Fabrication of hierarchical Bentonite/Chitosan/NiFe₂O₄ ternary nanocomposite and its efficiency in the removal of Pb(II) from aqueous medium

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ABSTRACT

Lead is the most globally bountiful harmful substantial component in nature. Its utilization can be followed to verifiable occasions. The current work archives the combination, portrayal, and utilization of bentonite/chitosan/NiFe₂O₄ ternary novel nanocomposite. Nanostructured nickel ferrite was joined with chitosan and bentonite to frame a novel ternary nanocomposite. The nanocomposite was portrayed by FT-IR, powder XRD, VSM, and TEM investigation. All the logical outcomes affirm the fruitful arrangement of ternary composite. The as-blended nanomaterial was applied as nano-adsorbent for the evacuation of lead (II) from water. The ideal composite portion was 50 mg/100 mL was seen as profoundly powerful for the evacuation of 82% Pb (II) particles from 200 ppm watery arrangement. This ferromagnetic, minimal effort, the eco-accommodating nanocomposite may end up being helpful for the expulsion of lead from watery arrangement at the mechanical level.

Keywords: Ferromagnetic, Nanocomposite, NiFe₂O₄, Pb (II), Ternary

1. Introduction

Lead is the most all inclusive bountiful poisonous overwhelming component in the earth and is scattered in the various segments of nature. The sulling of lead with the earth is because of certain modern exercises like creation of the lead stockpiling cell, lead pipes, paints, clinical hardware, and distinctive rural practices. The poisonous impact of lead is because of its mediation with the biomolecules present in the living framework [1]. 10 mcg/dL (microgram per deciliter) is the lenient furthest reaches of lead in the living framework. Pb (II) particles can enter the living framework either through defiled water or air. When lead interacts with blood, it ties with the sulfur-containing protein and dispersed to the various tissues of the body. Lead is known to harm the kidney, liver, regenerative framework, mind work, and so on [2]. Sulling of lead in the drinking water is basically because of filtering of the metal, which may present genuine wellbeing perils [3]. Different

treatment procedures such as ion exchange, electrolyte removal, membrane filtration, reverse osmosis, solvent extraction and adsorption have been reported for effective removal of lead from water [4-7]. However adsorption technique is found to be the most successful and cost effective method for the removal of lead from water. Various materials either normally accessible or synthetically structured have been accounted for as adsorbents of lead from water. Be that as it may, the proficiency of such adsorbents in lead expulsion isn't up to the level [8]. Hence the requirement for more current progressively productive adsorbents for Pb(II) particles is viewed as a difficult task. As the procedure of adsorption is a surface marvel and henceforth it is expected that nanocomposite will be the best substitute and fruitful adsorbent for the expulsion of lead from water. As of late, the chance of getting ready metal ferrites based nanocomposites has opened another and energizing exploration field with progressive application in the field of water treatment [9] because of their nanometer size, superparamagnetic properties,

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and a high surface-to-volume proportion [10, 11]. More over the stability of spinel metal ferrite materials in acidic medium (pH 2.0-6.0) is a favorable position since they can use all through a wide pH extend [12]. Graphene based magnetic nanocomposite like iron oxide composed with reduced graphene oxide has been reported to be an effective adsorbent for removal of lead from water [13]. In the last few years polyacrylic acid processed with bentonite clay nanocomposite have been used as an adsorbent for lead removal [14]. Excellent result in lead removal from water is obtained by using magnetite Dowex 50WX4 resin nanocomposite [15]. Efficient removal of lead ion from water has been reported by using rare earth oxide, alumina nanocomposite [16]. Manganese ferrite - graphene oxide magnetic nano hybrid found to exhibit enhanced lead and arsenic adsorption capacity from water [17].

In this current examination, a ternary nanocomposite made out of nickel ferrite - chitosan - bentonite has been incorporated and its productive evacuation of lead (II) from water has been researched spectrophotometrically. Consolidation of nickel ferrite in the composite as adsorbent is due to its easy removal from aqueous solution applying magnetic field. To accomplish an enormous surface zone it is created in the nanoscale. Chitosan is a direct polysaccharide made out of D-glucosamine, and N-acetyl D-glucosamine. It additionally can shape hydrogen bonds with the oxygen particle of ferrite and hydrogen atom of its amino groups [18]. Chitosan has high adsorption limits and shows great properties for substantial metals expulsion from watery arrangements [19]. Clay and clay minerals are among the most plentiful regular and modest inorganic filler materials that have been broadly examined on account of their solid adsorption and complexation capacity [20]. Bentonite is fundamentally expandable montmorillonite clay which has the smallest crystal size and subsequently the biggest inside also, outside surface zones for cation exchange. The extending layer of aluminosilicates with various surface interchangeable cations, for example, calcium, sodium, magnesium, and potassium gives the proclivity and limit with respect to bentonite adsorption which is a favored technique in water and wastewater treatment attributable to its basic procedure, modest, and bounty flexibility of adsorbent, and phenomenal adsorbent properties rendering outstanding expulsion proficiency toward target contaminations [21]. The adsorption limit of chitosan is additionally improved by impregnating bentonite in the composite [22]. Considering all these extraordinary highlights of individual parts; they are consolidated into the ternary composite to achieve a powerful adsorption limit.

2. Experimental

2.1. Materials

Iron (III) nitrate nanohydrate $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$, nickel nitrate hexahydrate $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$, polyethylene glycol (PEG) 400, lead nitrate $(\text{PbNO}_3)_2$, sodium hydroxide (NaOH) are purchased from Merck. Chitosan, bentonite and Eriochrome black T are purchased from Sigma-Aldrich. All chemicals were used without further purification.

2.2. Preparation of Nickel ferrite

Nickel ferrite $(\text{NiFe}_2\text{O}_4)$ nanoparticles were synthesized via

co-precipitation followed by calcinations in muffle furnace. The synthetic procedures required analytical grade 4 M solution of sodium hydroxide (NaOH) which was slowly added to salt solutions of 5 mM ferric nitrate $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$ and 2.5 mM nickel nitrate $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$. The pH of the solution was adjusted to 12 by drop wise addition of NaOH solution. 1 gm (2.5 mM) of PEG 400 was added to the solution as the stabilizing agent. The precipitate so formed was washed with distilled water and ethanol and dried in an oven at low temperature. The dried precipitates were finally calcined in a muffle furnace at 500°C for 5 h.

2.3. Preparation of Bentonite/chitosan/ NiFe_2O_4 Ternary Nanocomposite

The ternary nanocomposite was prepared in two phases. First, binary composite of nickel ferrite and chitosan was prepared which was the further transformed to ternary composite by adding bentonite to this binary composite. In a typical experiment 0.5% W/V chitosan solution was prepared by stirring in a 1% V/V aqueous acetic acid solution at room temperature. The solution was kept for overnight and then centrifuged [23]. To this solution 70 mg of finely powdered nickel ferrite was added. The mixture was sonicated for 30 minutes to get uniform dispersion. Finally the solid binary mixture was isolated by centrifugation. In a separate experiment 50 mg of bentonite was dissolved in 50 mL of deionised water and stirred magnetically. To this solution the solid binary composite was added and stirred for 12 h. Finally the ternary composite is collected by centrifugation.

2.4. Spectrophotometric Investigation of Lead Adsorption by the Composite

Standard lead solution of three different concentrations 600 ppm, 400 ppm, 200 ppm was prepared using lead nitrate and different amount of composite was added to the solution. The composite and lead nitrate solutions were thoroughly agitated in a mechanical shaking machine and were removed after 24 h contact time by filtration using whatman 40 filter paper. The absorbance of each solution was monitored using Eriochrome black T as complexing indicator in basic medium.

3. Characterization of Synthesized Materials

FT-IR analysis of synthesized nickel ferrite was carried out on IR Affinity-1 M/S. Shimadzu, Japan. X – ray diffraction analysis (XRD) of the nickel ferrite and the ternary composite was carried out on Xpert³ MRD XL with $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) with a scan speed of $2^\circ/\text{min}$. Transmission electron microscope (TEM) image were obtained by JEM-100 CX II (20-100 KV in 20 KV steps). The magnetic property was analyzed using vibrating sample magnetometer (VSM, Lakeshore-7,410 series). The spectrophotometric investigation of the lead removal from water by the composite was carried out using UV- visible spectrophotometer (JASCO-V730). EDX of the used composite after lead removal was performed on OXFORD XMX N equipment.

4. Results and Discussion

4.1. FTIR Studies

FT-IR spectrum as shown in the Fig. 1 confirmed the formation of spinel nickel ferrite structure in the prepared nickel ferrite. The broad band at 3443.3 cm^{-1} corresponds to the O-H stretching vibration of water molecule of moisture absorbed in the sample. The two prominent bands at 566.3 cm^{-1} and 633.5 cm^{-1} are assigned to the intrinsic stretching vibration of Metal – Oxygen bond in the tetrahedral and octahedral site of the spinel ferrite respectively [24]. The band at 1386.2 cm^{-1} attributed to a stretching vibration of the C-O-C ether band, which indicates the presence of $-\text{CH}_2\text{-O-CH}_2-$ group in poly ethylene glycol (PEG) [25]. The band at 1462.1 cm^{-1} corresponds to the vibration mode of O-H band.

Fig. 1. FT-IR spectra of nickel ferrite.

4.2. XRD Studies

Powder X-ray pattern of nickel ferrite and ternary composite is shown in the Fig. 2. The XRD pattern shows that nickel ferrite is formed in a single phase spinel structure. The diffraction peaks indexed at [311], [400], [440] reflection plane corresponds to 2θ angle 32° , 45.5° and 75.5° respectively. The average crystallite size calculated by using Scherer's equation [26] was at was found to be 28 nm. Considerable broadening of the peaks attributed to the presence of very small crystallites in the composite. Chitosan is a semi crystalline polymer which has characteristic scattering peak at around 19° to 22° in accordance with the literature [27] matched with the present findings. The presence of bentonite in the composite is confirmed by two peaks, one most intense peak at around 27° and another at low intensity around 51° which are also supported by the literature [28]. The most intense and prominent peak of nickel ferrite corresponds to (311) plane shifted at slight higher wave length with subsequent decrease in the intensity. This is attributed to introduction of amorphous component in the composite.

Fig. 2. Powder XRD pattern of nickel ferrite and ternary composite.

4.3. VSM Studies

The hysteresis curve obtained from vibrating sample magnetometry is shown in Fig. 3. The magnetic parameter namely Coercivity (H_{ci}), Magnetization (M_s) and Retentivity (M_r) are found to be 145.66 Oe, 0.33,830 emu/ g and 16.272 emu/ g respectively. The magnetic property of the composite is compared with the standard hysteresis curve of nickel ferrite. The data obtained confirmed the soft ferromagnetic character of the composite. Relatively low value of saturation magnetization (M_s) may be due to the composite formation with non magnetic chitosan polymer and bentonite present in the composite which binds the outsized surface area of

Fig. 3. Hysteresis curve of composite obtained from VSM analysis.

nickel ferrite [29]. Also the decline in the value of the coercivity (H_c) may be due to decrease in surface anisotropy [30]. The relatively low value of magnetization indicates that the separation of composite will not be possible by the external magnetic field.

4.4. TEM, HR-TEM and SAED Studies

The morphology and structure of the composite was investigated by transmission electron microscopy. The TEM image of the composite is shown in the Fig. 4

Fig. 4. TEM images of the nano composite. (a), (b) HR-TEM, (c) and SAED pattern (d) of the nanocomposite.

The Fig. 4(a), (b), shows chitosan - bentonite encapsulated nickel ferrite particles is almost spherical with average particle size 30 to 40 nm. The HR-TEM image of the composite is displayed in Fig. 5(c). The SAED pattern confirms the crystalline nature of the composite. Lattice fringes 0.340 nm corresponds to the [211] plane of bentonite, 0.437 nm corresponds to the [110] plane of chitosan and 0.254 nm corresponds to the [222] plane of nickel ferrite. These three significant planes in this composite confirm the ternary nature of the composite.

4.5. Removal of lead by Bentonite/Chitosan/ NiFe_2O_4 Ternary Nanocomposite

The spectrophotometric analysis of lead removal by the composite from water was carried out using Eriochrome black T as complexing agent. The absorbance of the different lead solution was monitored and the results are analyzed.

The absorption of the 600 ppm lead solution was found to be 0.0,181 at 566 nm. However for the same solution from which lead is removed by using 150 mg composite, the absorption noticeably increases to 0.0,223 at same wave length. Similarly for other two solution of same concentration from which lead is removed

by using 100 mg and 50 mg of composite, the absorption was found to increase. This sudden increase in absorbance of lead solution having 150 mg of composite confirms the decrease in concentration of lead in the solution and proportionate increase in the concentration of complexing reagent Eriochrome black T. Similar trends of result is observed in case of 400 ppm and 200 ppm lead solution. The percentage of Pb (II) removal was calculated by using the Eq. (1).

$$\text{Removal Efficiency (\%)} = \left(\frac{C_0 - C_e}{C_0} \right) \times 100 \quad (1)$$

Where C_0 and is the initial concentration of the Pb solution and C_e is the equilibrium metal concentration after removal. It is observed that with the decrease in the composite amount the efficiency of Pb (II) removal increase. This is due to aggregation of adsorbent and consequently the available adsorption sites decreases gradually [31]. In case of 200 ppm solution maximum amount of lead removal was achieved when 50 mg of the composite was used in 100 mL of the solution. The overall trend of the lead removal indicated that composite dose should be optimum. Similar trends were observed in reported literatures [32, 33]. The graphical representation of percentage of lead removal against the composite dose for three different solutions is shown in Fig. 5.

Fig. 5. Graphical illustration of percentage of lead removal against composite dose for different solution (B. 600 ppm, C. 4,000 ppm, D. 200 ppm).

Clearly, by intensifying the adsorbent doses, the removal efficiency diminished and this may be because of the fact that every single active site was completely uncovered at lower doses, while just a small amount of the dynamic sites were uncovered at higher dosages [34]. Hence, higher adsorbent doses may cause accumulation, which diminishes the all-out surface area of adsorbent, prompting a reduction in adsorption [35].

4.6. Comparison with Other Composite for Pb^{2+} Removal from Aqueous Solution

Ternary nanocomposite based on cobalt ferrite is not yet inves-

Table 1. Comparison of Performance of Nickel Ferrite/ Chitosan/ Bentonite Ternary Composite with Some Reported Adsorbent for Lead Removal from Aqueous Solution

Sl. No	Composite	Dose	Time	Efficiency	Reference
1.	Cobalt Ferrite / Zeolite	100 mg/L	24 h	98%	[36]
2.	Manganese Ferrite/ Biochar	0.25 g/L	24 h	96%	[37]
3.	Nickel Ferrite/ SiO ₂	300 mg/L	24 h	87%	[38]
4	Nickel Ferrite/ Chitosan/ Bentonite	50 mg/100mL	24 h	82%	This Work

tigated too much particularly to contemplate the lead removal proficiency from aqueous solution. Some binary nanocomposite of metal ferrite are accounted for which has superior proficiency in lead evacuation [36, 37, and 38]. Our designed ternary nanocomposite based on cobalt ferrite is novel in this regard. Some reported works in lead removal are listed herewith.

Ternary nanocomposite based on cobalt ferrite is not yet investigated too much particularly to contemplate the lead removal proficiency from aqueous solution. Some binary nanocomposite of metal ferrite are accounted for which has superior proficiency in lead evacuation. Our designed ternary nanocomposite is novel in this regard.

4.7. Confirmation of the Removal of Pb (II) by EDX Studies

The exhausted composites were analysed by EDX method, which confirm the presence of lead in the composite. This justify that the composite efficiently removed lead ion from the aqueous solution. The EDX pattern of the exhausted composite is shown in Fig. 6.

Fig. 6. EDX spectrum of the exhausted composite.

5. Conclusion

In summary, we reported the synthesis of nickel ferrite – chitosan – bentonite ternary nanocomposite. The morphology and magnetic property of the composite were well established from VSM and TEM analysis. XRD analysis is also in good agreement with the composite formation. EDX analysis of the exhausted composite shows the presence of lead indicating its performance as efficient adsorbent of Pb (II) from water. The optimum concentration of the composite is 50 mg/100 ml, where the removal efficiency is 82%. Such composite material may prove to be useful for removal of lead from aqueous solution at industrial level.

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Author Contributions

D.G.T. (Ph.D Student) has conducted all experiments and prepare the manuscript, A.P. (Ph.D Student) helps in characterization process, K.C.D. (Ph.D Student) revised the manuscript along with correspondence and S.S.D. (Associate Professor) helps in entire supervision.

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