International Journal of Scientific Engineering and Research (IJSER)
ISSN (Online): 2347-3878
SJIF (2024): 6.623

# Iron Oxide Nanorod / Polyaniline Nanocomposite for Removal of Amido Black 10b Dye from Aqueous Solution

**Shalmali R. Zagade[1], Sandip T. Mali[2]**

[1]Department of Civil Engineering, SCOE, Vadgaon Bk., Pune, India

[2]Department of Civil Engineering, PCCOE, Pune, India

**Abstract:** ***Dyes used in industries are mutagenic and carcinogenic and have toxic effects on living organisms. The dye effluents need to be treated before discharge to the environment. Present study deals with synthesis of iron oxide nanorods by co - precipitation method. Further the iron oxide nanorods were synthesized with PANI to form nanocomposite and further evaluated for adsorption of hazardous dye Amido black. The FTIR, XRD and SEM analysis of nanocomposite were studied. The effect of adsorbent dose, dye concentration, contact time, temperature and pH and on the adsorption capacity of nanocomposite was investigated using batch experimentation. The maximum efficiency of nanocomposite was observed to be 99 %. The acidic nature of aqueous solution was more suitable for adsorption of dye using nanocomposite. The adsorption was the basic phenomenon involved in the treatment.***

**Keywords:** Polyaniline, ironoxide nanocomposite, amido black 10B dye, adsorption

## 1. Introduction

The presence of dyes in water even at very low concentration is highly visible and undesirable [[1]. These dyes are stable to light and non - biodegradable [2]. Over 70, 000 tons of approximately 10, 000 types of dyes and pigments are produced annually worldwide. Out of which about 20 - 30% dyes are wasted in industrial effluent during textile dyeing and finishing process [3]. Therefore, dye effluents need to be treated before discharge to the environment [4].

Many techniques like electrochemical coagulation, reverse osmosis, nano - filtration, adsorption using activated materials etc., are used for the removal of dye from wastewater. process for the treatment of dyeing industry effluent [5]. The nanoparticles in combination with organic or inorganic material are best and effective in adsorption of dye from aqueous solutions. The magnetic nanoparticles properties can be tailored by using functionalized polymers to impart surface reactivity [6].

The amido black dye has anionic nature and attach strongly to cationic groups in the fibre directly. The anionic dyes can be applicable to all kind of natural fibres like wool, cotton and silk as well as to synthetics like polyesters, acrylic and rayon. But they are not substantive to cellulosic fibres. They are also used in paints, inks, plastics and leather. When a person is exposed to amido black dye, shows symptoms of coughing and shortness of breath. When in repeated contact, causes skin & eye irritation, develops burns rashes with redness and pain. Inhalation of this dye leads to irritation and damage to respiratory tract [7].

Iron oxide magnetic nanomaterial’s have unique adsorption properties due to different distributions of reactive surface sites and disordered surface regions [8]. Iron oxides also have a selective adsorptive nature for anionic dye [9]. These magnetic nanoparticles properties can be tailored by using functionalized polymers to impart surface reactivity [14].

Polyaniline has been studied most extensively since it has a unique doping mechanism, excellent physicochemical properties and good stability and its raw material can be obtained easily [10]. Polyaniline is a selective adsorbent for the anionic dyes. These characteristics are mainly due to the interaction between the negatively charged anion of the dye and the positively charged Polyaniline backbone [11]. Therefore, Polyaniline has much potential as an inexpensive and effective adsorbent for almost all anionic dyes, because of its innate cationic nature.

## 2. Experimental

### 2.1 Materials

Ferrous sulphate, sodium hydroxide (5M), camphor sulfonic acid (CSA), aniline, ammonium peroxydisulphate (APS) were used for the synthesis of polyaniline/iron oxide nanocomposite. The polyaniline / iron oxide nanocomposite was synthesized by facile sonochemical route.

### 2.2 Synthesis of iron oxide nanoparticles

20g of ferrous sulphate ($FeSO_4.7H_2O$) was dissolved in 100ml of distilled waterto form solution. The solution was then kept for magnetic stirring and ultrasonication for 25 minutes. About 50ml NaOH (5M) solution was added drop - wise into the ferrous sulphate solution while ultrasonication. The precipitation so obtained was then filtered by using whatman filter paper to obtain precipitate of iron oxide. The precipitate obtained were washed with distilled water and kept for drying in oven at 100 $^oC$ for 2 - 3 hours. Calcination of iron oxide nanoparticles was done at 300 $^oC$.

International Journal of Scientific Engineering and Research (IJSER)
ISSN (Online): 2347-3878
SJIF (2024): 6.623

### 2.3 Synthesis of polyaniline/iron oxide nanocomposite

The Polyaniline/Iron oxide (PANIO) nanocomposite was prepared by chemical oxidative polymerization of aniline in the presence of iron oxide. Initially 0.003 moles of CSA were mixed with 0.05 moles of aniline in 200ml distilled water and the solution was stirred for 15 min on magnetic stirrer. Then the mixture was pre - cooled to ~ 0 $^{o}$C in an ice bath, to form homogeneous dispersion of aniline - CSA (Camphorsulphonic Acid) complex. The mixture was then kept for ultrasonication on probe sonicator along with ice bath. The 50ml of aqueous solution of APS was added drop wise into the emulsion while it was kept for ultrasonication. Then the precipitate was filtered and washed with distilled water and kept for oven drying at 50$^{o}$C.

### 2.4 Adsorption of amido black dye using nanocomposite

The batch study was conducted by dissolving 0.050g of adsorbent to 100ml of dye solution having 20mg/l concentration and then kept for magnetic stirring at normal speed in darkness and at room temperature. The concentration of amido black dye left after adsorption was analyzed using UV spectrophotometer at wavelength of 618nm. The parameters studied using batch experimentation were adsorbent dose, concentration of dye, contact time & pH variation.

## 3. Result and Discussion

### 3.1 Characterization of polyaniline/iron oxide nanocomposite

Fig.1 shows the FTIR (Fourier Transform Infrared Spectroscopy) spectra of iron oxide nanoparticles. The peaks observed at 640 to 418 cm $^{-1}$ are characteristics of Fe - O bond stretching. The Fig.2 shows the FTIR spectra of polyaniline iron oxide nanocomposite. The peak at 2881 to 2303 cm $^{-1}$ is due C - H stretch [12]. The peak at 1500 to 1444 cm $^{-1}$ is due to C - H bending. Peak at 1236 to 1174 cm $^{-1}$ is due to C - N bond in polyaniline/iron oxide nanocomposite [12]. The alkene=C - H bending is observed at 825 to 692 cm $^{-1}$ [12]. The peak at 4475 cm $^{-1}$ shows presence of Fe - O bond The SEM (Scanning Electron Microscopy) image of iron oxide nanoparticles before calcination is shown in Fig.3. It can be seen from figure that synthesized iron oxide exhibits uniform rod- shaped morphology. The Fig.4 shows the morphology of iron oxide after calcination. It was observed that the water molecules has been removed due to which there was reduction in the particle size. The Fig.5 shows the morphology of polyaniline iron oxide nanocomposite, where it was observed that the rod shape iron oxide was covered with polyaniline and smooth rods were formed. Fig.4 shows XRD (X - Ray Diffraction) pattern of iron oxide nanoparticles. The strong bands appear with maximum intensity at 32.4$^{o}$, 35.0$^{o}$, 53.3$^{o}$ and 62.4$^{o}$ representing Bragg reflections from (220), (311), (422) and (440) planes of iron oxide. These peak corresponds to the data in the JCPDS Card No.87 - 2334 [13].

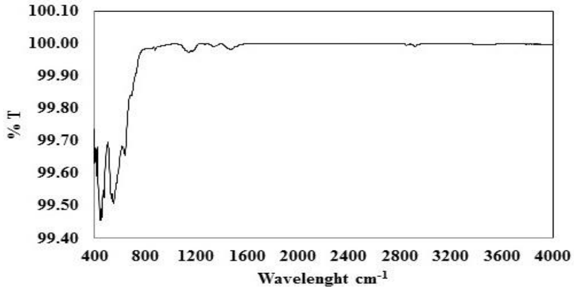


**Figure 1:** FTIR of iron oxide nanoparticles

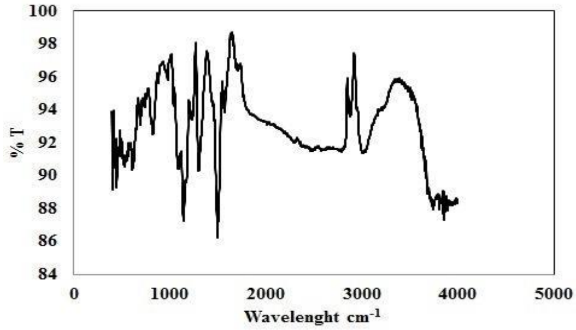


**Figure 2:** FTIR of polyaniline/iron oxide nanocomposite

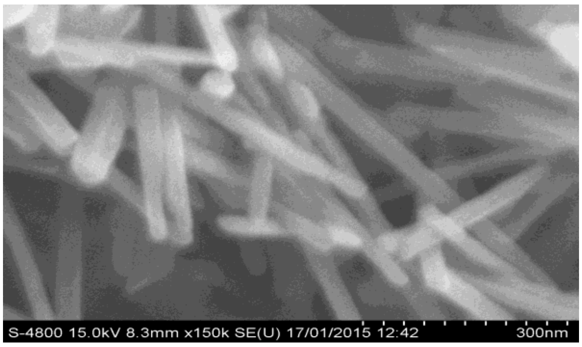


**Figure 3:** SEM image of iron oxide nanoparticles before calcinations

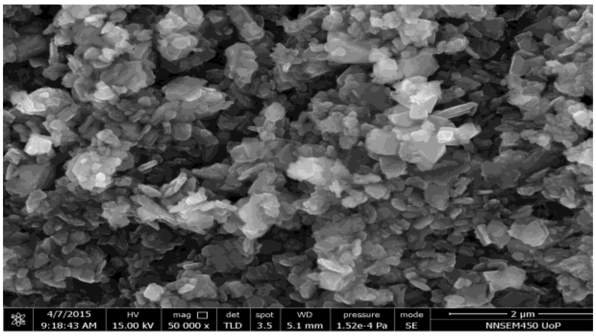


**Figure 4:** SEM image of iron oxide nanoparticles after calcination

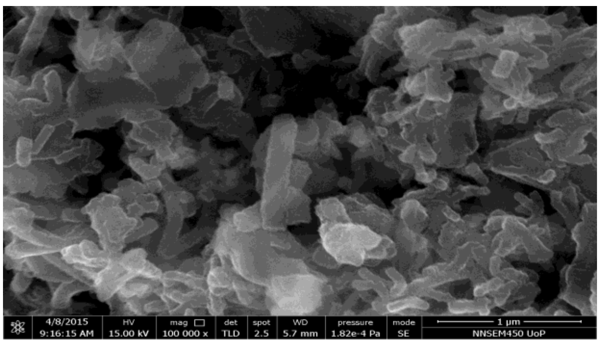


**Figure 5:** SEM image of polyaniline/ iron oxide nanocomposite after calcination

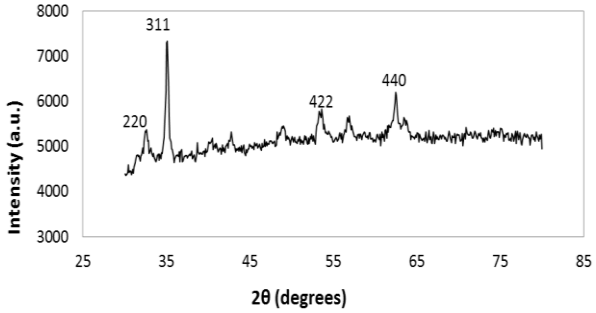


**Figure 6:** XRD of iron oxide nanoparticles

### 3.2 Effect of adsorbent dose

Increasing adsorbent dose increases the adsorption of dye. The maximum adsorption of dye is observed at the adsorbent dose of 50mg.

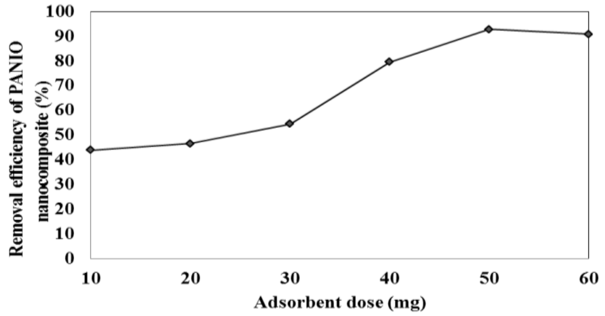


**Figure 7:** Effect of increasing adsorbent dose

### 3.3 Effect of concentration of dye

As the concentration of dye was varied from 10 to 100 mg/l with constant contact time and equal adsorbent dose of 50 mg for 100ml, the adsorption of dye was found to be decreased as shown in fig.6

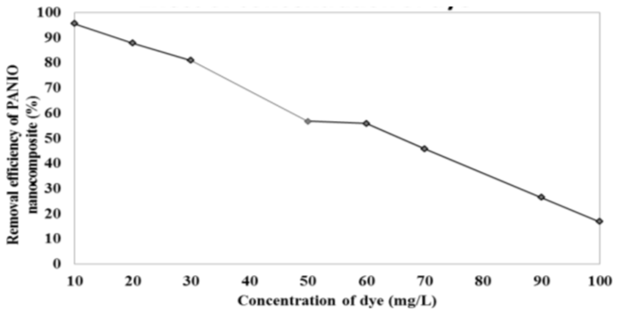


**Figure 8:** Effect of increasing concentration of dye

### 3.4 Effect of contact time

The Fig.7 shows the concentration of dye adsorbed by the adsorbent. It was observed that with increase in contact time the adsorption of dye also increases. Initially the adsorption was fast. Then the slower adsorption phase was observed where adsorption of dye was relatively small and then the equilibrium was achieved.

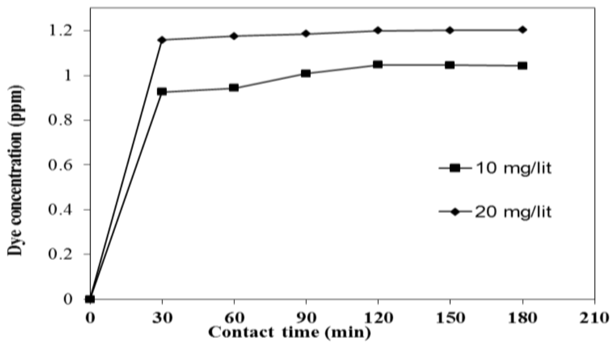


**Figure 9:** Effect of variation of contact time

### 3.5 Effect of pH variation

The adsorbent dose of 50mg and 20 mg dye concentration were selected as optimum range for analysis of pH variation. It was found that the removal efficiency was higher in acidic medium while lower in alkaline medium as shown in Fig.8. In acidic medium the $H^+$ ion concentration is more and thus the polyaniline/iron oxide nanocomposite acquires positive charge. The surface of nanocomposite attracts the anionic dye.

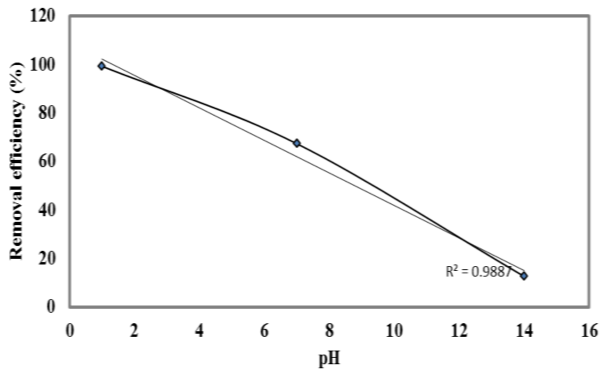


**Figure 10:** Effect of pH

### 3.6 Effect of temperature

The temperature of 100ml of 10 mg/l dye solution was kept different as 10°, 30° and 50°C. The 50 mg dose of adsorbent was added to each sample and kept for stirring. It was found that the dye solution having more temperature gives maximum efficiency and that having low temperature gives less efficiency for adsorption as shown in fig.11

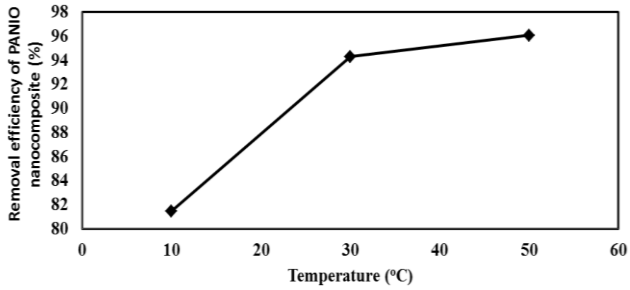


**Figure 11:** Effect of temperature

### 3.7 Adsorption Mechanism

In the present study adsorption isotherm models for Langmuir and Freundlich isotherm were evaluated using contact time parameter.

### 3.7.1 Langmuir Isotherm

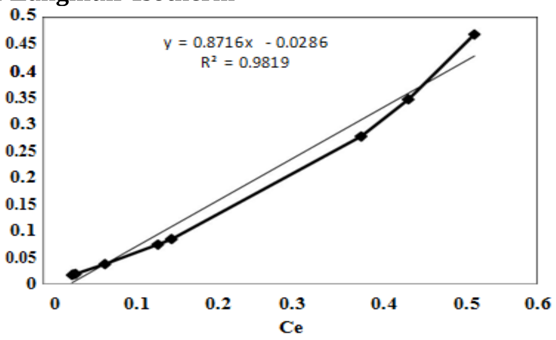


**Figure 11:** Langmuir Isotherm

According to Langmuir isotherm the $R^2$=0.9812, which is equal to unity. This shows that Langmuir isotherm is best fitted for adsorption phenomenon. Thus the monolayer adsorption occurs on the polyaniline iron oxide nanocomposite. The maximum adsorption capacity of nanocomposite is 4.052 mg/g.

### 3.7.2 Freundlich Isotherm

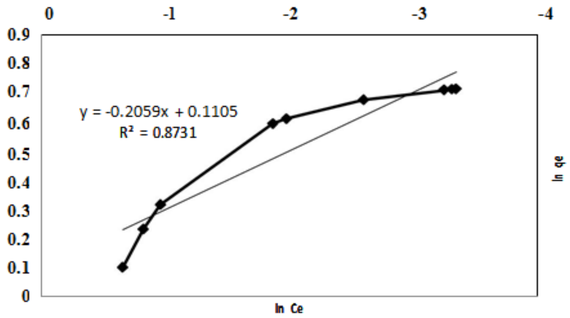


**Figure 12:** Freundlich Isotherm

The value of $R^2$ = 0.87 indicates that the Freundlich isotherm is not suitable for adsorption.

## 4. Conclusion

The ironoxide nanorods were coated with polyaniline to form rod shaped nanocomposite particles. The result shows that rod shaped polyaniline/iron oxide nanocomposite is effective adsorbent for adsorption of amido black dye. A 50mg polyaniline /iron oxide nanocomposite can decolorize as much as 100ml of amido black dye solution of 20mg/l concentration in 2 hours. The removal of amido black is pH dependent and maximum removal is attained at lower pH. The maximum efficiency observed with optimum dose of adsorbent and lower pH is about 99%. The higher temperature favors the rate of adsorption. The data obtained from adsorption isotherms are well fitted with Langmuir model which suggests the monolayer coverage of the dyes on surfaces of polyaniline/iron oxide nanocomposite.

**Acknowledgment**
The author generally acknowledge to the Savitribai Phule Pune University for SEM and XRD studies and thanks to Sir Parshurambhau College, Pune for FTIR studies.

## References

[1] Jain, R., & Sikarwar, S. (2006). photacatalytic and adsorption studies on removal of dye congo red from wastewater. *int. j. environmental pollut.*, 158 - 178.
[2] Joshi, S. K., & Srivastava, V. (2010). *Int J Chem Tech Research 2 (1)*, 427 - 435.
[3] Patil, M. R., & Shrivastava, V. S. (2014). Photocatalytic degradation of carcinogenic methylene blue dye by using polyaniline - nickel ferrite nanocomposite. *Pelagia research library*, 8 - 17.
[4] Vera, G., Aleksandra, V., & Marana, S. (2005). *Dyes and Pigments67*, 93 - 97.
[5] Sivakumar, P., & Palanisamy, P. N. (2008). Low - cost non - conventional activated carbon for the removal of reactive red 4: kinetic and isotherm studies. *Rasayan J. Chem.*, 871 - 883.
[6] Bao, X., Qiang, Z., Chang, J. H., Ben, W., & Qu, J. (2014). Synthesis of carbon - coated magnetic nanocomposite (Fe3O4[at]C) and its application for sulfonamide antibioticsremoval. *Journal of Environmental Sciences 26*, 962–969.
[7] Chemiland21http: //www.chemicalland21. com/specialtychem/finechem / AMIDO%20BLACK%2010B. ht)
[8] Tiwari, D. K., Behari, J., & Sen, P. (2008). Application of nanoparticles in wastewater treatment. *World Applied Science Journal3 (3)*, 417 - 433.
[9] Stipp, S., Hansen, M., Kristensen, R., Hochella, M. F., Bennedsen, L., Diderikesen, K., et al. (2012). *Chem. Geol.190*, 321 - 337.
[10] Dhawan, S. K., Singh, N., & Rodrigues, D. (2003). *Science Technology Adv. Material 4*, 105 - 113.
[11] Mahanta, D., Madras, G., Radhakrishnan, S., & Patil, S. (2009). *J. Phys. Chem. B 113*, 2293 - 2299.
[12] Rais, A. K., & Kumar, R. (2010). Conducting polyaniline/iron oxide composite: A novel adsorbent for removal of amido black 10B. *Journal of chemical engineering* Data 55 (9), 3489 - 3493.
[13] Xiaonan Liu, Fanghua Zhu, Wei Wang, Jiehong Lei, Guangfu Yin (2016). Synthesis of Single - Crystalline Iron Oxide Magnetic Nanorings as Electrochemical Biosensor for Dopamine Detection. *International journal of electrochemical science*, 9696 – 9703.
[14] Bao, X., Qiang, Z., Chang, J. H., Ben, W., & Qu, J. (2014). Synthesis of carbon - coated magnetic nanocomposite ($Fe_3O_4$[at]C) and its application for sulfonamide antibiotics removal. *Journal of Environmental Sciences 26*, 962 - 969.