

Research Paper

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## Removal of Toxic Substances From Textile Dyeing Industry Effluent Using Iron Oxide Nanoparticles

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The present study deals with the removal of toxic substances from textile dyeing industry effluent using iron oxide nanoparticles. Iron Oxide Nanoparticles were successfully synthesized by simple and cost effective precipitation method without any other additives. The Nanoparticles were characterized by using SEM, EDAX, FTIR and XRD. SEM image of Iron Oxide Nanoparticles was observed at the wavelength range from 11.27 mm, 11.27 and 11.28mm. EDAX spectrum recorded on the Iron Oxide Nanoparticles is shown as three peaks located between 2keV and 10keV. The FTIR spectrum of Iron Oxide Nanoparticles was analyzed in the range of 400-4000cm-1 and spectral bands were observed. 100mg of Iron Nanoparticles is introduced in textile dyeing industry effluent and exposed to sunlight for a period of one hour. The pH, electrical conductivity, total dissolved solids, COD, chloride, sodium, potassium, calcium, zinc and nickel of the effluent was decreased when exposed to Iron Oxide Nanoparticles under sunlight. The synthesized Iron Oxide Nanoparticles was found to be an effective and economically cheap absorbent in the removal of toxic substances from Textile dyeing Industry effluent.

## **KEYWORDS**

Removal, toxic, textile dye industry effluent, iron oxide, nanoparticles.

## INTRODUCTION

One of the most serious environmental problems is the existence of hazardous and toxic pollutants in the environment. Effluents from industrial or municipal sources may contain hundreds to thousands of chemicals, but only a few are responsible for aquatic toxicity. Among industries, textile dyeing industries have provided a wide range of color & bright hues. Presence of these dyes in water even at very low concentration is highly visible and undesirable. Over 70,000 tons of approximately 10,000 types dyes and pigments are produced annually world wide of which about 20 - 30% are wasted in industrial effluents during the textile curing and finishing processes. (Manohar and Shrivastava. 2015). Use of synthetic dyes has an adverse effect on all forms of life. The presence of even very low concentrations of dyes in effluent is highly visible and degradation products of these textile dyes are often carcinogenic.Various techniques for removing dyes from effluents have been developed, including electrochemical treatment, sonochemical treatment, photo catalytic treatment and adsorption. Absorption by Iron Oxide Nanoparticles is a promising and attractive alternative for treatment of azo containing effluents and are inexpensive and readily available. Among the various techniques nanoparticles have accepted significant attention due their outstanding physico-chemical properties. Among the various oxide Nanoparticles, Iron Oxide Nanoparticles have unique properties of small size, larger special surface are and Magnetic property. Due to the high surface area, good absorbing capacity the synthesized Iron Oxide Nanoparticles was used in the removal of toxicity from textile dying industryeffluent. The work related to the use of Iron Oxide Nanoparticles for the removal of toxic substances from Dyeing industry effluent is totally wanting. Hence the present study was carried out.

## Synthesis of iron oxide nanoparticles

The Precipitation method was adopted for synthesis of iron oxide nanoparticles. 0.03 mole of 5.96g FeCl<sub>2</sub> were dissolved into 150ml of distilled water and stirred vigorously using magnetic stirrer for 20 minutes. Precipitation was achieved by adding 100ml of 1 M NaOH solution in drop wise under vigorous stirring. The initial pH was observed as 3 and it was increased to pH 12 using 1M NaOH. Then precipitating process was continued until dark black color precipitate obtained. Then the Fe<sub>3</sub>O<sub>4</sub> precipitate was taken into centrifuge tube and centrifuged at 1500 rpm for 20 minutes. The centrifuged process continued with water and two times with ethanol. Then the precipitate was dried. Finally, iron oxide nanoparticles (Fe<sub>3</sub>O<sub>4</sub>) is formed.

#### CHARACTERIZATION SEM analysis

The morphology and composition of  $\rm Fe_3O_4$  nanoparticles were examined by Scanning Electron Microscopy (SEM) using a LEO 1455 VP equipped with energy dispersive.

## Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) is used to measure the vibration modes of functional groups of molecules and is sensitive to molecular structure, conformation and environment. Therefore, in the current study it is possible to directly relate the intensities of the absorption bands to the concentration of the corresponding functional groups. FTIR spectroscopy was analyzed in the range of 4000 – 400 cm<sup>-1</sup>. The FTIR spectra of synthesized iron oxide nanoparticle were analyzed for knowing the possible functional groups. The measurement was carried out by JASCO (FTIR-6200) spectrum.

### Energy Dispersive X-Ray Spectroscopy (EDAX)

A minute drop of nanoparticle solution was cast on alumi-

num foil and subsequently dried in air before transferring it to the microscope. An energy dispersive X-ray detection instrument (EDAX) (HORIBA 8121-H) was used to examine the elemental composition of the sample.

#### X-ray diffraction (XRD)

X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a given material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized and average bulk composition is determined.

#### Physico-chemical parameters of the effluent:

The physico – chemical parameters such as pH, electrical conductivity, total solids, total dissolved solids, total suspended solids, hardness, sodium, potassium, calcium, magnesium, sulphate, chloride, dissolved oxygen, BOD, COD, copper, nickel and zinc were determined using standard methods (APHA,2012).

# Role of iron oxide nanoparticles on physico-chemical parameters of textile dyeing effluent:

The role of iron oxide nanoparticles on the physico-chemical parameters of textile dyeing industry effluent such as colour, pH, electrical conductivity, COD ,calcium and heavy metals were estimated after exposing the effluent in the sunlight for a period of one hour with 100 mg of iron oxide nanoparticle.

### **RESULTS AND DISCUSSION**

The Physico-chemical characteristics of the textile dyeing industry effluent is presented in Table 1. The chemical method has been successfully used to synthesize the magnetite nanoparticles. This approach provides a simple, general and economical method for the preparation of monodisperse magnetic nanoparticles with a diameter range of 20-42 nm.(Hasany et al., 2012). The precipitation synthesis of iron oxide nanoparticle is high concentration starting material. The nanoparticle formed from slow titration of FeCl<sub>3</sub> with aqueous have controlled growth and controlled crystal structure depend on the temperature of the synthesis. (Tharani and Nehru 2015). Nanoparticles were successfully synthesized using chemical method to remove toxic substances from textile dyeing industrial effluent and characterization made by using SEM, EDAX, FTIR and XRD. The morphology of obtained nanoparticles shown by SEM image (Fig.1) of Fe $_3O_4$  that composed of small particles, having the diameter size of 5-20nm. The SEM image showing the high density chemical synthesized Fe<sub>3</sub>O<sub>4</sub> further confirmed the development of iron oxide nanostructures. Obtained nanoparticles showed that hexagonal and spherical in nature. The microscopic image shows that the Fe<sub>3</sub>O<sub>4</sub> nanoparticles did not appear as discrete particles but form much larger dendrite flocks whose size could reached micron scale size range about 11.27mm (scale bar 1µm), 11.27mm (scale bar 2µm), 11.28mm (scale bar 5µm).Characterization of Fe<sub>3</sub>O<sub>4</sub> EDX spectra (Fig.2)showed the strong peaks of Fe and O. The composition components of Fe  $_{3}O_{4}$  formed by co-precipitation method of synthesis Fe was 65.72% and O was 27.54% These results demonstrate the purity of the synthesis results. XRD patters of the Fe<sub>3</sub>O<sub>4</sub> shows six characteris

Table 1. Physico-chemical characteristics of textile dyeing industry effluent

Parameters Colour		Values	
		Green	
pH		8.57	
Electrical Conductivity	Ms./cm	3200	
Total Solids	mg/l	9200	
Total Dissolved Solids		6900	
Total Suspended Solids "		2200	
Total Hardness		1320	
Chloride "		3770	
Sulphate		740	
Dissolved Oxygen	**	2.902	
Dissolved carbon dioxide "		5.32	
BOD "		26.45	
COD "		462	
Sodium "		8.125	
Potassium		22.529	
Calcium "		10.01	
Magnesium		14.72	
Copper		0.942	
Nickel		0.692	
Zinc		1.692	

#### All the values are average of ten individual observations FIGURE 1.SCANNING ELECTRON MICROSCOPY IMAGE ON IRON OXIDE NANOPARTICLES

All the values are average of ten individual observations

# FIGURE 1.SCANNING ELECTRON MICROSCOPY IMAGE ON IRON OXIDE NANOPARTICLES





1µm of iron oxide nanoparticles



c) 5µm of iron oxide nanoparticles

FIGURE 2.ENERGY DISPERSIVE X-RAY SPECTROSCOPY (EDX) IMAGE



## FIGURE 3. XRD ANALYSIS OF IRON OXIDE NANOPARTICLE



Fourier Transform Infrared spectroscopy measurements were carried out to identify the possible functional groups responsible for the reduction of the Fe ions in chemically synthesized iron oxide nanoparticles. The FTIR spectrum of iron oxide nanoparticles was analyzed in the range 4000-400cm<sup>-1</sup> (Fig.4) and bands observed at 3790.02, 2985.33, 1599.78, 1283.99, 864.50 and 570.64.

#### FIGURE 4. FOURIER TRANSFORM INFRARED SPECTROSCO-PY (FTIR) IMAGE



The decolourization efficiency for various initial dye concentrations shows that the efficiency gradually decreases with an increase in initial dye concentration.(Kumaravel Dinesh et al., 2014). The concentration of dye increases from 20-80 mg/l, percentage of removal of dye by PANI-Fe<sub>2</sub>O<sub>2</sub> decreases from 98.5-90% for absorbent dose 10 mg/l (Patil and Shrivastava 2015). In the present study similar observation was made that absorption by Iron Oxides increases in diluted dye effluent(1:1) than raw dye effluent and absorption was quick and high with increased % of removal. The efficiency of dye absorbed is rapid in initial stage up to 60 min and after that remains almost constant due to saturation of the active site which do not allow further absorption to take place. The optimal contact time to attain equilibrium was experimentally found to be about 60 min.(Neeraj Jain et al., 2015).

Colour removal also increased with increasing contact time. The equilibrium time at 30 min and after equilibrium time the colour removal was stable. In the optimum condition absorption of colour removal was 24.40%. This condition colour removal is optimum. So that extra time does not increase the colour removal. Karthik et al( 2014) reported that Fe<sub>3</sub>O<sub>4</sub> powder used for removal of color from textile dye wastewater by batch experiments and adsorption equilibrium is attained within 45 minutes. The dye is rapidly absorbed in the first 40 min up to 80% and then absorption rate decresed gradually and reached equilibrium in about 90 min up to 98.5%.(Patil and Shrivastava 2015). Same observation made and that shows absorption rate is high at 0-30 min and slowly decreased with increase in the time. Rmoval of dye decreased from 88 to 37 % when the pH was increased from 2 to 9. Since the maximum removal of dye is achieved at a pH of 2. Acidic condition is favourable for Fe<sub>2</sub>O<sub>4</sub> powder. (Neeraj Jain et al., 2015). Decolourization of the BBG dye aqueous solution was carried out at various pH values, and observed that initial solution pH (2-4) has a remarkable influence on the decolourization efficiency (Kumaravel et al., 2016). The pH of dye solution plays an important role in the whole absorption process and particularly on the colour removal. The most colour removal was 25.19% at pH solution 5. All the absorption was highly pH dependent. The optimal pH for removal of Cr(IV), Cu(II) and Ni(II) were 2.5,6.5 and 8.5 by magnetite nanoparticles.

The pH value of the aqueous solution that affects the oxidation of organic substances both directly and indirectly. The pH of the solution was adjusted using either 0.1N HCl or 0.1N NaOH and the reaction time was 60 min. The colour removal of the dye had exhibited a different trend showing higher colour removal both at pH 3(acidic) and 11(alkaline) (Kumaravel et al., 2016). Same observation made and colour removal high at the pH of 2(acidic).After the decolorization of the solution, the COD decreased sharply, reaching a plateau that corresponds to the oxidation of most stable compounds indicating the almost complete mineralization of intermediates has occurred. (Abdul Raheem Giwa et al., 2012). The TDS and COD of the dye efflluent were very high. Experiments carried out for effluent with different dilutions 1:100, 1:10, 1:1 and for raw textile effluent. Highest (88%) COD removal was observed for the 1:100 (v/v) diluted effluent and lowest(41%) COD removal for raw effluent after 1h of treatment. (Hemapriyamvada and Sivasankar 2016) In the present study highest (55%) COD removal was observed in the 1:1 (v/v) and lowest (30%) in raw sample. The capacity of  $Fe_3O_4$  NPs and its contact time was recognized in the simulated wastewater. The obtained results showed the relationship between the amount of absorbent and required time in order to achieve removal efficiency higher than 90% in the wastewater sample.(Nasser et al., 2011). Waste water from textile industry dye effluent was treated successfully by using iron oxide nanoparticles at a laboratory scale and ambient temperature . The result indicated that iron oxide nanoparticles was effective to reduce the colour, pH, electrical conductivity, COD, calcium, total dissolved solids ,heavy metals so chemically synthesized iron oxide nanoparticles can be used for the removal of contaminants present in the textile dyeing industry effluent.



b)50% (50ml effluent+50ml distilled water) effluent treated with 100 mg nanoparticles



#### **REFERENCES:**

Abdul Raheem Giwa., Peter Obimma Nkeonye., Kasali Ademola Bello and Kasali Ademola Kolawole.(2012) Photocatalytic Decolonization and Degradation of C.I. Basic Blue 41 using TiO<sub>2</sub> Nanoparticles. Journal of Environmental Protection, 3:1063-1069.

- APHA. 2012. Standard methods for examination of water and waste water. 21st Edition. American Public Health Association, Washington, DC.
- Hasany,S,F., I.Ahmed., Rajan,J and A.Rehman(2012) systematic Review of the preparation Techniques of Iron Oxide Magnetic Nano particles. Nanoscience and Nanotechnology, 2(6):148-158.
- Hemapriyamvadha, R and T. Sivasankar.(2014) Sonophotocatalytic treatment of methyl orange dye and real textile effluent using synthesized nano-zincoxide. Colouration Technology, 131:1-10.
- Karthik.V.,K.Saravanan., P.Bharathi., V.Dharanya and C.Meiaraj.(2014) An overview of treatments for the removal of textile dyes. Journal of Chemical and Pharmaceutical Science, 301-307.
- Kumaravel Dinesh.G., S.Anandan and T.Sivasankar (2014) Sonophotocatalytic treatment of Bismark Brown dye and real textile effluent using synthesized novel Fe(O)- doped TiO<sub>2</sub> catalyst. Royal Society of Chemistry,5:10440-10451.
- Kumaravel Dinesh.G., S.Anandan and T.Sivasankar (2016) Synthesis of Fe/ ZnO composite Nanocatalyst and its sonophotocatalytic activity on acid yellow 23 dye and real textile effluent. Clean Techn Environ Policy,1-15
- Manohar R.P and V.S.Shrivastava (2015) Adsorption removal of Carcinogenic acid violet19 dye from aqueous solution by polyaniline-Fe<sub>2</sub>O<sub>3</sub> magnetic nanocomposite. Journal of Materials and Environmental Science, 6(1):11-21.
- Nasser Dalali., Mahboobe Khoramnezhad., Mina Habibizadeh and Mohammad Faraji.(2011) Magnetic Removal of Acidic dyes from wastewaters using surfactant- coated Magnetite Nanoparticles. IPCBEE, 15:89-93.
- Neeraj Jain., M.K.Dwidedi., Rashi Agarwal and Pragati Sharma.(2015) Removal of Malachite Green from Aqueous solution by zeolite-Iron oxide Magnetic Nanocomposite. IOSR Journal of Environmental Science, 9:42-50.
- Tharani, K and L.C.Nehru (2015) Synthesis and characterization of iron oxide nanoparticle by precipitation method. International journal of Advanced Research in Physical Science, 2:47-50.