

Eco-friendly Magnetic Biopolymer Nanocomposites for Removal of Organic Dye/Heavy Metals from Waste Water

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ABSTRACT

Chitosan is studied as a natural polysaccharide which has a remarkable unique combination of chemical, physical and mechanical properties. Chitosan, an amino polysaccharide obtained from deacetylation of chitin, is a natural polymer that has a plethora of applications in various fields including water purification, drug designing, cosmetics, food and packaging etc. Being a natural polymer it has less residual impact on the environment, and hence serves as a potential alternative to the chemically hazardous precursors. The present work focusses on deploying the useful chemical properties of chitosan to design a solution for removal of organic dye such as methylene blue and heavy metals from industrial waste water. In order to achieve this, a composite system consisting of chitosan, magnetic ferrite (Fe_3O_4) nanoparticles and graphene oxide was synthesized to obtain the synergistic combination of the desired properties. The ferrite nanoparticles were synthesized by alkalisation of Fe^{+2} and Fe^{+3} species using co-precipitation method in the presence of a suitable surfactant. The CFG (chitosan/ Fe_3O_4 nanoparticles/graphene oxide) nanocomposite was synthesized through a facile chemical route. The characterization of the synthesized composite was carried out using X-ray diffraction (XRD), Vibrating Sample Magnetometer (VSM) and microstructural analysis. Adsorption ability of CFG nanocomposite for methylene blue, potassium dichromate and cobaltous chloride was evaluated by using UV-Visible spectroscopy studies.

Key words: Chitosan, Fe_3O_4 nanoparticles, graphene oxide, water treatment.

1. INTRODUCTION

Water, the blue liquid- the backbone of all forms of life, has been sustaining the environment and supporting beings as long as life can be traced back on earth. However, it is a matter of great concern that, this wonderful blessing is depleting at an alarming rate. In the early 21st century, prospects of human development are threatened by a deepening global water

crisis (Seckler *et al.*, 1999). About 71% of the global population (4.3 billion people) face moderate to severe water scarcity at least once a year. About 66% (4.0 billion people) lives are under acute water scarcity at least once a year. Of these 4.0 billion people, about 25% people live in India and another 0.9 billion live in China. By 2025, half of the world's population will be marked as living in water-stressed areas. Such regions do not have enough water resources to maintain 1990 levels of per capita food production from irrigated agriculture, despite having high levels of irrigation efficiency, in addition to meeting reasonable water needs for domestic, industrial and environmental purposes by 2025. People living in these regions will have no other choice but to reduce water use in agriculture and transfer it to other sectors, reducing domestic food production and resulting in more food import.

This depletion is mainly caused by the contamination of available water resources. Industrial waste products released into lakes, rivers, and other water bodies are an important reason for the problem (Singh *et al.*, 2018, Akpor *et al.*, 2014; Saidi, 2010). Industrial waste comprises of waste produced by industrial activity which includes any material that is rendered useless during a manufacturing process such as that of factories, industries, mills, and mining operations. Various waste materials are chemical solvents, paints, sandpaper, paper products, industrial by-products, heavy metals, and radioactive wastes (Goodwill & Somtochukwu, 2017). This research work solely deals with designing of an efficient technique for the removal of heavy metals/organic dyes from wastewater to make it suitable for further use.

The term 'Heavy metal' refers to any metallic element that has a relatively high atomic density, at least five times the specific gravity of water, and is toxic or venomous even at lower concentrations (Sutirman *et al.*, 2018; Pugazhenthiran *et al.*, 2016). Heavy metals include lead (Pb), cadmium (Cd), zinc (Zn), mercury (Hg), arsenic (As), silver (Ag), chromium (Cr) etc. Even though heavy metals are natural components of the earth's crust, they are a major cause of environmental pollution. Heavy metals are introduced in the ecosystem via a vast range of processes including agricultural and industrial activities, waste disposal and even through some natural processes like weathering of rocks and volcanic eruptions. When the concentrations of these metals go over the permissible limits in the ecosystem, they become hazardous to living organisms. Once high concentrations of heavy metals enter the food chain, they tend to accumulate in living tissues and cause serious health issues such as organ damage, reduced growth and development, etc. Hence, it is important to minimize these heavy metals from water sources in the interest of public health and the environment.

Till now various methods have been used for the removal of heavy metals from wastewater including chemical oxidation, chemical precipitation, ion exchange, reverse osmosis, membrane separation, electro dialysis, etc. (Barakat, 2011). Unfortunately, majority of them suffer from significant disadvantages like high capital and operational costs, production of toxic sludge, incomplete removal, high energy inputs (Fu & Wang, 2011). The negative impact of these materials on the environment cannot even be estimated correctly. Therefore, environment-friendly solutions having low-cost capital investment and less adverse environmental impact should be adopted. Among various approaches adopted so far, adsorption method of removal of toxic wastes from waste water has an edge as it offers advantages such as low costs, removal of different types of pollutants, less chemical use, etc. (Mehdinia *et. al.*, 2015).

Chitin a naturally abundant mucopolysaccharide is a white, inelastic, hard and nitrogenous compound that is a by-product of the fishery industry and is considered as a regenerating raw material which is only second to cellulose in terms of abundance (Miretzky & Cirelli, 2009). Its natural abundance amounts to more than 1000 tons per year and about 70% of which comes from marine species. Chitosan, the main derivative of chitin, is a polysaccharide found in shells of shrimp and other crustaceans (Islam *et. al.*, 2016).

The chemical structure of chitin and chitosan is quite identical to that of cellulose which consists of several hundreds to more than thousand β -(1-4) linked D-glucose units (Figure 1). The hydroxyl group at C-2 position of cellulose is replaced by acetamide group in the structure of chitin and chitosan. Chitosan, β -(1-4) linked 2-amino-2-deoxy- β -Dglucopyranose, is an N-deacetylated derivative of chitin obtained by transforming the acetamide groups into primary amino groups (Marroquin *et. al.*, 2013).

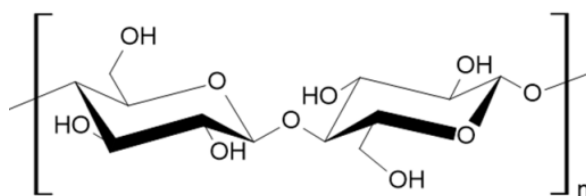


Figure 1: Chemical structure of cellulose polymer

However, deacetylation of chitin is more or less never complete and chitosan or deacetylated chitin still contains acetamide groups to some extent. Unlike cellulose, chitin and chitosan contain 5–8 % nitrogen, which in chitin is in the form of acetylated amine groups and in chitosan, in the form of primary aliphatic amine groups, which makes chitin and chitosan

suitable for various reactions of amines. However, chitosan is chemically more reactive than chitin due to the presence of primary and secondary hydroxyl groups on each repeat unit, and the amine group on each deacetylated unit (Figure 2).

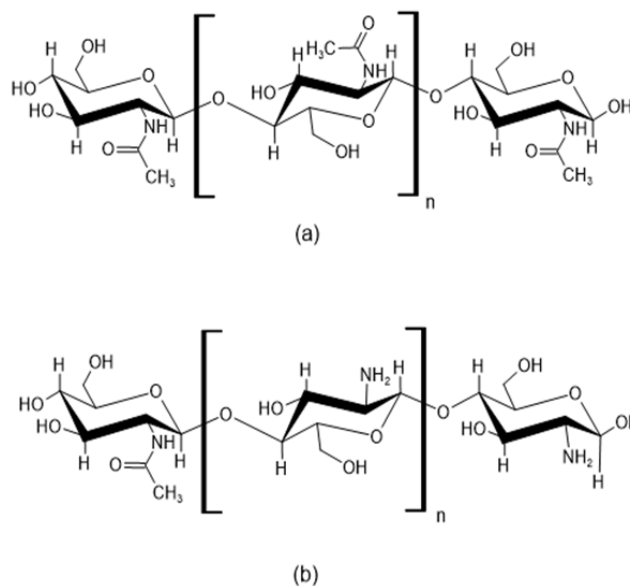


Figure 2: Structural differences between (a) chitin and (b) chitosan

Chitosan has emerged as the most versatile biopolymer for a broad range of applications involving water treatment due to its biodegradability, biocompatibility and anti-bacterial properties. Biopolymers, because of their ability to lower transition metal ion concentrations to sub-part per billion concentrations, are becoming appealing to industries. Another striking feature of biopolymers is that they have a number of functional groups like hydroxyl and amine groups which act as chelating and reaction sites (Tran *et. al.*, 2017).

Another major quantum leap in the field of wastewater treatment is from the field of nanotechnology, which provides an opportunity for effective water purification by developing advanced materials (Abbas *et. al.*, 2015). Nanoparticles, due to their small object effect and quantum size effect have superior properties as compared to bulky materials. Fe_2O_3 (magnetite) is one of the magnetic nanoparticles. Fe_2O_3 , can be shown as $(\text{FeO} \cdot \text{Fe}_2\text{O}_3)$. Magnetite differs from most other iron oxides since it contains both Fe^{2+} and Fe^{3+} ions. Fe_3O_4 has a cubic inverse spinel structure that consists of a cubic close packed array of oxide ions, where tetrahedral site is occupied by Fe^{3+} ions surrounded by four O atoms, while octahedral site is occupied with both Fe^{2+} and Fe^{3+} ions surrounded by six oxygen atoms. Investigations have shown that Fe_2O_3 magnetic nanoparticles are superparamagnetic and have high a coercive nature and low Curie temperature. They are also non-toxic and biocompatible

which makes them immensely suitable for water treatment. Nano composites are the new age smart solutions with a unique combination of properties (Ma *et al.*, 2014; Chaki *et al.*, 2015). The main advantage of using magnetic nanoparticles is that, these can be recovered efficiently to be used innumerable times, without any appreciable loss of material.

In recent years, significant attention has been focused on graphene based nanomaterials because of their great potential in applications such as catalysis, enzyme adsorption, biosensors and even water treatment (Lee *et al.*, 2013). Graphene is an intercalation compound of graphite and is used in wastewater treatment owing to its structure and antimicrobial properties. In particular,

Graphene Oxide (GO) chemically exfoliated from oxidised graphite is considered to be a promising material, exhibiting excellent mechanical, electrical and thermal properties (Geim & Novoselov, 2007).

Adsorption and filtration are two methods in which graphene oxide can be used in water purification. Magnetic separation technology is also being widely used in the fields of separations and adsorptions nowadays. The introduction of magnetic nanoproperties into graphene or GO will combine their high adsorption capacity and the separation convenience of the magnetic materials. The preparation of graphene-based magnetic nanocomposite has arisen as a next generation resources with versatile applications for the removal of various toxic elements and compounds (Rodil *et al.*, 2009). Another way graphene finds use in wastewater treatment is in the form of nanosheets used for filtration which possess good tensile strength. It can be used to prepare nanosheets of various pore sizes (Li *et al.*, 2008). Graphene-based materials act as adsorbents for numerous pollutants present in wastewater owing to their large surface area and other properties as well (Yang *et al.*, 2018). Magnetic biopolymer nanocomposites incorporate the exceptional features of both nanoparticles and biopolymer: High interfacial reactivity of nanoparticles, outstanding compatibility and mechanical properties owing to biopolymer matrix, and also potential to regeneration and reuse.

In view of the above, an attempt has been made to design a composite system for the removal of heavy metals from waste water samples. The reported work illustrates the detailed synthesis steps of the formation of chitosan/Fe₃O₄ nanoparticles/graphene oxide composite. The schematic details of the synthesis steps are included in the text.

2. MATERIALS AND METHODS

2.1 Experimental procedure for the synthesis of ferrite nanoparticles

For the synthesis of ferrite magnetic nanoparticles, inorganic salts of Fe^{2+} and Fe^{3+} were used. The two salts used were ferrous sulphate (FeSO_4), and ferric chloride (FeCl_3) taken in a molar ratio of 2:1 respectively. Ferrous sulphate and ferric chloride in required amounts were taken in separate beakers and dissolved completely in 100 ml of distilled water. The two solutions were mixed in a separate beaker, and this mixture was magnetically stirred. A reddish-brown solution obtained after mixing the two solutions indicated complete mixing of the two solutions. The pH was found to be around 6.5-7.0. To incorporate alkaline nature in the solution NH_4OH solution was added drop-wise to the solution with continuous stirring. The colour of the solution gradually changed from light brown to jet black and pH value reached 11. After the required pH was obtained, Sodium Lauryl Sulfate was added to homogenise the medium. The black ferrite particles were magnetically separated and washed repeatedly with distilled water and until pH was 7 (Figure 3). With each washing, the pH and conductance of the supernatant liquid was measured. After washing of the nanoparticles was completed, the nanoparticles were dried in an oven at 60°C . The nanoparticles were kept in desiccator for further use.

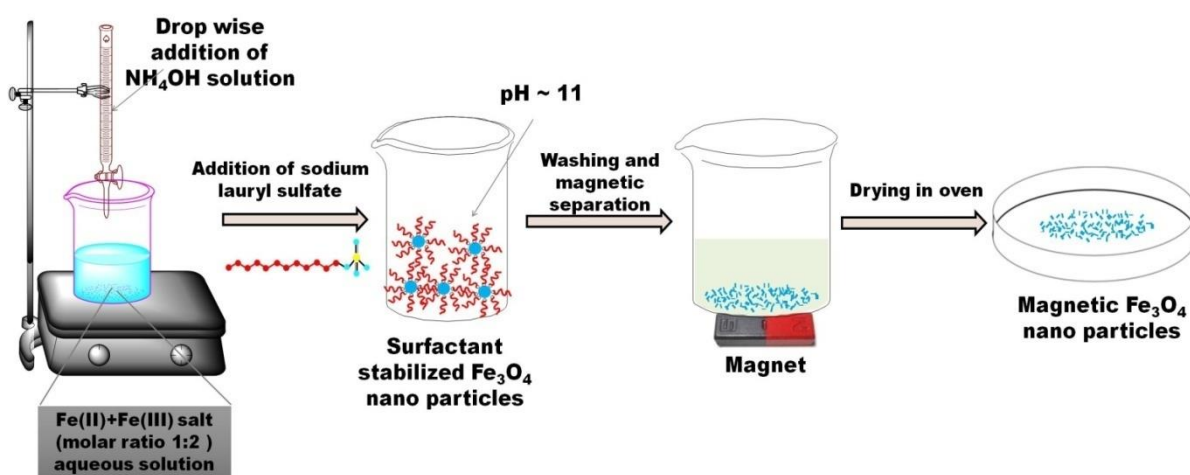


Figure 3: Schematic representation of synthesis of Fe_3O_4 nanoparticles using co-precipitation method

2.2 Experimental procedure for the synthesis of chitosan solution

Chitosan is a natural polycationic linear polysaccharide derived from chitin. Its solubility in neutral and alkaline solution is very less, therefore for the preparation of chitosan solution, acidic conditions were used. Chitosan solution was prepared by the dissolution of 1 wt% of chitosan in acetic acid solution. The solution was stirred vigorously with the help of a magnetic stirrer, and the stirring was continued overnight to ensure complete mixing, and to obtain a pale coloured clear solution of chitosan (Figure 4).



Figure 4: Aqueous Chitosan solution obtained after overnight stirring at room temperature

2.3 Experimental procedure for the synthesis of CFG (Chitosan/Fe₃O₄/Graphene oxide) nanocomposite

CFG (Chitosan/Fe₃O₄/graphene oxide) nanocomposite was synthesized using ferrite nanoparticles, chitosan solution and stable dispersion of graphene oxide in water. The aqueous suspension of Fe₃O₄ nanoparticles were homogenized in an ultrasonicator for about 20 minutes, followed by the addition of these nanoparticles to the chitosan solution under vigorous stirring conditions. The stirring continued for about 30 minutes, after which the aqueous suspension of graphene oxide was added drop-wise.

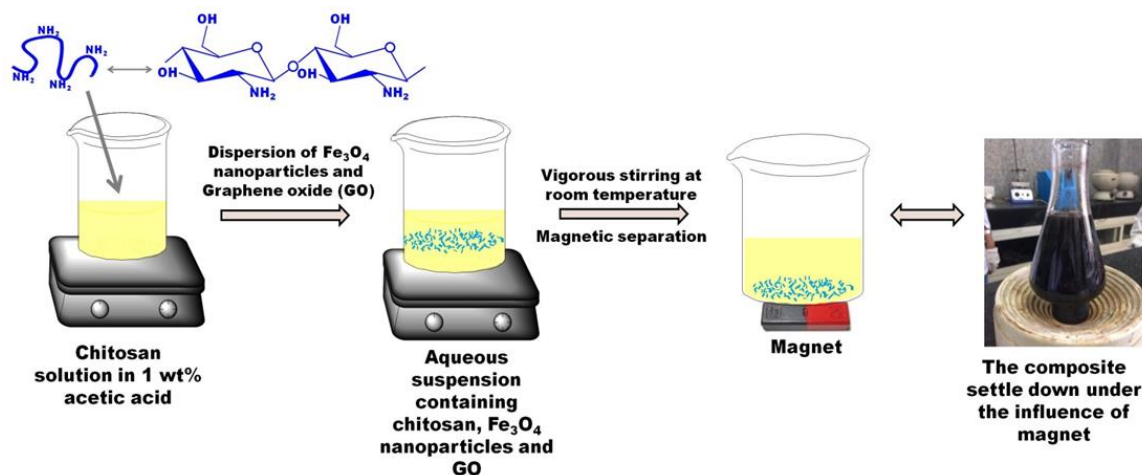


Figure 5: Scheme showing synthesis of Chitosan/Fe₃O₄/graphene oxide nanocomposite

The stirring process was continued for about 3 hours to ensure proper mixing of the constituents of the composite. The nanocomposite particles are retrieved by magnetic separation method. The particles were dried at room temperature, followed by heating at 60°C in an oven. The scheme of synthesis of CFG nanocomposite is shown in Figure 5.

3. RESULTS AND DISCUSSION

The synthesized CFG nanocomposites were characterized using X-Ray diffraction (XRD) technique, vibrating sample magnetometer (VSM) analysis, Transmission Electron Microscopy (TEM) and UV-visible spectroscopy. Adsorption studies with the waste water samples containing different contaminants such as methylene blue, Co (II) ions and Cr VI ions are also carried out using UV-visible spectroscopy technique.

3.1 X-Ray Diffraction analysis

Figure 6 shows the X-ray diffractogram of the synthesized Fe₃O₄ nanoparticles. Diffraction peaks at 2θ (2 Theta) values at 30.1°, 35.7°, 43.2°, 53.6°, 57.2°, 62.7° and 74.3° corresponding to d_{hkl} planes (d is the interplanar distance and h, k, l are integers) 220, 311, 400, 422, 511, 440, 533, respectively match with the standard pattern of Fe₃O₄. The crystallinity of the samples is indicated by sharper diffraction peaks at respective diffraction angles. The average particle diameter was also calculated using Debye-Scherrer formula and the average size of the ferrite particles was found to be 26.7 nm.

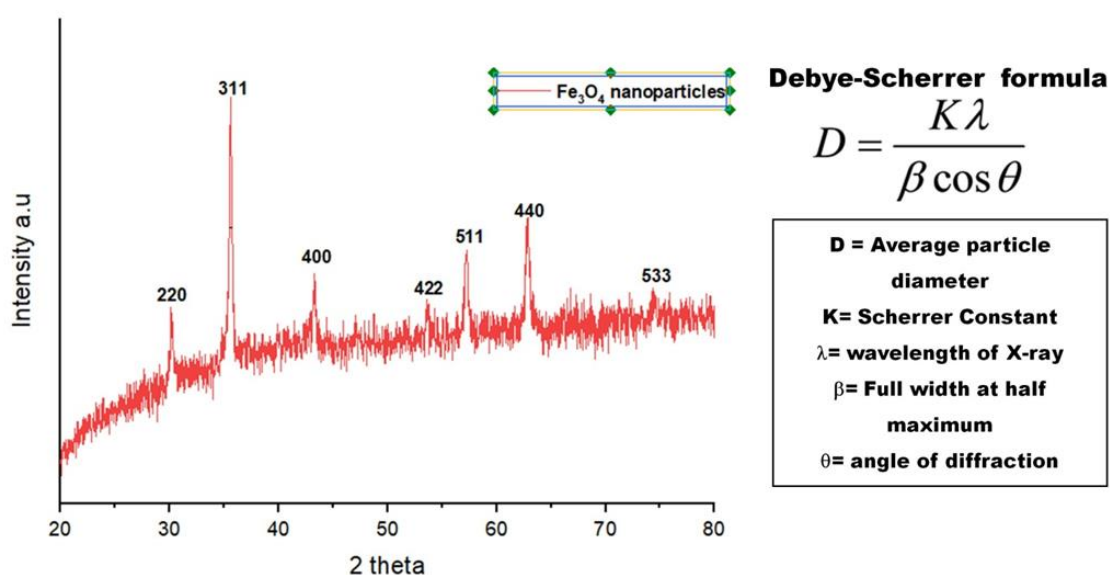


Figure 6: X- Ray Diffraction pattern of Fe₃O₄ nanoparticles

3.2 Vibrating Sample Magnetometer (VSM) Analysis

Figure 7 shows the VSM curves of Fe₃O₄ nanoparticles and CFG composite. The VSM studies exhibit the magnetic properties of Fe₃O₄ and CFG nanocomposite. The M-H loop shows saturation magnetization of Fe₃O₄ nanoparticles, 74.9 emu/g. However, incorporation of Fe₃O₄ nanoparticles in chitosan/GO matrix, the saturation magnetization of CFG composite is reduced to 61.5 emu/g. This is attributed to decrease in the degree of magnetization of magnetic Fe₃O₄ nanoparticles when present in the non-magnetic chitosan/GO

matrix. The amount of saturation magnetization in the composite is sufficient for the smooth recovery of nanocomposite from the water sample through an external magnet.

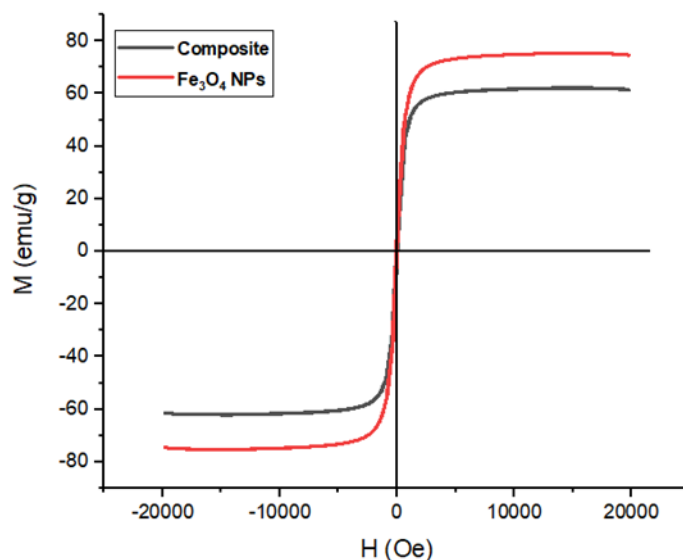


Figure 7: Vibrating Sample Magnetometer (VSM) curve of Fe₃O₄ nanoparticles and CFG nanocomposite

3.3 Microstructural Analysis

Figure 8 shows the Transmission Electron Microscopy (TEM) micrograph of CFG composite. The micrograph of CFG composite indicates that Fe₃O₄ particles are found to be uniformly and well dispersed in chitosan/GO matrix which suggests good compatibility between Fe₃O₄ particles and bio-organic chitosan/GO matrix as shown in TEM (Figure 8).

3.4 UV-Visible spectra of untreated and treated samples

CFG (Chitosan/Fe₃O₄/GO) nanocomposite is found to be an excellent adsorbent material for the removal of organic cationic dye such as methylene blue. Figure 9 shows the UV-visible spectra of untreated and treated solution of methylene blue. UV-visible spectra of untreated solution of methylene blue showed two absorption peaks at around 665 nm, which is due to the presence of chromophores and auxochrome and 296 nm due to $\pi-\pi^*$ transition (extend of conjugation length) in methylene blue molecules. The intensity of these peaks was significantly decreased (hypochromic shift) when the methylene blue solution was treated with CFG nanocomposite.

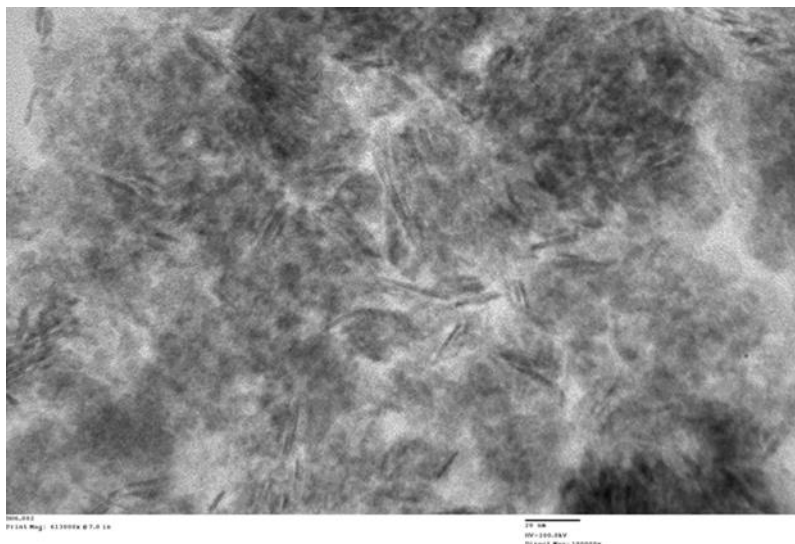


Figure 8: Transmission Electron Microscopy (TEM) micrograph of CFG composite

The UV -Visible spectrum of the treated solution clearly indicates the occurrence of adsorption phenomenon. The change in the intensity of the colour is also evidenced by the inset photographs in Figure 9. It was found that graphene oxide in CFG nanocomposite, contains many oxygen containing functional groups such as carboxyl ($-\text{COOH}$), hydroxyl ($-\text{OH}$) and ketone ($>\text{C}=\text{O}$) and it is hydrophilic in nature with a large negatively charged surface that supports the effective adsorption of cationic dye such as methylene blue.

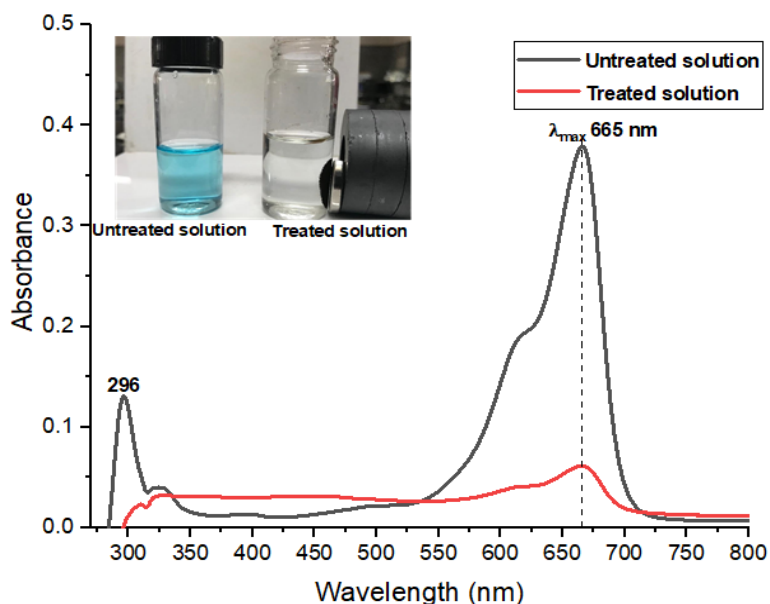


Figure 9: UV-visible spectra of untreated and treated solution of methylene blue

Similarly, CFG nanocomposite was also employed for adsorption of Co(II) , and Cr(VI) ions. UV-visible spectra of untreated solution of cobalt chloride revealed the characteristic

UV visible absorption peaks at around 514 nm, while intensity of this peak significantly decreased when the cobalt chloride solution was treated with CFG composite (Figure 10).

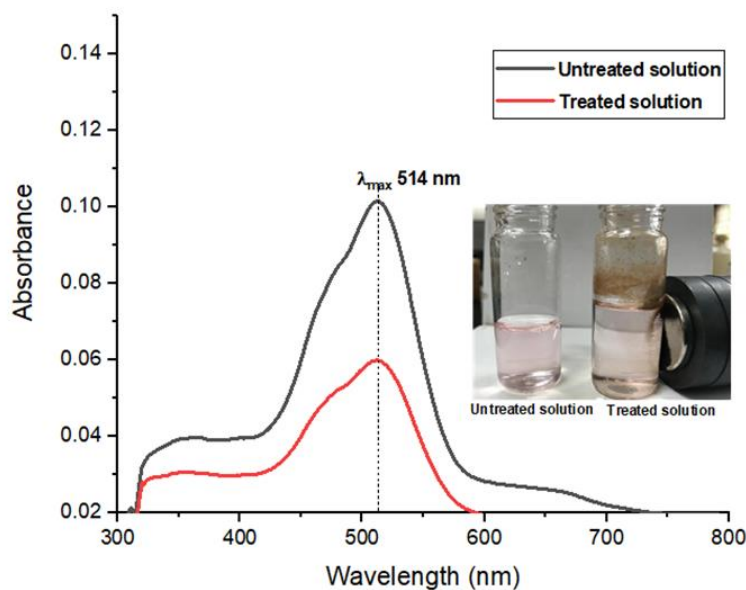


Figure 10: UV-visible spectra of untreated and treated solution of cobalt (II) chloride

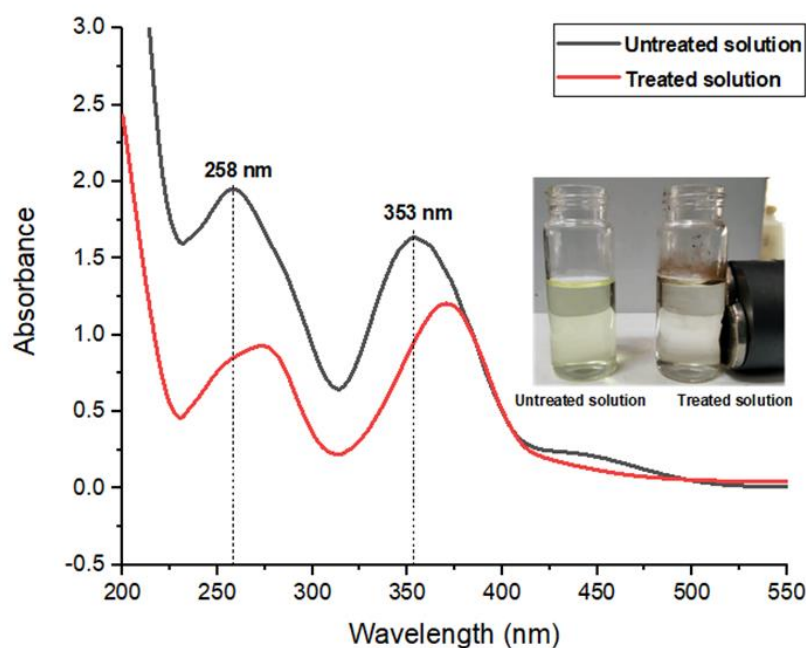


Figure 11: UV-visible spectra of untreated and treated solution of potassium dichromate

Results clearly indicate the adsorption of cobalt ions by CFG nanocomposites. Moreover, when the solution of potassium dichromate ($K_2Cr_2O_7$) was treated with CFG nanocomposites, the intensity of UV-visible absorption peaks (observed at 258 nm and 353 nm) was also found to decrease (Figure 11). In addition to this, shifting of absorbance peaks

towards higher wavelength (bathochromic shift) was also noticed in UV-visible spectra of CFG treated potassium dichromate solution. This could be due to some chemical interaction or complex formation phenomenon during the adsorption process.

4. CONCLUSION

Magnetic Fe₃O₄ nanoparticles were successfully synthesized using co-precipitation method. Synthesis of CFG (Chitosan/ Fe₃O₄/graphene oxide) nanocomposite was carried out in the presence of ferrite nanoparticles, chitosan solution and graphene oxide solution. Formation of ferrite nanoparticles was confirmed by XRD studies. The magnetic behaviour of ferrite nanoparticles was studied by VSM analysis. VSM studies also revealed the formation of magnetic CFG nanocomposite as the magnetic property was introduced in chitosan/GO matrix by incorporation of magnetic ferrite nanoparticles. Compatibility of Fe₃O₄ particles with bio-organic chitosan/GO matrix was indicated by TEM studies of CFG nanocomposites. The adsorption behavior of synthesized CFG nanocomposite was evaluated by UV-visible spectroscopy and the results show that CFG nanocomposites exhibited excellent adsorption properties with methylene blue dye, potassium dichromate and cobalt (II) chloride solutions. The nanocomposite was efficiently recovered by using a simple magnet. These studies clearly reveal that the magnetic CFG nanocomposite has excellent adsorption property for methylene blue and toxic heavy metals such as cobalt and chromium ions and it can be considered as a potential material for removal of cationic organic dyes and heavy metals from waste water.

5. CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding this research article.

6. SOURCE OF FUNDING

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