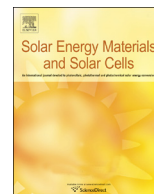




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Contents lists available at ScienceDirect

Solar Energy Materials & Solar Cells

journal homepage: www.elsevier.com/locate/solmat

Effects of fabricated technology on particle size distribution and thermal properties of stearic–eicosanoic acid/polymethylmethacrylate nanocapsules

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ARTICLE INFO

Article history:

Received 8 January 2013

Received in revised form

16 July 2013

Accepted 19 September 2013

Available online 5 October 2013

Keywords:

Phase change materials

Thermal energy storage

Nanocapsule

Fatty acid eutectic

Chemical technique

ABSTRACT

Polymethyl methacrylate (PMMA) encapsulated eicosanoic–stearic acid (EA–SA) eutectic nanocapsules were prepared by ultraviolet photoinitiated emulsion polymerization and various characterization techniques were employed to investigate the influence of preparation methods on thermal properties and particle size distribution (PSD). The results show that the particle size decreased and PSD narrowed with the increase of agitation speed, reduction of initiator and monomer concentration, existence of cross-linking agent and stabilized at agitated speed higher than 5000 rpm and monomer concentration lower than 0.15 mol/L. However, latent heats of the capsules decreased with the increase of monomer and initiator concentration. Type of emulsifier in emulsion has significant effects on PSD and phase change properties of EA–SA/PMMA and nonionic emulsifier is suitable for reducing particle size and enhancing heat storage ability. Morphology and chemical characteristic analysis indicate that spherical nanocapsules with average diameter of 46 nm were successfully fabricated and its maximum encapsulation ratio is 68.8 wt% without leakage of core material. The melting and crystallizing temperatures and latent heats of capsules were determined as 56.9 °C and 54.5 °C, 126.4 J/g and 128.3 J/g, respectively. Accelerated thermal cycling test shows that the nanocapsules have good thermal and chemical reliability after repeated thermal cycling. Besides, the super-cooling problem of PCMs reduced dramatically by forming nanocapsules.

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1. Introduction

The current energetic and environmental situation reveals the urgent need for a more rational and efficient use of energy. Thermal energy which could be obtained from clean sources such as the sun, geothermal fields, oceans and waste heat from many man-made devices is one of the main sources of energy. Therefore, there is a large interest to develop efficient systems or materials to scavenge thermal energy conveniently and reuse it either directly or convert it to suitable form such as electricity which is as important as developing new sources of energy [1–3]. Latent heat thermal energy storage (LHTES) has been considered as an important advanced energy technology due to the insignificant temperature variation between energy storage and release and considerable latent heat per unit volume of phase change materials (PCMs). As renewable and clean energy storage materials, PCMs have been demonstrated as an acceptable energy storage

in various applications, such as thermo-regulated building materials [4,5], heat transfer media [6], heat and solar energy storage devices [7,8] and thermo-regulating or intelligent textiles [9].

So far, a large number of inorganic and organic compounds have been intensively investigated, such as salt hydrates, paraffin, fatty acids, fatty acid esters and their binary and ternary mixtures. Among the investigated PCMs, fatty acids are preferred due to their high energy storage density, excellent chemical stability and nontoxicity, proper thermal characteristics of constant temperature and smaller volume change during the phase transition. However, there is a risk of PCMs leakage when PCMs melt and crystallize repeatedly during direct use in practical. Additionally, low thermal conductivity which leads to large temperature gradients during heat transfer in or out of the material, and reduces heat transfer rates is the other problem [1,10,11]. Therefore, it seems that encapsulation is an effective solution for the inherent problems of PCMs [12,13]. Microencapsulated phase change materials (MEPCMs) are very small bicomponent particles consisting of a core material, the PCMs, and an outer shell or capsule wall. By microencapsulating PCMs into inorganic or polymer shells, their heat transfer area is enlarged, and thus their

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heat transfer efficiency can be greatly increased. Meanwhile, the MEPCMs can be used directly without extra encapsulation because of the reactivity of PCMs towards the outside environment can be reduced and the change in volume during the phase transition can be controlled [14].

Lots of works have been done in fabricating and characterizing of microcapsules using different shell materials include silicon dioxide [15,16], polystyrene (PS) [17], melamine-formaldehyde (MF) [18], urea-formaldehyde (UF) [19], polymethyl methacrylate (PMMA) [20,21], polycarbonate (PC) [22], styrene-methyl methacrylate copolymer [23] and rigid polyurethane (PU) [24]. Latest literature survey on MEPCMs indicated that the microencapsulation of PCMs using PMMA as shell, which is a thermoplastic, transparent and commercially available acrylic resin with the merits of non-toxic, easy handling and processing, relative chemical resistance and high impact strength, have attracted more and more attention [20,21,25–27]. From this point of view, PMMA is a versatile material and promising polymer used as shell material for LHTES application. Several physical and chemical methods have been developed for producing of PMMA shell microcapsules. Wang [26] introduced self-polymerization method to encapsulate capric acid (CA)–lauric acid (LA), CA–MA (myristic acid), CA–SA and LA–MA eutectics with PMMA in the ratio of 50/50 wt% and investigated their potential application in building. Loxley [27] introduced phase separation method to encapsulate *n*-hexadecane with PMMA and obtained the capsules with shells of various thicknesses by altering the concentration of polymer in the oil phase. Alkan et al. [25] prepared a series of fatty acids/poly (methyl methacrylate) (PMMA) form-stable PCMs by entrapping stearic acid (SA), palmitic acid (PA), myristic acid (MA), and lauric acid (LA) into PMMA polymer matrix using solution casting method. C. Alkan also microencapsulated *n*-octacosane [28], *n*-heptadecane [29], docosane [30], and *n*-eicosane [20] with PMMA as the shell by emulsion polymerization, in which methylmethacrylate, allyl methacrylate and tertbutylhydroperoxide were used as shell-forming monomers, cross linking agent and initiator, respectively. Qiu [31] synthesized *n*-octadecane containing microcapsules with different crosslinked methylmethacrylate-based polymer as shells by suspension-like polymerizations and they also investigated the influences of the type and amount of cross-linking agent, the type of initiator and polymerization temperature on the properties of as-prepared microencapsulated phase change materials. Shirin-Abadi [1] investigated the in situ encapsulation of *n*-hexadecane with PMMA shell through mini-emulsion polymerization and the shell thickness, void fraction of capsules were estimated. The results showed that the weight fractions of pure polymeric particles for the prepared samples were 12–17 wt%, and the shell thickness of the capsules was about 12 nm, the void fractions for these samples were 69–77%. Wang [21,32] and Ma [33] microencapsulated phase change material by emulsion polymerization utilized irradiation-initiated method and the validity of UV irradiation to fabricate MEPCMs was identified.

Using ultraviolet (UV) light initiated polymerization has many significant advantages, such as the polymerization process can be easily varied by controlling the light intensity and the exposure time of irradiation. Another advantage of UV initiated polymerization is that the radical flux is independent of temperature, whereas, a certain temperature is necessary for significant decomposition of the initiator in chemical initiation [34,35]. There is limited precedence for producing microencapsulated PCMs by UV initiated emulsion polymerization, but the previous reports in literatures [21,32,33] are focused on the preparation, characterization and thermal properties of PMMA shell MEPCMs. The special investigations about the influence of fabrication technology on the particle size distribution and thermal energy storage property of microencapsulated PCMs are ignored. As we all know, particle

diameter and thermal energy capability are the vital parameters for MEPCMs in practical application. For example, in thermal fluids, the large particles of microencapsulated PCMs cause the increase in a fluid's viscosity and often crush during pumping as well.

On the basis of this understanding, the encapsulation of stearic–eicosanoic acid eutectic mixtures in PMMA for the preparation of MEPCMs through UV photoinitiated emulsion polymerization has been studied in the current work. Also, the processing parameters on particle size distribution and heat storage ability were investigated. In addition, Fourier transform infrared spectroscopy (FT-IR), scanning electronic microscope (SEM), differential scanning calorimetry (DSC), accelerated thermal cycling test were employed to determine the chemical characteristics, microstructure and thermal performance, chemical and thermal reliability of the MEPCMs which prepared in the optimum technical conditions.

2. Experimental

2.1. Materials

Phase change materials, core materials, including eicosanoic acid (EA, AR) and stearic acid (SA, industrial grade) were supplied by Aladdin Chemical Reagent Co. Ltd. and Beijing Chemical Co. Ltd., respectively. Methyl methacrylate (MMA, AR) purchased from Tianjin Kermel Chemical Reagent Co. Ltd. was used as monomer in the composition of the shell. MMA was washed by 5% NaOH solution ($V_{\text{MMA}}:V_{\text{NaOH}}=5:1$) in a separatory funnel for several times to remove the polymerization inhibitor hydroquinone and then purified by double atmospheric distillation before use. Polyethylene glycol octyl phenylether (TX-100), cetyl trimethyl ammonium bromide (CTAB), sodium dodecyl sulfate (SDS) and poly(vinyl alcohol) (PVA) were obtained from Shanghai Chemical Reagent Co. Ltd. and used as surfactant and dispersing agent, respectively. Ethylene glycol dimethacrylate (EGDMA) and 1, 4-butanediol dimethacrylate (BDDMA) were bought from Shanghai Chuanghe Chemical Reagent Company and used as crosslinking agent. Oil-soluble initiator, 2-hydroxyl-2-methyl-1-phenyl acetone (1173) was supplied from TCI (Shanghai) Chemical Co. Ltd. Deionized water (DI) consumed in all polymerization recipes was self-made by our laboratory. The chemical structures of TX-100, EGDMA and BDDMA crosslinking agent and 1173 photosensitive solicitation reagent are shown in Fig. 1.

2.2. Preparation of stearic–eicosanoic acid eutectic mixture

Eutectic mixture, a composition of two or more components, has the lowest melting temperature and the best thermal reliability [36]. In order to determine the eutectic mixture ratio, a series of the binary systems of SA and EA in different weight

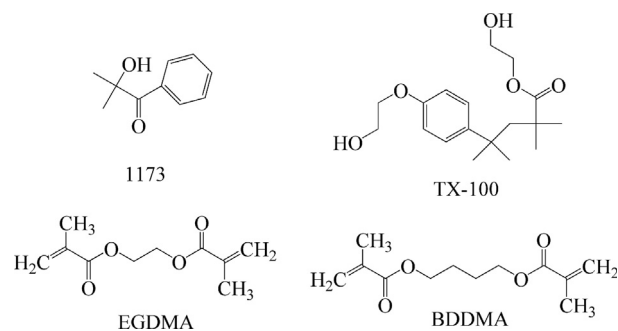


Fig. 1. Chemical structures of TX-100, EGDMA, BDDMA and 1173.

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