Particuology 22 (2015) 157–162

Contents lists available at ScienceDirect

Particuology

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

Fabrication of submicron barium sulfate aggregates in the presence of ethylenediaminetetraacetic acid anions

Linna Hu [a,*], Guangxiu Wang [a], Chun Yang [a], Rong Cao [b]

[a] School of Chemical Engineering and Technology, Hebei University of Technology, Tianjin 300130, China
[b] Baoding Lucky Innovative Materials Co., Ltd., Baoding 071000, China

ARTICLE INFO

Article history:
Received 4 August 2014
Received in revised form 27 October 2014
Accepted 2 November 2014

Keywords:
Barium sulfate
EDTA anion
Particle morphology
Self-assembly
pH value

ABSTRACT

Barium sulfate aggregates with an average size of 0.5 μm were synthesized at pH 7, directed by ethylenediaminetetraacetic acid (EDTA) anions. The particle morphology, chemical composition, and size distribution of the $BaSO_4$ aggregates were characterized. The as-synthesized $BaSO_4$ particles were spherical and comprised many interconnected nanoballs, of which the surface properties were affected by the EDTA anions. The adsorption of EDTA anions reversed the charge and weakened the surface polarity of $BaSO_4$, instigating the formation of aggregates by a self-assembly and transformation process. The resulting $BaSO_4$ particles at pH 9–10 were ellipsoidal and featured smooth surfaces. Based on the zeta potential of $BaSO_4$, variations in the morphology induced by changes in pH were closely related to the adsorption of mono- and multi-valent anions onto the electrical double layer of $BaSO_4$.

© 2015 Chinese Society of Particuology and Institute of Process Engineering, Chinese Academy of Sciences. Published by Elsevier B.V. All rights reserved.

Introduction

Recently, there has been increasing interest in using barium sulfate particles as reinforcement in polymer matrices (Mann & Ozin, 1996; Mathur, Shen, & Altmayer, 2007). The addition of barium sulfate in polymers can result in materials possessing higher stiffness, strength, and corrosion resistance when compared with that of conventional filled polymer composites (Wang, Wu, Ye, & Zeng, 2003; Zeng & Bai, 2005). However, a key problem remains: can the particles achieve a stable dispersion in the polymer? Small particles can easily agglomerate into larger particles because of the high surface activity and high surface energy during the preparation process. Thus, controlling the particle size and dispersivity and avoiding the agglomeration of small particles during the preparation process are current hot topics in the field of solid state materials (Sun, Zhang, Wu, & Zhu, 2014; Wang, Liu, & Zhang, 2009; Wong, Jaworski, & Nienow, 2001; Zhao et al., 2011).

EDTA can form stable complexes with various metal cations and is widely used in many industrial and academic fields. It is well known that EDTA can interact with Ba^{2+} cations in solution, thereby reducing the number of free ions available to participate in the crystallization process and further enhancing the crystal growth of BaSO4 (Jones et al., 2007; Zhang et al., 2009a; Zhang, Zhang, Li, Yin, & Guo, 2011). However, it is shown in this paper that EDTA does not operate in this manner only, and that direct EDTA–surface interactions are also important to consider. Spherical BaSO4 aggregates with a good dispersion were fabricated in the presence of EDTA that was added to control the microenvironment during the preparation process. The EDTA consumption was very small and EDTA could be recycled during the synthesis process (Liu, Tang, Lv, Li, & Ding, 2014; Pieper, Aman, & Tomas, 2012). The as-obtained aggregates were of comparable sizes with those reported in the literature (Jones et al., 2007; Zhang et al., 2009a; Zhang et al., 2011).

In a particular system, the growth units do not aggregate randomly, but in an ordered manner, subsequently forming regular geometric structures. Furthermore, the aggregation of small crystals often leads to porous and hollow structures that are desirable attributes for several potential applications such as water purification, solar energy conversion, and drug carrier systems (Liu, Bai, & Sun, 2011; Li et al., 2007). Spherical BaSO4 particles are ideal core materials because of their low cost, non-toxicity, thermal stability (melting point, 1580 °C), and chemical inertness. Furthermore, spherical particles with a narrow size distribution can be easily obtained. For example, submicron spherical BaSO4 particles have been coated with nano Y_2O_3:Eu^{3+} phosphor layers by a wet chemical method. The $BaSO_4$–Y_2O_3:Eu^{3+} core-shell particles showed a

* Corresponding author. Tel.: +86 022 60204744; fax: +86 022 60438201.
E-mail addresses: hln@hebut.edu.cn, 18002136882@163.COM (L. Hu).

http://dx.doi.org/10.1016/j.partic.2014.11.009
1674-2001/© 2015 Chinese Society of Particuology and Institute of Process Engineering, Chinese Academy of Sciences. Published by Elsevier B.V. All rights reserved.

red emission under ultraviolet excitation (Zhang et al., 2009b). Therefore, spherical BaSO4 aggregates have attracted great interest owing to their importance in both fundamental research and potential industrial applications.

In the current study, a new type of spherical BaSO4 aggregates were successfully prepared by a self-assembly and transformation process controlled by EDTA anions. An interesting advantage of this simple fabrication method was that the particle size and morphology could be easily tuned. Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), and laser particle size analysis were used to understand the effect of the EDTA anions on the morphology and size distribution of the BaSO4 particles. Furthermore, the zeta potential of the particle surface was investigated. The adsorption of mono- and multi-valent anions in the electrical double layer (Hang, Shi, Feng, & Xiao, 2009) of the barium sulfate particles had an obvious influence on particle morphology. New insights and mechanism schematic models were proposed.

Experimental

Synthesis of BaSO4 aggregates

A facile synthesis procedure for the preparation of BaSO4 aggregates was performed as follows. The reaction temperature was maintained at 45 °C using a temperature controlling instrument. BaCl2·2H2O (14.66 g, Sinopharm Chemical Reagent Co., Ltd, analytical reagent) and disodium ethylenediaminetetraacetate (22.33 g, Sinopharm Chemical Reagent Co., Ltd, analytical reagent) were mixed in distilled water (100 mL). The solution pH, as monitored by a pH meter, was adjusted to the desired value using NH3·H2O (Sinopharm Chemical Reagent Co., Ltd, analytical reagent). Then, Na2SO4 solution (100 mL, 0.6 mol/L, Sinopharm Chemical Reagent Co., Ltd, analytical reagent) was added dropwise to the above solution at a rate of 60 drops/min. After several minutes, the mixture gradually turned turbid and a white precipitate formed. After the reaction was complete, the mixture was kept under static conditions for 8 h. Finally, the white precipitate was filtered and washed several times with distilled water and dried in an oven at 100 °C for 4 h. The desired product was lightly ground and passed through a 220-mesh sieve for subsequent studies.

Characterization

The particle size distribution (PSD) was evaluated on a laser particle size analyzer (LS-POP, OMEC, China) using water as a suspending medium. The morphology of the particles was examined on a Hitachi S-4800 (Japan) scanning electron microscope (SEM) operating at 15 kV. Prior to SEM analysis, the samples were dispersed in absolute ethanol, dropped onto carbon-coated copper grids placed on a filter paper, and dried at room temperature (Hu, Dong, & Zhen, 2009; Wang et al., 2007). FTIR spectra were recorded in the range of 400–4000 cm^{-1} on a VECTOR-22 FTIR spectrometer (Bruker, Germany) with a resolution of 8 cm^{-1} by embedding the prepared powders in KBr pellets. X-ray diffraction (XRD) patterns of the powdered samples were recorded on a D/MAX-2000/PC diffractometer (Rigaku Corporation, Japan) equipped with Cu Kα radiation at 40 kV and 200 mA. Zeta potential analysis of the BaSO4 particles prepared at varying aging times was conducted on a Zetasizer 3000 HS (Malvern, UK) equipped with a microprocessor unit. The unit automatically calculated the electrophoretic mobility of the particles for conversion to zeta potential values using the Huckel and Smoluchowski equations.

Results and discussion

Particle size distribution

Typically, the formation of BaSO4 includes a precipitation step (0.5 h), an aging step (8 h), and a drying step (4 h). The PSD and mean

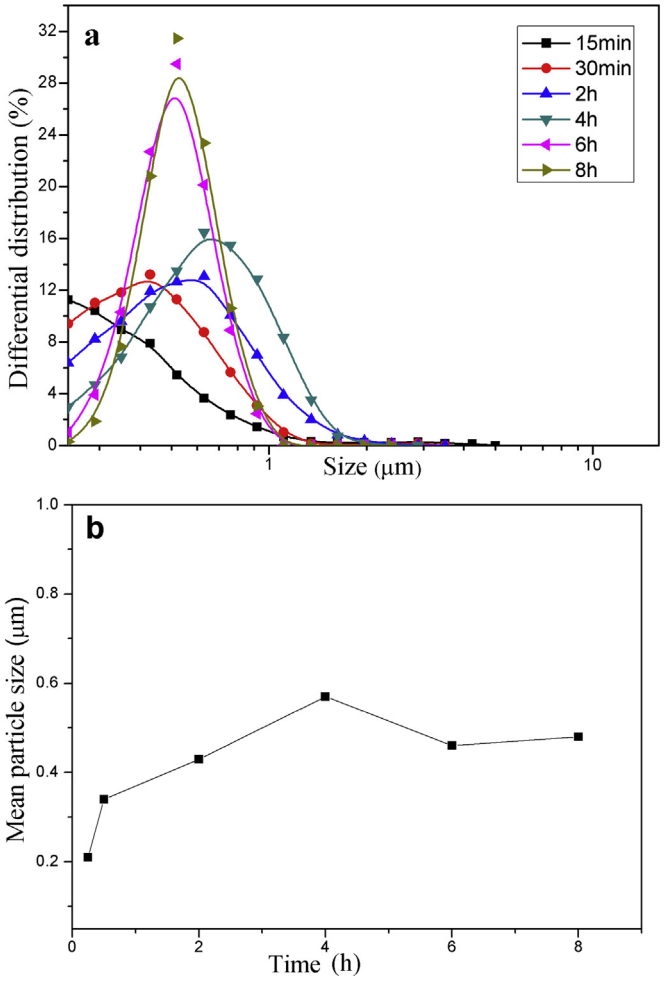

Fig. 1. (a) Particle size distributions and (b) mean particle sizes of the BaSO4 samples obtained from a precipitation and aging stages in the presence of EDTA.

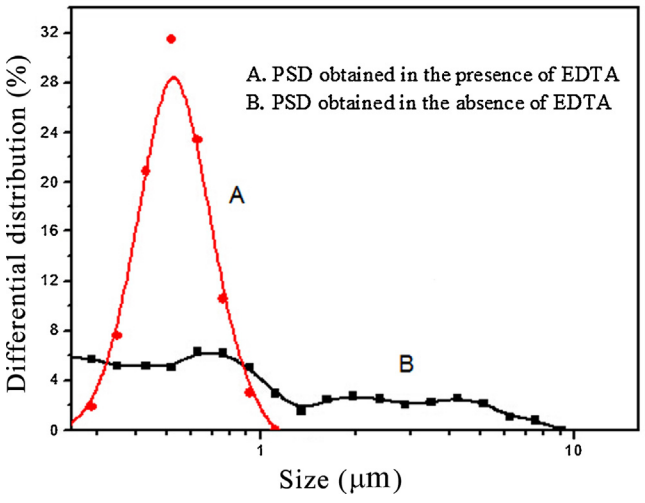

Fig. 2. Particle size distributions of the BaSO4 samples, prepared in the presence (Curve A) and absence (Curve B) of EDTA, measured after the drying process.

Download English Version:

https://daneshyari.com/en/article/671867

Download Persian Version:

https://daneshyari.com/article/671867

Daneshyari.com