

## SYNTHESIS, CHARACTERIZATION AND SELECTED APPLICATION OF CHITOSAN-COATED MAGNETIC IRON OXIDE NANOPARTICLES

M. A. Radwan, Mohamed Adel Rashad, M. A. Sadek, Hany A. Elazab

Chemical Engineering Department  
The British University in Egypt, BUE, Cairo, Egypt  
E-mail: elazabha@vcu.edu

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### ABSTRACT

*In this research, we report an efficient method to prepare highly active chitosan-coated magnetic iron oxide nanoparticles by implementing co-precipitation technique which is used for waste water treatment applications. Chitosan-coated magnetic iron oxide nanoparticles were synthesized and characterized using TEM, XRD, and XPS. A comparative study has been made between chitosan powder and chitosan coated with magnetic nanoparticles with respect to the rate of adsorption of copper from aqueous solution. It was also experimentally verified that chitosan-coated magnetic iron oxide nanoparticles are much more effective in adsorption of heavy metals than chitosan powder and it takes just few minutes instead of hours. Moreover, the super paramagnetic behavior of the chitosan coated with iron oxide nanoparticles enabled the easy removal through applying an external magnetic field and hence leaving the solution free from copper.*

*Keywords:* contaminants, heavy metals, chitosan, magnetic iron oxide nanoparticles, wastewater treatment.

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### INTRODUCTION

Industrial wastewater is considered as one of the most significant sources of pollution that could affect the water environment. During the last century huge amounts of industrial waste water were released into rivers, lakes and costal zones [1 - 12]. Unfortunately, this led to several pollution issues in the water environment and initiated a negative impact to the eco-system. Consequently, pollution caused by heavy metals has emerged as one of the most significant research efforts due to the huge growth of industrial activities [6, 7, 10, 13 - 21]. There are several sorts of waste water as a direct impact of the different industries and this will lead to different types of harmful wastes [5 - 12, 17 - 26]. The expanding rates of industrial wastewater in developing nations are thought to be much higher than those in developed nations. This predicts that industrial waste water pollution, as a main environment pollution issue, will be expected to move from developed nations

to developing nations in the early 21st century.[6, 17, 27 - 31] Waste water is considered as a very critical issue due to the presence of several toxic metals such as copper, nickel, mercury, lead, zinc, arsenic etc. and those metals are widely and commonly used in the industry and are produced by dental procedure, electroplating, tanning, textile, paper and pulp industry and are possibly toxic to people. Unfortunately, those kinds of metals are very common in most of the industries so the industrial wastes will contain large amounts of those metals, and therefore the waste water should be treated and cleaned out of those toxic metals before being discharged due to its severe effect on humans, environment and generally on health [32 - 37]. Effective ways of removing those toxic metals from waste water has been under study for years [17 - 21, 26].

Some of those studies have been developed in order to remove toxic poisonous metals from waste water through physical treatment that can additionally be utilized to remove other tiny concentrations of hazardous

substances dissolved in water [38 - 40].

The present physico-chemical procedures for heavy metal removal like precipitation, reduction, ion-exchange etc. are very costly and inefficient in handling massive quantities. They additionally cause metal bearing sludge that is hard to dispose [41 - 43]. Most of these out-dated methods are also exceptionally very costly, so it would be inefficient and especially in the developing countries which have limited resources, capabilities, low budget and large amounts of those wastes are generated and discharged. Thus, there is an obligation for innovative and operative approaches and methods which are indeed economical and eco-friendly [44 - 51]. One of the most usually utilized methods involves the procedure of adsorption. Bio-adsorption is an attractive substitute to common and usual processes. Extensively obtainable biopolymers are additionally being utilized for adsorption generally because they are inexpensive resource or a freely obtainable resource [32, 37, 41].

In this work, the adsorption technique based on magnetic nanotechnology was adopted as a potential route through which we have developed an efficient method to prepare highly active chitosan-coated magnetic iron oxide nanoparticles by implementing co-precipitation technique.

## EXPERIMENTAL

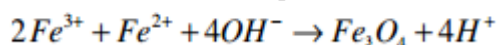
### Chemicals

All chemicals were purchased and used as received without further purifications. Ferrous chloride ( $\text{FeCl}_2$ , 98 %), ferric chloride ( $\text{FeCl}_3$ , 98 %), chitosan, were provided by Sigma Aldrich, Chemie GmbH (Munich, Germany). Sodium hydroxide was provided by El Gomhoria Co., Cairo Egypt.

Synthesis of magnetic  $\text{Fe}_3\text{O}_4$  nanoparticles (MNPs)

Samples were prepared by implementing the co-precipitation technique. This method is probably one of the simplest and the most efficient chemical routes to prepare magnetic particles. A mixture of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  salts in the molar ratio 1:2 is used in the synthesis of  $\text{Fe}_3\text{O}_4$  magnetic nanoparticles (MNP).

The reaction in this process is as follows:



In the typical synthesis, 50 grams of a mixture containing both  $\text{FeCl}_3$  and  $\text{FeCl}_2$  was prepared with the molar

ratio of 1:2. This mixture was then added to 700 mL distilled water. After an efficient mixing of  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  and  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  together into one solution, NaOH (1.5 M) is added to the solution drop wise, under vigorous stirring and mechanical agitation at a rate 0.5 mL per second. Then, a dark-black precipitate was formed immediately. The solution was decanted and then the prepared  $\text{Fe}_3\text{O}_4$  was washed with distilled water for several times and then washed with acetone and dried in a furnace for about five hours at  $80^\circ\text{C}$ . After drying, the sample was grinded into the smallest possible particle size.

### Synthesis of chitosan-coated magnetic $\text{Fe}_3\text{O}_4$ nanoparticles (MNPs)

The suspension cross-linking technique was used for the preparation of chitosan-coated magnetic iron oxide nanoparticles. In this specific procedure, 5% chitosan (25 grams) solution was prepared using a 2 % aqueous acetic acid solution, (10 ml acetic acid in 500 ml deionized water), containing 1 g  $\text{Fe}_3\text{O}_4$  dry magnetic nanoparticles. And then, this solution was poured, drop-wise, into the dispersion medium, which was composed of 140 ml paraffin and 2.5 ml span-80.

During this process, the dispersion medium was stirred using a strong ultrasonic agitation at room temperature. Next, an additional 15 ml of 25 % glutaraldehyde solution was added to the dispersion medium and then solution was stirred for further 5 h. Then, the chitosan-coated magnetic iron oxide nanoparticles were recovered from the reaction mixture by using a permanent magnet as a strong external magnetic field; the products were washed with ethanol and dried in a vacuum oven at  $80^\circ\text{C}$  for 3 hours.

### Catalyst Characterization

A JEOL JEM-1230 electron microscope operated at 120 kV and equipped with a Gatan UltraScan 4000SP 4K X 4K CCD camera was used to obtain TEM images. TEM samples were prepared by placing a droplet of the prepared catalyst dissolved in ethanol on a 300-mesh copper grid (Ted Pella) which was then left to evaporate in air at room temperature. The X-ray photoelectron spectroscopy (XPS) analysis was performed on a Thermo Fisher Scientific ESCALAB 250 using a monochromatic Al KR X-ray. The X-ray diffraction patterns were measured at room temperature using an X'Pert PRO PANalytical X-ray diffraction unit.

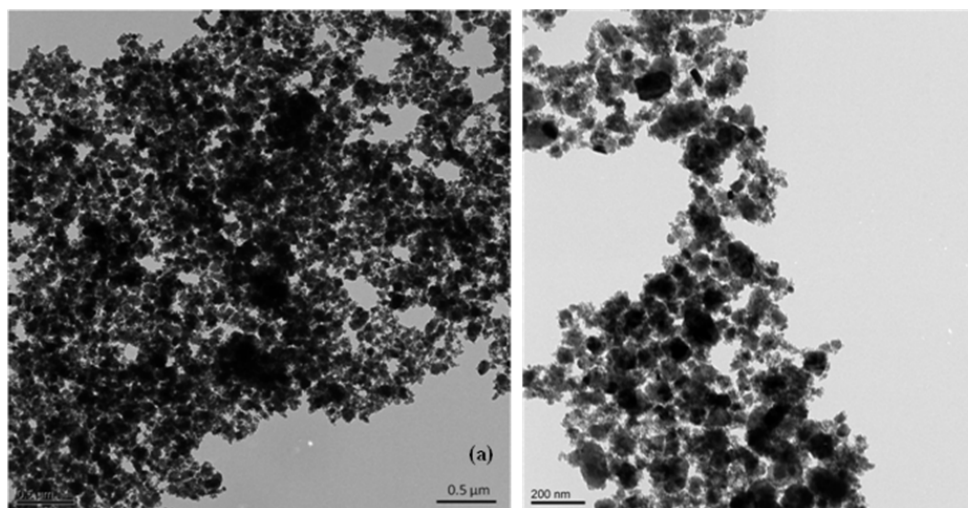


Fig. 1. TEM of (a) uncoated  $\text{Fe}_3\text{O}_4$  nanoparticles, (b) chitosan-coated  $\text{Fe}_3\text{O}_4$  nanoparticles.

#### **Determination of chitosan rate of adsorption of $\text{Cu}^{++}$ Chitosan powder (without magnetic iron oxide nanoparticles)**

0.1 gram of chitosan was added to 100 ml of copper solution in a flask equipped with a magnetic stirrer at room temperature ( $25^\circ\text{C}$ ) and a velocity of 150 r.p.m, with maintaining the pH of solution to be around 7. Eight samples of the above mixture were prepared and then stirred for different times starting with three hours and keep adding another three hours for the next mixture until the 8<sup>th</sup> one was stirred for twenty four hours. Then, chitosan was left to settle. The  $\text{Cu}^{++}$  concentration in the water sample was measured by atomic adsorption apparatus.

#### **Chitosan powder (chitosan-coated magnetic iron oxide nanoparticles)**

0.1 gram of coated chitosan nanoparticles was added to 100 ml of copper aqueous solution in a test tube and then shaken well for different times. 10 samples of the above mixture were prepared and shaken for different times starting with one minute till 10 minutes for the 10<sup>th</sup> sample with one minute increment for each sample. Then, a magnet was used to collect the chitosan-coated magnetic iron oxide nanoparticles. The solution was then filtered to remove any remains of the chitosan nanoparticles and the concentration of  $\text{Cu}^{++}$  was measured using atomic absorption apparatus.

#### **Measurement of heavy metal concentration Cu (II) in aqueous solution at different periods**

The capacities of  $\text{Cu}$  (II) removal by chitosan-coated

magnetic iron oxide nanoparticles samples were measured by using atomic absorption spectrophotometer (Z-5000, Hitachi, Japan). The concentration of  $\text{Cu}$  (II) in copper aqueous solution in the prepared samples was measured by using atomic absorption spectroscopy (AAS) as a spectro-analytical method for the quantitative determination of chemical components utilizing the absorption of optical radiation (light) by free atoms in the vaporous state. This procedure was implemented for confirming the concentration of a specific component (the analyte) in the sample to be analyzed.

## **RESULTS AND DISCUSSION**

### **Characterization**

Fig. 1 displays a typical TEM image of the chitosan-coated  $\text{Fe}_3\text{O}_4$  nanoparticles, most of the nanoparticles have spherical shapes with diameters between 1 - 20 nm.

Fig. 2 displays the XRD patterns of the Chitosan coated  $\text{Fe}_3\text{O}_4$ . The Chitosan coated  $\text{Fe}_3\text{O}_4$  nanoparticles show the characteristic peaks for the spinel  $\text{Fe}_3\text{O}_4$  phase (ICCD-00-003-0863). Magnetic nanoparticles were synthesized by precipitating iron salts with a molar ratio of 1:2 in an alkaline medium under constant stirring. The crystalline structures of unmodified magnetic nanoparticles and the prepared chitosan coated nanoparticles were studied by XRD.

For the chitosan and chitosan coated  $\text{Fe}_3\text{O}_4$  nanoparticles, six characteristic peaks for magnetite ( $2\theta = 30.1^\circ, 35.5^\circ, 43.1^\circ, 53.4^\circ, 57.0^\circ$  and  $62.6^\circ$ ), corresponding to the plane (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0), were observed for the prepared samples.

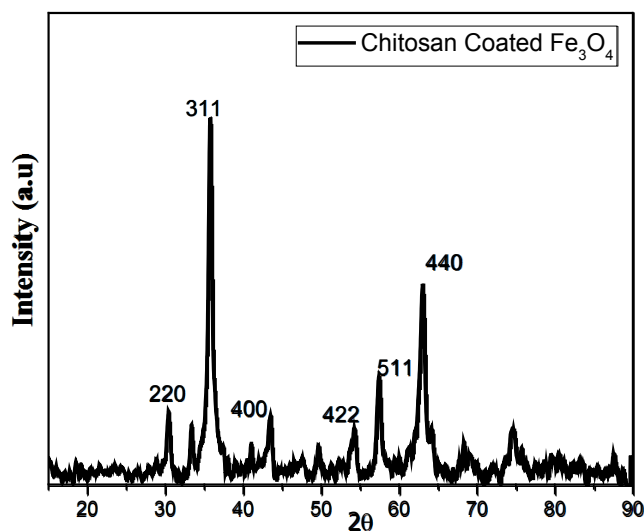


Fig. 2. XRD of chitosan-coated  $\text{Fe}_3\text{O}_4$  nanoparticles.

The crystalline patterns of the prepared samples were found to coincide very well. This indicates that the outer shell of chitosan did not change the typical magnetite XRD pattern.

To characterize the surface composition, we carried out XPS measurements as shown in Figure 3 for the Chitosan coated  $\text{Fe}_3\text{O}_4$ . Fig. 3 displays the XPS patterns of the chitosan coated  $\text{Fe}_3\text{O}_4$ . The data reveals the presence of Fe(III) as indicated by the observed peaks at 724.2 eV and 710.5 eV corresponding to the binding

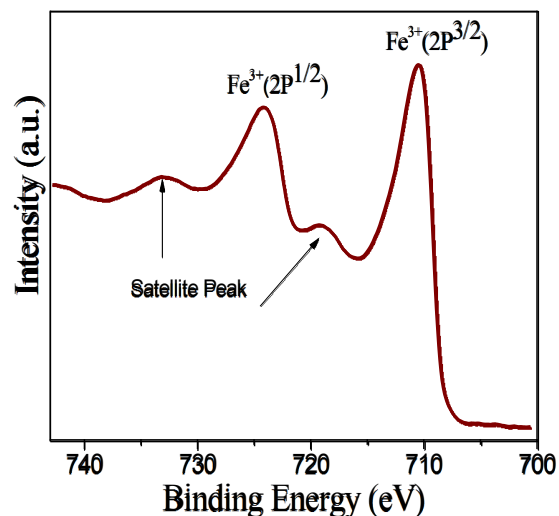


Fig. 3. XPS of chitosan-coated  $\text{Fe}_3\text{O}_4$  nanoparticles.

energies of the  $2p^{1/2}$  and  $2p^{3/2}$  electrons, respectively. The broad Fe(III)  $2p^{3/2}$  peak centered at 710.5 eV most likely contain contributions from the Fe(II)  $2p^{3/2}$  which normally occurs at  $\sim 708$  eV. The main advantage of using iron oxide nanoparticles in coating chitosan is that its magnetic properties make it easy to perform separation from the reaction mixture via applying an external magnetic field. The experimental process of chitosan-coated magnetic iron oxide nanoparticles in a Cu aqueous solution is shown in Fig. 4.

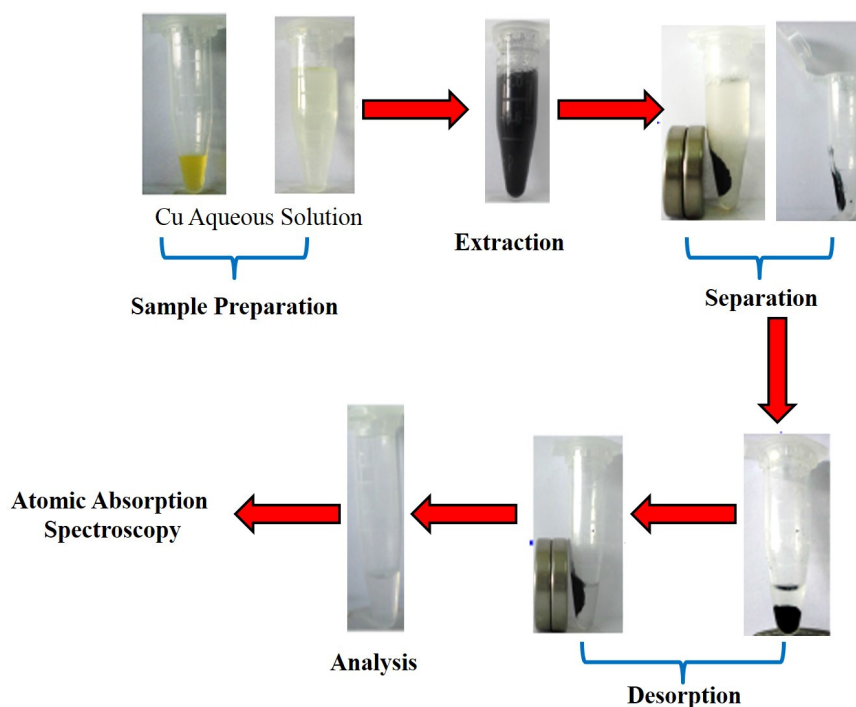


Fig. 4. The experimental process of chitosan-coated magnetic iron oxide nanoparticles in a Cu aqueous solution.

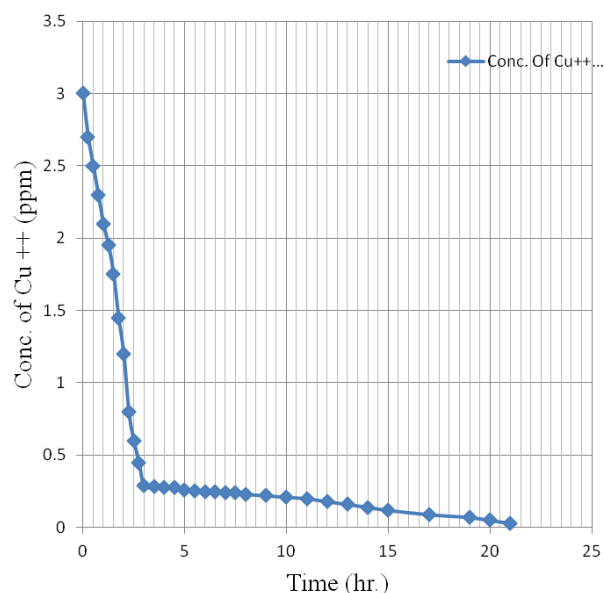


Fig. 5. Concentration of Cu in aqueous solution per time (chitosan powder).

#### Dependence of heavy metal adsorption rate by chitosan in aqueous solution on time

The concentration of chitosan is 0.1 gram per 100 ml of Cu<sup>++</sup> aqueous solution. It was found after measuring the concentration of Cu<sup>++</sup> in aqueous solution with atomic absorption spectroscopy (AAS), starting from  $t = 0$  till  $t = 23$  hours, It was found that the concentration went down from 3 particle per million to 0.29 particle per million after 3 hours and after 15 hours went to 0.12 particle per million then after 21 hours the concentration is down to 0.03 particle per million and it remains almost constant with time increase to 22 and 23 hours as shown in Fig. 5.

#### Dependence of heavy metal adsorption rate by chitosan-coated magnetic iron oxide nanoparticles in aqueous solution on time

The concentration of chitosan-coated magnetic iron oxide nanoparticles is 0.1 gram per 100 ml of Cu<sup>++</sup> aqueous solution, while the ratio of chitosan to Fe<sub>3</sub>O<sub>4</sub> is 4:1 which means the amount of chitosan alone in 0.08 grams.

After measuring the concentration of Cu<sup>++</sup> in aqueous solution with atomic absorption spectroscopy (AAS) starting from  $t = 0$  till  $t = 60$  minutes with 0.5 minutes increment, it was found that after 3 minutes the concentration went down from 3 particle per million to 0.63 particle per million and after 10 minutes went to

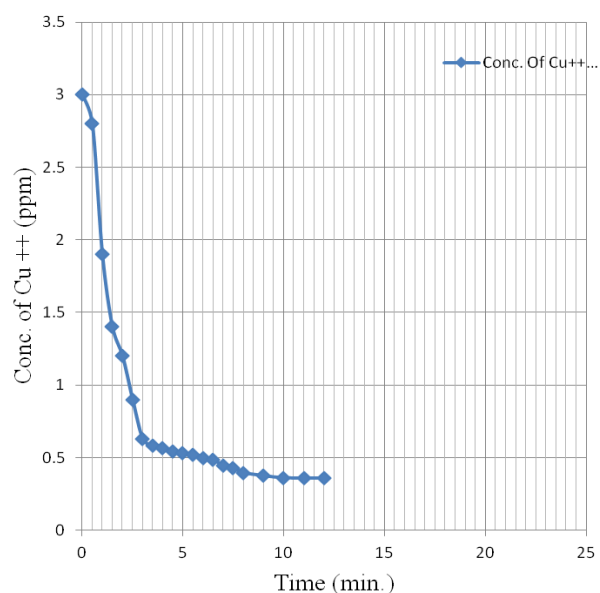


Fig. 6. Concentration of Cu in aqueous solution as dependence on time (chitosan-coated magnetic iron oxide nanoparticles).

0.36 particle per million and it remains almost constant with time increase to 15, 20, 30, and 60 minutes as shown in Fig. 6.

It was found from experimental results that the more the chitosan is available in the Cu<sup>++</sup> aqueous solution, the more the concentration of C<sup>++</sup> is decreasing and the same trend was observed for chitosan-coated magnetic iron oxide nanoparticles which mean that the chitosan is working properly and efficiently.

While comparing the results of the two experiments; chitosan powder alone and chitosan-coated magnetic iron oxide nanoparticles; it is clear that chitosan-coated magnetic iron oxide nanoparticles is much more effective and faster in adsorption the Cu<sup>++</sup> in a very short time period and this is due to the high surface area of the nanoparticles.

#### CONCLUSIONS

In conclusion, we adopted here one of the adsorption techniques that is implementing the concept of using magnetic nanotechnology as a remediation technology. We have developed an efficient method to prepare highly active chitosan-coated magnetic iron oxide nanoparticles by implementing co-precipitation technique. Chitosan-coated magnetic iron oxide nanoparticles were synthesized and characterized using TEM, XRD, and XPS. A comparison study has been made between chitosan

powder and chitosan coated with magnetic nanoparticles with respect to the rate of adsorption of copper from aqueous solution.

The magnetic iron oxide nanoparticles are coated with chitosan which develops surface functionalized groups that form miniature aggregates in aqueous suspensions. The sorption of metals like Cu (II) in the contaminated water takes place on those aggregates.

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