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Particuology

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Advanced microemulsion synthesis and characterization of wollastonite (CaSiO₃)/polystyrene one-dimensional nanorods with core–shell structures

Aniruddha Chatterjee*, Prashant Khobragade, Satyendra Mishra**, Jitendra Naik

University Institute of Chemical Technology, North Maharashtra University, Jalgaon 425001, Maharashtra, India

ARTICLE INFO

Article history:

Received 29 August 2015
Received in revised form 19 April 2016
Accepted 23 April 2016
Available online xxx

Keywords:

Core–shell structures
Atomized microemulsion
One-dimensional nanorods

ABSTRACT

In this work, one-dimensional core–shell nanorods (CSNRs; 185–250 nm wide and 1–1.5 μm long) consisting of triethoxyvinylsilane-modified wollastonite (CaSiO₃) nanorods (MWNRs) as a core and polystyrene as a shell with uniform size were successfully synthesized using an advanced microemulsion technique. The effect of varying the surfactant cetyltrimethylammonium bromide (producing CSNRs_{CTAB}) or sodium dodecyl sulphate (producing CSNRs_{SDS}) upon the size and morphology of the CSNRs was investigated by field-emission scanning electron microscopy (FE-SEM). X-ray diffractometry and Fourier transform infrared spectrophotometer revealed the existence of a strong interaction between the MWNRs and polystyrene, which implies that the polymer chains were successfully grafted onto the surface of the MWNRs. The CSNRs were blended with polypropylene by melt processing, and the effect of the CSNRs upon the morphological properties of the polypropylene matrix was investigated by FE-SEM and atomic force microscopy. It was observed that the polystyrene chains that grafted onto the CaSiO₃ nanorods interfered with the aggregation of CaSiO₃ nanorods in the polypropylene matrix and thus improved the compatibility of the CaSiO₃ nanorods with the polypropylene matrix. Furthermore, the compatibility of CaSiO₃ nanorods with polypropylene of CSNRs_{SDS}/polypropylene was superior to that of CSNR_{CTAB}/polypropylene.

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Introduction

Core–shell nanomaterials have recently attracted considerable research attention owing to their large variety of applications in

chemistry, biotechnology, and materials science. These core–shell nanomaterials have been designed and developed to exhibit novel and excellent optical, electrical, magnetic, chemical, mechanical, rheological, and catalytic properties with multifunctional and multidimensional structures by varying the compositions (Ma, Zhu, Li, & Cao, 2008; Radev, Hristov, Samuneva, & Ivanova, 2009). For inorganic nanoparticles, the encapsulation of the nanoparticles made of materials such as titanium dioxide (TiO₂) (Choi, Kim, & Kwak, 2007; Faridi-Majidi & Sharifi-Sanjani, 2007; Jiang et al., 2006; Li, Liang, Zheng, Lin, & Cai, 2014; Rong, Chen, Wu, & Wang, 2005; Yang & Dan, 2005), silver (Ag) (Chen et al., 2015), gold (Au) (Tang, Liu, Sun, Zheng, & Cheng, 2007; Zawada, Tomaszewski, & Megiel, 2014), zinc oxide (Tang, Cheng, & Ma, 2006), silica (Avolio, Gentile, Avella, Capitani, & Errico, 2010; Chakkalalal, Alexandre, Boschetti-de-Fierro, & Abetz, 2012; Chevigny, Gignes, Bertin, Jestin, & Boué, 2009; Chuayjuljit & Boonmahitthisud, 2010; Chuayjuljit & Luecha, 2011; Kwon, Im, & Kim, 2011; Zhu, Cai, Zhou, & Shi, 2008), calcium carbonate (CaCO₃) (Bhanvase, Pinjari, Gogate, Sonawane, & Pandit, 2011; Ma, Rong, Zhang, & Friedrich, 2005), halloysite (Liu et al., 2011), antimony trioxide (Xie, Li, Liu, & Mai, 2004), alumina (Rong, Ji, Zhang, & Friedrich, 2002; Zeng, Yu, & Guo, 2005), and

Abbreviations: 1D, one-dimensional; AFM, atomic force microscopy; APS, ammonium persulfate; CSNRs, core–shell nanorods; CTAB, cetyl trimethyl ammonium bromide; DI, deionized; DSC, differential scanning calorimetry; EDS, energy-dispersive X-ray spectroscopy; FE-SEM, field-emission scanning electron microscopy; FTIR, Fourier transform infrared spectroscopy; HR-TEM, high-resolution transmission electron microscopy; MWNRs, surface-modified wollastonite (CaSiO₃) nanorods; nPS, polystyrene nanoparticle; PMMA, polymethyl methacrylate; PP, polypropylene; PS, polystyrene; SDS, sodium dodecyl sulphate; TEM, transmission electron microscopy; TEVS, triethoxyvinylsilane; TGA, thermogravimetric analysis; WCA, water contact angle; WNRs, wollastonite (CaSiO₃) nanorods; XRD, X-ray diffraction.

* Corresponding author. Present address: Maharashtra Institute of Technology, Aurangabad, Maharashtra, India. Tel.: +91 2402375260; fax: +91 2402375275.

E-mail addresses: aniruddha.chatterjee2006@yahoo.co.in, aniruddha.chatterjee@mit.asia (A. Chatterjee).

** Corresponding author.

E-mail addresses: profsm@rediffmail.com (S. Mishra).

<http://dx.doi.org/10.1016/j.partic.2016.04.002>

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Please cite this article in press as: Chatterjee, A., et al. Advanced microemulsion synthesis and characterization of wollastonite (CaSiO₃)/polystyrene one-dimensional nanorods with core–shell structures. *Particuology* (2016), <http://dx.doi.org/10.1016/j.partic.2016.04.002>

ferric oxide (Bach, Islam, Kim, Seo, & Lim, 2012), using polymers such as poly(methylmethacrylate) (Avolio et al., 2010; Bach et al., 2012; Bhanvase et al., 2011; Chakkalakal et al., 2012; Jiang et al., 2006; Kwon et al., 2011; Liu et al., 2011; Tang et al., 2006; Xie, Li, Liu, & Mai, 2006; Yang & Dan, 2005; Zhu et al., 2008) and polystyrene (Chen et al., 2015; Chevigny et al., 2009; Choi et al., 2007; Chuayjuljit & Boonmahitthisud, 2010; Chuayjuljit & Luecha, 2011; Faridi-Majidi & Sharifi-Sanjani, 2007; Rong et al., 2002, 2005; Tang et al., 2007; Zawada et al., 2014; Zeng et al., 2005), have been studied intensively and have received interest in a wide range of industrial fields. Core-shell nanoparticles consisting of an inorganic core and a polymer shell sometimes combine the desired properties of the different constituent materials (Toskas et al., 2011; Zhong & Maye, 2001). For example, the inner inorganic core exhibits high rigidity, thermal resistance, and mechanical strength while the outer polymer shell can increase the nanoparticle stability and tenability with heat stability, high strength and chemical resistance to the inorganic part and good flexibility and mouldability to the organic part. Inorganic nanoparticles, however, have a tendency to agglomerate because of their high surface area and surface energy. Traditionally, low-molecular coupling agents or surfactant treatment of the filler surface have been shown to be reasonably effective. However, low molecular weight compounds have a tendency to migrate out from the interface, and the consequent changes in the mechanical and physical properties of the composites are not desirable. Therefore, there is high demand for new technologies for core surface treatments, wherein encapsulation of nanoparticles by a polymer is a promising way to overcome the problem. In addition to agglomeration, a major problem related to nanocomposites is their dispersibility within the polymer matrix. Surface modification (Li et al., 2014), which comprises alteration of the nanoparticle surface from hydrophilic to hydrophobic via polymer grafting, is one of the best ways to obtain a uniform and high level of dispersion of inorganic nanoparticles in the polymer matrix.

Among inorganic nanoparticles, calcium silicate (wollastonite) has shown great importance in biomedical (drug delivery, tissue engineering, bone and dental related applications), polymer, and coating applications owing to its superior bioactivity and biodegradability, chemical compatibility, and antibacterial properties (Ding, Shie, & Wang, 2009; Guo et al., 2013; Kalaivani, Singh, Ganesan, & Kannan, 2014; Lu, Zhu, Ao, Qi, & Chen, 2012) compared with other inorganic materials. However, the porous nature (Kang, Huang, Yang, Yang, & Lin, 2011; Wu et al., 2012) and micron-scale size of the wollastonite (CaSiO_3) particles weakens their mechanical properties and other features, and it is therefore a great challenge to maintain the properties of these particles (Wu et al., 2012). Researchers have developed nano- CaSiO_3 and modified nano- CaSiO_3 particles of different shapes and sizes to overcome these difficulties, particularly for biomedical and polymer applications (Guo et al., 2013; Luyt, Dramićanin, Antić, & Djoković, 2009; Motisuke, Santos, Bazanini, & Bertran, 2014; Ruiz-Hitzky, Aranda, Darder, & Ogawa, 2011). Surface modification and dispersion of CaSiO_3 nanorods remains a significant challenge for researchers (Radev, Hristov, Fernandes, & Salvado, 2010; Obare, Jana, & Murphy, 2001). Recently, researchers have reported the effect of poly(ethylene glycol) particles and polymethyl methacrylate (PMMA) beads upon the pore structures of CaSiO_3 for in vivo bone-formation applications (Xu et al., 2008; Wang et al., 2012). A polymeric layer on the inorganic surface reduces the particle surface energy as well as promotes dispersion of the particles and interfacial adhesion, leading to enhanced mechanical properties, toughness, and heat resistance of the resulting composites.

Recently we have synthesized polystyrene (PS) and PMMA nanoparticles by a modified microemulsion process (Mishra & Chatterjee, 2011a) as well as a novel atomized microemulsion

process (Mishra & Chatterjee, 2011b), and isolated PS and PMMA nanoparticles were subsequently blended with polypropylene (PP) (Mishra & Chatterjee, 2011a) and linear low-density polyethylene (Mishra, Chatterjee, & Rana, 2011a) to study their rheological, thermal, and mechanical properties. In our previous work, we have successfully prepared core-shell particles comprising calcium carbonate nanoparticles (nano-CaCO_3)/PS (Chatterjee & Mishra, 2013a; Mishra, Chatterjee, & Singh, 2011b) as well as (nano-CaCO_3)/PMMA (Chatterjee & Mishra, 2012, 2013b) with CaCO_3 as the core and PS or PMMA as the shell by an atomized polymerization technique, whereupon isolated core-shell nanoparticles were incorporated in PP and high-impact PS matrices. The antibacterial activity, cytotoxicity, and sustained drug release behaviour of PMMA/biosurfactant core-shell nanoparticles have also been studied by Hazra, Kundu, Chatterjee, Chaudhari, and Mishra (2014). They have also synthesized biosurfactant-templated calcium sulphate (CaSO_4) nanorods by an ultrasonication method (Hazra et al., 2014b) and investigated the thermophysical, mechanical, and biodegradation properties of biosurfactant-graft- CaSO_4 /PS nanocomposites (Kundu, Hazra, Chatterjee, Chaudhari, & Mishra, 2014).

In a continuation of our previous work, this study aims to develop an efficient and faster process for production of CaSiO_3 /PS CSNRs by an advanced atomized microemulsion process. The hydrothermal microemulsion technique (Chatterjee, Khobragade, & Mishra, 2015) has been used to synthesize one-dimensional (1D) CaSiO_3 nanorods, which were then encapsulated by PS using cetyl trimethyl ammonium bromide (CTAB) and sodium dodecyl sulphate (SDS) as surfactants via an atomized microemulsion technique. To the best of our knowledge, limited studies have been reported on the encapsulation of CaSiO_3 nanorods using a microemulsion-assisted route, which consumes less reaction time with a high percentage of grafting. This has motivated us to use this route with the advanced atomized microemulsion technique, and the objective of this work was therefore to explore the synthesis of 1D CaSiO_3 /PS CSNRs through this advanced atomized microemulsion technique within a very short time (1.5 h). Furthermore, we also focused on improving the performance of the (CaSiO_3 /PS CSNRs)/PP nanocomposites by enhancing the interfacial adhesion by adding a small amount (1.5 wt%) of encapsulated CaSiO_3 nanorods. Thus, this approach could improve the compatibility of the CaSiO_3 nanorods with the polymer matrix through the lipophilic polymer layer (PS layer) grafted onto the surface of the CaSiO_3 nanorods. The polymer shell coatings not only prevented aggregation of the nanorods but also produced excellent compatibility between the core particles and the polymer matrix (Chatterjee & Mishra, 2013a; Mishra et al., 2011b).

Experimental

Chemicals and reagents

The wollastonite nano-rods (WNRs), with an average diameter of 150–200 nm and length of 1–1.5 μm , were synthesized by the hydrothermal microemulsion technique (Chatterjee et al., 2015). The nanorods were subsequently modified by the silane coupling agent, triethoxyvinylsilane (TEVS). The materials, including the monomer styrene, the initiator ammonium persulfate (APS), and the surfactants cetyl trimethyl ammonium bromide (CTAB) and sodium dodecyl sulphate (SDS) were procured from Sigma Aldrich (Mumbai, India). The styrene was treated with a 5% NaOH aqueous solution to remove the inhibitor and was distilled under reduced pressure in a nitrogen atmosphere prior to polymerization. All other materials were of analytical grade, and were used without further purification. Deionized (DI) water was used throughout

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