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One-pot synthesis and characterization of biopolymer – Iron oxide nanocomposite

A.Jegan [1]
A.Ramasubbu[2]
S.Saravanan[1]
S.Vasanthkumar[3,*]

[1]*Department of Chemistry, Sona College of Technology, Salem, India.*
[2]*Department of Chemistry, Government Arts College (Autonomous), Coimbatore, India*
[3]*Department of Chemistry and nanotechnology, Karunya University, Coimbatore, India*

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* Corresponding author:
S.Vasanthkumar
Department of Chemistry and nanotechnology, Karunya University, Coimbatore, India
Tel +91 422 2614484
Fax +91 422 2615615
Email kumar2359@yahoo.com

ABSTRACT

The magnetite (Fe_3O_4) – agar nanocomposite was prepared by co-precipitation of Fe (III) and Fe (II) ions for the first time. The obtained samples were characterized by x-ray diffraction, Fourier-transform infrared spectroscopy, scanning electron microscopy and transmission electron microscopy. FT-IR results confirm the formation of Fe_3O_4 nanoparticles in agar matrix. The XRD results revealed the presence of magnetite nanoparticles. The SEM results confirm that the magnetite nanoparticles are dispersed in the agar matrix. TEM micrograph clearly illustrates that the magnetite nanoparticles sizes varies from 50 – 200 nm.

Keywords: *Magnetite; Agar; Nanomaterials; X-ray diffraction*

INTRODUCTION

In recent times naturally available polymers based nanocomposites attract great attention by all researchers. This is because natural polymers are environmentally benign substances and also possess great potential to be developed for industrial and medical applications by themselves or in combination with other supplementary organic or inorganic compounds. The use of natural polymers has been extended to the nanotechnology and biomedical fields, due to its biocompatibility for in vivo applications, as well as its stabilization of nanostructures. Several authors have used natural polymers for surface modification of nanomaterials, preparation of nanoparticles and nanocomposites and also as stabilizing or capping agents [1-4]. Agar agar or agar is a gelatinous, nontoxic and biodegradable substance derived from marine algae. Agar is composed of Agaropectin and Agarose [5]. Agar dissolves in boiling water and when cooled it forms a gel between 32° and 43°C.

Agar is approximately 80% fiber, so it can serve as an intestinal regulator and as an impression material in dentistry [6]. In the food industry, agar can be used as a stabilizer and thickener. Magnetite (Fe_3O_4) nanoparticles are important material and they have been used in various applications, like magnetic storage media [7], biosensor applications [8], target - drug delivery [9], contrast agents in magnetic resonance imaging and hyperthermia treatment of cancers [10]. A wide variety of methods have been reported to synthesize Fe_3O_4 nanoparticles, including co-precipitation [11], sol gel-method [12], Flow injection synthesis [13], electrochemical methods [14], solvothermal synthesis [15], hydrothermal synthesis method [16], microwave-assisted synthesis [17] and thermal decomposition [18]. Magnetite (Fe_3O_4) biopolymer nanocomposites have been given much attention due to their unique properties and potential applications. Herein we report a one pot green and simple synthesis of agar – Iron oxide nanocomposite using coprecipitation method and the prepared agar iron oxide sample was characterized by FT-IR, XRD, SEM and TEM.

EXPERIMENTAL

Materials

Agar Agar was purchased from HiMedia (India), Ferric chloride hexahydrate ($FeCl_3 .6H_2O$), ferrous sulfate heptahydrate ($FeSO_4. 7H_2O$) and Sodium hydroxide were obtained from Qualigens, India. Agar was purified by washing with double distilled water repeatedly.

Synthesis of Agar – Iron oxide nanocomposite

About 1 g of purified agar was dissolved in 100 ml of distilled water under magnetic stirring at 95ºC. 100 ml mixture of 2:1 molar ratio Fe^{3+}/ Fe^{2+} solution was prepared by using 50 ml of 0.2 M ferric chloride hexahydrate ($FeCl_3 .6H_2O$) and 50 ml of 0.1 M ferrous sulfate heptahydrate ($FeSO_4. 7H_2O$).The iron solution was added drop wise in the Agar solution at 65ºC under magnetic stirring. During the reaction Agar solution pH was maintained at 9-10 using 0.1 M NaOH. The brown-black color of the iron solution changed to dark black, implying the synthesis of the magnetite nanoparticles. After completing the reaction, a black precipitate was obtained, this was washed repeatedly with distilled water. The obtained precipitate was dried at 40ºC in an Oven.

Characterization of Agar – Iron oxide nanocomposite

The obtained sample was prepared as a crushed powder weighing approximately 1mg and mixed together with 100 mg of crushed KBr. FTIR spectra (Perkin-Elmer Spectrometer) was taken between 400 and 4000 cm^{-1}. X-ray powder diffraction (XRD) data were collected using an X-ray diffractometer (Bruker, AXS) with Cu-Kα radiation (0.15406 nm).The surface morphologies of the particles were mapped by a scanning electron microscope (SEM) (model JEOL 63690). The particle size and morphology were examined using a transmission electron microscope (TEM, Philips EM). For TEM analysis, the powder sample was ultrasonically dispersed in distilled water to form a very dilute suspension, and then a few drops were deposited on the carbon-coated copper grids.

RESULTS AND DISCUSSION

FT-IR study of the nanocomposite

The prepared black precipitate was washed with distilled water repeatedly to remove the biopolymer. The purified composite was dried and examined using FT-IR spectroscopy. Figure 1 shows the FT-IR spectrum of the iron oxide agar nanocomposite. The absorption bands at 563 cm^{-1} were attributed to the vibrations of Fe–O functional group [19]. In addition, the bands located at 929 and 1021 cm^{-1} can be ascribed to 3, 6-Anhydro-galactose bridge vibration of agar [20]. Moreover, the other absorption bands at 1640 cm^{-1} and 1562 cm^{-1} correspond to amine function deformations vibrations and the peptide link vibrations respectively, revealing the presence of hexagonal Fe_3O_4 coated with agar [21]. The broad adsorption band at 3429 cm^{-1} can be attributed to O–H stretching of the polysaccharide.

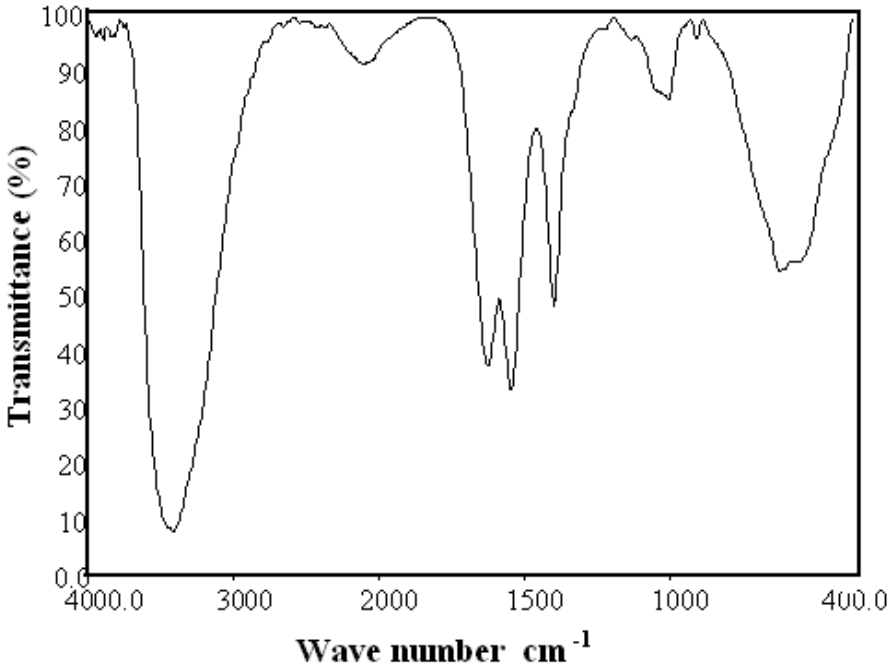

Fig.1. FT-IR spectrum of prepared Agar– Fe_3O_4 nanocomposite

X-ray diffraction

XRD patterns of agar-Fe_3O_4 nanocomposite are shown in Figure 2. The patterns of agar-Fe_3O_4 showed diffraction peaks at 2 Θ = 30.54, 36.00, 43.54, 53.82, 57.66 and 63.28 which can be indexed to (220), (311), (400), (422), (511) and (440) planes of magnetite respectively. The results are in good agreement with the previous reports [16, 17]. No other impurity related peaks were detected. The XRD result confirms the formation of magnetite nanoparticles.

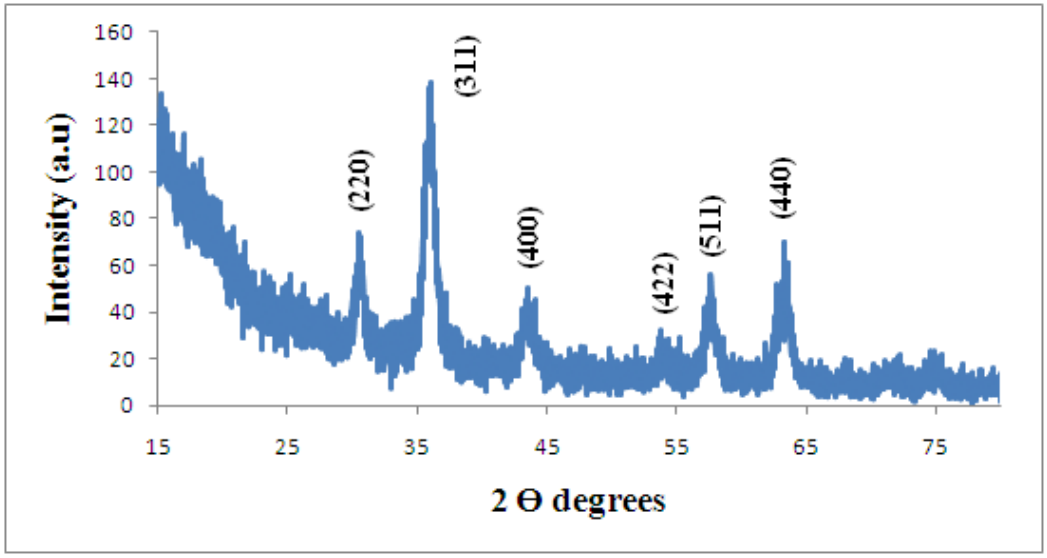

Fig.2. XRD pattern of the as prepared Agar– Fe_3O_4 nanocomposite

Scanning electron microscope

The morphology of the prepared sample was investigated by scanning electron microscope. Figure 3 (a), (b) and (c) represents a typical SEM image of the prepared agar – iron oxide nanocomposite at different magnifications. The micrographs of prepared composite clearly illustrates that the magnetite nanoparticles are formed in the agar matrix. The magnetite nanoparticles are well dispersed in the natural polymer and consist of hexagonal, cubic and spherical shape particles. The SEM images show that the particles size varies from 50 – 200 nm.

Transmission electron microscope

Additional structural characterization was carried out using TEM. Figure 4 shows the TEM image of synthesized magnetite – agar nanocomposite. From the TEM image, it can be noted that the composite mainly consists of spherically shaped nanoparticles, which are well dispersed in the agar matrix. The TEM images also confirm that the particle sizes varies from 50 – 200 nm.

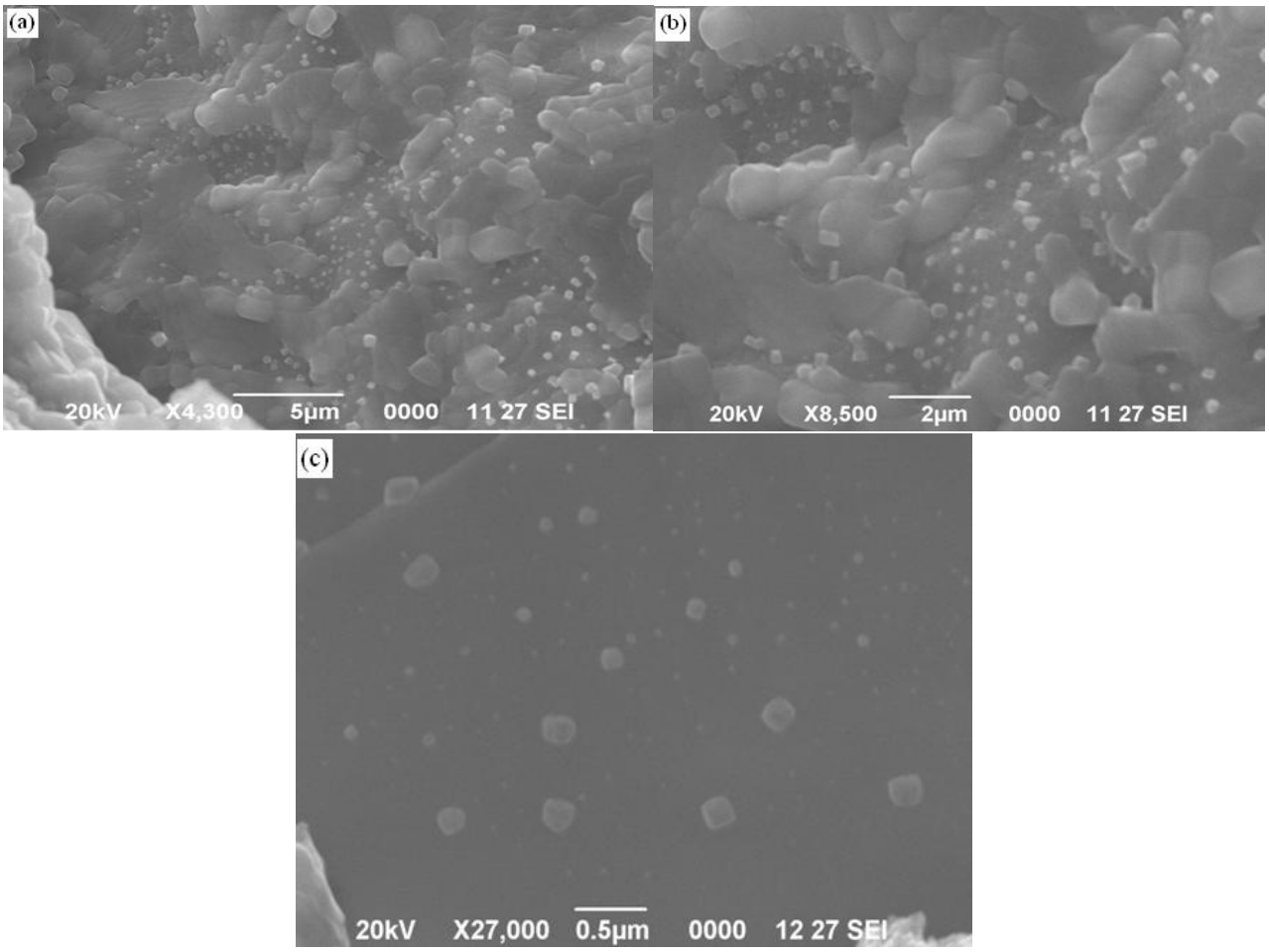

Fig.3. (a, b and c). SEM images of prepared Agar– Fe_3O_4 nanocomposite at different magnifications

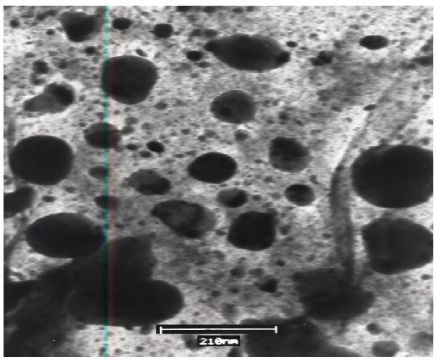

Fig.4. TEM image of prepared Agar– Fe_3O_4 nanocomposite

CONCLUSION

Successful synthesis of magnetite – agar nanocomposite by a simple co-precipitation method was accomplished for the first time. The FT-IR spectrum reveals that the magnetite nanoparticles are present in the organic substance. The XRD results clearly prove the formation of magnetite nanoparticles. The SEM and TEM images show the shape and size of the synthesized nanocomposite which varies from 50 – 200 nm.

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