In-situ core-shell polymerization of magnetic polymer nanocomposite (PAAc/Fe$_3$O$_4$) particles via gamma radiation

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Abstract Gamma radiation as a novel approach to synthesize core-shell magnetic polymer nanocomposite (PAAc/Fe$_3$O$_4$) particles was investigated. The Fe$_3$O$_4$ nanoparticles were primarily prepared by in situ precipitation-oxidation and radiation polymerization of polyacrylic acid PAAc. The polymerization process was carried out by dissolving two salts of Fe$^{2+}$/3+ sulfates into a 20% solution of PAAc followed by exposing them to gamma radiation at a dose of 30 kGy. Subsequently, an appropriate amount of ammonium (NH$_4$OH) solution was added to the PAAc/Fe$^{2+}$/3+ polymer solution, yielding a black precipitate of PAAc/Fe$_3$O$_4$. Transmission electron microscopy revealed that the in situ polymerization of core-shell magnetic polymer nanocomposites resulted in sphere-like particles of PAAc/Fe$_3$O$_4$ with diameters of 30 nm. Hysteresis loops indicated that these particles are ferromagnetic in nature.

Keywords Gamma radiation, Nanocomposite, PAAc/Fe$_3$O$_4$, Core-shell particles, Ferromagnetic

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Introduction

Radiation-induced polymerization has been investigated for a long time as an interesting and novel method for controlling polymerization reactions under irradiation conditions by conventional free radical mechanisms. Conditions of radical radiation polymerization in which no special initiator is necessary and polymerization is mainly initiated by OH radicals that are the radiolysis product of water. Radical radiation of chain polymerization occurs by C–H bond scission with C–C covalent bond formation in the molecules causing crosslinking of the polymer.

As shown in Scheme 1, the first stage of the polymerization process is the dissociation of 

**Scheme 1**

Fe$^{2+}$/3+ sulfates into a 20% solution of PAAc followed by exposing them to gamma radiation at a dose of 30 kGy. Subsequently, an appropriate amount of ammonium (NH$_4$OH) solution was added to the PAAc/Fe$^{2+}$/3+ polymer solution, yielding a black precipitate of PAAc/Fe$_3$O$_4$. Transmission electron microscopy revealed that the in situ polymerization of core-shell magnetic polymer nanocomposites resulted in sphere-like particles of PAAc/Fe$_3$O$_4$ with diameters of 30 nm. Hysteresis loops indicated that these particles are ferromagnetic in nature.

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In this article, in situ core-shell polymerization of PAAc/Fe$_3$O$_4$ nanocomposites via gamma radiation at a dose of 30 kGy has been carried out. The black fine powder obtained, consisting of nanoparticles with a diameter of approximately 30 nm, was characterized by dynamic light scattering (DLS), delivery and microwave absorbers. A major difficulty in the preparation of these polymer nanocomposites is the dispersion of the nanoparticles in the polymer matrix. Nanoparticles tend to agglomerate and form separate phases in a polymer matrix. This causes unfavorable thermal and mechanical properties. To enhance dispersion and polymer-filler interaction, the nanoparticles must be uniformly dispersed in the polymer matrix. In this study, PAAc/Fe$_3$O$_4$ nanocomposites were synthesized by an in situ core-shell polymerization process. Magnetic oxide Fe$_3$O$_4$ was used as a core and PAAc polymer as a shell.

The thermal and magnetic properties of the nanocomposites were investigated to reveal the interaction between Fe$_3$O$_4$ particles and PAAc matrix. Magnetic polymer nanocomposite particles have been considered as an important material for a number of applications. Examples include, immunoassay methodologies, the isolation of nucleic acid sequences, micro-organisms, and in fluorescent biological imaging etc.

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Fourier transform infrared (FTIR), Thermogravimetric analysis (TGA) and X-ray diffraction (XRD). Magnetic characterization was also performed and revealed that the nanocomposite polymer particles are ferromagnetic in nature.

**Materials and methods**

All chemicals used in this research were of analytical grade and obtained from commercial sources. FeSO₄·7H₂O, Fe₂(SO₄)₃·4H₂O, ammonium hydroxide (NH₄)OH and poly acrylicacid (PAAc) were all obtained from Sigma-Aldrich, Germany. Deionized water was obtained from Milli Q system and used throughout.

**Synthesis of PAAc /Fe₃O₄ nanocomposite**

A new approach for radiation synthesis of PAAc/Fe₃O₄ was carried out in two steps. First, 1 g of FeSO₄ and Fe₂(SO₄)₃, with a mole ratio of 1.7:1, was added while stirring to 20% of PAAc dissolved in 18 ml of acetone-water (20:80) solvent with the pH of the solution carefully adjusted up to four (Figure 1). This solution was stirred for about 1 h until stable. The mixture was poured in a test tube and homogenized for 20 min using a sonication bath, and then injected with N₂ inert gas for 3 min.

The tube was then sealed and exposed to gamma radiation at a dose of 30 kGy, after which the polymerization process was finished. Next, ammonium solution was added to the mixture until the pH reached 10 and the mixture was stirred at a temperature of 50 °C. The core-shell nanoparticles that formed were then washed with distilled water until the pH reached 7. After that, the black powder obtained was filtered and rinsed with distilled water and then methanol. The powder was then dried in an oven at a temperature of 50 °C for 24 h.

**Characterization**

Fourier transform infrared (FTIR) spectroscopy was carried out by a Brüker FTIR-6000 (Brüker, Germany) using an attenuated total reflectance (ATR) unit to investigate the interaction of functional groups in PAAc with the magnetic nanoparticles Fe₃O₄. The crystallographic characterization of the magnetic polymer nanocomposite PAAc/Fe₃O₄ was done by a powder X-ray diffraction (XRD) spectrometer (Schimadzu XRD-6000, Japan). Transmission electron microscopy (TEM) images were obtained to reveal the morphology and size of the nanoparticles. The mean hydrodynamic diameter of the nanoparticles was measured using dynamic light scattering (DLS) (Malvern).
To make the sample suitable for DLS measurements, aqueous suspensions were prepared as follows. 0.1 mg of PAAc/Fe₃O₄ sample was dispersed in 10 ml distilled water. Electron-spin resonance (ESR) tests were carried out at ambient temperature using a Brüker EMX spectrometer (X-band). The magnetic properties of PAAc/Fe₃O₄ measured using a vibrating sample magnetometer (VSM, LakeShore 7400) at 25 °C. Thermal analysis was performed using a TGA of TA Waters Instruments (USA). All measurements were conducted under a nitrogen atmosphere.

Results and discussion

FTIR-ATR of PAAc/Fe₃O₄ nanocomposite

The FTIR-ATR spectra of crosslinked PAAc composites with Fe₃O₄ nanoparticles are shown in Figure 2. The broad peak found at 3440 cm⁻¹ is due to H–bonding between –OH and –COOH groups in the polymer. The band observed at 2972 cm⁻¹ is attributed to C–H bands. The band at approximately 1729 and 1650 cm⁻¹ is due to C=O and C–O stretching, respectively. The peak at 1080 cm⁻¹ is characteristic of –CH₂–, while skeletal vibration involving C–O–C bridge stretching of the glucosamine residue is responsible for the band at 1107 cm⁻¹. The 1025 cm⁻¹ band is likely to be related to CH–OH bonds in the cyclic compounds. The peaks that appeared at 582 and 562 cm⁻¹ are indicative of stretching, and the variation modes of Fe–O confirms the presence of crystalline Fe₃O₄. The FTIR studies indicated that a physical interaction is present between Fe₃O₄ nanoparticles coated with PAAc polymer.

Particle size distribution of PAAc/Fe₃O₄ nanocomposite

TEM images confirm the formation of core-shell particles of magnetic polymer nanocomposites (Figure 3). The Fe₃O₄ core, after being coated with the PAAc shell, appears much darker than the polymer shell and seems to be spherical with a diameter of approximately 30 nm. The average nanoparticle diameter was found to be around 110 ± 5 nm using dynamic light scattering (DLS) measurements (Figure 3). The particle size obtained by DLS is much larger than by TEM because in the DLS method the nanoparticles are dispersed in water, which causes an increase in size as water molecules are absorbed by the PAAc. Some particle aggregation is also observed due to magneto-dipole interactions.

XRD of PAAc/Fe₃O₄ nanocomposite

As shown in Figure 4 the XRD patterns of the magnetic polymer nanocomposite PAAc/Fe₃O₄ had six diffraction peaks as shown in Figure 3 at 2θ = 30.04°, 35.28°, 43.06°, 53.32°, 57.02°, and 62.63°, which corresponds to the (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) crystal planes, respectively. These are the characteristic peaks of standard Fe₃O₄ crystals.
and demagnetize. The curve is typical for magnetite. The saturation magnetization ($B_s$) is 88 emu/g and at this point all domains are aligned in the same orientation as the $B_s$ and $H_c$ (coercive force), i.e. the force required to remove the residual magnetization from the material, is 230 Oe and $B_r$ (retentive force), i.e. the level of residual magnetization, is 6.75 emu/g. This is a clear indication that the Fe₃O₄ particles have a strong magnetic response to an external magnetic field even when they are embedded into a PAAc matrix.

At ambient temperature, a sharp and strong single asymmetric resonance is observed in the ESR spectra (Figure 4(b)) at a field of around 3442 G for PAAc/Fe₃O₄. The line width of the signal is about 540 G and this line broadening may arise from dipolar interactions between super-paramagnetic nanoparticles with values of $g// = 1.99$ and $g⊥ = 2.25$. The $B–H$ curve and ESR spectrum confirm the ferromagnetic behavior of the investigated PAAc/Fe₃O₄ nanocomposites.

Thermal stability of PAAc/Fe₃O₄ nanocomposite

TGA is typically used to assess the filler content in polymers nanocomposites. Nanofillers also typically have a significant impact on the thermal properties of the polymer composites. The mass loss curve of the PAAc/Fe₃O₄ nanocomposite is compared to neat Fe₃O₄ and reported in Figure 5. The mass loss over a temperature range from 100 to 700 °C for pure Fe₃O₄ is about 12%. This might be due to a loss of residual water in the Fe₃O₄ sample. The TGA result in Figure 5 indicates that on the PAAc/Fe₃O₄ undergoes thermal degradation between 116 and 270 °C with a total mass loss of 20%. This is attributed to the fact that the PAAc/Fe₃O₄ nanocomposite can absorb a certain amount of moisture.

Magnetic characterization of PAAc/Fe₃O₄ nanocomposite

The aim of this study was to synthesize a composite material with a strong magnetic response, where the hydrophilic character may be used as an environmental memory alloy. Here the PAAc/Fe₃O₄ nanocomposites exhibit the hydrophilic because of the poly acrylicacid (PAAc) together with magnetic properties as indicated in Figure 4(a) by the $B–H$ curves (hysteresis loop), showing clearly ferromagnetic properties as reflected by the sigmoidal curved shape which perfectly fits that of a soft magnetic material that is easy to magnetize and demagnetize. The curve is typical for magnetite. The $B_s$ (saturation magnetization) is 88 emu/g and at this point all domains are aligned in the same orientation as the $B_s$ and $H_c$ (coercive force), i.e. the force required to remove the residual magnetization from the material, is 230 Oe and $B_r$ (retentive force), i.e. the level of residual magnetization, is 6.75 emu/g. This is a clear indication that the Fe₃O₄ particles have a strong magnetic response to an external magnetic field even when they are embedded into a PAAc matrix.

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under ambient conditions. Thermal degradation finishes at 700 °C with a total mass loss of 55.9%, which indicates that the Fe₃O₄ content in the PAAc/Fe₃O₄ nanocomposites is about 56%. Moreover, TGA also revealed that Fe₃O₄ could be used to increase the thermal stability of PAAc in the same way as many other nanofillers.31–34

Conclusions
Magnetic polymer nanocomposites were synthesized as core-shell particles using in situ polymerization induced by gamma radiation. In order to overcome agglomeration of Fe₃O₄, PAAc was used as a polymeric shell. PAAc/Fe₃O₄ nanocomposites were characterized by various techniques, including FTIR, TEM, TGA, ESR, and DLS. TEM imaging indicated that these nanoparticles had a diameter of approximately 30 nm and were well dispersed. The thermal stability of the PAAc/Fe₃O₄ nanocomposites was significantly enhanced by the addition of Fe₃O₄ nanoparticles as filler. This might be the result of a nanocoagulation effect, which reduces the mobility of polymer chain due to interactions between the nanoparticle and the polymer chains. A study of the magnetic properties of the prepared PAAc/Fe₃O₄ nanocomposites showed that these nanoparticles were ferromagnetic in nature.

Disclosure statement
No potential conflict of interest was reported by the author.

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