

## The synthesis and properties of Fe<sub>3</sub>O<sub>4</sub>/ Sodium acetate / CMS ternary nanocomposites as electrorheological fluid

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**Abstract:** In this paper have been studied a ternary nanocomposite as rheological fluid based on Carboxymethyl starch and sodium acetate with nano metallic oxides. These nano composite was prepared by the two – step composite method .Firstly, the polar sodium acetate compound was directly intercalated into the interlayer of nano ferric oxide and then the intercalated complex was composite with CMS by the solution method. The composites, thus synthesized have been characterized by Fourier transfer infrared (FTIR) spectrophotometer and X-ray diffraction. The morphology of these composites was studied by scanning electron microscopy. [The Journal Of American Science. 2007;3(4):30-34]. (ISSN: 1545-1003).

**Keywords:** electrorheological fluid, ternary system, nanocomposite, CMS,

### Introduction

Electrorheological materials are typical suspensions composed of micrometer size particles and dielectric liquids[1]. The material parameters of these nanocomposites such as viscosity, yield stress and shear modulus can change obviously and reversibly upon the application of an external electric field[2]. These characteristics find practical applications in many fields[3,4].

Fe<sub>3</sub>O<sub>4</sub> is a mineral as nano particles (10-12 nm). This material is as guest species in sodium acetate (CH<sub>3</sub>COONa) layers. Only a limited number of polar guest species, such as N-methylformamide (NMF) and dimethylsulfoxide (DMSO), can directly be intercalated[5,6]. Because of small particle size and intercalation properties, Fe<sub>3</sub>O<sub>4</sub> /organic nanocomposites exhibit novel hybrid synergetic properties derived from two components[7]. Nanocomposites have become the subject of considerable interests for the design of high performance engineering materials with enhanced stiffness, strength, two dimensional stability, mechanic, magnetic, thermal, electric and optical characteristics. The choice of intercalation Fe<sub>3</sub>O<sub>4</sub> with CH<sub>3</sub>COONa is aimed at modifying the dielectric and polarization properties of Fe<sub>3</sub>O<sub>4</sub>, so as to improve its electrorheological activity, reduce cost and attain the high cost performance[8,9]. As opposed to starch, CMS is a cold water-soluble starch that can form a transparent solution in water. CMS, as an anionic polyelectrolyte[10].

In this study, The Fe<sub>3</sub>O<sub>4</sub>/ CH<sub>3</sub>COONa/CMS nanocomposite is fabricated according to the physical and chemical design of the electrorheological material. The polar compound (CH<sub>3</sub>COONa) is directly intercalated into the interlayer of ferric oxide, and then the intercalated complex is interacted with CMS by solution method[11]. The dielectric and conductivity properties of ternary nanocomposite are improved enormously, which results in the strength of the particle polarization and a large enhancement of the electrorheological effect[12].

The experiment results show that by the design and control of the molecular chemical structure, the physical design for dielectric properties is achieved and thus the characterization of ternary nanocomposite is optimized[13].

### Experimental Materials

Nano particles of Fe<sub>3</sub>O<sub>4</sub> were purchased from nanotechnology center of Baku State University. The particles have an average of 10-12 nm. The sodium acetate sample employed in this work was obtained from Merck Chemical Company and used to prepare the nanocomposites without further purification. The cornstarch, sodium hydroxide and chloroacetic acid was purchased from Merck Company.

### **Instruments**

The images of nanoparticles were investigated using Philips XL30 scanning electron microscope. The Fourier transfer infrared (FTIR, Bruker) spectroscopy was used to identify the polymer on the Fe<sub>3</sub>O<sub>4</sub> nano particles surface. Spectra were obtained in the wave number range of 400-4000 cm<sup>-1</sup>. Spectra of the nanocomposite were recorded from KBr in 1:10 (wt/wt) ratio. Powder X-ray diffraction (XRD) patterns were obtained with a Phillips diffractometer using CuK $\alpha$  radiation with a scanning rate of 2 min<sup>-1</sup>.

### **Preparation of carboxymethyl starch (CMS).**

Firstly, the 0.5 g cornstarch and 120 ml 2-propanol were placed in a 500 ml vessel and stirred for 2 h. The 5 g sodium hydroxide was added and reacted for 1 h at 78-80°C. After that, the 10 g chloroacetic acid was added to the vessel and stirred for another 2 h at 50°C. The product was filtered and washed several times with ethanol, then dried under vacuum. The resulting carboxymethyl starch (CMS) was crushed in a mortar [degree of substitution (DS) = 0.49][14].

### **Preparation of Fe<sub>3</sub>O<sub>4</sub>/ sodium acetate intercalate**

Fe<sub>3</sub>O<sub>4</sub> (3 g) was dispersed in 40 ml ethanol and stirred for 3 h. Then 2.25 g sodium acetate (mass ratio Fe<sub>3</sub>O<sub>4</sub>: CH<sub>3</sub>COONa is 1: 0.75) were added drop by drop into the Fe<sub>3</sub>O<sub>4</sub> suspension. When sodium acetate solution was dropped, the temperature was increased to 50 °C for the purpose of evaporation of ethanol. The sample was sealed in a weighting bottle and placed in an oven for 14 h at 80 °C and the resulting material was got.

### **Preparation of Fe<sub>3</sub>O<sub>4</sub>/ sodium acetate/ carboxymethyl starch nanocomposite.**

1.8 g CMS and 50ml distilled water were mixed and stirred for 10 h in a 100 ml vessel, then the appropriate amount of Fe<sub>3</sub>O<sub>4</sub>/ sodium acetate intercalation was added slowly into the vessel and stirred for 12 h at room temperature. After approximately 180 min, the product was sprayed into a liquid nitrogen bath cooled down to 77° K, resulting in frozen droplets. These frozen droplets were then put into the chamber of the freeze-dryer. In the freeze-drying process, the products are dried by a sublimation of the water component in an iced solution. The ternary nanocomposite was crushed in a mortar.

## **Results and discussion**

### **Scanning electron micrography (SEM).**

SEM of Fe<sub>3</sub>O<sub>4</sub>/ CH<sub>3</sub>COONa/CMS nanocomposite synthesized by chemical oxidative is shown in Figure 1. Fe<sub>3</sub>O<sub>4</sub>/ CH<sub>3</sub>COONa/CMS nanocomposite is very sensitive to the temperature. Due to the interaction electron and sample. Scanning electron micrography images were obtained from a diluted solution of the nanocomposite particle. The white spots are Fe<sub>3</sub>O<sub>4</sub> and CH<sub>3</sub>COONa nano particles. The SEM image shows the presence of spherical Fe<sub>3</sub>O<sub>4</sub> particles in CMS matrix, which are homogeneously distributed throughout the composites, which is also confirmed from XRD studies.

### **X-ray diffraction**

The crystallinity of the formed composites was followed with X-Ray diffraction (XRD) as a function of weight percent inorganic component. Figure 2 shows X-ray diffraction pattern of Fe<sub>3</sub>O<sub>4</sub>/CH<sub>3</sub>COONa/CMS nanocomposite. Diffraction of Fe<sub>3</sub>O<sub>4</sub>/ CH<sub>3</sub>COONa/CMS nanocomposite have a very intense, sharp and narrow peak at about 2 $\theta$  = 8.35°, which is a characteristic peak of nanocomposite. We can observe that the X-ray diffraction pattern of the original Fe<sub>3</sub>O<sub>4</sub> modifies dramatically.

### **Fourier transfer Infrared spectra**

Figure 3-a shows the FT-IR spectrum of carboxymethyl starch (CMS), where the % of transmittance is plotted as a function of wave number (cm<sup>-1</sup>). The wide peak around 3411 cm<sup>-1</sup> is attributed to the O-H stretching vibrations of CMS. The peaks at 1597 and 1417 cm<sup>-1</sup> attribute to the COO<sup>-</sup> unsymmetrical and symmetrical stretching vibration respectively. The FT-IR spectra of the ternary

nanocomposite in Figure 3-b shows that those peaks associated with intercalation have a small change. But the peaks of the  $\text{COO}^-$  unsymmetrical and symmetrical stretching vibrations are moved to  $1580$  and  $1424$   $\text{cm}^{-1}$  respectively, and the results show that the  $\text{COO}^-$  groups of CMS have a strong interaction with the group of  $\text{Fe}_3\text{O}_4$ .

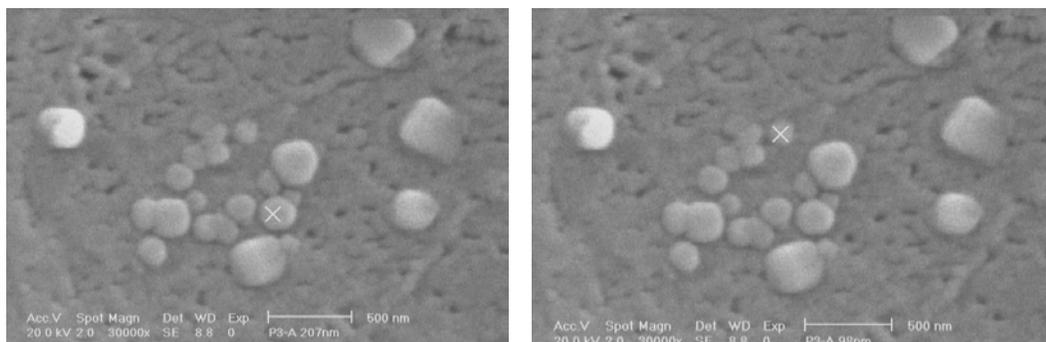


Figure 1. Scanning electron micrograph of  $\text{Fe}_3\text{O}_4$ / sodium acetate/ carboxymethyl starch ternary nanocomposite

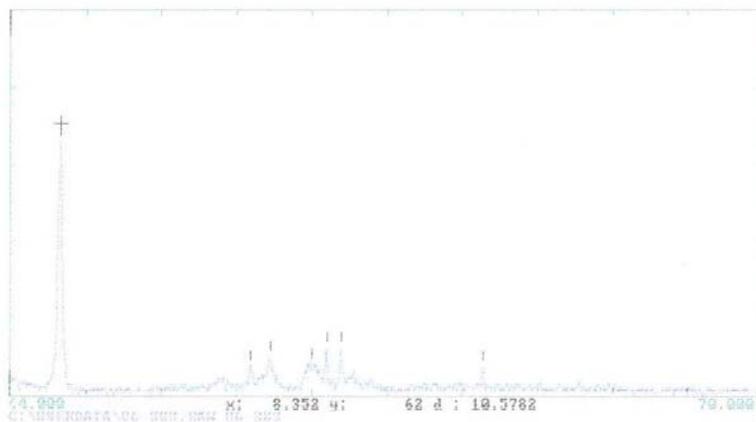


Figure 2. XRD spectra of  $\text{Fe}_3\text{O}_4$ / sodium acetate/ carboxymethyl starch ternary nanocomposite

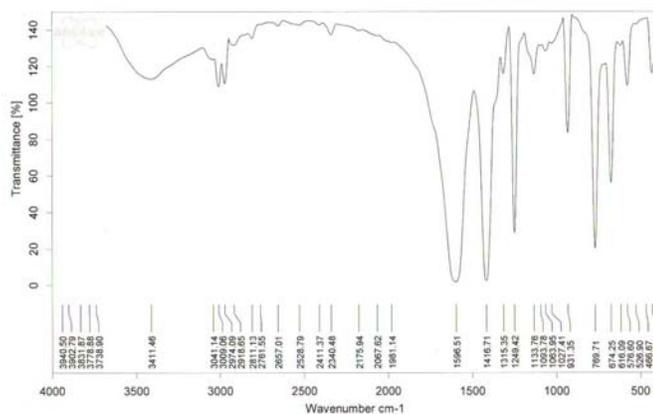


Figure 3-a. FT-IR spectra of pure carboxymethyl starch (CMS)

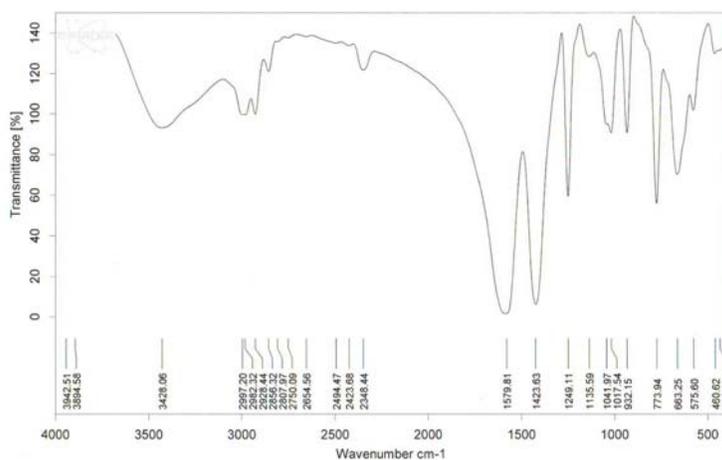


Figure 3-b. FT-IR spectra of  $\text{Fe}_3\text{O}_4$ / sodium acetate/ carboxymethyl starch ternary nanocomposite

### Conclusions

We synthesized the two – step nanocomposite by organic /inorganic materials. The results showed that the polar species such as sodium acetate intercalated in the interlayer of  $\text{Fe}_3\text{O}_4$  with different sodium acetate content. The  $\text{Fe}_3\text{O}_4$ /  $\text{CH}_3\text{COONa}$ /CMS nanocomposite is fabricated according to the physical and chemical design of the electrorheological material. The polar compound ( $\text{CH}_3\text{COONa}$ ) is directly intercalated into the interlayer of ferric oxide, and then the intercalated complex is interacted with CMS by solution method. The experiment results show that by the design and control of the molecular chemical structure, the physical design for dielectric properties is achieved and thus the characterization of ternary nanocomposite is optimized.

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