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Article

Preparation, Characterization of Magnetic Chitosan Nanoparticles Coated with Fe₃O₄ Prepared by Ex-Situ Co-Precipitation Method

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Abstract: The suspension cross-linking technique has been utilized to create the magnetic chitosan nanoparticles, which were then applied to the magnetic carrier method at 80°C. First the FeCl₂ and FeCl₃ solution co-precipitated and synthesized Fe₃O₄ for utilization in the formation of magnetic chitosan nanoparticles, it has been characterized by utilizing DLS (Dynamic light scattering), XRD (X-ray diffraction spectrometer), FTIR (Fourier transform infrared spectroscopy), which identified structure, size, FE-SEM (Field Emission Scanning Electron Microscope) and HR-TEM (High-resolution transmission electron microscopy) which identified structure and particle size whereas magnetic behavior of chitosan nanoparticles was determined by VSM (Vibrating sample magnetometer). According to the results collected, the magnetic chitosan nanoparticles have been spherical in form and size ranging from around 250 to 400 nm.

Keywords: magnetic nanoparticles; chitosan; XRD; FESEM; FTIR; DLS; VSM; applications

1. Introduction

A cationic polysaccharide is chitosan, it is a macromolecule formed by the repetition of D-glucosamine which was easily obtained by partial deacetylation of chitin, chitin is a widely occurring bio-polymer in nature after cellulose [1] a variety of eukaryotic organisms, including crustacea, fungi, and insects, it is a polymer of N-acetyl-D-glucosamine.[2] Given that heavy metal ions are poisonous and non-biodegradable, which represent a severe danger to ecological systems and public health[3], the adsorption technique has been utilized to remove ions of the heavy metal by the water solutions. Therefore, it is essential to eliminate such harmful metal ions in the wastewater.[4] In this investigation, Fe₃O₄ magnetic chitosan nanoparticles have been created, assessed, and used to remove metals from a water solution.[5] We have created a chitosan magnetic nano-adsorbent employing chitosan as ionic exchange groups and Fe₃O₄ nanoparticles as the cores.[6] Most chitosan-based adsorbents are sub-micron to micron-sized, and to offer a sufficient surface area for adsorption, they need large internal porosities. The adsorption rate and the available capacity are reduced by the diffusion constraint inside the particles. This particular kind of magnetic adsorbent, which has favorable compatibility, a cheap price, quick and simple extraction or regeneration, and convenient operation, can be helpful for future study and actual metal removal applications.[7] Several benefits have led to the adsorption process being regarded as the most widely utilized approach (efficient, eco-friendly, economical, simple, and reversible).[8] Here, chitosan-coated with magnetic nanoparticles of Fe₃O₄ was effectively created as a new nano-adsorbent. Metals' adsorption kinetics and absorption capability were examined.[9] We systematically looked at how adsorbent dosage, pH, and contact duration affected the results. Additionally, the metals adsorbent's reusability and adsorption process were investigated. Additionally, the adsorbent's ability to remove metals from a water solution was assessed.[10]

2. Materials and methods

2.1. Materials

The chitosan form of a shrimp shells purchased from Sigma-Aldrich, which was about 75% (deacetylated) and ranged in molecular weight from 100 kDa to 1200 kDa, depending on the production method and product quality.[11] acetic acid, hydrochloric acid (ACS reagent, 37%), additionally, analytical grade reagents that are readily accessible on the market, such as FeCl_2 and FeCl_3 , were utilized without additional purification, along with double-distilled water for preparing the solution.

2.2. Preparation of magnetic chitosan nanoparticles

In order to obtained ferrogel solution for examined, 0.65 g of chitosan was first dissolved in 100mL of 1 % (v/v) acetic acid to prepared 0.6 % w/v chitosan solution.[12] This solution was then continuously stirred for an 1 hour at room temperature having the pH of 4 increased with 10 N solution of NaOH, and 10 mL of Fe_3O_4 in aqueous solution (by the mixing of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ (1.90 g) and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (4.50 g) salts in de-ionized water of 50 mL) was then dropwise added into chitosan solution and then sonicated for half an hour.[13] After adding tripolyphosphate, the (Tripolyphosphate) TPP was combined for a further hour after being dissolved in water to a concentration of 1 percent under magnetic stirring at 2,000 rpm. (The weight-to-volume ratio of chitosan to TPP is 5:1 and 2:1, respectively.) [14][15] The particles have been incubated for 30 minutes at room temperature. All magnetic chitosan nanoparticles were resuspended in ultrapure water, the products have been rinsed with ethanol, and they have been drying in a vacuum oven at a temperature of 500°C for 24 H after being filtered by centrifugation at 9,000 g for 50 min at 5° [16]. [17] The electrostatic attraction among the positively charged chitosan ion and the negatively charged area around the surface of Fe_3O_4 led to the chitosan-coated Fe_3O_4 . Then, tripolyphosphate and sulphate were used to create an ionic crosslinked chitosan complex, which delayed the formation of a Fe_3O_4 coating and improved the biodegradability & biocompatibility of the chitosan nanoparticles. The combination of these crosslinking created chitosan- Fe_3O_4 nanoparticles with a spherical morphology, and interactions between the two occurred through electrostatic forces.[18] After drying, the nanoparticles were characterized to look at their physical character and magnetic properties.

2.3. Characterizations of magnetic chitosan nanoparticles

TEM 200CX, Quanta 400 FESEM, and EDS have been utilized to study the shape and size of dried chitosan nanoparticles. [18] The electrons are launched in a strong electric field gradient after being liberated from a source of field emission. The socalled primary electrons are focused and refracted bent into a narrow scan beam within the high vacuum column, where it bombards the target. A detector then picks up the secondary electrons and produces an electronic signal, [19] and signal displays the FESEM images. Emitted electrons from an electron source at the top of the microscope's column fill a vacuum.[20].

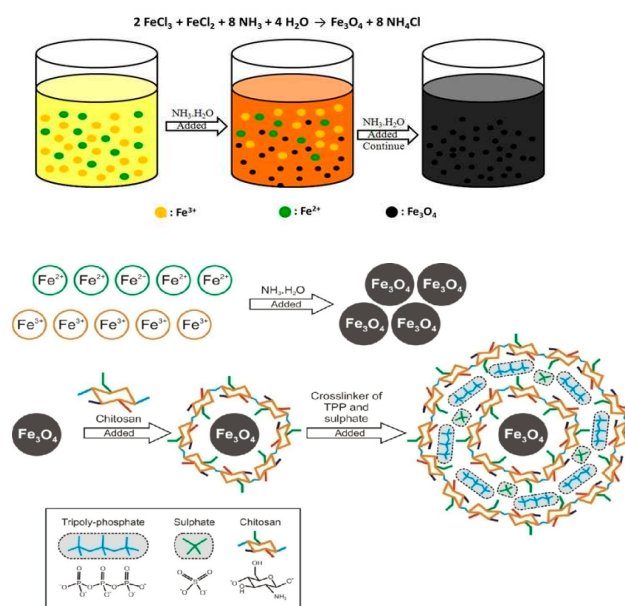


Figure 1. The formation mechanism of chitosan- Fe_3O_4 nanoparticles by ex-situ method.

The electrons are focused using electromagnetic lenses into a very narrow beam that is then directed into the specimen of interest. An energy-dispersive spectrometer (EDS) within the TEM may also detect X-ray emission as the findings of the main electron beam's interaction using the sample.[21] In DLS analysis, a sample suspension is irradiated through a beam of laser, which causes the laser light to scatter in all directions. A alternative name for DLS is photon correlation spectroscopy.[22] Over time, the light dispersion is seen at a certain angle. The randomized Brownian motion of the particles is what causes the signal fluctuation.[23] The Stokes-Einstein equation uses the angular intensity distribution to calculate the particle size. On the basis of Faraday's Law of Induction, the VSM is a scientific tool used to detect magnetic characteristics.[24] The VSM works on the premise that when the sample material is exposed to a homogeneous field of magnet, In the sample, a dipole moment is produced that is proportional to the sample susceptibility product as well as the applied field.[25]

3. Results and discussions

3.1. Nanoparticles with magnetic properties are described

3.1.1. Morphological characterization

With an almost limitless depth of focus, the FESEM gives information on topography and elements at magnifications of

10x to 300,000x. [26] FESEM focuses on the capability to evaluate smaller-area contamination areas at electron accelerating voltages compatible with EDS and with mapping, as shown in Figure 2. [27] Because electrons have a shorter de Broglie wavelength than light, HRTEM can image at a much greater resolution than light microscopes. [28] The goal of TEM imaging of nanomaterials is to capture many calibrated transmission electron pictures that faithfully depict the nanomaterial on the HRTEM images in Figure 3.[29]

3.1.2. Magnetic properties

The magnetic characteristics of materials like a function of temperature, time, and magnetic field are measured using VSM (vibrating sample magnetometer) instrument, as shown in Figure 4 [30] The magnetization of Fe_3O_4 chitosan nanoparticles was shown, and the VSM predicted the improved magnetic behaviour of nanoparticles. All samples were shown to possess superparamagnetic

capabilities.[31] The graph displays the Fe_3O_4 chitosan nanoparticles' saturation magnetization values, all of which are sufficient for a practical magnetic separation. The VSM is the tool used to measure the magnetic moment, which is the most basic property of solid sample magnetism. The magnetization spiral for magnetite nanoparticles at 25°C temperature is shown in Figure 4. [32]

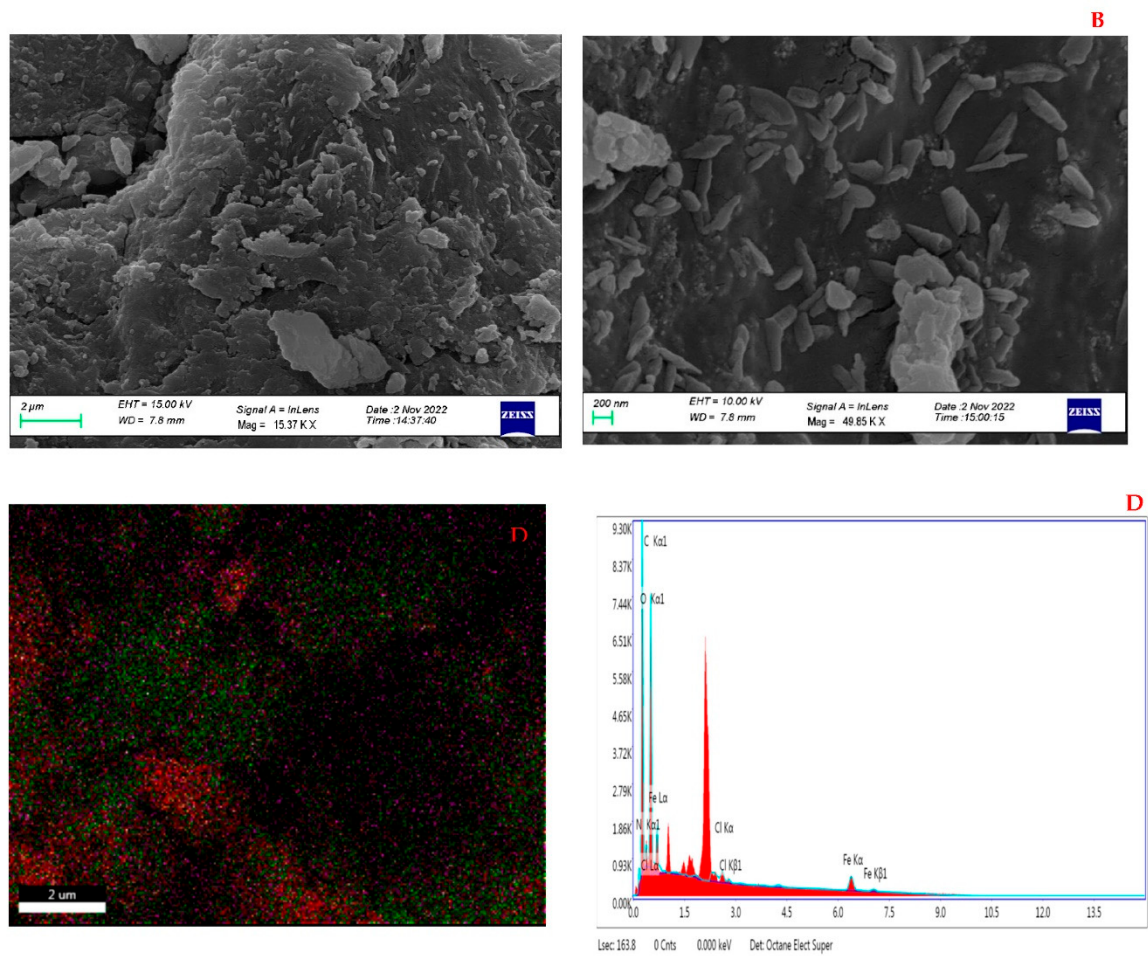


Figure 2. FESEM images (A and B) of magnetic chitosan nanoparticles and mapping (C) with EDS (D).

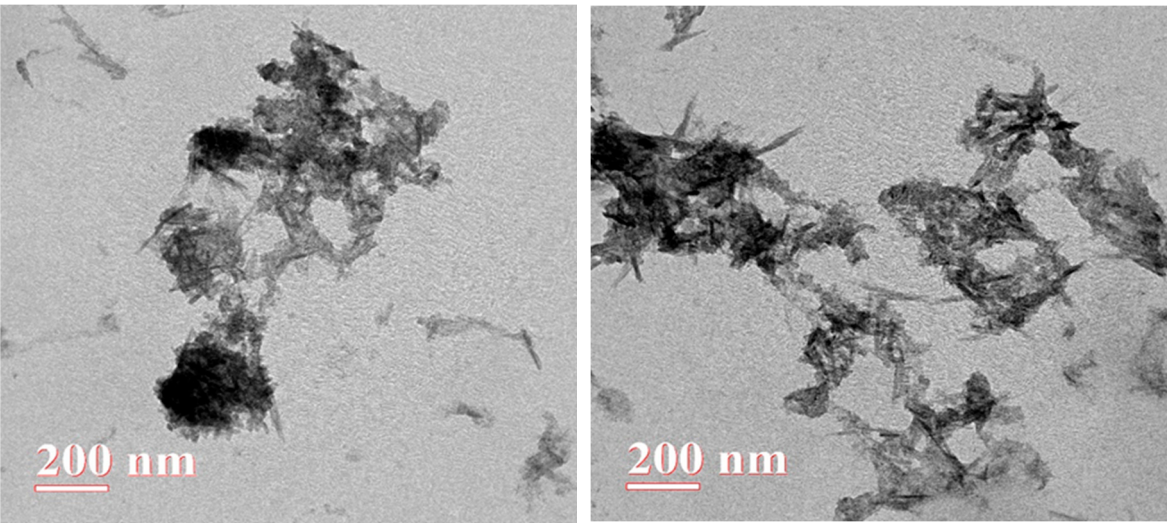


Figure 3. HRTEM images of magnetic chitosan nanoparticles.

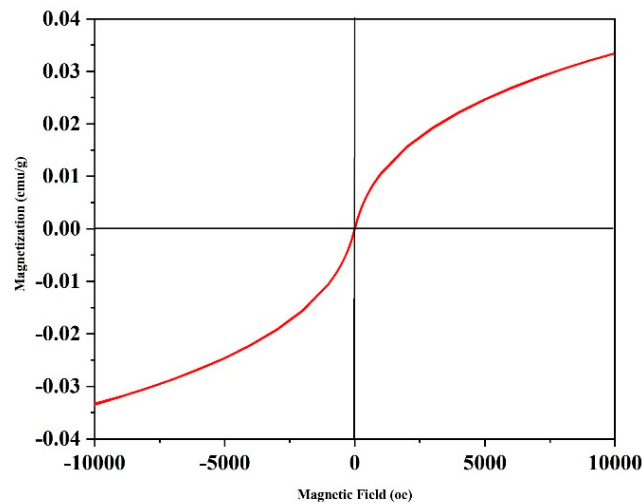


Figure 4. Magnetic properties of magnetic chitosan nanoparticles in VSM curve.

3.1.3. DLS analysis

Particle size may be determined by observing the inconsistent changes in light intensity that are scattered from a suspension or solution.[33] As seen in Figure 5, this approach is often in relation as DLS. The most popular approach for analyzing nanoparticles and figuring out their size distribution is DLS.[34] DLS is a very efficient and quick method for determining the particles size distribution in the fields of physics, chemistry, and biochemistry, particularly for monomodally dispersed spherical particles. By combining DLS with methods like AFM and TEM, one can quickly gain a thorough understanding of the analyte's size distribution. [35]

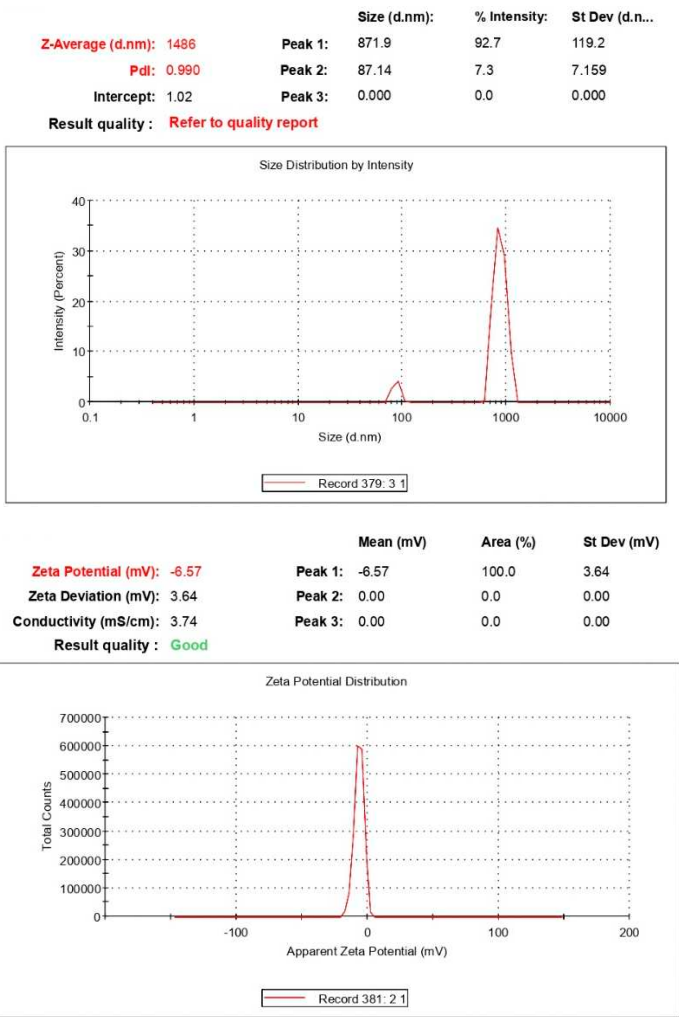


Figure 5. Size distribution and Zeta potential distribution of magnetic chitosan nanoparticles by DLS.

3.1.4. XRD analysis

Particles of Fe₃O₄ with magnetic characteristics and the ability to be utilized for magnetic separation are shown by the XRD patterns of the pure magnetic chitosan nanoparticles displayed in Figure 6. Three prominent distinctive peaks for magnetic chitosan nanoparticles were identified by XRD at 2θ = 9.4°, 20.0°, and 35.4° in the graph. [36].

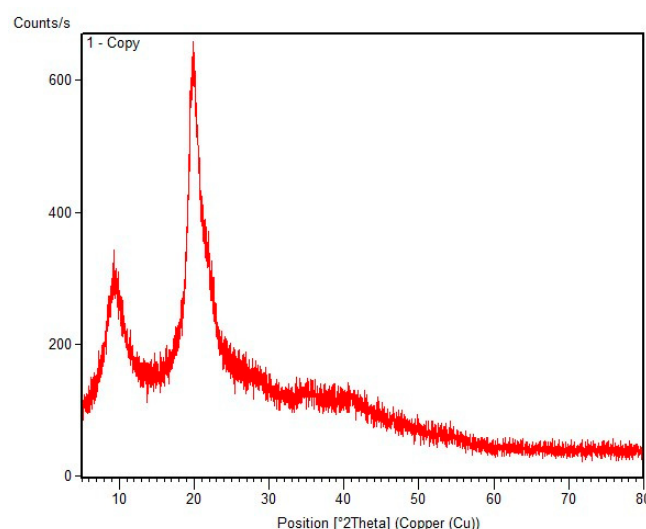


Figure 6. Chitosan nanoparticles with a magnetic XRD pattern.

3.1.5. FTIR analysis

Spectra of FTIR for pure chitosan and magnetic chitosan nanoparticles have been plotted to verify the presence of the surface Fe_3O_4 coating. [37] For chitosan, the peak at 3438.9 cm^{-1} is attributed to O-H stretching vibration, and the peak at 2924 cm^{-1} is expressed for C-H stretching vibration [38]. In contrast, 1113.7 cm^{-1} was observed for primary alcoholic group C-O stretching, 1638.2 cm^{-1} was observed for $-\text{NH}_2$ in amide group, and 3438.9 cm^{-1} was also seen for N-H bond in chitosan. [39] However, for magnetic chitosan nanoparticles, O-H stretching vibration and C-H stretching vibration peak at 3427.4 cm^{-1} and 2918.7 cm^{-1} , respectively [40]. For $-\text{NH}_2$ in amide group, magnetic chitosan nanoparticles peak at 1636.3 cm^{-1} . [41],[15]

FTIR spectroscopy examined by nicolet iS50 FTIR tri-detector, which was gold flex spectrometer-gold optics with 0.09 cm^{-1} resolution and using the DLaTGS detector with KBR window and scanning range was $4000\text{--}400\text{ cm}^{-1}$. especially Far-IR is typically defined as radiation between range of 500 cm^{-1} and 20 cm^{-1} , whereas mid-IR between 4000 cm^{-1} and 500 cm^{-1} and NIR as usually between $10,000$ and $4,000\text{ cm}^{-1}$

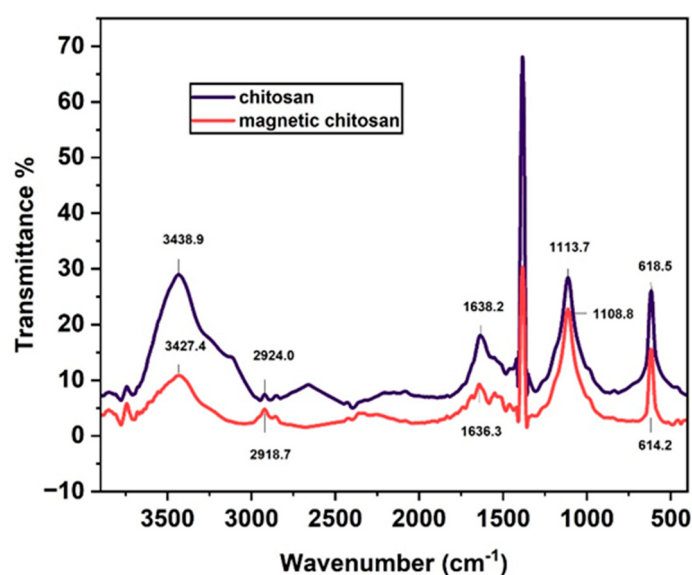


Figure 7. FTIR spectra of chitosan and magnetic chitosan nanoparticles.

3.1.6. Applications of magnetic chitosan nanoparticles

The magnetic chitosan nanoparticles are a unique material with optimal heavy metal adsorption behavior, [42] Because of their efficient elimination of heavy metals by magnetic fields and strong metal chelating capacity, magnetic chitosan nanoparticles are a significant class of heavy metal adsorbents. [43] Chitosan and Fe_3O_4 were combined in a sufficient ratio to create chitosan magnetite particles through the amine group via reverse-phase suspension cross-linking technique, which is relevant in a field's varieties, like wastewater treatment. In comparison to other separation techniques, [44] advances in magnetic separation are efficient, quick, and economical. The magnetic chitosan's adsorption efficiency on the basis of the utilizing the chemical groups for the extracting the heavy metals like arsenic and mercury. [45] This method has made the separation process simpler since it eliminates the need for centrifugation or filtering, making it simple to separate magnetic adsorbents from samples of any size.[46] Capacity of adsorption, the active sites numbers, and the adsorbent surface area are used to measure the effectiveness of heavy metal removal. As adsorbent concentrations were raised, the adsorption capacity went up, then dropped.[47] The adsorbent surface area is increased and abundance of adsorption sites for heavy metals including arsenic, mercury, and lead are responsible for this. Additionally, strong magnetization and adsorption capability of magnetic chitosan nanoparticles towards heavy metals from wastewater are shown.[48]

4. Conclusions

Chitosan has been linked to the Fe_3O_4 nanoparticles surface to create the magnetic Fe_3O_4 chitosan nanoparticles. Chitosan is highly effective in nanoparticles production or magnetic nanoparticles formation. The magnetic chitosan nanoparticles exhibit very steady magnetization capabilities, as shown in Figure 3. In this research, ionic gelation was used to create magnetic chitosan nanoparticles. Size, magnetic properties and morphology of the magnetic chitosan nanoparticles have been assessing by utilizing the FESEM, TEM, DLS and VSM methods. In FESEM the energy dispersive spectroscopy (EDS) or elemental mapping method popularly utilized for qualitative evaluation of materials but it is able to give the semi-quantitative results as well. The HR-TEM investigations showed the presence of Fe_3O_4 nanoparticles with lateral sizes of about 10 to 200 nm. The magnetic chitosan nanoparticles, which ranging size from 200 to 400 nm, seemed to be separated. Additionally, more chitosan and Fe_3O_4 increased the molecular weight of the magnetic nanoparticles, which in turn increased their size. DLS is often utilized to examine the Zeta potential as well as size of magnetic nanoparticles, which was 871.9 nm, despite the fact that the rise in Fe_3O_4 concentration likewise improved the "magnetic property of the magnetic chitosan nanoparticles. The FTIR spectrum of magnetic chitosan nanoparticles revealed that the peak of O-H stretching vibration was considerably broader than pure chitosan, and the XRD patterns of the magnetic chitosan nanoparticles" analysed two primary peaks.

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