SYNTHESIS & CHARACTERIZATION OF IRON OXIDE NANOPARTICLES AND THEIR APPLICATION IN THE TREATMENT OF INDUSTRIAL WASTEWATER

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5. Introduction/Background: Nanoscience is one of the most important research and development frontiers in modern science. Nanotechnology is now widely used throughout the pharmaceutical industry, medicine, electronics, robotics, and tissue engineering. The use of nanoparticles offers many advantages due to their unique smaller size. The heavy metal pollution is becoming one of the most serious environmental problems. Therefore various methods for heavy metal removal from wastewater have been extensively studied during the past decades, such as chemical precipitation, electrochemical techniques, membrane filtration, ion exchange, and adsorption. To date, considerable research attention has been paid to the removal of heavy metals from contaminated water via adsorption process. In theory, the adsorption process can offer flexibility in design and operation and in many cases will produce high-quality treated effluent. In addition, as the adsorption is sometimes reversible and adsorbent can be regenerated by suitable desorption process, various types of adsorbents have found application in the removal of heavy metals, including activated carbon, carbon nanotubes, polymeric adsorbents, metal oxides, and bio-adsorbents. Among these adsorbents, iron-based magnetic nanoparticles have distinguished themselves by their unique properties, such as larger surface area-volume ratio, diminished consumption of chemicals, and no secondary pollutant. However, with another special property of this kind magnetic materials are realized and utilized in the context of environmental remediation. Iron oxides exists in many forms in nature, with magnetite (Fe₃O₄), hematite (α-Fe₂O₃) and maghemite (γ-Fe₂O₃), being most probably common and important technologically. It has also been noted that the
adsorption capacities of adsorbents rely largely on the available surface areas, and the increase of the surface area is normally obtained by the decrease of the particle size of adsorbents. As a result, there is a need to synthesize such absorbents with proper particles sizes for the removal of heavy metals from industrial wastewater. Up to now, there are several methods that can be used to synthesize iron-oxide-based nanoparticles. These methods include hydrothermal synthesis, thermal decomposition, co-precipitation, sol-gel method and colloidal chemistry method. Among these synthesis methods, co-precipitation has proven to be the most promising method for the production of nanoparticles as the procedure is relatively simple and the particles can be obtained with controlled particle size.

6. Objectives:
- To synthesize Iron oxide Nanoparticles using Co-precipitation method.
- To characterize these nanoparticles using Scanning Electron Microscopy(SEM), X-ray Diffraction(XRD) and Fourier Transform Infrared spectroscopy(FTIR)
- After synthesis & Characterization of iron oxide nanoparticles, these are used as adsorbents for the removal of toxic heavy metals (such as Cu(II), Cr(VI)) from their synthetic solution of wastewater.
- To study the effect of process parameters like pH, temperature, iron oxide dosage, contact time and initial heavy metal ion concentration.
- To test which adsorption isotherm (Langmuir or Freundlich) fits the data.

7. Methodology:
- Iron oxide nanoparticles are synthesized using Co-precipitation method.
  - This method is probably the simplest and most efficient chemical pathway to obtain magnetic nanoparticles. Iron oxides (Fe₃O₄) are usually prepared by dissolving stoichiometric mixture of ferrous (FeSO₄) and ferric (FeCl₃) salts in aqueous medium with a ratio of 2:1 (Fe³⁺/Fe²⁺). 100ml of 0.4 mol/L solution of FeCl₃ and 100ml of 0.2mol/L solution of FeSO₄ were mixed and dissolved in distilled water. Then 2 mol/L of Sodium hydroxide(NaOH) was added into the above solution and the pH value was maintained between 10-11 with continuous stirring using a magnetic stirrer for 1 hour and a dark precipitation was formed.
  - The produced precipitation was filtered & after drying at 150° C for 2 hours the powder will be obtained. Particles with sizes ranging from 5 to 100 nm will be obtained using this method. The chemical reaction of Fe₃O₄ formation may be written as
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    \text{Fe}^{2+} + 2\text{Fe}^{3+} + 8\text{OH}^- \rightarrow \text{Fe}_3\text{O}_4(s) + 4\text{H}_2\text{O}
    \]
  - After the synthesis of iron oxide nanoparticles, it was characterized using Scanning Electron Microscopy(SEM), X-ray Diffraction(XRD) and Fourier Transform Infrared spectroscopy(FTIR)
  - After synthesis & Characterization of iron oxide nanoparticles, these are used as adsorbents for the removal of toxic heavy metals (such as Cu(II), Cr(VI)) from their synthetic solution of wastewater.
Batch experiment will be carried out at room temperature.

The effect of process parameters like pH, temperature, iron oxide dosage, contact time and initial heavy metal ion concentration are studied.

Langmuir and Freundlich adsorption isotherms are tested for the obtained data.

8. Results and Conclusions:
- Iron oxide (Fe₃O₄) Nanoparticles are synthesized with particle size ranging between 8.69nm-56.58nm (<100nm).
- The size and shape of Fe₃O₄ was confirmed using SEM, XRD and FTIR
- Batch studies were conducted for the removal of toxic Heavy metals like Copper(II) and Chromium(VI)
- According to the results, maximum metal removal Efficiency (%) for Cu(II) & Cr(VI) were obtained at: pH=6 for Cu(II) and pH=2 for Cr(VI); Contact time=60 minutes; Fe₃O₄ dosage=0.2 g; Temperature=30°C; Metal concentration=10ppm for Cu(II) & 20ppm for Cr(VI).
- The sorption data fitted for both Langmuir and Freundlich isotherms out of which Langmuir Adsorption model was found to have the highest regression value for both Cu(II)(R²=0.997) & Cr(VI)(R²=0.996) and hence the best fit.

9. Scope for Future work: In the coming years, despite all the recent progresses made, it is still a challenge to be faced that synthesis of high-quality functionalized magnetic iron oxide NPs with a tunable sizes and shapes in a controlled manner. Moreover synthesis and surface engineering of iron oxide NPs involves complex chemical, physical, and physicochemical multiple interactions, it is the another challenge to understand the synthetic mechanisms in detail. However, the magnetic properties and function of uncoated and surface functionalized iron oxide NPs depend upon their physical properties: the size and shape, their microstructure, and the chemical phase in which they are present. Luckily, several physicochemical techniques have been developed to determine these parameters. Therefore, how to improve the stability and availability of functionalized iron oxide NPs in extreme environmental conditions, how to develop an efficient and orderly magnetic micro- or nano-assembly structures, and how to realize large-scale or industrial synthesis, these problems are urgent to be solved for obtaining a ideal functionalized iron oxide materials. For all that, we still believe the surface functionalization and modification of magnetic iron oxide NPs to introduce additional functionality will attract more and more attention. Furthermore, multifunctional magnetic iron oxide composite nanoparticle systems with designed active sites will promise for a various applications, such as catalysts, magnetic recording, bioseparation, biodetection, etc. The future work in this area must be focused on the research of the toxicity and degradability of uncoated and surface functionalized iron oxide NPs, and preparing it via green chemistry for reducing the environmental pollution in the removal of toxic heavy metals from the industrial wastewater as much as possible. Successful development in this area will aid the growth of the various scientific researches or industrial applications as well as improving the quality of life in the population.