

Thermal and Magnetic Characteristics of Cellulose Acetate- Fe_3O_4

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Abstract

Fe_3O_4 nanoparticles were synthesized via a simple chemical reaction between $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ under nitrogen atmosphere at room temperature, and then nanoparticles were added to cellulose acetate (CA) polymer. The influence of nanoparticles on the thermal properties of CA polymeric matrix was studied using thermogravimetric analysis (TGA). Nanostructures were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and Fourier transform infrared (FT-IR) spectroscopy. The magnetic properties of the samples were also investigated using an alternating gradient force magnetometer (AGFM). We found that the Fe_3O_4 nanoparticles exhibit a ferromagnetic behavior with a saturation magnetization of 59 emu/g and a coercivity of 105 Oe at room temperature.

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1. Introduction

The enormous use of polymeric materials is attributed to their extraordinary properties such as low weight and ease of processing. However for improvement of some properties like thermal and mechanical stability, large numbers of additives were added to the polymeric matrix and formed

polymer matrix composite. In the most polymeric composite, one phase is usually continuous and called the matrix, while the other phase called the dispersed phase. Polymeric composite can be processed at a much lower temperature compared to other composite. Scientists for improvement the properties of composite materials investigated composites with lower fillers size, leading to the

development of nanocomposites. Nanocomposite refers to a composite which one phase has nanoscale morphology. Together with numerous advantages that polymeric materials provide to society in everyday life, there is one obvious disadvantage related to their high flammability. Therefore, improving the thermal behaviour of polymers is a major challenge for extending their use for most applications. Various flame retardant additives, such as halogenated compounds are limited with respect to the environmental requirements. Cellulose acetate is a well known derivative of cellulose and is widely used in a broad field of applications such as adhesive, film base in photography, synthetic fibre and separation processes (filtering, reverse osmosis) [1–5]. CA is produced either by heterogeneous or homogeneous acetylation of cellulose. In this work first Fe_3O_4 nanoparticles were synthesized under nitrogen atmosphere, and then the nanoparticles were incorporated in CA polymer in order to obtain thermal stable and magnetic nanocomposite.

2. Experimental

2.1. Materials and characterization

All the chemicals were of analytical grade and were used as received without further purification. Cellulose acetate (MW: 100000) was purchased from Acros company. XRD pattern was recorded by a Philips, X-ray diffractometer using Ni-filtered Cu K_α radiation. SEM images were obtained on LEO instrument. A Galaxy series FTIR5000 spectrometer for FTIR tests were used. TGA was carried out using an instrument (Shimadzu TGA-50H) with a flow rate of 20.0 mL min^{-1} and a heating rate of $10 \text{ }^\circ\text{C min}^{-1}$ under nitrogen atmosphere. Room temperature magnetic properties

were performed using a Meghnatic Daghigh Kavir Company AGFM device in an applied magnetic field sweeping between $\pm 8000 \text{ Oe}$.

2.2. Synthesis of Fe_3O_4 nanoparticles

In a typical procedure, $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ (0.02 mol) $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.04 mol) were dissolved in 100 ml distilled water under nitrogen atmosphere. Then 12 ml of NH_3 (3M) was added to the solution under ultrasonic waves at room temperature. After 30 min stirring, the black precipitate was formed. The precipitate was centrifuged, washed with distilled water and absolute ethanol. The black solid were dried in a vacuum oven at $50 \text{ }^\circ\text{C}$ for 4 h and stored for further investigation.

2.3. Synthesis of CA- Fe_3O_4 nanocomposite

CA (0.9 g) was dissolved in 15 ml of acetone. Fe_3O_4 nanoparticles (0.1 g) were dispersed in 15 ml of acetone with ultrasonic waves. Then the dispersion of magnetic nanoparticle was added slowly to the polymer solution. The solution was mixed under stirring for 5 h. After stirring, the product was casted on a glass plate and after solvent evaporation; the nanocomposite was placed in the vacuum oven for another 4 h for the removal of residual traces of solvent.

3. Results and discussion

The XRD pattern of as-synthesized CA/ Fe_3O_4 nanocomposite is shown in Fig. 1. The XRD pattern of the as-prepared iron oxide is indexed as a pure cubic Fe_3O_4 structure; in agreement with the literature value (JCPDS card No. 03–0863). The peaks of Fe_3O_4 in the XRD pattern of

nanocomposite confirm existence of the nanoparticles in the polymeric matrix.

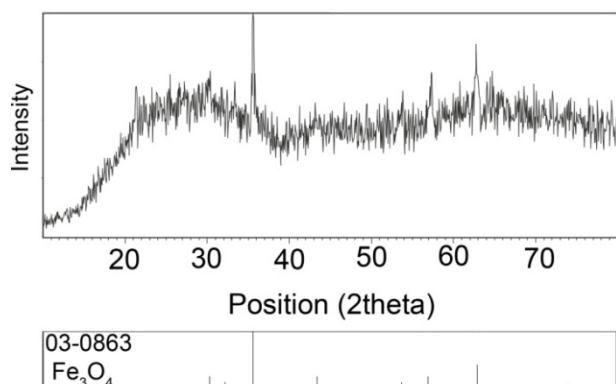


Fig. 1. XRD pattern of CA/Fe₃O₄ nanocomposite.

SEM images of Fe₃O₄ nanoparticles and CA/Fe₃O₄ nanocomposite are shown in Fig. 2a and Fig.2b respectively. Fig.2b shows that Fe₃O₄ nanostructures obviously are dispersed in the polymeric matrix.

The IR spectrum of the Fe₃O₄ is shown in Fig. 3a. Fig. 3b is FT-IR spectrum of CA/Fe₃O₄. An absorption peak occurs at 1755 cm⁻¹ that is related to the C=O bond in CA. Absorptions at 1039 and 1162cm⁻¹ are attributed to the C-O bonds in CA polymer. Also, the absorptions at 3487cm⁻¹ are referred to the stretching vibration of O-H bond. The absorption at 430 cm⁻¹ at nanocomposite confirms the existence of Fe₃O₄ nanoparticles in the polymeric matrix.

The TGA curves of pure CA and CA/Fe₃O₄ nanocomposite in N₂ atmosphere are illustrated in Fig.4a and 4b respectively. The thermal decomposition of CA/Fe₃O₄ nanocomposite shifts towards higher temperatures in the presence of Fe₃O₄ nanostructures. Therefore polymer chains well adsorb on the surface of the Fe₃O₄ nanostructures.

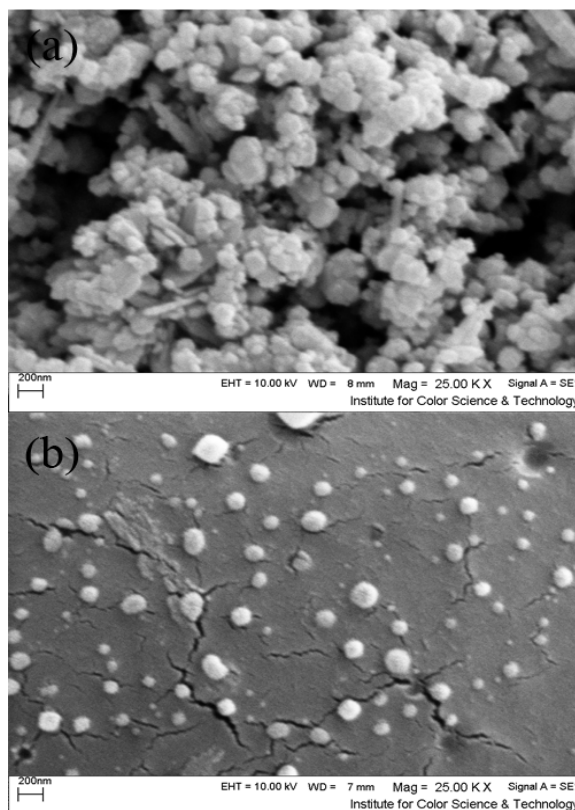


Fig. 2. SEM images of (a) Fe₃O₄ and (b) CA-Fe₃O₄.

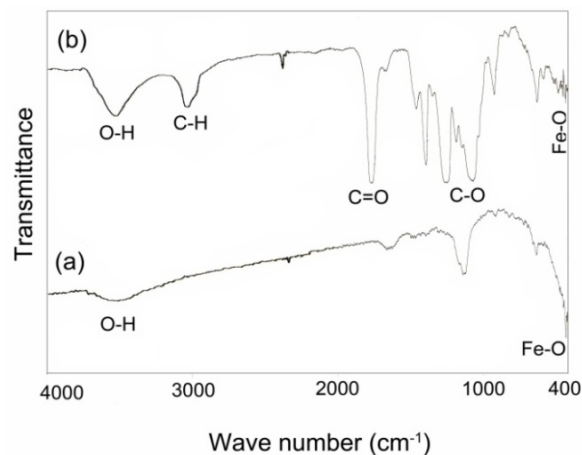


Fig. 3. FT-IR spectrum of (a) Fe₃O₄ and (b) CA/Fe₃O₄ nanocomposite.

Adsorption of polymer chains on the surface of nanostructures results in restriction of segmental mobility and serves to suppress redistribution and chain transfer reactions. Also, Fe₃O₄ has barrier effect to slow down mass transport (volatilization)

and thermal transport during decomposition of the polymer.

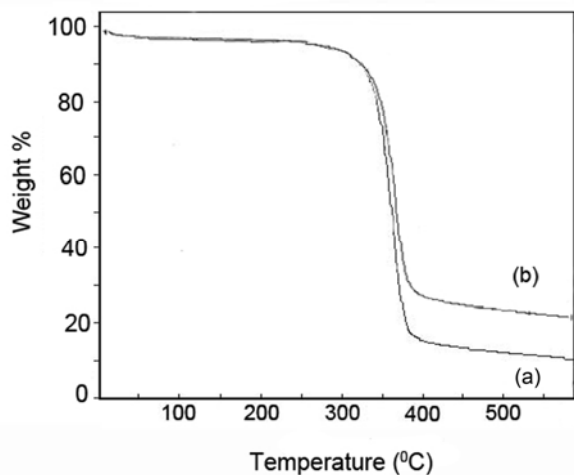


Fig. 4. TGA spectrum of (a) pure CA and (b) CA/Fe₃O₄ nanocomposite.

Magnetic properties are strongly influenced by many parameters, including crystallinity, size, shape and crystal defects. Room temperature magnetic properties of our samples are studied using an AGFM device. Hysteresis loops for Fe₃O₄ nanoparticles and CA/Fe₃O₄ (10%) nanocomposite are depicted in Fig. 5a and 5b respectively. The hysteresis loops of the magnetic nanoparticles and nanocomposites show ferromagnetic behavior. Clearly, the saturation magnetization of Fe₃O₄ nanoparticles is much higher than which is obtained for CA/Fe₃O₄ nanocomposite. The coercivity of CA/Fe₃O₄ (10%) nanocomposites is also higher than which of Fe₃O₄ nanoparticles. This result can be attributed to the interactions between polymer chains of CA and Fe₃O₄ nanoparticles.

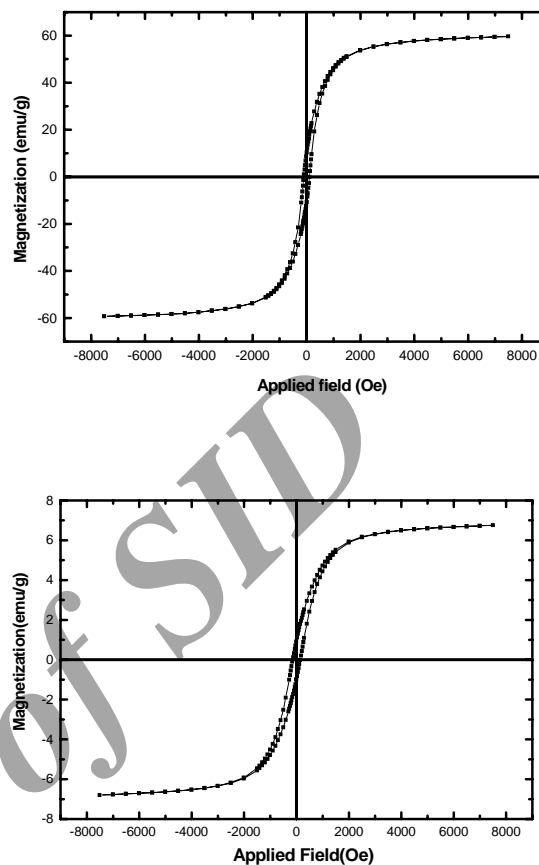


Fig. 5. Room temperature magnetization curves of (a) Fe₃O₄ nanoparticles (b) CA/Fe₃O₄ (10%) nanocomposite.

4. Conclusion

Magnetic nanoparticles were synthesized via a simple reaction under ultrasonic waves. Nanoparticles were then added to CA polymer. The influence of magnetic nanoparticles on the thermal properties of CA matrix was studied using thermogravimetric analysis. The thermal decomposition of the nanocomposite shifted towards higher temperature in the presence of the Fe₃O₄ nanoparticles. Adsorption of polymer chains on the surface of Fe₃O₄ nanoparticles results in to slow down product volatilization and thermal transport during combustion of the polymer. We found that the as-obtained Fe₃O₄ nanoparticles exhibit a

ferromagnetic behavior with a saturation magnetization of 59 emu/g and a coercivity of 105 Oe at room temperature.

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