

Preparation and Characterization of Poly(divinylbenzene) Microcapsules Containing Octadecane

Preeyaporn Chaiyasat^{*}, Amorn Chaiyasat^{**}, Waraporn Boontung, Supaporn Promdsorn, Sutanya Thipsit

Department of Chemistry, Faculty of Science and Technology, Rajamangala University of Technology Thanyaburi, Thanyaburi, Thailand
Email: ^{*}p_chaiyasat@mail.rmutt.ac.th; ^{**}a_chaiyasat@mail.rmutt.ac.th

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ABSTRACT

Poly (divinylbenzene) (PDVB) microcapsules containing octadecane (OD) (PDVB/OD) used as heat storage material were synthesized by suspension polymerization at 70°C using benzoyl peroxide and polyvinyl alcohol as initiator and stabilizer, respectively. Thermal properties and stability of PDVB/OD microcapsules were determined using differential scanning calorimeter (DSC) and thermogravimetric analyzer. The morphology and structure of microcapsules were characterized by optical microscope, scanning electron microscope and fourier transform infrared spectrophotometer. From DSC analysis, the melting temperature of encapsulated OD (28°C) was almost the same as that of bulk OD (30°C) while it was quite different in the case of the solidification temperature (19°C and 25°C for encapsulated and bulk OD, respectively). The latent heats of melting (184.0 J/g-OD) and solidification (183.2 J/g-OD) of encapsulated OD were reduced from those of bulk OD (241.7 and 247.0 J/g, respectively). However, the prepared PDVB/OD microcapsules are able to be used for heat storage applications.

Keywords: Microencapsulation, Microcapsule, Heat Storage Material, Octadecane, Suspension Polymerization, Poly (Divinylbenzene)

1. Introduction

The utilization of a latent heat storage system using heat storage materials or phase change materials (PCMs) is an effective method of advanced energy technologies, preserving available energy and improving its consumption. The heat storage materials are able to change the status with a convinced temperature range. They can absorb and emit the energy when the temperatures of the materials overpass the temperature of phase change during heating and cooling processes, respectively [1-10]. The thermal energy storage systems typically select the application temperatures approach phase transition temperatures of PCMs. Paraffin waxes (the linear chain hydrocarbons or n-alkanes) such as tetradecane (TD), hexadecane (HD), octadecane (OD), nonadecane (ND), and eicosane are useful as one group of numerous numbers of heat storage materials that melt and solidify at a wide range of temperatures, making them attractive for many applications [11]. These PCMs are nontoxic, non-corrosi-

ve, chemically inert, easily obtained, and have no unpleasant odor. They have different phase change temperatures, melting (T_m) and solidification (T_s), depending on the number of carbons in their structures [9,12]. They have moderate thermal energy storage but low thermal conductivity. Therefore, they require large surface areas in applications. Encapsulation of these materials is associated with many advantages such as provide large heat transfer area, control the volume change of the storage materials as phase change occurs and protect the PCM against the influences of the outside environment [2,6,10,13]. Therefore, it is more practical than the traditional PCMs in many applications such as thermal insulation [7], heat transfer [14], building materials [15] and the wall energy storage capacity [16]. A numerous number of studies have been carried out on micro and nanocapsulation of PCMs using different polymers as the capsule shell materials. The *in situ* polymerization to fabricate the microcapsules and nanocapsules containing

OD core with melamine-formaldehyde shell [17], resorcinol-modified melamine-formaldehyde shell [18] were reported. The encapsulation of n-TD core with urea-formaldehyde polymer shell were also carried out by *in situ* polymerization [19]. Three types of paraffin waxes (n-HD, n-OD and n-ND) were encapsulated through complex coacervation of natural and biodegradable polymers, gum arabic-gelatin mixture [20]. Paraffin waxes encapsulated with styrene and methyl methacrylate copolymer were prepared by suspension polymerization [21]. The preparation of polystyrene (PS) microcapsules containing n-HD using casein instead of commercial stabilizer to emulsify the mixture of core materials, monomers and initiators during the suspension polymerization were also reported [22]. For textile applications, melamine formaldehyde microcapsules containing eicosane were prepared by *in situ* polymerization [23,24] and PS microcapsules containing paraffin wax were synthesized by suspension like polymerization [25]. Polymethylmethacrylate microcapsules containing n-octadecane [26] and heptadecane [27] were prepared by emulsion polymerization. The capsules of PS as the shell and n-OD as the core were prepared by miniemulsion polymerization [28]. Microencapsulated n-OD with polyurea shells having different soft segments was synthesized with interfacial polycondensation [29]. To improve the shell strength, crosslinked polydivinylbenzene (PDVB) microcapsule particles with encapsulated n-HD prepared by micro-suspension polymerization utilizing the Shirasu Porous Glass membrane emulsification technique for the preparation of comparatively monodisperse DVB/HD droplets were prepared [30-32]. OD is an attractive PCM giving good capability of heat absorption and emission in appropriate phase change temperature range (23 - 28°C) comfortable for the human body. Moreover, its latent heat (241.2 J/g) is higher than that of the other PCMS with similar phase change temperature range [33]. As seen from the litera-

tures above, a numerous of different polymers were used to prepare microencapsulated PCMs but there is no any report study on microencapsulation of OD with PDVB (the high mechanical properties polymer) shell (PDVB/OD capsules). Therefore, in this work, OD and PDVB were chosen to use as heat storage material and particle shell, respectively, of the microcapsules prepared by suspension polymerization. The morphology, chemical structure, thermal stability and thermal properties of PDVB/OD capsules were characterized by scanning electron microscope (SEM), optical microscope (OM), fourier transform infrared spectrophotometer (FT-IR), thermogravimetric analyzer (TGA) and differential scanning calorimeter (DSC), respectively.

2. Experimental

2.1. Materials

DVB (Aldrich; purity, 80%) was washed with 1N sodium hydroxide (NaOH) and distilled water to remove polymerization inhibitors before use. Poly (vinyl alcohol) (PVA) (Aldrich; degree of saponification, 87 - 90%) was used as received. Reagent-grade benzoyl peroxide (BPO) was purified by recrystallization. OD (Merck; 99.5%) was used as received.

2.2. Microcapsules Preparation

The microcapsules of PDVB/OD were prepared by suspension polymerization as the procedure shown in **Figure 1**. The homogeneous organic phase of DVB and OD at the ratio of 50:50% wt/wt (approximately 10% wt of aqueous solution) were mixed with BPO (8% wt of monomer) and then added to the aqueous phase containing PVA (1.5 g of PVA in 150 g of water). Emulsification was carried out by high shear rate at the speed of 5,000 rpm for 5 min resulting in the organic phase droplets dispersed in the aqueous phase. The resulting emulsions were transferred to the reactor and polymerized at

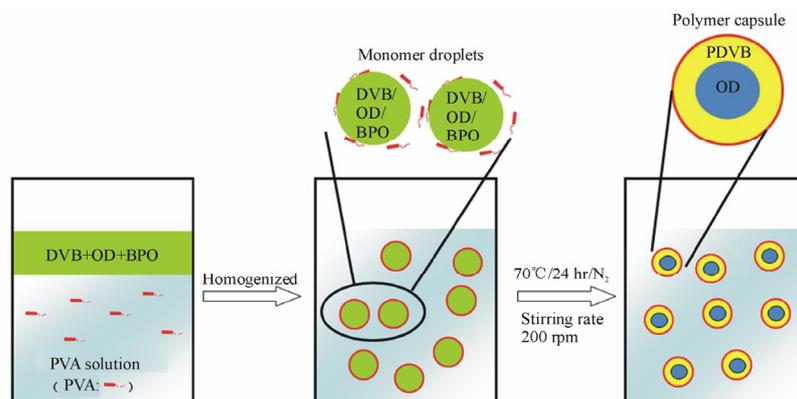


Figure 1. Schematic of the preparation process of PDVB/OD capsules with a suspension polymerization.

70°C for 24 h under N₂ atmosphere.

2.3. Characterization of Microcapsules

The particle diameters of microcapsules were obtained from OM (SK-100EB & SK-100ET, Seek, Seek Inter Corporation Ltd., Thailand) photos calculated from 200 particles by VIS Plus software. An OM and SEM (JSM-6510, JEOL, JEOL Ltd., Japan) were used to investigate the morphology of the surface and the inner structure of the microcapsules. For SEM observation, one drop of the microcapsule dispersion was placed on a nickel SEM stub and dried before Au-coated. Thermal stability of OD, PDVB and microcapsules was studied with TGA (TGA/SDTA 851°, Mettler-Toledo, Mettler-Toledo Ltd., Switzerland) using heating rate of 20°C/min. The chemical structure of the polymer was determined by FT-IR spectroscopy (PerkinElmer System 2000, PerkinElmer, USA). The dispersion sample was filtered and then kept in vacuum oven overnight. The dried sample was ground with dried potassium bromide (KBr) powder and compressed into a disc. The pure OD was directly mixed with KBr before measurement.

2.4. Thermal Properties Measurement

The latent heats of solidification (H_s), melting (H_m), T_s , and T_m of OD encapsulated in microcapsule particles in aqueous solution (solid content: ca 10%) were measured in an aluminum pan with a DSC (DSC 822, Mettler-Toledo, Mettler-Toledo Ltd., Switzerland) under a N₂ flow with the scanning temperature range and rate of 0 - 40°C and 5°C/min, respectively. To compare H_s and H_m of the encapsulated OD having different wt% in the capsule particles and also pure OD, the H_s and H_m values were used in the unit of joule per 1 g of encapsulated OD (J/g-OD). They were calculated from the cooling/heating peak area of DSC thermogram and OD content obtained from TGA analysis using the following equation (1).

$$\text{J/g-OD} = (A/B) \times 100 \quad (1)$$

where

$A = H_s$ or H_m of encapsulated OD in microcapsule dispersion obtained from DSC thermogram (J/g-sample) $B = \% \text{ OD}$ in microcapsule dispersion obtained from TGA thermogram

3. Results and Discussion

3.1. Microcapsule Formation

The preparation process of the PDVB capsules containing OD as heat storage material by suspension polymerization was shown in **Figure 1**. Aqueous phase containing PVA as stabilizer was added with oil phase consisting of OD, DVB and BPO. PVA molecules strongly adsorb

onto the oil-water interface and stabilize the oil droplets generated by high shear rate. The polymerization took place within the oil droplets after the temperature reached to 70°C. OD is well miscible with DVB monomer in the early stage of polymerization resulting in homogeneous phase in monomer droplets. When the PDVB was formed, the oil phase became gradually less compatible until reach the critical chain length of PDVB, phase separation occurred. Because the interfacial tension of PDVB-water is lower than that of OD-water, the PDVB move to the interface of oil droplets to form the polymer shell. At the end of polymerization, the polymer capsules of OD core encapsulated by PDVB shell were obtained. When the dispersions were kept for a while, all of the PDVB/OD capsules floated on the top of the dispersion solution because the total density of the capsules is lower than that of water. This indicates that the PDVB/OD capsules were successfully prepared.

3.2. Morphology of Microcapsule and Particle Size

The PDVB/OD microcapsules were observed by OM and SEM as shown in **Figure 2**. The SEM photograph indicates that most of the particle shapes were spherical with smooth outer surface even the microcapsule sizes were polydisperse consistent with the result from the optical micrograph. This is general characteristic of the polymer particles obtained from conventional suspension polymerization. Moreover, the OD core was completely encapsulated with PDVB shell as shown in the optical micrograph. The average diameter of the microcapsules is approximately 28 μm , measured from the optical micrographs calculated from 200 particles by VIS plus 3.0 software. Because quite small microcapsules were obtained, PDVB/OD capsules therefore afford a large surface area per unit volume. This is the reason why the microcapsules containing the heat storage material are more attractive than the pure one.

3.3. Chemical Structure Characterization of Microcapsule

FT-IR spectra of OD, PDVB and PDVB/OD microcapsules were shown in **Figure 3**. In the case of pure OD (**Figure 3(a)**), the characteristic absorption peak at 720 cm^{-1} corresponds to the in-plane rocking vibration of the methylene group. The absorption peak at 1460 and 1360 cm^{-1} are associated with C-H stretching vibrations of methylene bridges. The alkyl C-H stretching vibrations of methyl and methylene groups are observed at 2920 and 2850 cm^{-1} . In the case of PDVB spectrum (**Figure 3(b)**), the characteristic absorption peaks at 700 - 750 cm^{-1} are benzene ring deformation vibration. The absorption

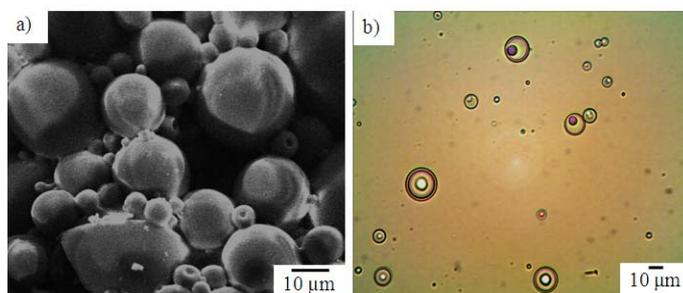


Figure 2. SEM photo (a) and optical micrograph (b) of PDVB/OD prepared by suspension polymerization.

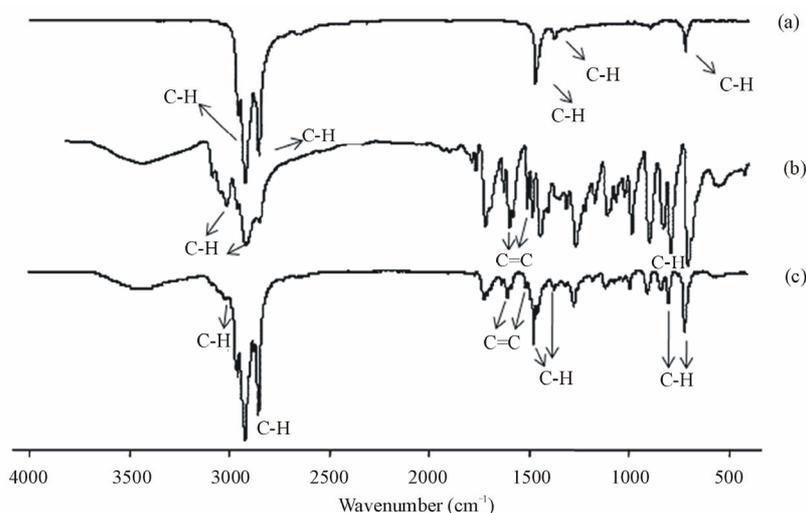


Figure 3. FT-IR spectra of OD (a), PDVB (b) and PDVB/OD capsule (c).

peaks at 1500, 1600 cm^{-1} are associated with benzene ring C = C stretching vibration. The aliphatic C-H stretching vibration is observed at 2920 cm^{-1} whereas 3030 cm^{-1} showed the characteristic peaks of aromatic C-H stretching vibration. All the above characteristic peaks of bulk OD and PDVB are able to observe in the spectrum of the PDVB/OD microcapsules (Figure 3(c)), and no new peak is observed. This result indicates that the OD was incorporated in the PDVB.

3.4. Thermal Stability of PDVB/OD Capsule

The TGA thermograms of bulk OD, PDVB and PDVB/OD capsule were shown in Figure 4 and Table 1. The decomposition of OD started at approximately 165°C and completely lost its weight at 305°C while the decomposition temperature range of PDVB is 330°C to 495°C. The TGA curves of OD and PDVB were sharp and showed only one step. In the case of PDVB/OD capsules TGA curve, it was mainly consisted of three steps: 50 - 124, 180 - 295 and 350 - 480°C corresponding to the decomposition of water, OD and PDVB respectively. In comparison with OD, the decomposition tem-

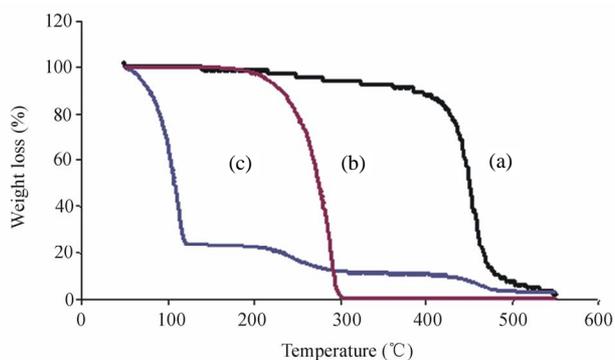


Figure 4. TGA thermograms of (a) PDVB; (b) OD and (c) PDVB/OD microcapsule.

perature of encapsulated OD was slightly higher than that of bulk OD due to the encapsulation.

3.5. Thermal Properties of Microcapsule

The thermal properties of the OD encapsulated in PDVB microcapsules were measured using DSC. The H_m (184.0 J/g-OD) and H_s (183.2 J/g-OD) of encapsulated OD were lower than those of the bulk OD (241.0 and 247.0 J/g of

Table 1. TGA data of OD, PDVB and PDVB/OD microcapsule.

	Degradation interval (°C)	Weight loss (%)
OD	162-305	100
PDVB	330-495	92
PDVB/OD	50 - 124 (1 st step)	76
	180 - 295 (2 nd step)	12
	350 - 480 (3 rd step)	10

H_m and H_s , respectively) as shown in **Figure 5**. This seems to be general case for PCMs microcapsules consistent with the other reports [17,18,22,26,27,29-32,34,35]. The possible reason of the reduction of H_m and H_s of encapsulated PCMs is that the phase separation between the polymer shell and PCM core was incomplete as in the case of PDVB/HD microcapsules prepared by suspension polymerization [32]. The capsules may have some unexhausted monomers or oligomer incorporated at the interface between polymer shell and PCM core as in the case of PS/HD microcapsules prepared by suspension polymerization [22]. They may act as the compatibilizer and help the miscibility between polymer and PCM material leading to the decreasing of the phase separation in oil phase. However, to overcome this problem, the copolymerization with more hydrophilic monomers is the good idea as copolymerization of PDVB with methyl acrylate, ethyl acrylate and butyl acrylate [32]. In the case of phase transition temperature, T_m of encapsulated OD (28.3°C) was almost the same as that of bulk OD (30°C). In contrast, T_s was shifted to the lower temperature comparing to bulk OD. This occurrence is namely supercooling. Supercooling leads to the reduction of the T_s , resulting in the releasing of latent heat at a lower temperature or a wider temperature range. This effect may limits to the applications. To prevent supercooling, the nucleating agents were incorporated in the PCMs core. However, increasing nucleating agent content decreased the latent heat [36,37]. At the present, there is still no good solution to overcome this problem. As the total surface area of the microcapsules increased comparing to bulk OD, these PDVB/OD capsules are quite acceptable for energy storage applications even having a slightly lower latent heats.

4. Conclusions

PDVB/OD microcapsules were successfully prepared by suspension polymerization. The optical micrographs showed that OD had been completely encapsulated inside the PDVB microcapsules with average size of 28 μm . The regular spherical particles with smooth outer surface were observed by SEM. All the characteristic peaks of OD and PDVB were observed in the FT-IR spectrum of

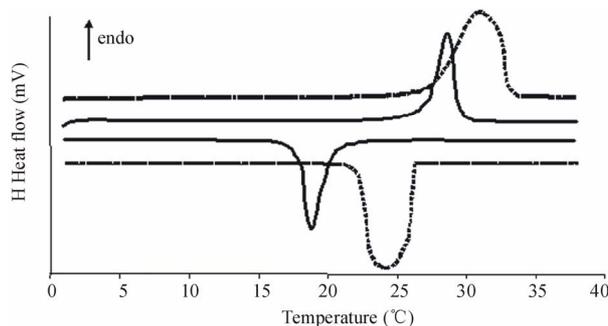


Figure 5. DSC thermograms of encapsulated OD in PDVB/OD capsule (solid line) and bulk OD (dotted line) (scanning rate 5°C/min).

the PDVB/OD capsules without any new peaks. The latent heats, H_m and H_s , of OD in the capsule were slightly lower than those in bulk OD. As a result, the prepared PDVB/OD microcapsules are able to be used for heat storage applications because of their large surface area and still remain good thermal properties of encapsulated OD.

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