DEVELOPMENT OF NANO CRYSTALLINE MAGNETITE USING SOL-GEL METHOD

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Abstract

Magnetite (Fe_3O_4) nano particles were successfully synthesized by sol–gel method, and the material was sintered at a temperature of 200°C for 1hr. The phase structures, morphologies, particle size, and chemical composition of Fe_3O_4 nanoparticles were characterized by X-ray diffraction, field emission scanning electron microscopy and the results indicated the nanosize of Fe_3O_4 particles. The particle size estimated from Scherrer equation was obtained to be 28nm. Inductively Coupled Plasma-OES (ICP-OES) was used to study the effect of the Fe_3O_4 for the treatment of water for the removal of the arsenic. 0.1mg of the sample was added to 1 litre of water procured from various industrial areas in Hyderabad. A concentration of 0.004mg/l of arsenic in water was reduced to a value lower than that of the permissible concentration of 0.001mg/l wth a few minutes of stirring.

Key words: Sol-Gel, Scherrer equation, Inductively Coupled Plasma-OES (ICP-OES)

Introduction

Nanoscience is an upcoming topic of the research interest in the field of Material Chemistry. Optimization of the synthesis, parameters to obtain nanomaterials size in the range of 1-100nm which is the latest development in the nanotechnology. Adsorption is a conventional, but efficient technique to remove heavy metals from the aqueous solution. Many types of adsorbents for water waste water treatment have been commercialized and further being rapidly developed. In most of the cases these adsorbents are highly non porous materials providing inadequate surface are for adsorption. Thus, developing an adsorbent with large surface area is of a great significance. Various types of magnetic nano particles have been successfully synthesized and have received considered attention to solve environmental problems. The presence of heavy metals in the ground water and the surface water is becoming a severe environmental and public health issues.

Apparently, the magnetic nanoparticles posses the advantages of large surface area, high number of active sites and high magnetic properties, which lead to high adsorption effiency, high removal rate of contaminants and easy and rapid separation of adsorbates from solution due to magnetic field. In addition,to this it is possible that the harmful components can be removed, and the magnetic particles after magnetic separation by the external magnetic field can be reused. These research studies have sufficiently demonstrated the superior performance of the nanoparticles on the removal of heavy metals. Magnetic nanoparticles as a novel adsorbent are expected to yield an attractive and inexpensive method of removal of heavy metals based on its simple synthesizing method and high surface area. Thus, the objectives of this study are to assess the performance of magnetite nanoparticles for the selective removal of these heavy metals to achieve the possible regeneration of magnetite nanoparticles for reuse and to explore the mechanism of metal adsorption[2].

Keeping the above applications in view, the study is aiming at developing mesoporous nanomaterials by the SOL-GEL method. Among various chemical synthetic methods of preparation of metal oxides, sol-gel method offers several advantages over the other methods. Sol-gel is the most simple and sophisticated method in which various material parameters such as powder morphology, surface area, average crystalline size and phase structures are controlled. Magnetic nanoparticals with a diameter of 28nm prepared by the SOL-GEL method have been applied to adsorb the arsenic ions in water. A concentration of 0.004mg/l of arsenic in water was reduced to a value lower than the permissible concentration of 0.001mg/l after few minutes of stirring.

Iron oxides are the most widely used materials due to their saturation magnetization, their biocompatibility, their stability under ambient conditions and simplicity in their preparational processes. Among the iron oxide materials Magnetite Fe_3O_4 has attracted wide field of research. Magnetite was reported to get adsorb arsenic ions from the contaminated water. Nanoparticles were collected by using a magnet and the arsenic concentration in the water sample was determined by Inductively Coupled Plasma-OES (ICP-OES). By adjusting the amount of Poly Ethylene Glycol, the size and morphology of the product can be modified. It has been reported that PEG is easily adsorbed at the surface of metal oxide and when the surface of the metal oxide adsorbs PEG, the colloidal activities will also greatly decrease and the growth rate of the particle will be confined[3].

Method of Analysis

i. Materials

Ferric nitrate, Poly Ethylene Glycol-200 used for the synthesis were analytical grade. All the aqueous solutions were prepared with deionised water.

i. Synthesis of magnetite Nanoparticals

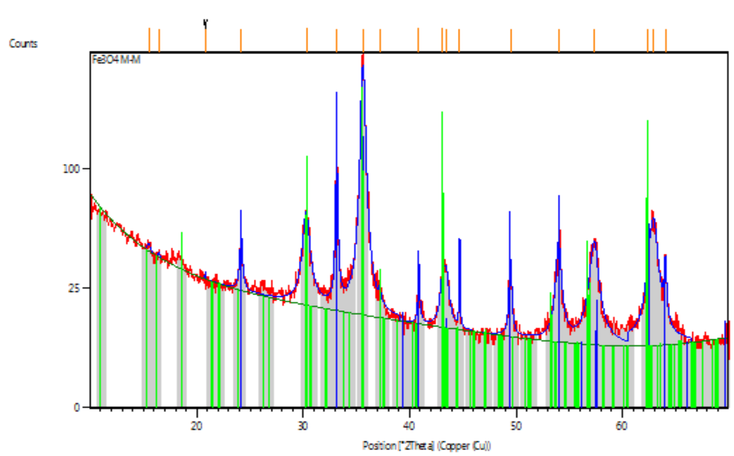
SOL-GEL method offers several advantages over the other method. Sol-gel method is developed for the preparation of magnetite nanoparticles by using Poly Ethylene Glycol-200 (PEG-200). In this method ferric nitrate (2gms) is taken in a beaker and diluted with distilled water and kept for stirring. Poly ethylene glycol-200 is also taken in another beaker and diluted with distilled water and kept for stirring. After mixing the two, the mixture is heated for 2hrs at 40^0c and then aged for 2hrs. The gel is kept in oven and then will dry at 60^0C over night. The obtained powder was then calcined at 200^0C for required time in a muffle furnace and then characterized by XRD which confirmed the presence of Fe_3O_4 nanoparticles[1].

ii. Characterization and Applications

X-ray powder diffraction (XRD) measurements are performed using a Bruker D8 Advance diffractometer operating at 40 kV, 30 mA equipped with Cu Kα radiation (1.78901 Å) for angles in the range 2h = 20–70 using an angle step size of 0.02. The broad peaks indicate the formation of very fine particles in the nanoscale range. The crystallite size of the nanoparticles is calculated by Scherrer formula:

$$D = \frac{K \cdot \lambda}{\beta \cdot \cos\theta}$$

Where, K is a dimensionless shape factor, λ is the X-ray wavelength, β is the line broadening at half the maximum intensity (FWHM) and θ is the Bragg angle[1].



Magnetite (Fe_3O_4) nanoparticles are successfully synthesized by sol–gel method. The phase structures, morphologies, particle sizes, chemical composition, and magnetic properties of Fe_3O_4 nanoparticles are characterized by X-ray diffraction, field emission scanning electron microscopy and the results indicate that the size of Fe_3O_4 nanoparticles is 28nm. Inductively Coupled Plasma-OES (ICP-OES) is used for the treatment of water for the removal of the arsenic.

S.No	Sample Code	As (mg/l)
1	HSW-WS	0.004
2	HSW-S	0.001
3	BW-WS	0.003
4	BW-S	B.D

B.D: Below detection limit

Magnetite is able to remove the arsenic element completely from the hardwater collected from an industrial area in Hyderabad.

Conclusion

Among the kinds of adsorbents, particularly magnetic iron oxides such as magnetite (Fe_3O_4) have been investigated intensively for environmental and Bio-Applications. Fe_3O_4 nanoparticles show convenient magnetic properties, low toxicity and price affordable, high surface area to volume ratio, which are associated to their ability for surface chemical modification and show the enhanced capacity by removing toxic elements in water treatment[1].

References

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