/g)	ΔH_a^e (J/g)	Content of <i>n</i> -alkanes (wt%)
C18	31	222	21	222	222	–
MicroC18	28	125	17	127	126	56.8
C19	36	219	26	221	220	–
MicroC19	34	118	23	118	118	53.7
C20	40	240	30	241	240.5	–
MicroC20	41	136	27	136	136	56.6

^a Melting point.

^b Melting enthalpy.

^c Crystallization point.

^d Crystallization enthalpy.

^e $\Delta H_a = (|\Delta H_m| + |\Delta H_c|)/2$.

of the majority of MicroC18. The formation of concaves is caused by the density difference between monomers and polymers, and the density difference between melted and crystallized *n*-octadecane in the fabrication process of MicroC18 [28]. The diameters of microcapsules are about 80–100 μm; and the heat absorbing and evolving properties of MicroPCMs are listed in Table 1. The content of *n*-alkanes in the microcapsule can be calculated according to the measured enthalpies,

$$x = \frac{|\Delta H|}{|\Delta H_0|} \times 100\% \quad (1)$$

where $|\Delta H|$ is the enthalpies of MicroPCMs; $|\Delta H_0|$ is the melting enthalpy of *n*-alkanes.

2.3. Fabrication of PU foams containing MicroPCMs

The predetermined masses of TMN-3050, MicroPCMs, AK-6618, H₂O, MC, stannous octoate and TEDA were added in a beaker and mixed vigorously with strong stirring to obtain a formulated mixture. The predetermined masses of TDI were then added in the same beaker and strong stirring was applied for 7–10 s. The obtained mixture was immediately poured into a rectangular mold and left undisturbed for about 1 min, allowing the formation and growth of the foam. Then it was immediately moved into an oven and kept at 80 °C for about 40 min.

2.4. Characterization of the foams

The surface morphologies of foams were obtained by using a scanning electron microscope (SEM, Quanta-200). The specimens were coated with gold.

The thermal properties of foams were measured using a differential scanning calorimeter (DSC, Perkin-Elmer) at a rate of ±5 °C/min in a nitrogen atmosphere.

The densities of foams were tested with the National Standard (GB) of The People's Republic of China, GB/T 6343-1995.

The tensile strength and elongation at breakage of foams were tested with GB/T 6344-1996.

The permanent deformations of foams were tested with GB/T 6669-1986.

3. Results and discussion

3.1. Effects of MicroC18 on the fabrication of PU foams

Table 2 shows the effects of the content of stannous octoate on the formation of PU foams. In the process of PU foam formation, the use of filler can influence the surface tension and viscosity of the foaming system and further cause negative effect on the foam process. Increasing the content of organic stannum not only reduces the surface tension but also increases the rate of gelling reaction [29]. The appearance of the foam can be improved by increasing

Table 2
Effects of stannous octoate on foaming.

TMN-3050 (g)	AK-6618 (g)	TEDA (g)	Stannous octoate (g)	H ₂ O (g)	TDI (g)	MicroC18 (g)	Appearance of foam
100	1.2	0.15	0.2	3	41.5	40	Coat clammy
100	1.2	0.15	0.25	3	41.5	40	Coat clammy
100	1.2	0.15	0.3	3	41.5	40	Excellent
100	1.2	0.15	0.35	3	41.5	40	Excellent
100	1.2	0.15	0.4	3	41.5	40	Cell closed

Table 3
Effects of TDI on foaming.

TMN-3050 (g)	AK-6618 (g)	TEDA (g)	Stannous octoate (g)	H ₂ O (g)	TDI (g)	MicroC18 (g)	Appearance of foam
100	1.2	0.15	0.3	3	40	40	Loose
100	1.2	0.15	0.3	3	41	40	Loose
100	1.2	0.15	0.3	3	41.5	40	Excellent
100	1.2	0.15	0.3	3	42	40	Excellent
100	1.2	0.15	0.3	3	42.5	40	Shrinking

Table 4
Foaming formulation after adjusting the content of additives.

TMN-3050 (g)	AK-6618 (g)	TEDA (g)	Stannous octoate (g)	H ₂ O (g)	TDI (g)	MC (g)	MicroC18 (g)	MicroC18 content (wt%)
100	1.2	0.2	0.25	3	40	–	0	0
100	1.2	0.2	0.25	3	40	–	10	6.4
100	1.2	0.2	0.25	3	40	–	20	12.1
100	1.2	0.2	0.25	3	40	–	30	17.1
100	1.2	0.2	0.3	3	41.5	–	40	22.1
100	1.2	0.2	0.3	3	41.5	–	45	23.6
100	1.5	0.2	0.35	3	42.5	10	50	25.4

the content of stannous octoate when the content of MicroC18 is higher. However, when a higher content of stannous octoate is used, the closed-cell rate will be accelerated. This is explained by the gelling reaction rate being excessively accelerated. Thus the intensity of liquid film will be enhanced sharply during the foaming process, which results in the formation of closed-cells.

The effects of the content of TDI on foaming are listed in Table 3. The shell of MicroC18 might absorb some amount of moisture, so the content of TDI has to be increased when adding more MicroC18 in the foaming system.

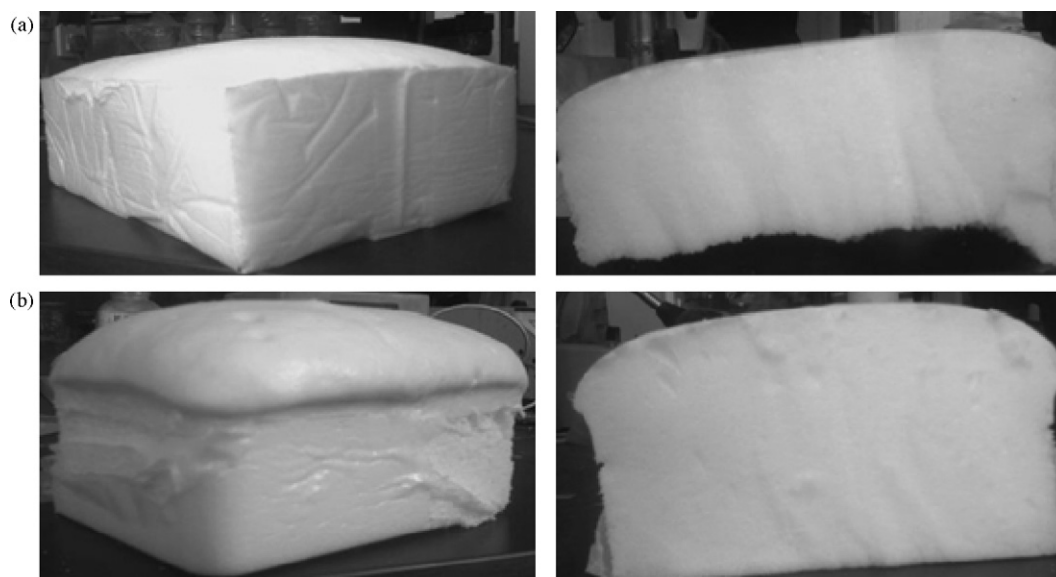
In order to improve the quality of the foams, the contents of additives should be adjusted as shown in Table 4. When the content of MicroPCMs was higher than 23.6 wt%, the viscosity of the

foaming system was too high to obtain qualified foam. The MC was added into the foaming system to reduce the viscosity of the mixture. Fig. 2 illustrates that the appearances of foams with different contents of MicroC18 have no obvious difference after adjusting the contents of additives.

3.2. Morphology of PU foams

SEM micrographs of PU foam are presented in Fig. 3. Microcapsules were evenly inserted inside the foam matrix and well-compatible with the polyurethane matrix.

Fig. 4 shows SEM micrographs of PU foam cell structure. When MicroC18 were added in the foaming system, the cell diameter was

**Fig. 2.** Appearances of PU foams with different MicroC18 contents: (a) 6.4 wt%; (b) 23.6 wt%.

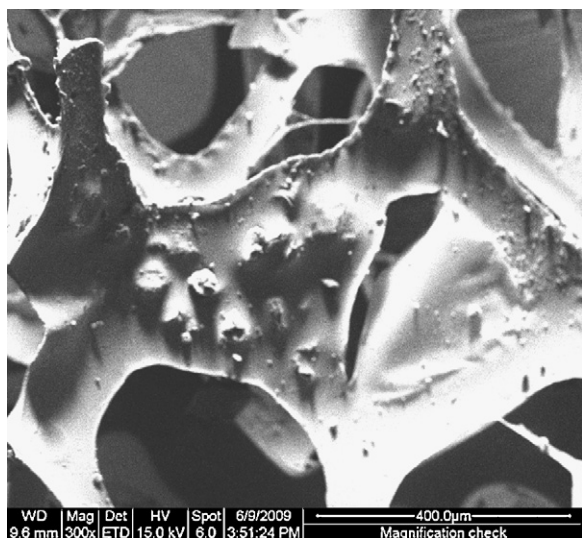


Fig. 3. SEM micrographs of PU foam containing MicroC18.

smaller than that without MicroC18. The phenomenon is induced by the “nucleation” function and accords the rule that, in general, added powder filling will reduce the cell diameter. However, the diameters of the cells became larger when the content of MicroC18 is 27.2 wt%. This was caused by the adding of MC.

3.3. Heat properties of PU foams containing MicroPCMs

Fig. 5 shows DSC curves of PU foam with various contents of MicroC18. Compared to pure PU foam, the foam containing MicroC18 has an endothermic peak and an exothermic peak in the process of heating and cooling in the range of 5–40 °C. The heat absorbing and evolving properties of the foams are listed in Table 5. The heat storage and thermo-regulated ability of foam were enhanced by an increase in the content of MicroC18. The measured average enthalpy is lower than the calculated value, which is probably caused by the poor thermal conductivity of the foam [21,30].

Fig. 6 presents DSC curves of PU foams containing MicroC18, MicroC19 and MicroC20, respectively. The endothermic peak and exothermic peak of PU foams containing MicroPCMs moved in the direction of higher-temperature with the increase of carbon atom number in *n*-alkanes – as has been reported in the literature [31]. The multiple-peaks in the cooling curves are assigned to the heterogeneous and homogeneous nucleation [32].

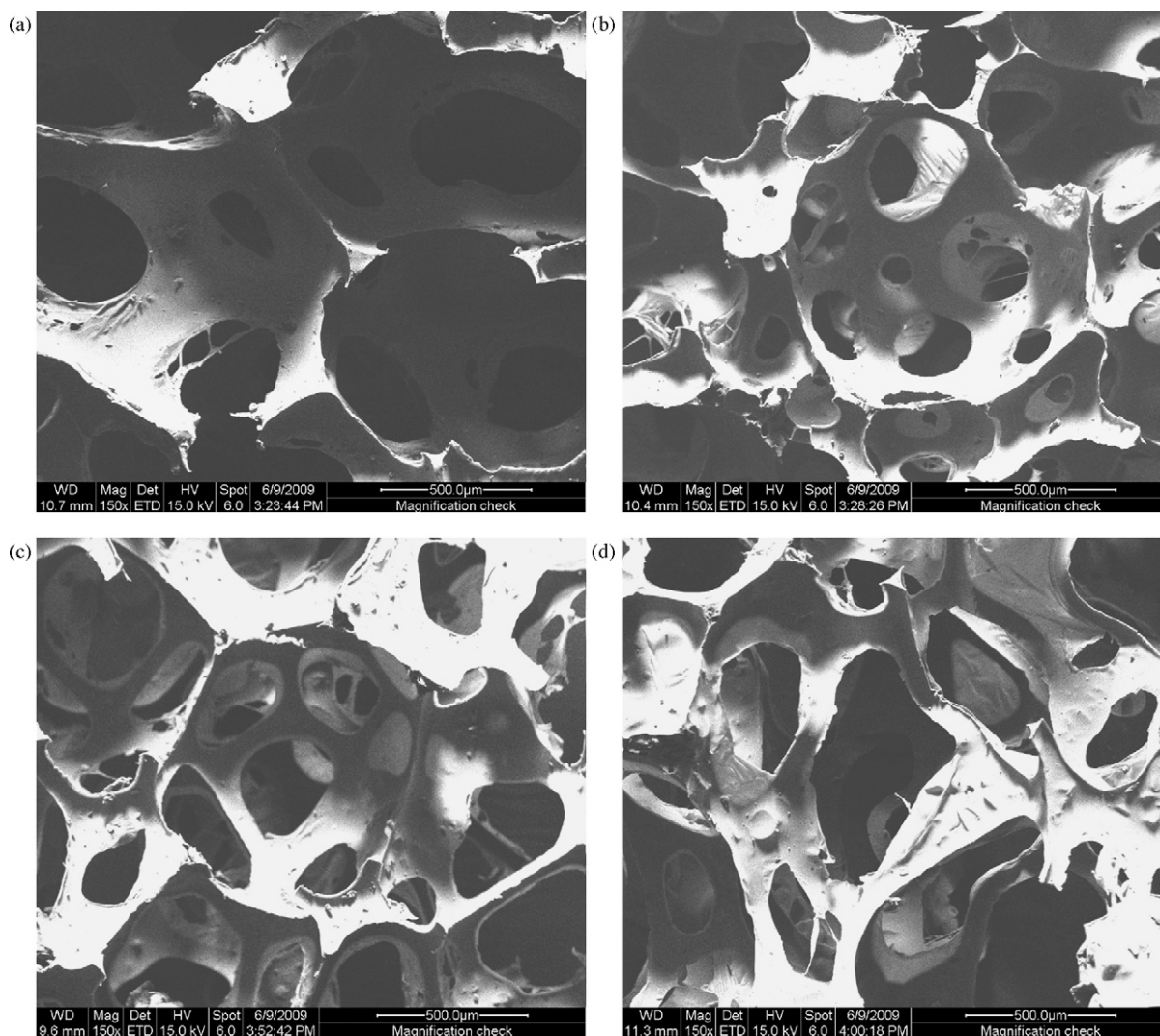


Fig. 4. SEM micrographs of PU foams with different MicroC18 contents: (a) 0 wt%; (b) 12.1 wt%; (c) 22.1 wt%; (d) 25.4 wt%.

Table 5
Heat properties of PU foam containing MicroC18.

MicroC18 (g)	MicroC18 content (wt%)	T_m^a (°C)	ΔH_m^b (J/g)	T_c^c (°C)	ΔH_c^d (J/g)	ΔH_a^e (J/g)	ΔH^f (J/g)	Efficiency of enthalpy ^g (%)
0	0 (PU)	–	0	–	0	0	–	–
10	6.4	28	5	21	5	5	8.0	62.5
20	12.1	28	11	21	11	11	15.2	72.3
30	17.1	28	16	21	17	16.5	21.5	76.7
40	22.1	28	21	21	22	21.5	27.8	77.3
45	23.6	28	25	21	26	25.5	29.7	85.9
50	25.4	28	28	21	28	28	32.0	87.5

^a Melting point.

^b Melting enthalpy.

^c Crystallization point.

^d Crystallization enthalpy.

^e $\Delta H_a = (|\Delta H_m| + |\Delta H_c|)/2$.

^f Calculated enthalpy from the composition of the foam.

^g Efficiency of enthalpy was calculated by $\Delta H_a/\Delta H$.

Table 6
Heat properties of PU foam containing different types of MicroPCMs.

MicroPCMs	MicroPCMs content (wt%)	T_m^a (°C)	ΔH_m^b (J/g)	T_c^c (°C)	ΔH_c^d (J/g)	ΔH_a^e (J/g)	ΔH^f (J/g)	Efficiency of enthalpy ^g (%)
MicroC18	23.6	28	25	21	26	25.5	29.0	85.9
MicroC19	23.6	33	27	25	26	26.5	27.9	94.9
MicroC20	23.6	38	31	30	31	31	32.0	96.8

^a Melting point.

^b Melting enthalpy.

^c Crystallization point.

^d Crystallization enthalpy.

^e $\Delta H_a = (|\Delta H_m| + |\Delta H_c|)/2$.

^f Calculated enthalpy from the composition of the foam.

^g Efficiency of enthalpy was calculated by $\Delta H_a/\Delta H$.

The heat absorbing and evolving properties of the foams are listed in Table 6. For the same MicroPCMs content, the efficiency of enthalpy of the foams fabricated with MicroC18 is lower than that of the foams fabricated with MicroC19 and MicroC20. By selecting the appropriate content of MicroPCMs, the PU foams can satisfy various required conditions.

3.4. Comparison of some mechanical properties of PU foams synthesized with different polyethers

Table 7 shows some mechanical properties of PU foams synthesized with combination of polyether polyols and general polyether,

respectively. The elongations at breakage decrease with increased content of MicroC18. In addition, it has no obvious effect on tensile strength of the foams when the MicroC18 content is less than a certain value. When a higher content of MicroC18 was used, however, the tensile strength decreases. The tensile strengths decrease noticeably when the MicroC18 contents are higher than 17.1 and 22.1 wt% for these two different polyethers.

Fig. 7 shows the permanent deformations of the foams. The permanent deformations of the foams are enhanced with an increase in the content of MicroC18. The reason might be that MicroC18 are incompressible particles and the deformations are difficult to recover after being compressed. However, the influence of

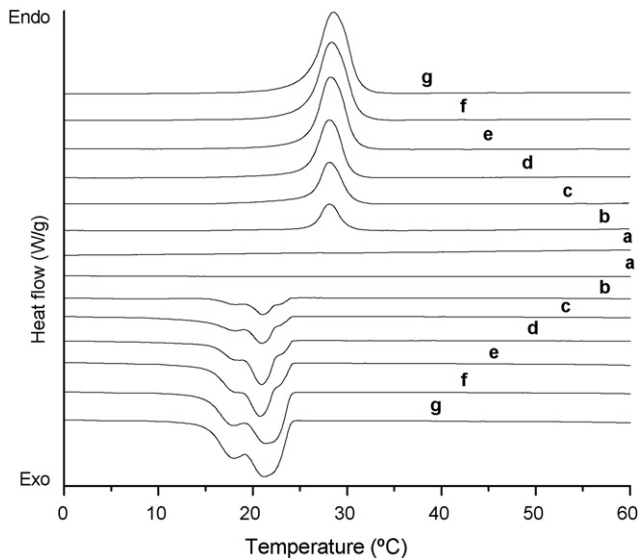


Fig. 5. DSC curves of PU foam with various contents of MicroC18: (a) 0 wt%; (b) 6.4 wt%; (c) 12.1 wt%; (d) 17.1 wt%; (e) 22.1 wt%; (f) 23.6 wt%; (g) 25.4 wt%.

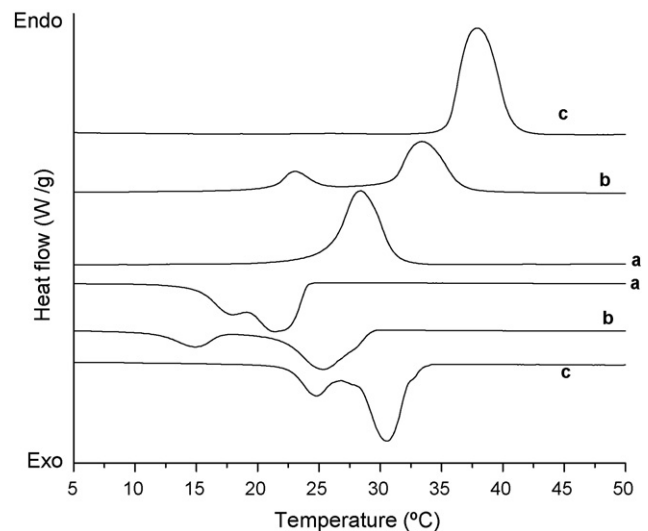


Fig. 6. DSC curves of PU foams with different types of MicroPCMs: (a) MicroC18; (b) MicroC19; (c) MicroC20.

Table 7
Mechanical properties of PU foams synthesized with combination of polyether polyols and general polyether.

MicroC18 content (wt%)	Combination of polyether polyols		General polyether	
	Elongation at break (%)	Tensile strength (kPa)	Elongation at break (%)	Tensile strength (kPa)
0	105	104	127	114
6.4	60	99	101	110
12.1	38	102	81	111
17.1	34	101	73	108
22.1	22	94	66	104
23.6	18	80	60	96
25.4	16	67	55	87

Table 8
Effects of H₂O and MC contents on the densities of foams.

TMN-3050 (g)	AK-6618 (g)	TEDA (g)	Stannous octoate (g)	H ₂ O (g)	MC (g)	TDI (g)	MicroC18 (g)	Foam density (kg/m ³)
100	1.2	0.2	0.25	3.0	0	40	45	51.9
100	1.2	0.2	0.25	2.5	0	35.5	45	68.6
100	1.2	0.2	0.25	2.0	0	30.5	45	86.3
100	1.2	0.2	0.25	3.0	0	40	45	51.9
100	1.2	0.2	0.25	3.0	5	40	45	44.3
100	1.5	0.2	0.25	3.0	10	40	45	38.2
100	1.8	0.2	0.25	3.0	12	40	45	35.2

MicroC18 on the permanent deformation of foam synthesized with general polyether is smaller than that synthesized with combination of polyether polyols.

According to the above-mentioned analysis, the mechanical properties of PU foams synthesized with general polyether are better than that synthesized with a combination of polyether polyols. The result reflects the difference of the structure of the polyol being used. The polymer chain of general polyether is unbranched. In this study, however, the polyether polyols is made of polymer polyether polyols (POP). The chain of POP is branched, and was fabricated by grafting modification with vinyl monomers. The components of POP include grafted polyether, unreacted polyether and vinyl polymer. The grafting modification and the existence of vinyl polymer are equal to the added organic filler in polyether. Therefore, the influence of MicroC18 on the mechanical properties of foam synthesized with a combination of polyether polyols is greater than that of synthesized with general polyether.

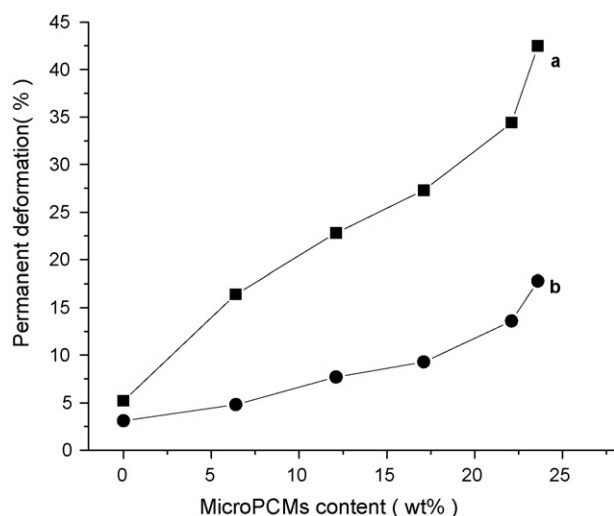


Fig. 7. Permanent deformations of foams: (a) synthesized with combination of polyether polyols; (b) synthesized with general polyether.

3.5. Fabrication of PU foams with various densities

The densities of foams are increased when the contents of MicroC18 increase as shown in Fig. 8. The components of the combination of polyether polyols are fixed and unknown. Thus the attempt at changing the densities of foams synthesized with a combination of polyether polyols is difficult. However, the densities of foams synthesized with general polyether can be changed easily by increasing or decreasing the content of the foaming agent.

Table 8 shows the influence of the content of H₂O on the density of foam. The density of foam is increased when the content of H₂O is decreased. It is caused by the reduction of gas during the foaming process. In addition, the qualified foam cannot be fabricated when the content of H₂O is less than 2 g. By contrast, the lower-density foam cannot be fabricated by increasing the content of H₂O. A higher content of H₂O can increase the dosage of TDI, which results in an obvious decrease in the content of MicroC18. In this study, MC was used as the physical foaming agent to reduce the density of foam. As shown in Table 8, the densities of foams

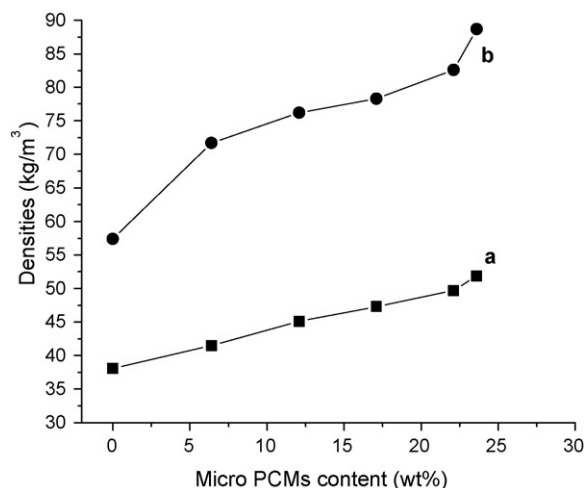


Fig. 8. Densities of foams: (a) synthesized with combination of polyether polyols; (b) synthesized with general polyether.

decrease with an increase in the content of MC. However, the qualified foam cannot be fabricated when the content of MC is higher than 12 g.

4. Conclusions

The foams containing 23.6–25.4 wt% microencapsulated *n*-octadecane, *n*-nonadecane and *n*-eicosane were fabricated using a combination of polyether polyols and general polyether as raw materials. The foaming formulation should be adjusted when various contents of MicroC18 are added to the foaming system. As the MicroC18 content is increased, the heat storage and thermoregulated ability of foam become greater. For the same MicroPCMs content, the efficiency of the enthalpy of the foams fabricated with MicroC18 is lower than that of the foams fabricated with MicroC19 and MicroC20. The mechanical properties of PU foams synthesized with a general polyether are better than that synthesized with combination of polyether polyols. The densities of PU foams synthesized with general polyether can be changed by adjusting the content of the foaming agent.

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