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Studies on Leaf Extract Mediated Synthesis of Copper Nanoparticles for the Removal of Bromo Cresol Green Dye from Synthetic Waste Waters

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Abstract— **Present work describes the synthesis of copper nanoparticles using leaf extract which were used to prepare nano particles using green synthesis process for removing bromo cresol green (BCG) from aqueous solution. An adaptable technique was implemented for the synthesis of Copper Nanoparticle using leaves extract of bromo cresol green (BCG). The preparation of copper nanoparticles by using leaf extract has desired quality with low cost and convenient method. The leaf extract was mixed with copper salt solution by heating to a temperature of 60-70 ^{o}C and the reduction reaction was studied by observing the colour change. Equilibrium studies and isotherms were applied to correlate the preparation variables (time, pH, concentration and dosage) to the Decolourization of BCG from aqueous solution. The results obtained showed that the optimum conditions for preparing nano particles using BCG dye were contact time of 90 min and pH-4, concentration-20mg/L and dosage-0.5 g/L which resulted in 30.95 mg/g sorption capacity of BCG from aqueous solution at pH 4.0. The copper nano particles seems to be a good adsorbent for the decolorization of anionic dyes in particular BCG from synthetic waste water.**

Index Terms— **Copper Nanoparticle, Bromo Cresol Green (BCG), green synthesis, Isotherms, Kinetics, Sorption.**

1) INTRODUCTION

Nanotechnology is rapidly increasing field that making an impact on human life such as pharmaceutical, health, food, electronics, chemical industry, energy science, cosmetics, environmental science and space industries etc. There are many ways to synthesize nanoparticles such as sol gel method, chemical reaction, solid state reaction and co-precipitation. Compared to those methods green synthesis method is one of the best method for the synthesis of nanoparticles in recent years. This method have several advantages namely low cost, simple, use of less toxic materials, most important is eco-friendly. In this method, the plant extract has been used as reducing agent for the synthesis of copper nanoparticles **[01-05]**. In the present work we have developed green synthesis of copper Nanoparticles via a single-step, room-temperature reduction of dye using cassia occidentalis leaf extract **[06-10]**. In addition to providing enhanced research and development strategies, green chemistry can also play a prominent role in guiding the development of nanotechnology to provide the maximum benefit of these products for society and the environment.

2) EXPERIMENTAL PROCEDURE

The present experimentation is carried out in batch process, on adsorption of dye (Bromo Cresol Green BCG) from aqueous solutions by using Cassia Occidentalis leaves broth with copper nano particles (Co-Cu-NPS).

The experimental procedure consists of

1. Reagents and Materials
2. Preparation of Broth Solution and Nano particles formation
3. Preparation of 1000 mg/L dyes stock solutions
4. Characterization studies
5. Equilibrium Studies
6. Isotherms
7. Kinetics models

The dye considered for present study is Bromo Cresol Green

Fig.1.1 Dyes

Fig. 1.2 Chemicals

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1. Reagents and materials:

Analytical grade chemicals were used for experimentation and need no further purification. The sources of dye used were Bromo Cresol Green of analytical grad. Double distilled water is used to prepare all stock and synthetic solutions. From a stock solution containing 1000 mg of dye in 1.0litre, the synthetic solutions of dye were made. By addition of O.l M H_2SO_4 and O.l M NaOH solutions the pH of dye solutions were adjusted to the desired value.

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

*2.1Preparation of Cassia Occidentalis***:**

In this process 10 gm of fresh and cleaned leaves of CO are taken in a magnetic stirrer and to this 110 ml of distilled water is added and it is heated at 70^{o}C for 10min.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 yellow colour.

Fig.2.1 Broth solutions

2.2 Preparation of Nano Particles:

In this process 10 ml of broth solution is taken and to that 0.169 gms of $CuSO_4.5H_2O$ 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 pale yellow color is changed to light brown. This solution is used for various dye degradation process of different concentrations and different dosages.

Fig.2.2 Nano particles solutions (a) cassia occidentalis

3. Preparation of 1000 mg/L dye stock solutions:

To prepare 1000 ppm of Bromo Cresol Green –BCG were dissolved in 1.0 liter of double distilled water. From this stock solution synthetic samples of different concentrations of dye 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, 100, 150 and 200 ppm were prepared.

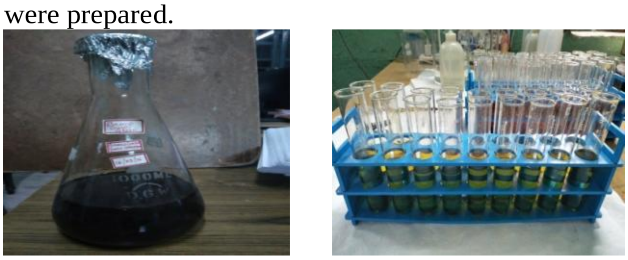

Fig. 3 Dye stock solutions (a) Bromo Cresol Green

RESULTS AND DISCUSSIONS:

In the present investigation, the prospectives of one adsorbents namely copper cassia occidentalis nano particals were evaluated to estimate their performance for the decolourization of Bromo Cresol Green dye present in aqueous solutions. The effects of parameters on decolourization of BCG dye were measured, data consisting of contact time, pH of the solution, initial concentration, dosage. The experimental data are obtained by conducting both Batch and Continuous experiments.

4. CHARACTERIZATION:

FESEM Analysis of Nano Particles:

- FESEM (Field Emission Scanning Electron Microscope) to measure and observe the small structures (as small as 1Nanometer = one billion of a millimeter) on the surface of cells**.**
- It provides high resolution and very high and stable probe currents for optimum imaging and analytical performance at high vacuum.

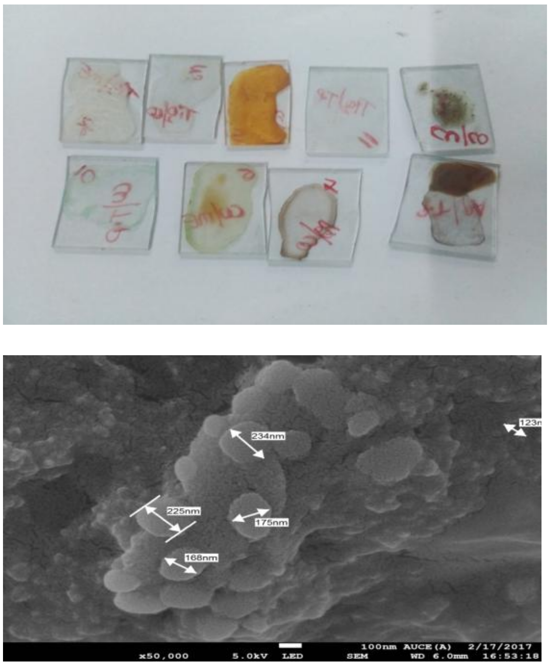

Fig.4 FESEM image of copper nano particles

Probe size **- sub-100 nm scale** and Magnification ranges from **25 to 1,000,000X**. With the integration of the in-the-lens acceleration and deceleration of the electron beam, low kV aberrations are reduced, yielding high resolution at the lowest accelerating voltages. Beam deceleration decreases charging while imaging non-conductive specimens, improves spot size at low kV, and enhances surface topography. The nano particles produced were in the range of 123 to 234 nm **[13–22]**.

5. Equilibrium studies on dye decolourization:

5.1 Effect of Equilibrium time

The decolourization of dye as a equilibrium studies using the dye Bromo Cresol green (BCG) at 303K (Room Temperature). The quantity of 20 ml of dye solution was taken along with 5ml of Cassia occidentalis broth copper nano particles (co-cu-nps) solution with varied time intervals ranging from 1-180 min. Initially, dye adsorbed and occupied selectively the active sites on the co-cu-nps solution. As the equilibrium time increased the active sites on the co-cu-nps were filled. The adsorption rate slowed down gradually and reached exhaust stage resulting a constant value. The % Decolourization of dye & dye uptake capacities were shown in fig. 5.1. The optimum time obtained is 90 min with the highest decolourization of dye was 65%. The rapid uptake of the dye indicates that the adsorption process could be ionic in nature where the anionic dye molecules attach to the various positively charged natural functional groups present on the surface of the co-cu-nps **[23-32]**.

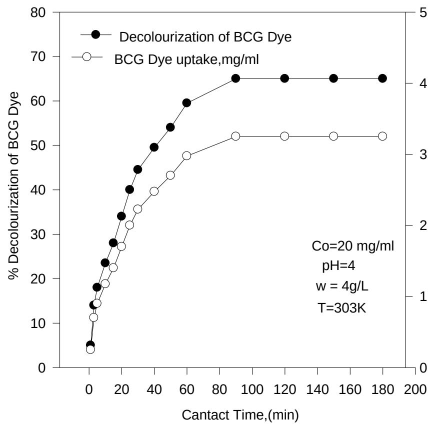

Fig.5.1 Contact Time with % dye decolourization of Bromo cresol green

5.2 Effect of pH

pH is one of the significant parameter which effects on the decolourization of the dye. The effect of pH was studied in the Dye Decolourization using the Cassia Occidentalis copper nanoparticles, the experiments was carried out and results are presented in Fig 5.2. The removal was increased from 59 % to 74.5 % as pH was increased from 2 to 4, The pH is varied for dyes using Cassia Occidentalis broth solution Cassia Occidentalis where as further increase in pH had a negative effect. The maximum % decolourization was found to be 74.5 % at pH 4 for BCG Dye. At Low pH depresses dye decolourization due to competition with H^+ ions for appropriate sites on the adsorbent surface. However, with increasing pH, this competition weakens and Bromo cresol green ions replace H^+ ions bound to the adsorbent. which causes a repulsive force between the dye and nano particles**[33-42]**.

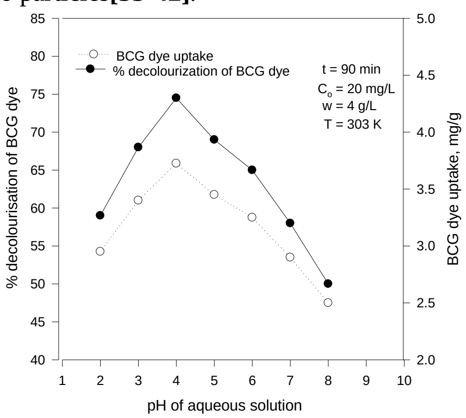

Fig.5.2 Effect of pH along with % dye decolourization of Bromo Cresol Green

5.3 Effect of initial concentration of dyes:

The initial concentration plays a key role in the decolourization of the dye. The different initial concentrations were taken and the results are studied and presented in the Fig 5.3. At the initial concentration of 20mg/l, the 75% decolourization of dye is obtained and different concentrations are taken by using Cassia Occidentalis copper nano particle solution and on further increase in concentration (200 mg/L), %dye decolourization has decreased. This is due to higher interaction between Cassia Occidentalis copper nano particles and the dye solution. The maximum decolourization of BCG Dye is 75%. **[43-52].**

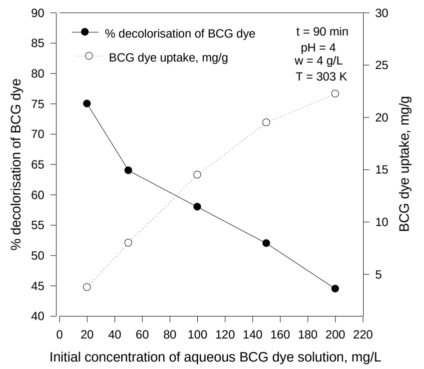

Fig.5.3 Initial concentration with % dye decolourization of Bromo cresol green

5.4 Effect of nano particle solution dosage:

The dosage of the Cassia Occidentalis copper nano particle was varied in the decolourization of the dye Bromo Cresol green(BCG) and the results are depicted in the Fig 5.4. and it showed that % Decolourization of Dye (BCG) increased and uptake decreased with increase in dosage. The maximum % decolourization of dye is attained at 0.5g and was almost constant at higher dosages. This trend could be explained as a consequence of partial aggregation. Therefore, the optimum dosage was selected as 0.5g for further experiments. The maximum % Decolourization of BCG dye is 88%.It may be due to more surface sites available for response with immediate dye to increase speed its decolorization. A decrease in overall adsorption capacity was observed with the increase in adsorbent dosage because adsorption of the dye did not increase in the proportion of increase in nano particles dosage **[53-62].**

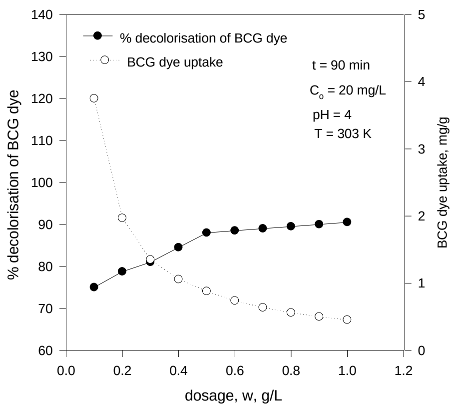

Fig. 5.4 % dye decolourization of Bromo cresol green on dosage

6. Isotherms

6.1. Langmuir isotherm*:*

Irving Langmuir developed an isotherm named Langmuir isotherm. It is the most widely used simple two- parameter equation. This simple isotherm is based on following assumptions:

Adsorbates are chemically adsorbed at a fixed number of well- defined sites, Each sit can hold only one adsorbate species, All sites are energetically equivalent, There are no interaction between the adsorbate species. The Langmuir relationship is hyperbolic and the equation is:

$q_e/q_m = bC_e/ (1+bC_e)$

The above Equation can be rearranged as

$(C_e/q_e) = 1/(bq_m) + C_e/q_m$

From the plots between (C_e/q_e) and C_e, the slope $\{1/ (bq_m)\}$ and the intercept (1/b) are calculated. Further analysis of Langmuir equation is made on the basis of separation factor, (R_L) defined as $R_L = 1/ (1+bC_e)$

$0<R_L<1$	indicates	favorable adsorption
$R_L>1$	indicates	unfavorable adsorption
$R_L = 1$	indicates	linear adsorption
$R_L = 0$	indicates	irrepressible adsorption

Langmuir isotherm is drawn for the present data and shown in Fig.6.1. The equation obtained 'n' $C_e/q_e = 0.0323\ C_e + 1.4294$ with a good linearity (correlation coefficient, R^2~0.9819) indicating strong binding of Bromo cresol green dye to the surface of *copper nanoparticles*.

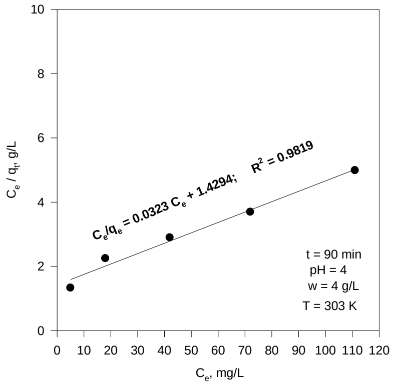

Fig. 6.1 Langmuir isotherm for % dye decolourization of Bromo cresol green

6.2. Freundlich isotherm:

Freundlich presented an empirical dye decolourization isotherm equation that can be applied in case of low and intermediate concentration ranges. It is easier to handle mathematically in more complex calculations.

The Freundlich isotherm is given by

$q_e = K_fC_e^n$

whereK_f(mg) represents the dye decolourization capacity when dye equilibrium concentration and n represents the degree of dependence of dye decolourization with equilibrium concentration Taking logarithms on both sides, we get

$\ln q_e = \ln K_f + n \ln C_e$

Freundlich isotherm is drawn between ln C_e and ln q_e in Fig.6.2 for the present data. The resulting equation ln q_e = 0.5946 ln C_e+ 0.3810; has a correlation coefficient of 0.9934.

The 'n' value in the above equations satisfies the condition of 0<n<1 indicating favorable dye decolourization.

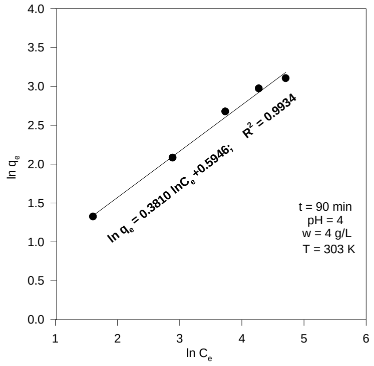

Fig. 6.2 Freundlich isotherm for % dye decolourization Bromo cresol green

6.3.Temkin isotherm:

Temkin and Pyzhev isotherm equation describes the behavior of many dye decolourization systems on the heterogeneous surface and it is based on the following equation

$q_e= RT \ln(A_TC_e)/b_T$

The linear form of Temkin isotherm can be expressed as

$q_e= (RT/ b_T) \ln(A_T) + (RT/b_T) \ln(C_e)$

where A_T= exp [b(0) x b(1) / RT]

b(1) = RT/ b_T is the slope

b(0) = (RT/ b_T) ln (A_T) is the intercept and

b= RT/b(1)

The present data are analysed according to the linear form of Temkin isotherm and the linear plot is shown in Fig.6.3. The equation obtained for Bromo cresol green dye decolourization is: $q_e = 6.1610 \ln C_e - 7.6232$ with a correlation coefficient 0.9621. The best fit model is determined based on the linear regression correlation coefficient (R^2). From the Figs 6.1,6.2 & 6.3, it is found that dye decolourization data are well represented by Freundlich isotherm with higher correlation coefficient of 0.9934, followed by Langmuir and Temkin isotherms with correlation coefficients of 0.9819 and 0.9621 respectively**[63–72]**.

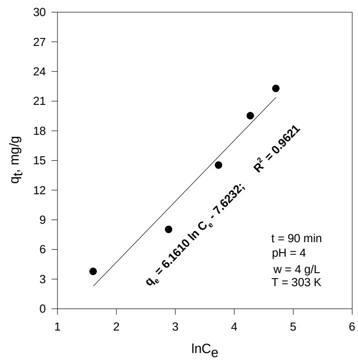

Fig. 6.3 Temkin isotherm for % dye decolourization of Bromo cresol green

Table - 1

Isotherm constant:

Langmuir isotherm	Freundlich isotherm	Temkin isotherm
q_m = 30.959 mg/g	K_f = 1.4637 mg/g	A_T = 0.2901 L/mg
R_L = 0.8984	n = 0.5946	b_T = 415.632
R^2 = 0.9819	R^2 = 0.9934	R^2 = 0.9621

7. Kinetics of dye decolourizations:

The order of adsorbate – adsorbent interactions have been described using kinetic model. Traditionally, the first order model of Lagergren finds wide application. In the case of dye decolourization preceded by diffusion through a boundary, the kinetics in most cases follows the first order rate equation of Lagrangen:

$(dq_t/dt)= K_{ad} (q_e – q_t)$

where q_e and q_t are the amounts adsorbed at t, min and equilibrium time and K_{ad} is the rate constant of the pseudo first order dye decolourization.

The above equation can be presented as

$\int (dq_t /(q_e – q_t))= \int K_{ad} dt$

Applying the initial condition $q_t = 0$ at $t = 0$, we get

$\log (q_e – q_t) = \log q_e – (K_{ad}/2.303) t$

$\log (q_e – q_t) = 0.4861-0.0159t$

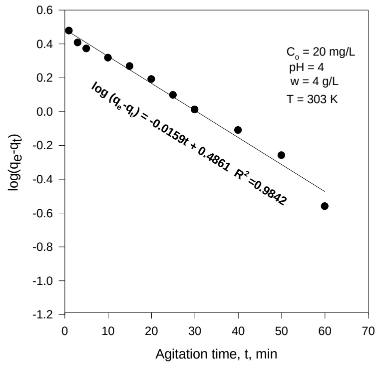

Fig. 7.1 first order kinetics for % dye decolourization of Bromo cresol green

Plot of $\log (q_e–q_t)$ versus‘t’ gives a straight line for first order kinetics, facilitating the computation of adsorption rate constant (K_{ad}). If the experimental results do not follow the above equation, they differ in two important aspects:

i) $K_{ad} (q_e – q_t)$ does not represent the number of available dye decolourization sites and

ii) $\log q_e$ is not equal to the intercept.

In such cases, pseudo second order kinetic equation: $(dq_t/dt) = K (q_e – q_t)^2$ is applicable,

Where ‘K’ is the second order rate constant.

The other form of the above equation is: $(dq_t /(q_e–q_t)^2)= Kdt$

let $q_e – q_t = x$

$dq_t = dx$

$1/ x = K x + C$

$C = 1/ q_e$ at $t = 0$ and $x = q_e$

Substituting these values in above equation, we obtain:

$1/(q_e – q_t) = Kt + (1/q_e)$

Rearranging the terms, we get the linear form as:

$(t/q_t) = (1/ Kq_e^{\,2}) + (1/q_e) t.$

$(t/q_t) = 0.2743 t + 4.9634.$

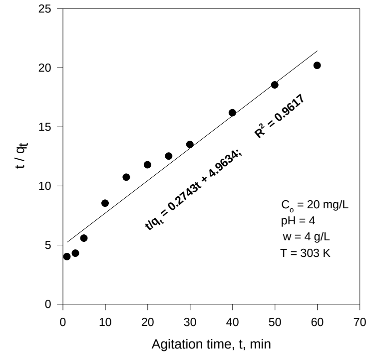

Fig. 7.2 second order kinetics for % dye decolourization of Bromo cresol green

The pseudo second order model based on above equation, considers the rate -limiting step as the formation of chemisorptive bond involving sharing or exchange of electrons between the adsorbate and adsorbent. If the pseudo second order kinetics is applicable, the plot of (t/q_t) versus ‘t’ gives a linear relationship that allows computation of q_e and K.

In the present study, the kinetics are investigated with 25 mL of aqueous solution (C_0= 20 mg/L) at 303 K with the interaction time intervals of 1 min to 180 min. Lagragen plots of $\log (q_e-q_t)$ versus contact time (t) for dye decolourization of Bromo cresol green the interaction time intervals of 1 to 180 min are drawn in figs.7.1 & 7.2 **[53-82]**.

Table – 2

Equations and rate constants:

Order	Equation	Rate constant	R^2
Lagergren first order	$\log (q_e-q_t) = –0.0159 t + 0.4861$	0.0366 min^{-1}	0.9842
Pseudo second order	$t/q_t = 0.2743 t + 4.9634$	0.0151 g/(mg-min)	0.9617

8. Conclusions

Investigations are carried out to find out the equilibrium conditions for the decolorization of BCG dye from an aqueous solution using cassia occidentalis copper oxide nano particle solution. The analysis of the experimental data results in the following conclusions:

The equilibrium contact time for cassia occidentalis dye is 90 minutes. % dye decolourization of Bromo Cresol Green dye from the aqueous solution increases significantly with increase in pH from 2 (59%) to 4 (74.5%) and later decreased. The maximum dye decolourization was obtained at a concentration of 20 mg/l (75% & 3.75mg/ml). The optimum dosage for dye decolourization is 0.5g (88 %). The maximum dye decolourization obtained when the processing parameters are set as: t = 90 min, pH = 4, w = 0.5 g, Co = 20 mg/l onto Co-Cu-NPS was observed. Hence the experimented Co-Cu-NPS is capable of decolorizing BCG dye.

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