

Synthesis of talc/ Fe_3O_4 magnetic nanocomposites using chemical co-precipitation method

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Abstract: The aim of this research was to synthesize and develop a new method for the preparation of iron oxide (Fe_3O_4) nanoparticles on talc layers using an environmentally friendly process. The Fe_3O_4 magnetic nanoparticles were synthesized using the chemical co-precipitation method on the exterior surface layer of talc mineral as a solid substrate. Ferric chloride, ferrous chloride, and sodium hydroxide were used as the Fe_3O_4 precursor and reducing agent in talc. The talc was suspended in deionized water, and then ferrous and ferric ions were added to this solution and stirred. After the absorption of ions on the exterior surface of talc layers, the ions were reduced with sodium hydroxide. The reaction was carried out under a nonoxidizing oxygen-free environment. There were not many changes in the interlamellar space limits (d-spacing = 0.94–0.93 nm); therefore, Fe_3O_4 nanoparticles formed on the exterior surface of talc, with an average size of 1.95–2.59 nm in diameter. Nanoparticles were characterized using different methods, including powder X-ray diffraction, transmission electron microscopy, emission scanning electron microscopy, energy dispersive X-ray spectroscopy, and Fourier transform infrared spectroscopy. These talc/ Fe_3O_4 nanocomposites may have potential applications in the chemical and biological industries.

Keywords: nanocomposites, Fe_3O_4 nanoparticles, talc, powder X-ray diffraction, scanning electron microscopy

Introduction

In the last two decades, inorganic materials with a layered structure have been used to improve the properties of polymer materials with the polymer molecular chain intercalating galleries of adjacent inorganic layers to form nanocomposites, which consist of novel metals and have important applications in medical identification, catalysis, sensors, optics, and electronics due to their shape and size.^{1–8} Moreover, magnetite (Fe_3O_4) nanoparticles have attracted increasing research attention in the field of environmental remediation.⁹

The application of Fe_3O_4 nanoparticles in the environmental field is mainly due to their much better adsorption and reduction activities than their traditional macro- or microparticle counterparts.¹⁰ In addition, Fe_3O_4 can be easily separated and collected by an external magnetic field. This extraordinary advantage is especially useful for the recovery or reuse of Fe_3O_4 nanoparticles.^{11,12} The application of small iron oxide particles in vitro diagnostics has been practiced for nearly 40 years. In the last decade, there has been an increase in the number of investigations using various types of iron oxides including $\gamma\text{-Fe}_3\text{O}_4$ (maghemite), Fe_3O_4 , and $\alpha\text{-Fe}_3\text{O}_4$ (hematite) particles, which have a core ranging from 5–20 nm in diameter. Of these, Fe_3O_4 is a very promising candidate

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since its biocompatibility has already been proven.^{13,14} Fe_3O_4 , is a common magnetic iron oxide that has a cubic inverse spinel structure with oxygen forming a face-centered cubic close packing and iron (Fe) cations occupying interstitial tetrahedral sites and octahedral sites.¹⁵

Talc, $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$, is a natural compound widely used in the form of a fine powder in several industrial products such as ceramics, putties, paper, paints, flame and ignition retardants, and as an active filler in polymer/silicates composites to improve mechanical characteristics (eg, strength, elasticity, shock resistance) and the nucleation of polymers.^{16,17} The structure of talc is the well-known 2:1 (T–O–T) layer configuration consisting of a octahedral magnesium (Mg) coordinated sheet (O) sandwiched between two tetrahedral silicon (Si) coordinated sheets (T). The talc structure does not present residual surface charge and the bonds between layers are provided by weak electrostatic and van der Waals forces.¹⁸ Talc is known as a two polytypic structure: a two-layer monoclinic 2M structure and a one-layer triclinic 1C1 polytype.^{19–22}

The co-precipitation method is widely used to prepare iron oxide nanoparticles, but the main achievement of this paper was to provide very fine particles (1.95–2.59 nm) with low agglomeration. These magnetic nanoparticles enable easy separation during material preparation and make them suitable for many applications. Moreover, surface decoration of talc with magnetic nanoparticles has led to a new class of nanocomposites materials, which could be also used for environmental purposes as absorbents of metal ions in wastewater treatment.^{23,24}

In this research, talc/ Fe_3O_4 nanocomposites were synthesized at room temperature on the exterior surface of talc layers in aqueous solution using ferric chloride (FeCl_3), ferrous chloride (FeCl_2), and sodium hydroxide (NaOH) as the iron precursor and reduction agent, respectively. Needless to say, to date, there has not been any research on talc/ Fe_3O_4 nanocomposites using the wet chemical reducing method (ie, lamellar polymeric silicate), which is the subject of this study.

Material and methods

All reagents in this work were of analytical grade and used as received without further purification. Ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and ferrous chloride tetrahydrate ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$) of 96% were used as the iron precursor and also, talc powder ($<10 \mu\text{m}$, $3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$) were obtained from Sigma-Aldrich (St Louis, MO, USA). NaOH of 99% was obtained from Merck KGaA (Darmstadt,

Germany). All these aqueous solutions were used with deionized water.

Synthesis of talc/ Fe_3O_4 nanocomposites

For the synthesis of talc/ Fe_3O_4 nanocomposites, 2.0 g of talc was suspended in 70 mL deionized water, and then the molar ratio in FeCl_3 solution was adjusted to 2:1 by adding a measured amount of Fe^{3+} and Fe^{2+} . The ion solution suspended with talc composites was stirred for 1 hour for impregnation by the external surface of talc layers to prepare talc/ Fe^{3+} – Fe^{2+} composites. The 15 mL freshly prepared NaOH (2.0 M) was added to talc/ Fe^{3+} – Fe^{2+} composites suspension under continuous stirring. The suspensions were finally centrifuged, washed twice with ethanol and double distilled water, and kept in a stove at 100°C . All the experiments were conducted at an ambient temperature and under a nonoxidizing oxygen-free environment through the flow of nitrogen gas.

Characterization methods and instruments

The prepared talc/ Fe_3O_4 nanocomposites were characterized by powder X-ray diffraction (PXRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), and Fourier transform infrared spectroscopy (FT-IR). The structures of the produced Fe_3O_4 nanoparticles in talc were examined using Philips X'pert PXRD (copper $\text{K}\alpha$ radiation; PANalytical, Almelo, The Netherlands). The changes in the interlamellar spacing of talc and nanoparticles in talc were also studied by using PXRD in the small-angle range of 2θ (5–15 degrees). In addition, the interlamellar space was calculated based on the PXRD peak positions using Bragg's equation. A wavelength of 0.15418 nm was used for these measurements. The PXRD patterns were recorded at a scan speed of 2 degrees/minute. The TEM observations were carried out using an H-7100 electron microscope (Hitachi Ltd, Tokyo, Japan), and the particle size distributions were determined using UTHSCSA ImageTool version 3.00 (The University of Texas Health Science Center, San Antonio, TX, USA). Furthermore, SEM and EDX were performed (XL 30; Philips, Eindhoven, The Netherlands) to study the morphology of talc and talc/ Fe_3O_4 nanocomposites. Moreover, the FT-IR spectra were recorded over the range of 200 – 4000 cm^{-1} utilizing the Series 100 FT-IR 1650 spectrophotometer (PerkinElmer, Waltham, MA, USA). After the reactions, the samples were centrifuged by using a high-speed centrifuge (Avanti® J-25; Beckman Coulter, Brea, CA, USA).

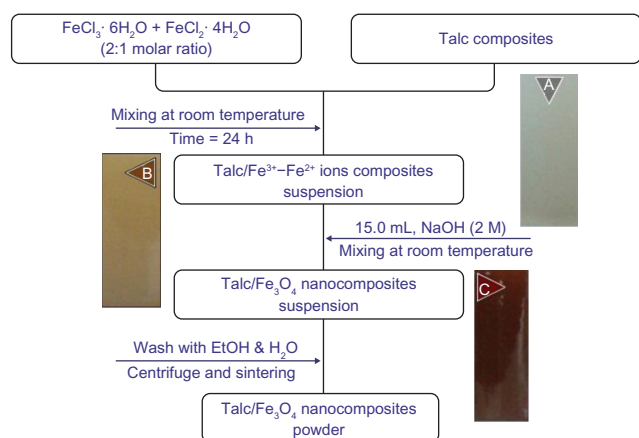
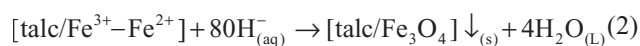
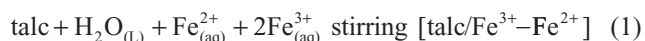


Figure 1 Flowchart of the synthesized talc/Fe₃O₄ nanocomposite powder in (A) talc and (B and C) talc/Fe³⁺-Fe²⁺ ion composites.

Abbreviations: EtOH, ethanol; Fe²⁺, ferrous ion; Fe³⁺, ferric ion; FeCl₂ · 4H₂O, ferrous chloride tetrahydrate; FeCl₃ · 6H₂O, ferric chloride hexahydrate; Fe₃O₄, magnetite; H₂O, water; NaOH, sodium hydroxide.

Results and discussion

The talc suspension was whitish gray in color, which turned to brown after the addition of FeCl₃ · 6H₂O and FeCl₂ · 4H₂O and then to a dark color after the addition of NaOH solution as a reducing agent suspension (Figure 1A–C). Conventionally, Fe₃O₄ nanoparticles are prepared by adding a base to an aqueous mixture of Fe³⁺ and Fe²⁺ chloride at a 2:1 molar ratio. The chemical reaction of Fe₃O₄ precipitation is given in Equations 1 and 2. The overall reaction may be written as follows:²⁵



The comparison between the PXRD patterns of talc and the prepared Fe₃O₄/talc nanocomposites under the chemical reduction route fell in the small-angle range of 2θ (9.35–9.50), which indicated the immobilized formation of Fe₃O₄ nanoparticles on the external surface of the talc layers. The TEM images and size distribution of talc/Fe₃O₄ nanocomposites showed that the mean diameter of the nanoparticles ranged from about 1.2–3.2 nm. Additionally, the SEM images indicated that there were no structural changes between the initial talc and talc/Fe₃O₄ nanocomposites. Furthermore, FT-IR spectra showed that there was no chemical interaction between the silicate layers and Fe₃O₄ nanoparticles in talc/Fe₃O₄ nanocomposites. The synthesized talc suspensions containing Fe₃O₄ nanoparticles were found to be unstable over a long period of time, displaying signs of precipitation.

PXRD

As shown in Figure 2, the original d-spacing (d_s) of talc at 2θ 9.35 degrees was 0.94 nm, which gradually decreased to 0.93 nm at 2θ 9.50 degrees by the formation of Fe₃O₄ nanoparticles on the surface of talc layers. This d_s value was direct proof that the Fe³⁺ ions were bound only on the external surfaces and edges of the talc external layer space. Consequently, the metallic nanoparticles formed only at the exterior layer location, causing a decrease in the basal spacing of talc.¹⁵ In this sample, the intensities of reflections and half-widths were constant; therefore, the parallel lamellar

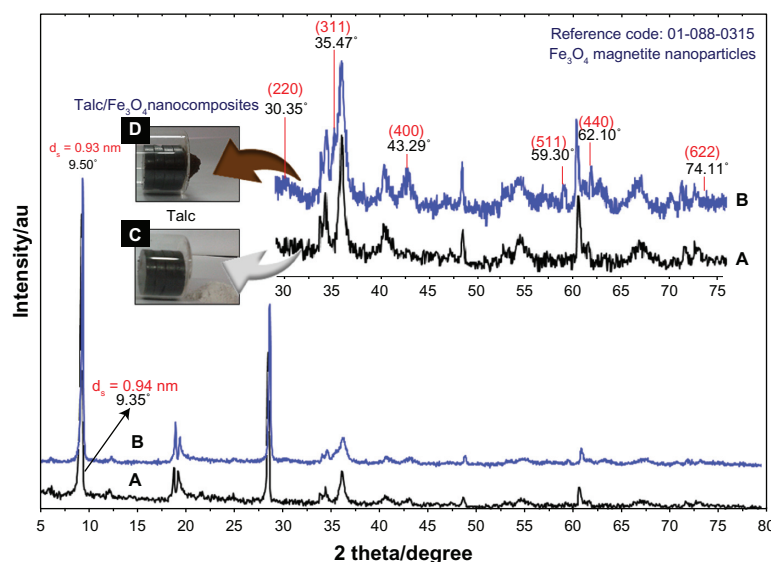


Figure 2 Powder X-ray diffraction of (A,C) talc and (B,D) talc/Fe₃O₄ nanocomposites with the related peaks.

Abbreviation: Fe₃O₄, magnetite.

structure of the mineral clay was not disrupted by the formation of nanoparticles.

The comparison between the PXRD patterns of the talc and Fe_3O_4 /talc nanocomposites in the small-angle range of 2θ (5–15 degrees) indicated the formation of the intercalated Fe_3O_4 nanostructure (Figure 2A and B). As shown in Figure 2B, the PXRD peaks in the wide-angle range of 2θ (30–80 degrees) ascertained that the peaks at 30.35, 35.47, 43.29, 59.30, 62.10, and 74.11 degrees related to the 220, 311, 400, 511, 440, and 622 AU crystalline structures of the Fe_3O_4 cubic nanocrystal with a spinel structure (reference code: 01-088-0315).^{26,27}

The effect of the magnetic field on the talc and talc/ Fe_3O_4 nanocomposite powder is shown in Figure 2C and D. These results confirm that there was a significant amount of Fe_3O_4 nanoparticles on the external layers of talc powder, because the Fe_3O_4 nanoparticles prepared in talc/ Fe_3O_4 nanocomposites were attracted by magnets.

The average particle size of Fe_3O_4 nanoparticles in talc substrate can be calculated using the Scherrer equation (3):

$$n = K\lambda/\beta \cos \theta \quad (3)$$

In this equation, K is the Scherrer constant with a value from 0.9–1 (shape factor), λ is the X-ray wavelength (1.5418 Å), $\beta_{1/2}$ is the width of the XRD peak at half height, and θ is the Bragg angle. From the Scherrer equation, the average crystallite size of Fe_3O_4 nanoparticles for Fe_3O_4 /talc nanocomposites is around < 5 nm, which is in line with the TEM and field emission SEM results discussed later.

TEM

Figure 3 demonstrates TEM images for the size distribution of talc/ Fe_3O_4 nanocomposites containing Fe_3O_4 nanoparticles. The TEM images and their size distributions revealed that the mean diameter \pm standard deviation of Fe_3O_4 nanoparticles was about 2.27 ± 0.32 nm. Importantly, no morphologic differences were observed between the initial talc and Fe_3O_4 nanoparticles. The Fe_3O_4 nanoparticles prepared on the external layer of talc composites are shown by TEM in Figure 3, which confirm that the structure of talc doesn't change in FT-IR spectroscopy. The number of Fe_3O_4 nanoparticles counted in the TEM images was around 374. Figure 4 illustrates the TEM images of pure talc and talc after impregnation with aqueous Fe^{3+} and Fe^{2+} ions. Figure 4A

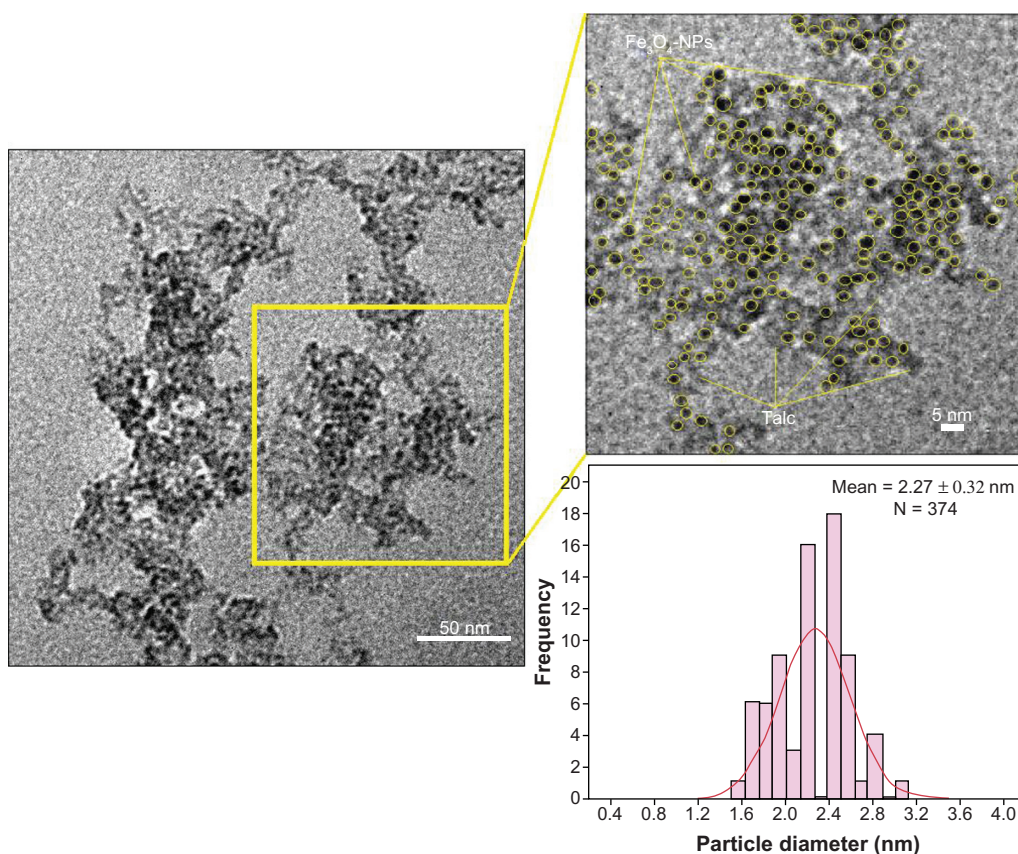


Figure 3 Transmission electron microscopy image and histogram of particle size distribution for talc/magnetite nanocomposites.

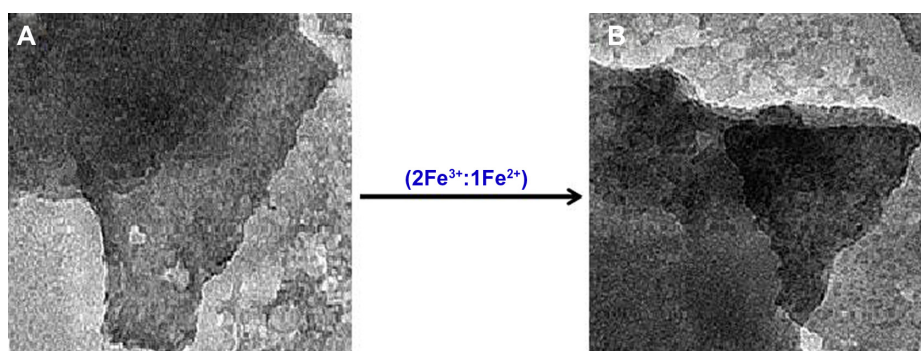


Figure 4 Transmission electron microscopy images of (A) pure talc and (B) talc/ Fe^{3+} – Fe^{2+} ion composites.
Abbreviations: Fe^{2+} , ferrous ion; Fe^{3+} , ferric ion.

demonstrates a typical talc clay image with homogeneously distributed clay flakes, and Figure 4B shows a TEM image for talc/ Fe^{3+} – Fe^{2+} composites without appearance of any nanoparticles.

SEM

The Fe_3O_4 nanoparticles prepared on the external layer of talc composites are shown by SEM in Figure 5A and B, which confirm that the structure of talc doesn't change in FT-IR spectroscopy. Figure 5A and B shows the SEM images for the talc/ Fe_3O_4 nanocomposites synthesized with the co-precipitation method. These results confirm that the exterior surface layer of talc as substrate can effectively control the shape and size of the Fe_3O_4 nanoparticles. The external surface of talc/ Fe_3O_4 nanocomposites with high magnification gradually become shinier due to the presence of small Fe_3O_4 nanoparticles that aggregate together and create large particles (Figure 4B).

EDX

Figure 6A shows the EDX fluorescence spectra for the talc; the peaks around 1.7, 2.4, 2.6, 2.9, 3.6, 4.0, 4.5, 5.0, 6.0, 6.4,

and 7.1 keV are related to the binding energies of talc. In Figure 6B, the peaks around 0.2, 0.8, 2.2, 6.4, and 7.0 keV are related to the Fe_3O_4 nanoparticle elements.²⁸ In addition, the EDX fluorescence spectra for the talc and talc/ Fe_3O_4 nanocomposites confirm the presence of elemental compounds in the talc and Fe_3O_4 nanoparticles without any impurity peaks. The results indicate that the synthesized Fe_3O_4 nanoparticles are of high purity.

FT-IR chemical analysis

Figure 7A and B shows the comparison of FT-IR spectra for the silicate host structure of talc and talc/ Fe_3O_4 nanocomposites with NaOH. The positions of vibrational bands in the region 400–1000 cm^{-1} corresponding to Si–O and other interlayer bonds remained unchanged, and a strong band at 980 cm^{-1} was associated with the stretching vibration of Si–O. The band at 663 cm^{-1} was also assigned to the stretching vibration of Si–O, which is usually taken as evidence for a three-dimensional amorphous silica phase. The band at 410–361 cm^{-1} was assigned to the Si–O–Si bending vibration. The FT-IR spectra indicated the rigidity of silicate layers and nonbonding chemical interaction

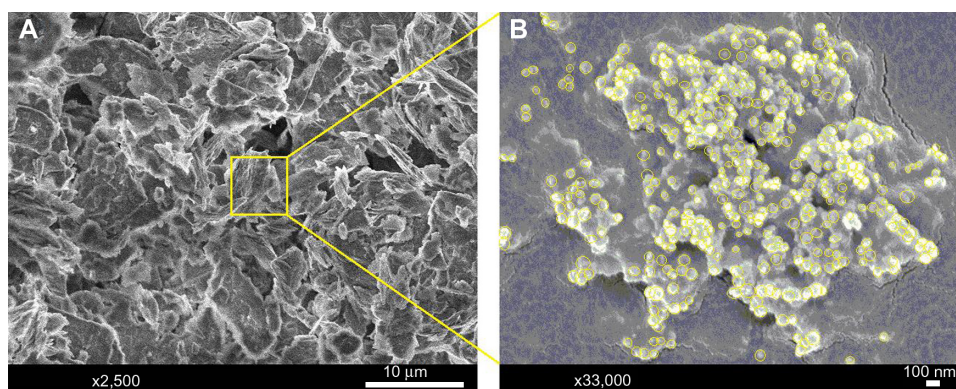


Figure 5 Scanning electron microscopy micrographs of talc/magnetite nanocomposites with (A) low and (B) high magnification.

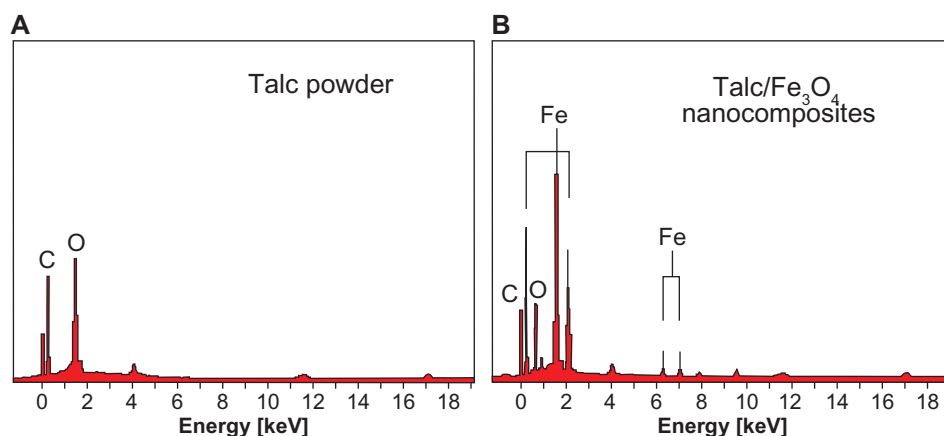


Figure 6 Energy dispersive X-ray spectroscopy of (A) talc and (B) talc/ Fe_3O_4 nanocomposites.

Abbreviations: C, carbon; Fe, iron; Fe_3O_4 , magnetite; O, oxygen.

between the silicate layers and Fe_3O_4 nanoparticles in talc/ Fe_3O_4 nanocomposites. A broad peak was due to the presence of van der Waals interactions between the hydroxyl groups of H_2O with an exterior layer of talc and the partial positive charge on the surface of Fe_3O_4 .¹⁵ These peaks, with the enhanced Fe_3O_4 in the talc/ Fe_3O_4 nanocomposites, shifted to low wave numbers and there was no change in peak intensity.

Conclusion

The Fe_3O_4 nanoparticles were successfully prepared by co-precipitation of Fe^{3+} and Fe^{2+} in talc as the substrate, with NaOH as the reducing agent. This method was demonstrated to be useful for the preparation of Fe_3O_4 nanoparticles. The Fe_3O_4 magnetic nanoparticles with a mean size of 1.95–2.59 nm on the talc layers were synthesized for the first time via a single-step co-precipitation reduction route. This is a cheap, facile, and environmentally friendly method which leads to the formation of Fe_3O_4 nanoparticles. Also,

the Fe_3O_4 nanoparticles prepared by this route were attracted by magnets. This indicates that the Fe_3O_4 nanoparticles are formed in the exterior surface of the talc layer.

Acknowledgments

The authors are grateful to the staff of the Department of Chemistry UPM for their help in this research, and Institute of Bioscience (IBS/UPM) for technical assistance.

Disclosure

The authors report no conflicts of interest in this work.

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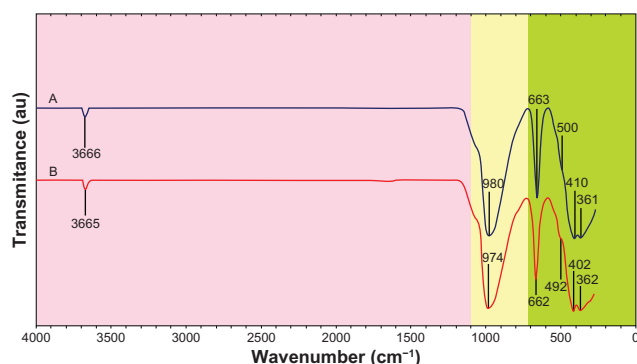


Figure 7 Fourier transform infrared spectra of (A) talc and (B) talc/magnetite nanocomposites.

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