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Green Synthesis of Iron Nanoparticles using Albizia lebbeck leaves for Synthetic Dyes decolorization

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Abstract**—**The innovative and impeccable nano technology is highly familiar for its wide applications. The present experimentation is carried out with albizia lebbeck leaves for removal of dyes using Iron nano particles. The operating parameters involved are contact time, pH, initial concentration, dosage of nano particle solution and temperature. The dyes studied are Congo red and Crystal violet. The characterization studies involved are FTIR and XRD. The optimum pH is 5 for crystal violet using albizia lebbeck nano particle solution. The equilibrium contact time obtained is 40 min. Similar trend also followed for Congo red dye. The positive attributes confirmed that albizia lebbeck leaves broth along with iron chloride formed iron nano particles and are capable of removing crystal violet and congo red dyes

Index Terms**—Dyes, pH, concentration, temperature, dosage, FTIR and XRD.**

I. INTRODUCTION

Textile industry effluents are highly toxic not only to humans but even microorganisms and plant species. These dyes in water stop the passage of sunlight to roots of the plants and sustainability of microorganisms. Hence these dyes from industries are to be pretreated before leaving them on to earth's surface. As there are several methods for pretreatment yet none of them are ecofriendly and free from chemicals. Hence search for alternate methods has risen and sought the attention towards nano technology. Hence the present study is focused on removal of dyes using nano particles. The present experimentation is designed in such a way that the operating parameters like contact time, pH, concentration, dosage and temperature will be optimized along with the confirmation through characterization using FTIR and XRD.

II. EXPERIMENTAL PROCEDURE

The present experimentation is carried out in batch process for removal of dyes (congo red and crystal violet) from synthetic solutions by using albizia lebbeck leaves broth with iron nano particles (al-fe-nps).
Analytical grade chemicals were used for experimentation and need no further purification. Double distilled water is used to prepare all stock and synthetic solutions. From a stock solution containing 1000 mg of dyes in 1.0 litre, the synthetic solutions of dyes were made. By addition of O.l M HCl and O.l M NaOH solutions the pH of dyes solutions were adjusted to the desired value.

Fig. 1 Dyes

Fig. 2 Dyes stock solutions(a)Congo Red(b)Crystal Violet

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2. Preparation of the Broth solutions and Nano particles formation:

2.1 Preparation of AlbiziaLebbeck broth :

In this process 25gm of fresh and cleaned leaves of AL are taken in a magnetic stirrer and to this 100 ml of distilled water is added and it is heated at 70^{o}C for 15min.After that the solution is filtered in 250 ml conical flask using whatmann's filter paper and it is kept aside for further process. The broth obtained is in pale brown colour.

Fig. 3 Albizia Lebbeck Leaves

2.2 Preparation of Nano Particles:

In this process 10 ml of broth solution is taken and to that 5 gms of Fecl3.6H2O is added in a 250 ml conical flask and is kept in an orbital shaker for 24 Hrs in order to obtain nano particles. The nano particles formation is noticed when the brown color is changed to black color. This solution is used for various dyes degradation process of different concentrations and different dosages.

Fig. 4 Nano particles solutions AlbiziaLebbeck

2.3 Preparation of 1000 mg/L dyes stock solutions:

To prepare 1000 ppm of congo red and crystal violet stock solution, 1.0 g of congo red and crystal violet were dissolved in 1.0 liter of double distilled water. From this stock solution synthetic samples of different concentrations of dyes were prepared by appropriate dilutions. 100 ppm dye solution was prepared by diluting 100 ml of 1000 ppm dye stock solution with distilled water in 1000 ml volumetric flask up to the mark. Similarly solutions with different dye concentrations such as 20, 50, 80, 120 and 150 ppm were prepared.

2.4 Characterization Studies: The characterization studied here are XRD AND FTIR. The prepared broth was centrifuged at 10,000 rpm for 2 hrs and obtained a thick paste which was dried in hot air over and further send to advanced analytical laboratory for FTIR and XRD study.

Fig. 5. Centrifuge samples for drying and Characterization analysis

(FTIR & XRD)

Fig. 6 equilibrium studies

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III. RESULTS AND DISCUSSION

3.1. CHARACTERIZATION

3.1.1 FTIR Spectrum

FTIR is a type of spectroscopy that measures absorption, emission and and photoconductivityof solids, liquids and gases. The peaks visible in the FTIR graph (Fig. 7) are due to the amide linkages between amino acid residues in the proteins.

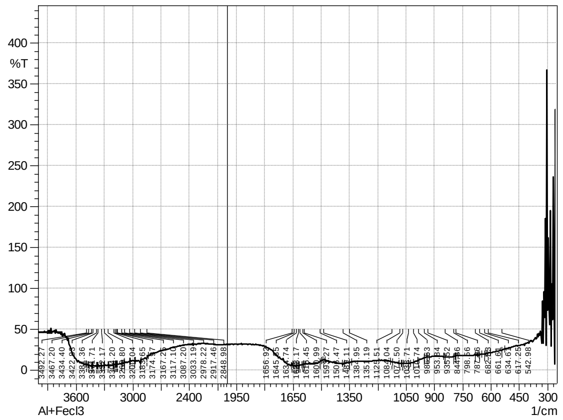

Fig 7 FTIR Spectrum of Albizia lebbeck nano particles

3.1.2 X–Ray Diffraction

X-ray diffraction is widely used for X-ray crystallography. Fig. 8 and Fig. 9 shows the XRD for the Iron nanoparticles using Albizia lebbeck broth and their matching compounds. The data shows diffraction peaks at 2θ values of albizia lebbeck are 0.7928,0.7901,0.7571,0.7891,0.7978,0.7405and 0.7636 corroborate the presence of Cs_3Sb_7,Cs_{23}.$O_{14.15}Sn_8$, BHO_4Zn_2, Ba_2SnTe_5, $Ag_5Fe_3Na_2O_{28}P_8$, Ba_3NaO_9, $Al_5Nd_{9.959}$O28. Their corresponding d-values are 16.049, 2.9612, 3.9932, 2.4136, 2.3230, 5.0953, 4.7827.

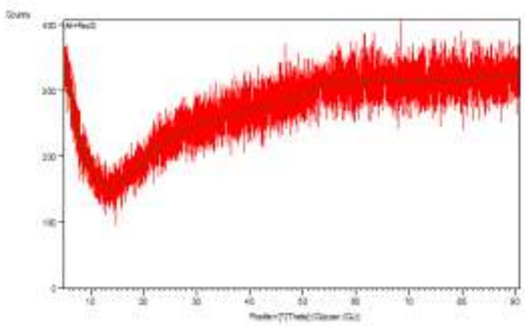

Fig 8 XRDpattern of Albizia lebbeck nano particles

Table 3.1 FTIR peaks & their bonds for Albizia Lebbeck

Sl No	Peaks in untreated powder, cm^{-1}	Bond and Functional group Description
1	542.98	C–Br stretch bands from alkyl halides
2	617.25	C–Br stretch bands from alkyl halides
3	634.61	C–Br stretch bands from alkyl halides
4	661.61	C–Br stretch bands from alkyl halides
5	682.83	C–Br stretch bands from alkyl halides
6	787.96	Aromatic C-H Bending
7	798.56	Aromatic C-H Bending
8	844.86	C–Cl stretch alkyl halides
9	935.52	S = O and C–S–O bandsfrom ester sulfonate
10	953.84	S = O and C–S–O bandsfrom ester sulfonate
11	986.63	S = O and C–S–O bandsfrom ester sulfonate
12	1010.74	S = O and C–S–O bandsfrom ester sulfonate
13	1038.71	–CH_2 bending vibrations
14	1071.50	– C–N Stretching
15	1084.04	Amide N-H bending vibrations
16	1125.51	Alkenyl C=C Stretch
17	1351.19	O-H bond
18	1384.95	Alkynyl C-H Stretch
19	1489.11	Amine N-H Stretch
20	1506.47	Amine N-H Stretch
21	1593.27	Amine N-H Stretch
22	1600.99	Amine N-H Stretch
23	1615.45	Amine N-H Stretch
24	1623.17	Amine N-H Stretch
25	1634.74	Amine N-H Stretch
26	1645.35	Amine N-H Stretch
27	1656.92	O-H bond
28	2848.98	Alkenyl C-H Stretch
29	2917.46	Alkenyl C-H Stretch
30	2978.22	Alkenyl C-H Stretch

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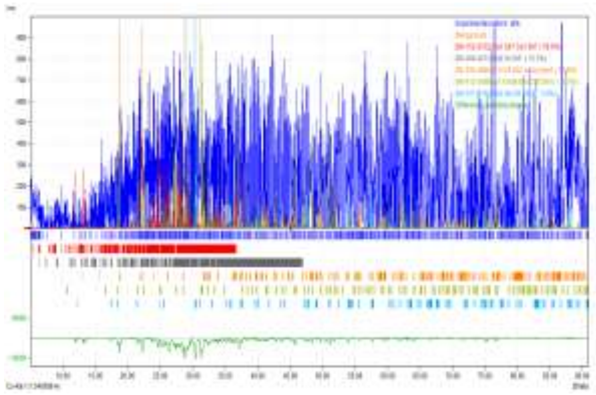

Fig 9 XRD pattern of Albizia lebbeck nano particles with matching compounds

3.2 Effect of Contact time:

Contact time is defined, as the time required for removal of Dyes CV, and CR using the broth Albizia Lebbeck. The Contact time is determined by plotting the % removal of Dyes CV against Contact time in Fig. 10 for 20 ppm of Dye solutions using Albizia lebbeck broth. The time intervals are observed from 1 min to 720 min. The % Removal of Dye CV for the 1 min is 14 % and it has been increased up to 60 % till the time 40 min and later it remained constant for the time up to 720 min from 40 min. The % Removal of Dye CR for the 1 min is 9 % and it has been increased up to 42 % till the time 50 min and later it remained constant for the time up to 720 min from 50 min. This suggests that rate of sorption of dye on the nanoparticle is quite fast. It is but obvious that the more number of nanoparticles, the more is the availability of catalyst for attacking the chromophoric system of the dye **[05, 07-08, 12-13]**

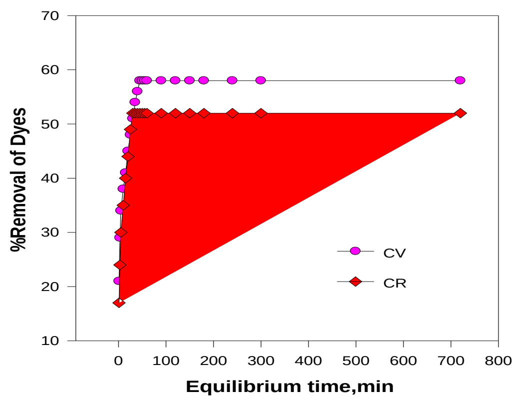

Fig. 10 Effect of Equilibrium time using AL leaves for % removal

3.3 Effect of pH:

pH of the aqueous solution plays a vital role in removal of Dyes. The pH of the Dye solution is dependent on the Contact time. In order to determine the optimal value, pH of the aqueous solution is varied from 2 to 8. The pH of aqueous solution is shown against % removal of Dye using Broth Albizia Lebbeck in Fig. 11 The % removal of Dyes CV and CR is increased from 42 % to 64 % as pH is increased from 2 to 5 and decreased beyond pH value of 5 for Dye CV and is increased from 24 % to 48% as pH is increased from 2 to 6 and decreased beyond pH value of 6 for Dye CRdue to competition with H^+ ions for appropriate sites on the Dye molecules. However, with increasing pH, this competition weakens and Fe^{2+}ions replace H^+ ions bound to the Broth. Variation in pH is one of the most significant factors which affect the extent of decolourisation by physicochemical treatment. Therefore the effect of pH of dye solution on adsorption capacity of the nanoparticle was investigated **[04, 05, 07-09]**.

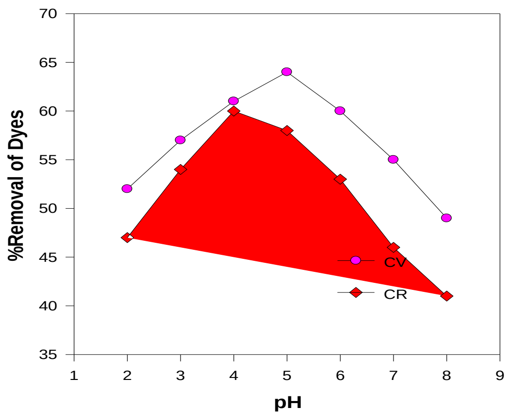

Fig. 11 Effect of pH using AL leaves for %removal

3.4 Effect of concentration :

The experiments were carried out using various concentrations of Dyes CV and CR From 2 ppm (mg/l) to 200 ppm (mg/l)at their respective pH values and at their equilibrium contact time. The effect of initial concentration of Dyes CV and CR in the aqueous solution are shown in Fig. 12. The % removal is decreased from 68% to 27% for CV dye and decreased from 64% to 23% for CR dye with an increase in Concentration from 20ppm (mg/L) to 200ppm (mg/L).The dye uptake capacity of the nanoparticle gradually decreases with an increase in dye concentration. This may be attributed to more dye molecules available for the equilibrium initially. However, there was a gradual decrease in % removal of the dye as the concentration of dye liquor was increased. It is probably because the increase in dye uptake was not in the proportion of increase in dye concentration**[1–2,7-9]**.

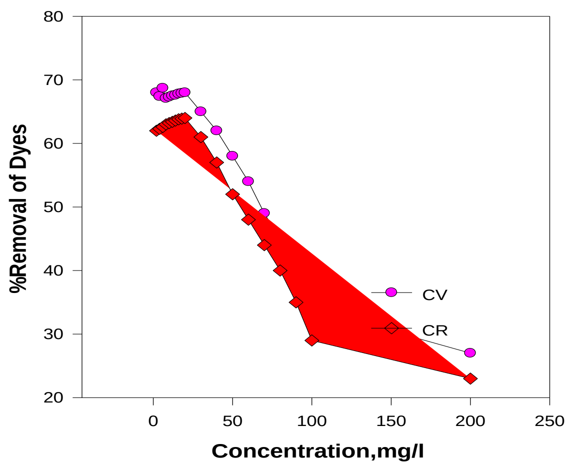

Fig. 12 Effect of concentration using AL leaves for %removal

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3.5 Effect of dosage:

The % removal of Dyes is drawn against dosage varying from 1ml to 10 ml for CV and CR dyes in Fig. 13. The removal of Dyes increases from 65% to 80% with an increase in dosage from 1 ml to 5 ml and later on it remainedconstant up to 10ml for CV and for removal of CR Dyes,% increased from 52% to 76% with increase in dosage from1 ml to 4 ml and later on it remained constant up to 10 ml. Similar trend was followed for the removal of CV & CR Dyes. The % removal increased from 52% to 84% with an increase in dosage from 1 ml to 6 ml and achieved a constant value up to 10ml for CV dye. The % removal of CR dye increased from 69% to 82% with increase in dosage from1 ml to 4 ml and attained constant value on further increasing the dosage up to 10 ml.From Figure 7, it is observed that as the nano particle solution dosage increased, the percentage removal also increased until it reaches a saturation point. Further increase in nano particle solution dosage did not change the percentage removal at all. An increase in sorption rate with nano particle solution dosage can be attributed to increased surface area and the availability of more sorption sites. It may be due to more surface sites available for reaction with dyes to accelerate its removal **[9,13-16]**.

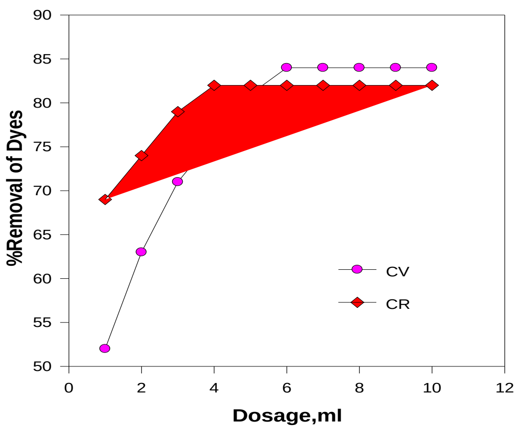

Fig. 13 Effect of Dosage using AL leaves for %removal

3.6 Effect of temperature:

As the experimentation is performed for various temperatures i.e. from 10 to 50^0C, the % removal of CV dyes for AL-Fe-NPS is plotted against five different temperatures of 10^0C, 20^0C, 30^0C, 40^0C, 50^0C. The % removal of the dyes is shown in Fig. 14. The % removal of CV dyes is increased on increasing the temperature i.e from 71% to 82% for CV dye **[13,17-20]**. The temperature of the solution plays an important role on the sorption capacity. If the sorption capacity increases with increasing temperature then the sorption is an endothermic process.

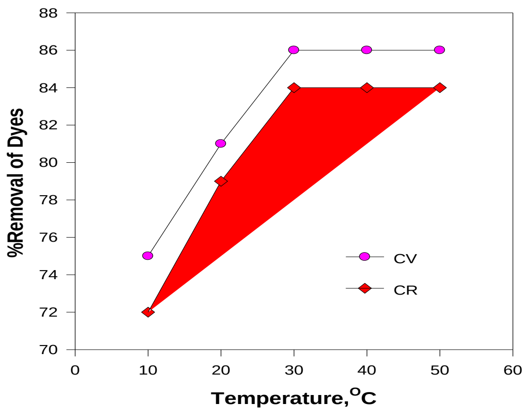

Fig. 14 Effect of Temperature using AL leaves for %removal

The above results and discussions clearly depicts that albizia lebbeck iron oxide nano particles solutions are capable of removing synthetic dyes from aqueous solutions.

IV. Conclusions

The analysis of the experimental data resulted in the following conclusions:

A)Removal of crystal violet with albizia lebbeck broth nano solution

The maximum removal of crystal violet dye (96.5926 %) onto AL broth Fe nano solution is observed when the processing parameters are set as: t = 40 min, pH = 5, w = 5 mg/L, Co = 20mg/L and T = 303 K.

B)Removal of congo red with albizia lebbeck broth nano solution

When the processing parameters are set as: pH = 6, w = 4 mg/L, Co = 20 mg/L and T = 303 K then, the maximum removal of congo red (96.5926 %) onto AL broth Fe nano solution is observed. With the above conclusions the authors confirm that the above mentioned leaves are capable of removing dyes using iron nano particles.

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