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JOURNAL OF RARE EARTHS, Vol. 32, No. 11, Nov. 2014, P. 1022

A novel multifunction material with both electrorheological performance and luminescence property

CHEN Mingxing (陈明星)¹, SHANG Yanli (商艳丽)², JIA Yunling (贾云玲)¹, LI Junran (李俊然)^{1,*}

(1. College of Chemistry and Molecular Engineering, Peking University, Beijing 100871, China; 2. College of Chemistry and Environmental Science, Hebei University, Baoding 071002, China)

Received 1 April 2014; revised 10 July 2014

Abstract: A multifunctional material with both electrorheological (ER) performance and luminescence property was synthesized by a simple coprecipitation. The tetrabutyl titanate, as well as the Tb(NO₃)₃·6H₂O and sulphosalicylic acid (C₇H₆O₆S·2H₂O, SSA) were chosen as starting materials. The composition, ER performance and luminescence property of the material were studied. The results showed that a novel material (TiTbSSA) with both ER performance and luminescence property was obtained. The relative shear stress $\tau_r(\tau_r=\tau_E/\tau_0, \tau_E \text{ and } \tau_0 \text{ were the shear stresses of the suspension with and without an applied electric field) of the suspension (30 wt.%) of the material in silicone oil reached 32.7 at a shear rate of 12.5 s⁻¹ and an electric field strength of 4 kV/mm (DC electric field). The material containing the rare earth (RE=Tb) complex exhibited fine luminescence performance and higher ER activity. Therefore, it is a novel multifunction material which would have wide application prospect.$

Keywords: multifunctional material; electrorheological performance; luminescence property; rare earth complex

Electrorheological (ER) fluid is an exceptional suspension, the dispersed particles in the suspension would be polarized and attracted to each other to form a fiber-shaped structure, consequently make an ER fluid increasing its viscosity, in an applied electric field^[1]. Furthermore, the viscosity change of an ER fluid is reversible with and without an applied electric field^[1]. In view of this reversible and quick response to external electric field, ER fluid has attracted much interest in the application in various mechanical devices, such as clutches, valves, damping devices, and so on^[1-3]. However, ER fluid has not been used wide because it does not have a high enough ER effect which is suited to the requirements of most of the applications. In order to get high active ER material, various types of ER materials, including ER materials doping the rare earth elements such as La, Ce, Pr, Y, Er, Gd based on inorganic compounds^[4-15] or organic polymer^[16-18], have been synthesized and studied. The majority of the inorganic compound materials have better ER performance. Yin and Zhao have obtained a cerium-doped TiO₂ ER material that has very high ER activity^[4]. However, up until now, an ER material based on titanium compound doping a kind of terbium complexes has not been studied.

Fluorescent material, particularly fluorescent rare earth complexes are of great interest owing to their broad applications in biochemistry, material chemistry, medicine and so on^[19]. However, the materials with both ER performance and luminescence property have not been reported except our research^[20,21]. In order to make a material have more broad application, which not only has better ER performance, but also has better luminescence property, we selected titanium compound as the principal component of our materials, and synthesized ER material with luminescence property. The composition, ER property and luminescence property of the material were studied in this paper.

1 Experimental

1.1 Preparation of TiTbSSA material

The TiTbSSA material was synthesized through the following process. First, a complex solution was prepared by mixing fully the ethanol solutions of Tb(NO₃)₃·6H₂O and sulphosalicylic acid (SSA), and then to add dropwise the complex solution into ethanol solution containing tetrabutyl titanate under vigorous agitation. The molar ratio of Tb(NO₃)₃·6H₂O, SSA and tetrabutyl titanate was 1:3:3. The sediment was separated and washed with absolute ethanol, then dried at 80 °C for 48 h. The yellow product was ground and finally dried in vacuum for 72 h at 50 °C. The material containing Tb(C₇H₅O₆S)₃ and TiO(OH)₂ was thus obtained.

Foundation item: Project supported by National Natural Science Foundation of China (10704041, 90922033, 21071008) and the National Basic Research Program of China (2013CB933401, 2010CB934601)

^{*} Corresponding author: LI Junran (E-mail: lijunran@pku.edu.cn; Tel.: +86-10-62753517) DOI: 10.1016/S1002-0721(14)60177-0

The dried particle material was mixed as quickly as possible with dimethyl silicone oil (density ρ =0.98 g/cm³ and viscosity η =98 mPa·s at 25 °C) under stirring, producing the ER fluid (suspension, 30 wt.%) sample. The ER fluid was then put in the gap between the cylinders of the apparatus as soon as possible for the ER measurement.

1.2 Characterization of material

The ER experiments were performed on a rotary viscometer (Type Physica McR501s, Anton Paar of Germany). In this study, the sample shear stresses and viscosities have been determined under different electric field strengths (E, dc field) at a given temperature (20 °C) and a shear rate (y) range of 0–300 s⁻¹. Infrared spectra (IR) of the materials were recorded with a Nicolet Magna-IR 750 spectrometer at 295 K. X-ray diffraction (XRD) analyses of the materials were fulfilled on a Bruker D8 ADVANCE X-ray diffractometer with Cu Ka radiation at a wavelength 0.15406 nm in a range of 10°-60°. The elemental analysis of the material was carried out using a German Elementar Vario EL instrument. The excitation and emission spectra of sample were determined on an Edinburgh FLS920 lifetime and steady state spectrometer. Excitation source is a Xe lamp with power of 450 W. The fluorescence lifetime of sample was obtained by using a microsecond flash lamp at a proper excitation wavelength and a frequency of 100 Hz. The micrograph of the material was obtained using a Desk scanning electron microscope (SEM) (Type PhenomproX, Phenom-World).

2 Results and discussion

2.1 Composition of material

The result from the elemental analysis of TiTbSSA material shows that the contents (wt.%) of carbon, nitrogen and hydrogen are C: 20.05, N: 0.78, H: 3.29, respectively, which is consistent with the composition $\left[\frac{1}{2}\text{Tb}(C_7\text{H}_5\text{O}_6\text{S})_3\right]\cdot[\text{TiO}(\text{OH})_2]\cdot2\text{H}_2\text{O}\cdot0.2\text{HNO}_3$, of which

the calculation contents (wt.%) are C: 20.11, N: 0.68 and H:2.71, respectively. The result mentioned above shows that the reaction scheme that corresponds to the synthetic procedure of the material is as follows:

$$Ti(C_{4}H_{9}O)_{4}+4H_{2}O=Ti(OH)_{4}+4C_{4}H_{9}OH$$

$$Ti(OH)_{4}=TiO(OH)_{2}+H_{2}O$$

$$3C_{7}H_{6}SO_{6}\cdot 2H_{2}O+Tb(NO_{3})_{3}\cdot 6H_{2}O=Tb(C_{7}H_{5}O_{6}S)_{3}+$$

$$3HNO_{3}+12H_{2}O$$

$$TiO(OH)_{2}+\frac{1}{3}Tb(C_{7}H_{5}O_{6}S)_{3}+0.2HNO_{3}+2H_{2}O=$$

$$1$$

$$\left[\frac{1}{3} \text{Tb}(\text{C}_7\text{H}_5\text{O}_6\text{S})_3\right] \cdot \left[\text{TiO}(\text{OH})_2\right] \cdot 2\text{H}_2\text{O} \cdot 0.2\text{HNO}_3$$

The result from elemental analysis indicates that there are water and nitric acid molecules in TiTbSSA material. Due to strong hygroscopicity of sulphosalicylic acid and rare earth nitrate, there are water molecules in the chemical formulae $[C_7H_6O_6S\cdot 2H_2O$ and $Tb(NO_3)_3\cdot 6H_2O]$ of sulphosalicylic acid and terbium nitrate as the original materials because the water molecules strongly bind with $C_7H_6SO_6$ or $Tb(NO_3)_3$ molecule by hydrogen bonds. Therefore, in the resultants of the coordination reactions of sulphosalicylic acid with rare earth nitrate, there are not only rare earth complex and HNO₃ molecules, but also H₂O molecules. Consequently, the HNO₃ and H₂O molecules, which have not been eliminated fully in our experimental conditions, appear in the TiTbSSA material due to the hydrogen bond action.

Fig. 1(a) illustrates IR spectra of TiTbSSA, SSA and Tb(NO₃)₃ in a range of 650–4000 cm⁻¹. Comparing with SSA, characteristic peak of –COOH group at 1668 cm⁻¹ disappears in IR spectra of TiTbSSA, and the characteristic peaks of –COO⁻ group at 1568 and 1473 cm⁻¹ appear in IR spectra of TiTbSSA^[22], which should be the result that the carboxyl group of SSA is coordinated to the Tb³⁺ ion with forming the rare earth complex by the reaction between SSA and the rare earth nitrate. To compare IR spectra of TiTbSSA with that of Tb(NO₃)₃, the characteristic peak of NO₃⁻ at 1383 cm⁻¹ appears in IR spectra of TiTbSSA material, which is consistent with the result of the elemental analysis. The pres-



Fig. 1 IR spectra of TiTbSSA, SSA and Tb(NO₃)₃

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