

RESEARCH PAPER

Sono-chemical synthesis of Fe₃O₄ nanostructures and its application in acrylonitrile-butadiene-styrene polymeric nanocomposite

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ABSTRACT

Sono-chemical synthesis is a fast procedure for preparing monodisperse nanoparticles. Magnetite nanoparticles were prepared via a sono-chemical reaction at room temperature. Fe₃O₄ nanostructures were then added to acrylonitrile-butadiene-styrene (ABS) copolymer. Nanocomposites are very appealing due to the fact that a small amount of nanostructure can lead to a great improvement in many properties such as mechanical and thermal properties. Nanostructures were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and vibrating sample magnetometer (VSM). The XRD pattern of nanoparticles is indexed as a cubic phase (space group: Fd-3m). Fe₃O₄ nanostructures exhibit a super paramagnetic behavior with a saturation magnetization of 54 emu/g and a coercivity of 11 Oe at room temperature. The effect of ultrasonic power on the shape and particle size was investigated and the result showed that at 150W by increasing power, the growth stage overcame the nucleation stage and nanoparticles with bigger diameters were obtained. The flame retardancy behavior of ABS-Fe₃O₄ was studied by UL-94 analysis.

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INTRODUCTION

Sono-chemical synthesis is a fast procedure for fabricating mono-disperse nanoparticles. Magnetic nanoparticles and nanoadditives have gained significant attention in composite industries due to the fact that adding a small amount of nanostructure to a composite can lead to a great improvement in physical and chemical properties of the composite. The key advantages of polymeric nanocomposites in comparison to many metallic alloys are thermal insulation, low density, and corrosion resistance. However the main drawback of most polymeric compounds is their high degree of flammability. Therefore, increasing the thermal stability and flame retardancy of polymers is an

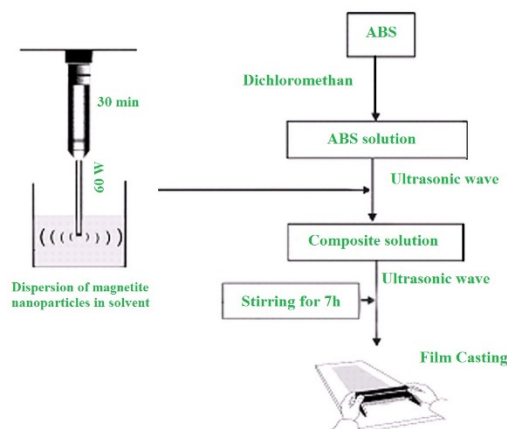
important subject for developing their applications. The conventional flame retardants (FR) that were used in polymeric matrices are toxic halogenated aromatic compounds, high loading of metal hydroxides, phosphorus-based compounds and layered silicates [1-4]. The higher level of flame retardancy of nanoparticles is because of their bigger surface to volume fractions which allow them to disperse into the polymeric matrix homogeneously, hence leading to the formation of a compact char during the combustion. Acrylonitrile-butadiene-styrene (ABS) is one of the most commonly-used polymers as an applicable industrial thermoplastic material due to its favorable characteristics including suitable mechanical properties, chemical resistance and processability. ABS is composed of

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Fig. 1. Preparation of ABS-Fe₃O₄ nanocomposite

a styrene-acrylonitrile copolymer matrix phase, with grafted butadiene chains. It has an appropriate balance between various physical properties such as toughness and chemical resistance of acrylonitrile, processability of styrene and impact resistance of butadiene [5–10]. However, this copolymer is flammable and is known as one of the most difficult polymers to be fire retardant. Nanocomposites are very attractive due to the fact that a small amount of nanostructure can lead to a considerable improvement in many properties such as mechanical and thermal properties [10–12].

In this work we have used the sono-chemical process to successfully synthesize Fe₃O₄ nanoparticles. Fe₃O₄ nanoparticles were then added to acrylonitrile-butadiene-styrene copolymer. The thermal stability and flame retardancy behavior of ABS filled with magnetite nanoparticles were investigated by UL-94.

EXPERIMENTAL

Materials and characterization

FeCl₃·6H₂O, FeCl₂·4H₂O and sodium hydroxide were purchased from Merck Company. All the chemicals were used as received, without further purification. X-ray diffraction (XRD) patterns were recorded by a Philips X-ray diffractometer using Ni-filtered CuK_α radiation. A multi-wave ultrasonic generator (Bandeline MS 73) equipped with a converter/transducer and titanium oscillator operating at 20 kHz, with a maximum power output of 150 W, was used for the ultrasonic irradiation. Scanning electron microscopy (SEM) images were obtained using a HITACHI instrument (Model

S-4160). Therefore, the samples were coated by a very thin layer of Au (BAL-TEC SCD 005 sputter coater) to make the samples' conducting surfaces obtain better contrast and prevent charge accumulation. In UL-94 a bar shape specimen of plastic 130 × 13 × 1.6 mm is positioned vertically and held from the top. A Bunsen burner flame is applied to the specimen twice (10 s each).

Synthesis of Fe₃O₄ nanoparticles

In a typical synthesis, 0.002 mol of FeCl₃·6H₂O and 0.001 mol of FeCl₂·4H₂O were dissolved in 100 ml distilled water. Under ultrasonic irradiation (50, 100 and 150W) sodium hydroxide (1M) was added to the solvent. The magnetite product was centrifuged, washed with alcohol and distilled water for several times, and dried in oven at 50 °C for 10 h.

Synthesis of ABS-Fe₃O₄ nanocomposite

4 g of ABS is dissolved in 15 ml dichloromethane solution. 1 g of Fe₃O₄ was dispersed in 10 ml dichloromethane solution with ultrasonic waves (30 min, 60W). The Fe₃O₄ dispersion was then slowly added to the polymer solution. After that, the new solution was mixed and stirred for 7 hours. In order to evaporate the solvent, the product was casted on a piece of glass template and was left for 24 hours (Fig 1).

RESULTS AND DISCUSSION

The XRD pattern of nanoparticles is shown in Fig. 2 and is indexed as a cubic phase (space group: Fd-3m). The experimental values are very close to

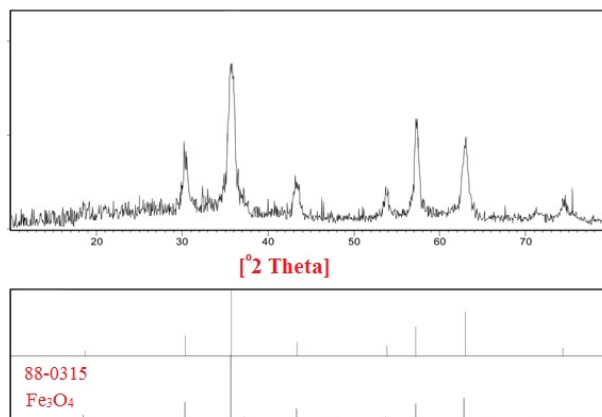


Fig. 2. XRD pattern of magnetite nanoparticles

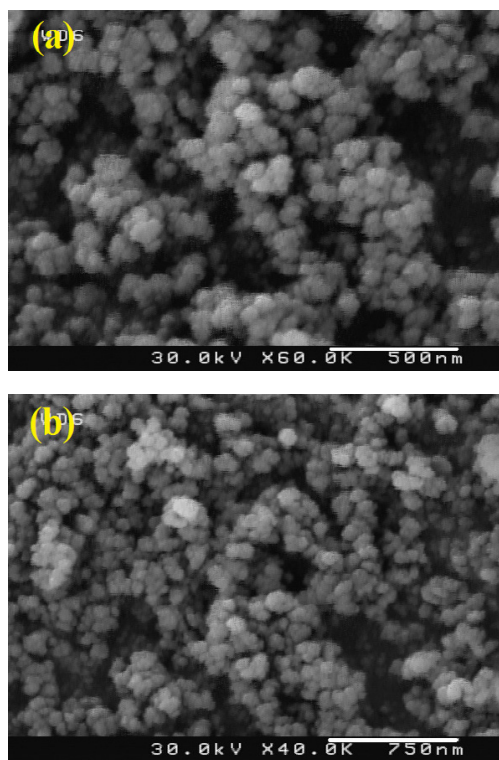


Fig. 3. SEM images of Fe₃O₄ synthesized at 50W

those of the literature (JCPDS No. 88-0315).

The crystallite size measurements were carried out using the Scherrer equation,

$$D_c = 0.9\lambda / \beta \cos\theta \quad (1)$$

β is the width at half maximum intensity of the observed diffraction peak, and λ is the X-ray wavelength (CuK _{α} radiation, 0.154 nm). The calculated crystallite size is about 40 nm.

The effect of ultrasonic power on the shape and particle size is shown in Fig 3. Nanoparticles at power 50W were obtained with the particle size of less than 30 nm.

Sonication and power effect was investigated by SEM image in Fig. 4. At 100W nanoparticles with the average diameter of less than 70 nm were prepared. The influence of higher power on the morphology of the product is shown in Fig. 5 in two different magnifications. At 150W by increasing

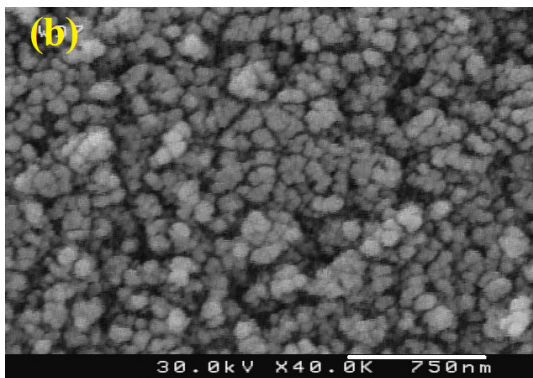
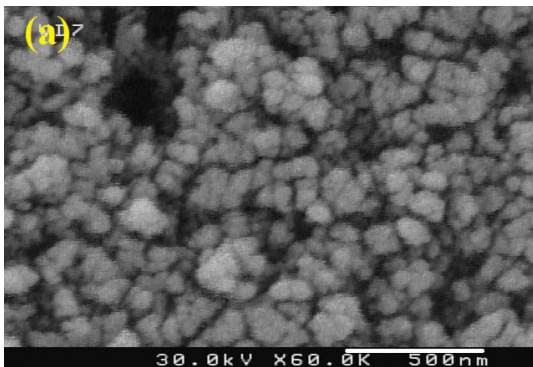


Fig. 4. SEM images of magnetite nanoparticles prepared at 100W

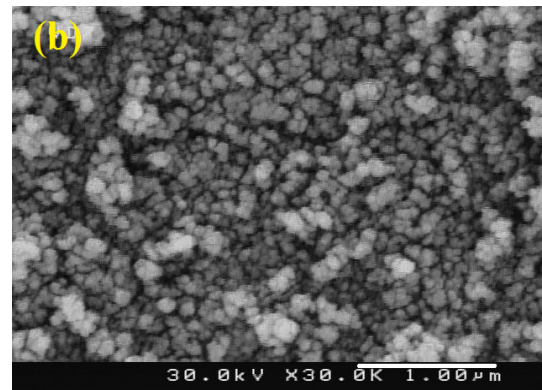
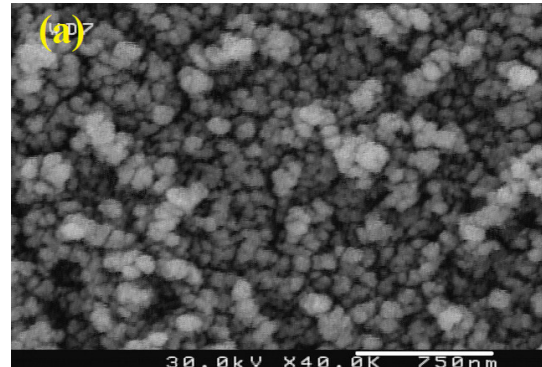


Fig. 5. SEM images of magnetite synthesized at 150W

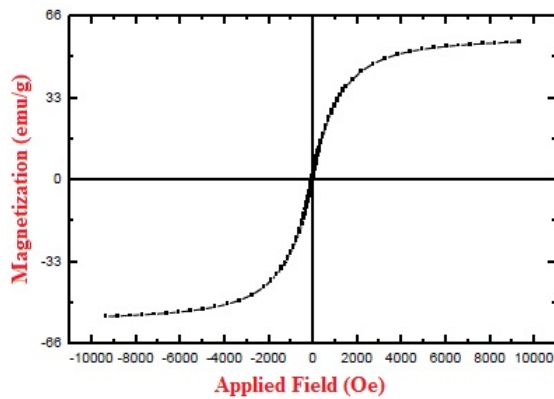


Fig. 6. Hysteresis loop of ABS-Fe₃O₄ nanocomposite

power, the growth stage overcame the nucleation stage and nanoparticles with larger diameters were obtained.

Hysteresis loop for ABS-Fe₃O₄ nanocomposite is represented in Fig. 6. Fe₃O₄ nanostructures exhibited a super paramagnetic behavior with a saturation magnetization of 54 emu/g and a coercivity of 11 Oe at room temperature.

Fe₃O₄ has a barrier effect to slow down the product volatilization and thermal transport

during decomposition of the polymer. Therefore, the residual amount of nanocomposites is higher than the pure polymer. It can be used as a char that prevent propagation of flame.

The effect of Fe₃O₄ nanostructure on the flame-retardant properties has been considered using UL-94 test. A V-0 classification is given to a material that is extinguished in less than 10 s after any flame application; drips of particles are allowed as long as they are not inflamed. A V-1 classification is

received by a sample with maximum combustion time lower than 30 s; drips of particles are allowed as long as they are not inflamed

The sample is classified as V-2 if it satisfies the combustion time criteria of V-1, but flaming drips are allowed. Materials are ranked as N.C. in UL-94 tests when the maximum total flaming time is above 50 s. The results of UL-94 test for both ABS and ABS-Fe₃O₄ nanocomposites is N.C. The results, according to this test, demonstrate that the Fe₃O₄ nanostructures can somewhat enhance the flame-retardant property of the ABS matrix. The flame-retardant property of the ABS-Fe₃O₄ nanocomposites was acceptable in comparison with other works [1, 7, 10, 12].

CONCLUSION

Magnetite nanostructures were synthesized via a sono-chemical reaction between FeCl₃.6H₂O and FeCl₂.4H₂O. Prepared nanoparticles were dispersed in ABS matrix appropriately. The influence of the inorganic phase on the thermal stability properties and flame retardancy of ABS matrix was studied.

CONFLICTS OF INTEREST

The authors announce that there are no conflicts of interest.

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