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Synthesis of hydrophobic zinc borate nanodiscs for lubrication

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Abstract

The crystal and hydrophobic zinc borate $(Zn_2B_6O_{11}*3H_2O)$ nanodiscs were successfully prepared by a wet method using Na₂B₄O₇*10H₂O and ZnSO₄*7H₂O as raw materials in situ aqueous solution, and oleic acid as the modifying agent. The microstructures and morphology of the asobtained samples were studied by X-ray diffraction (XRD), infrared spectra (IR), scanning electron microscopy (SEM) equipped with an energydispersive X-ray spectrometer (EDS) and thermogravimetric analysis (TGA). The measurement of the active ratio indicated that $Zn_2B_6O_{11}*3H_2O$ samples were hydrophobic. It had been found that the as-prepared materials displayed nanodisc morphology with average diameters from 100 to 500 nm and the thicknesses about 30 nm. Moreover, the friction coefficient of the base oil was decreased by the addition of hydrophobic zinc borate nanodiscs.

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Keywords: Zinc borate; Nanodiscs; Hydrophobe; Lubrication; Friction coefficient; Synthesis

1. Introduction

As borate salts, zinc borate has been the subject of significant research for applications including a polymer additive which serves as a fire retardant synergist, char promoter, anti-arcing agent, a preservative in wood composites, smoke and afterglow suppressant additive [1-15], and optical properties [16-23], and wear resistance [24].

Previous work about the synthesis of zinc borate included by reaction of zinc oxide and boric acid [25], and reactions of zinc salts and borate salts in hot water (≥ 60 °C) or using the ethanol supercritical fluid drying technique [26]. Important attributes of zinc borate include relatively low water solubility and a relatively high dehydration onset temperature [24]. The latter property permits processing in a wide range of polymer system. But zinc borate particles are hardly dispersed in a polymer matrix so that they prevent their uses in industry. To the best of our knowledge, studies of the preparation of zinc borate with nanostructures and the hydrophobic properties have been relatively

* Corresponding author. Tel./fax: +86 431 8499134. E-mail address: wangzc@mail.jlu.edu.cn (Z. Wang). few. Herein we report the synthesis of active zinc borate nanodiscs, which requires neither sophisticated techniques nor catalysts. Surface modification of zinc borate with hydrophobic properties would lead to a great expansion of its applications.

2. Experimental part

2.1. Materials

All the reagents used in our experiments were of analytical grade and were employed without any further purification and treatment. Distilled water was applied for all synthesis and treatment processes.

Table 1

Characteristics of synthesized $Zn_2B_6O_{11}\mbox{-}3H_2O$ powder at the different conditions

Sample no.	Reaction temperature (°C)	pН	The weight ratio of OA/ $Zn_2B_6O_{11}$ •7H ₂ O (wt.%)	Active ratio %
S1	70	5.8	0	0
S2	70	5.8	0.5	60.1
S3	70	5.8	1.0	99.9
S4	70	7.0	1.0	11
S5	70	8.0	1.0	0

⁰¹⁶⁷⁻⁵⁷⁷X/\$ - see front matter ${\ensuremath{\mathbb C}}$ 2006 Elsevier B.V. All rights reserved. doi:10.1016/j.matlet.2006.01.108



Fig. 1. XRD of: (a) pure $Zn_2B_6O_{11}$ • $3H_2O$ powders (sample S1, Table 1) and (b) hydrophobic $Zn_2B_6O_{11}$ • $3H_2O$ nanodiscs (sample S3, Table 1).

2.2. Synthesis of samples

In a typical procedure, a 500 mL three-neck round-bottom flask equipped with a thermometer, reflux condenser, and mechanical stirrer was charged with 100 mL of 0.1 mol dm⁻³ Na₂B₄O₇•10H₂O aqueous solution, 30.0 mL of absolute ethanol and a certain amount of oleic acid (OA) heated at 70 °C [26] and 20.0 mL of 1 mol dm⁻³ ZnSO₄•7H₂O aqueous solution was added dropwise to the first solution while being stirred for a period of about 0.5 h. After addition was complete, the mixture (pH=5.8) was continuously heated for about 6.5 h. The Different synthetic conditions and physical and chemical characteristics are shown in Table 1. The final mixture was then filtered, washed repeatedly with distilled water and absolute ethanol to remove unreacted reactants, oleic acid and by-products, and dried in the oven at 80 °C to obtain the final white zinc borate powders.



Fig. 3. The infrared spectra of (a) pure oleic acid, (b) pure zinc borate particles (Sample S1, Table 1), (c) hydrophobic zinc borate nanodiscs (sample S3, Table 1).

2.3. Characterization of the samples

The crystallinity and composition of as-prepared composites was characterized by X-ray power diffraction (XRD) (SHI-MADZU XRD-6000 diffractometer employing Ni-filtered Cu K_{α} radiation, at a scanning rate of 6°/min with 2 θ ranging from 20° to 45°) and an energy-dispersive X-ray spectrometer (EDS) (JEOL-6700F, Hitachi) attached to the SEM, respectively.

The morphology and size of the samples were observed using a Hitachi H-800 transmission electron microscope (TEM), at an accelerator voltage of 200kV and a Hitachi scanning electron microscope (SEM), a field-emission-scanning electron microscope (JEOL JSM-6700F, 3.0kV). The samples used for SEM and TEM characterization were dispersed in absolute ethanol and were ultrasonicated before observation.



Fig. 2. Typical EDS spectrum of the as-prepared sample (sample S1, Table 1).

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