

# Studies on Green Synthesis of Iron Nanoparticle for Solo Chrome Black (SCB) Dye Decolorization

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**Abstract**— The objective of this study was to minimize the threat to the environment by utilizing the green *Datura* leaf extract process and iron nanoparticles in the removal of solo chromo black (SCB) dye from aqueous solutions. In recent time rising an invincible interest to detect a cost-effective and eco-friendly material for the removal of hazardous chemicals from contaminated water. Among the different technologies, Nano technology studies emerge as a destructive technology leading to adsorption of dyes. *Datura* leaf extract and iron nanoparticles were successfully prepared. Photocatalytic activity effect of solo chromo black (SCB) was investigated. The present experimentation is designed in such a way that the operating parameters like contact time, pH, concentration, and dosage. The formation of nanoparticles was monitored by visualizing color changes and it was confirmed by UV Spectrophotometer and prepared iron samples were characterized by field emission scanning electron microscopy (FESEM). The produced nanoparticles calculated as 326-327 nm.

**Index Terms**— Iron Nanoparticles, *Datura* leaves, Solo Chromo Black (SCB), FESEM

## 1. Introduction

In the present era, concerns with environmental issues as water contamination has a major issue and it challenged the researchers to improve sustainable remediation processes[1]. Dye wastes from textile industries need adequate treatment before discharge into nature, the release of dye mixed wastewater into nearby water bodies generates considerable destruction to aquatic life, people and plants leads to water contamination[2]. Already there existed different traditional processes namely membrane separation, solvent extraction, ion exchange, adsorption and reverse osmosis to treat contaminated water, but these processes were not enough adequate for immensely contaminated water[5]. These methods were either energy-intensive or restricted due to high operational cost. Hence, rising an invincible interest to detect a cost-effective and eco-friendly biosynthesis material for the removal of hazardous chemicals from contaminated water [22]. A series of technological experiments were carried out to investigate the decolorization of dye. Among the different technologies, nano-technology studies were emerging as a stellar to adsorption of dye. Leaf extracts seem to be the best candidate for biosynthesis of nanoparticles. Nanoparticles produced by leaf extracts were more static, and the rate of reaction is quicker than the other organisms [6]. The iron magnetic nanoparticles have been successfully employed to remove the

solo chromo black (SCB) from aqueous media due to their magnetic properties, ease of functioning, high efficiency, abundantly availability on the surface of the earth, low toxicity and primarily low expensive [7]. The dye decolorization concentration was analysed in UV Spectrophotometer and prepared iron samples were characterized by field emission scanning electron microscopy (FESEM) to calculate the size of samples.

## 2. experimental Procedure:

- Collection of Chemicals and *Datura* leaves
- Preparation of Leaf extract
- Preparation of Iron Nanoparticles
- Characterization of Iron Nanoparticles
- Equilibrium Studies on Dye Decolorization
- Isotherms
- Kinetics

### A. Collection of Chemicals and *Datura* leaves:

- Collected solo chromo black (SCB) dye and prepared dye solution with concentration of 1000 ppm (1gm/1lt).



Fig. 2.1 Solo Chromo Black Dye (SCB)



Fig. 2.2 Dye Solution

- The Datura leaves were collected from Tenneti Park, Visakhapatnam.



Fig. 2.3 Datura Leaves

**B. Preparation of Leaf extract:**

The Datura fresh leaves were collected from Tenneti Park, Vishakapatnam and cleaned with fresh water to clear dust particles placed on the surface of leaves for several times. The Datura extract was prepared from 25gm of slashed fresh leaves in 100ml of distilled water at 70°C for 15 minutes. The extract was filtered off in 250 ml conical flask with filter paper by slowly cooled to ambient temperature. The incurred extract was in light yellow color.

**C. Preparation of Iron Nanoparticles:**

Diluted Iron particles into double distilled water and prepared iron solution. Added individually prepared both broth (Leaf extract) solution of 34ml and 66 ml of iron solution in a 250ml conical flask and preserved in a rotary shaker for one day in order to acquire iron nanoparticles. The nano particles formation is detected when the light yellow color is turned into black color. Mixed the iron nano particle solution with solo chromo black (SCB) dye solution and double distilled water in a test tube under the presence of light for photo catalytic activity for few hours.

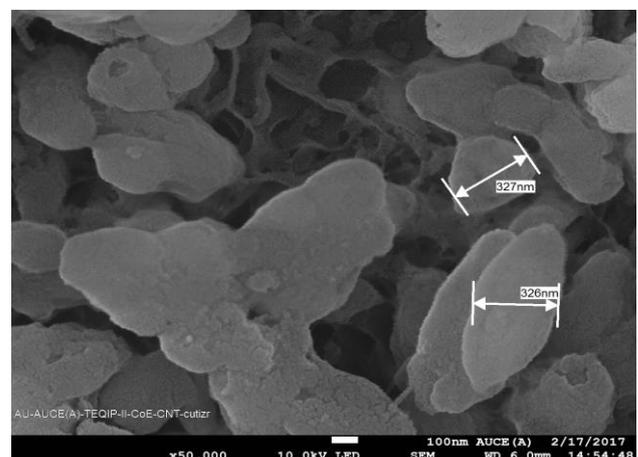


Fig 2.4. Datura Fe Nanoparticle solution

**3. Characterization:**

FESEM Analysis:

The nanoparticles size and structures on the surface of cells can be analyzed by Field Emission Scanning Electron Microscope (FESEM-Fig. 3.1). The small amount of sample was collected from the deposited iron nanoparticles solution test tubes on small glass plates and dried it under light till it dried and the dried samples were examined under FESEM analysis. FESEM analysis provides higher resolution images for low voltage.



**3.1. FESEM Analysis Results**

#### 4. Equilibrium Studies:

The present experimentation is designed in such a way that the operating parameters like contact time, pH, concentration, and dosage.

##### Effect of Contact Time:

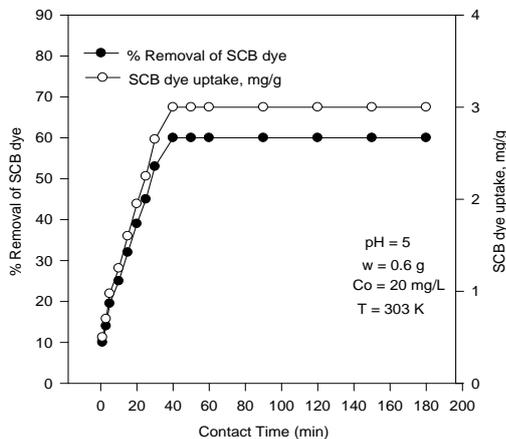


Fig. 4.1. % Removal of Dye vs Contact Time

The change in contact time is a valid parameter for % removal of dye was analyzed and it is determined by plotting the percentage removal of SCB dye against to contact time (Fig. 4.1). The adsorption capacity of solo chromo black (SCB) dye was observed from 1 to 180(1, 3, 5, 10, 10, 15, 20, 25, 30, 40, 50, 60, 90, 120, 150, 180) minutes and conclude that the % removal of SCB dye in the early stage was hiked drastically from 10% to 60% with respect to time up to 40 minutes and sustained a static position at 40 minutes onwards as time increases gradually. The natural tendency of adsorption was rapidly in the early stages due to a large number of active sites adhered on the surface of iron nanoparticles, after attained an optimum value the adsorption rate reduces with increase in contact time because of all available sites were covered and no active site was available for binding. Thus, the optimum value was evaluated at 40 minutes [4, 21, 28, 33].

##### Effect of pH:

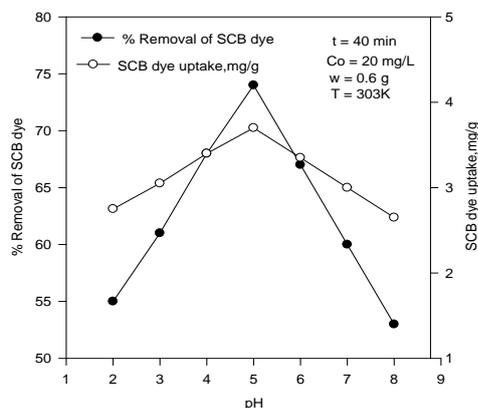


Fig. 4.2 %Removal of Dye vs pH

pH means potential of Hydrogen, itself it represents that the logarithm of the reciprocal of hydrogen ion concentration in gram atoms per liter and it provides measure on a scale from 0 to 14 of the acidity or alkalinity of a solution (where 7 is natural and less than 7 is more acidic and greater than 7 is more basic). The change in the concentration of pH is a significant parameter for a percentage of dye removal. The percentage removal of SCB dye with respect to pH parameter was studied. % removal of SCB dye versus pH of the solution is plotted in order to attain optimum value, pH of the aqueous solution was varied from 2 to 8 (2,3,4,5,6,7,8) and the fig 4.2 depicts that the % removal SCB dye gradually increased from 55% to 74% with pH 2 to 5 and the optimum adsorption capacity of the iron nanoparticle solution was attained at pH 5 (Acidic). Furthermore, as pH of the aqueous solution increases the % removal SCB dye decreases gradually. Generally, low pH solution results in an increase in the percentage of anionic dye removal because of the electrostatic attraction between the anionic dye and the positive surface charge of the adsorbent. At higher solution pH, electrostatic repulsion is found between the negatively charged surface and dye molecules, thus decreasing the adsorption capacity and percentage removal of anionic dye [4, 21, 28, 32, 33].

##### Effect of Initial Concentration:

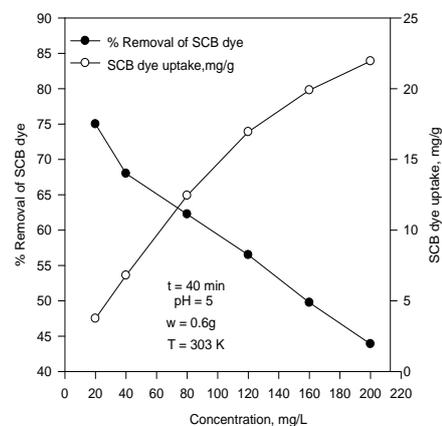
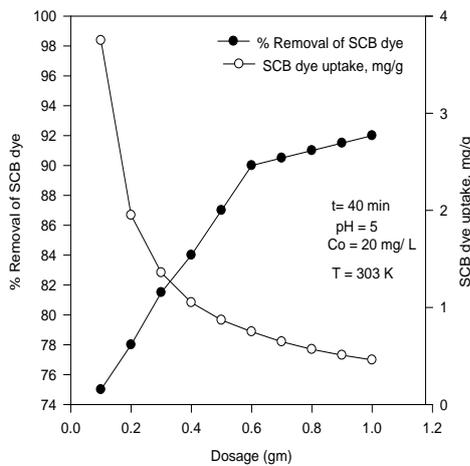


Fig. 4.3 %Removal of SCB dye vs Concentration

Initial concentration also one of the most key parameter for adsorption capacity and percentage removal of SCB dye from the aqueous solution. Generally, when the initial concentration is low the rate of adsorption capacity was high, because, the sunrays can easily penetrate into the nanoparticle solution. As the initial concentration increases lead to decrease the percentage removal of SCB dye which may be due to the saturation of adsorption sites on the adsorbent surface. The graph drawn (Fig. 4.3) between % removal of SCB dye versus initial concentration was observed for various concentrations from 20 to 200 (20, 40, 80, 120, 160, 200) mg/l and optimum dye concentration was detected at 20ppm [4, 21, 33, 34].

**Effect of Dosage:**



**Fig 4.4 % Removal of SCB dye vs Dosage**

The fig 4.4 plotted between % removal of SCB dye and dosage was studied. Generally, the percentage removal of SCB dye increases drastically up to a value, then onwards gradually increases with increase in dosages, because, the number of active sites increases with increase in the amount of dosage to adhere on the surface of iron nanoparticles. The % percentage removal of SCB dye was analyzed for different quantities of dosages ranging from 0.1 to 1 (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1) grams. From the graph, it can conclude, there was a rapid change in the adsorption capacity till 0.6 grams (from 75% to 90%) and onwards slightly decreases. Thus, the optimum value was considered at 0.6 grams [4, 33, 34].

**5. Isotherms:**

**5.1. Langmuir isotherm:**

Langmuir isotherm developed by Irving Langmuir. Generally, this is enormously used in the two-parameter equation. This simple isotherm is based on following assumptions:

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

$$QE/C_m = b_e / (1+bC_e)$$

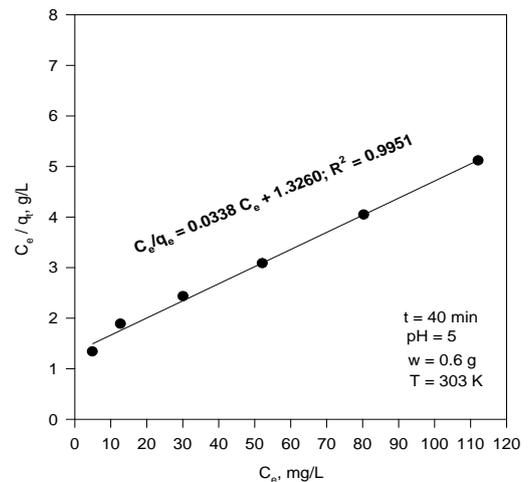
Above Equation can be rearranged as

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

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  $R_L = 1/ (1+bC_e)$  [28, 32].

$0 < R_L < 1$	favorable adsorption
$R_L > 1$	unfavorable adsorption
$R_L = 1$	linear adsorption
$R_L = 0$	irreversible adsorption

Langmuir isotherm is drawn for the present data and shown in Fig.6.1. The equation obtained ' $n$ '  $C_e/q_e = 0.0338 C_e + 1.3260$  with a good linearity (correlation coefficient,  $R^2 \sim 0.9951$ ) indicating strong binding of solo chromo black (SCB) dye to the surface of iron nanoparticles.



**Fig. 5.1 Langmuir isotherm for solo chromo black (SCB) dye**

**5.2. Freundlich isotherm:**

Freundlich presented an empirical dye decolorization isotherm equation that can be applied in case of low and intermediate concentration ranges. It is easier to handle mathematically in more complex calculations [28]. The Freundlich isotherm is given by

$$q_e = K_f C_e^n$$

where  $K_f$  (mg) represents the dye decolorization capacity when dye equilibrium concentration and  $n$  represents the degree of dependence of dye decolorization 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.5.2 for the present data. The resulting equation

$\ln q_e = 0.4337 \ln C_e + 0.5849$ ; has a correlation coefficient of 0.9874.

The ' $n$ ' value in the above equations satisfies the condition of  $0 < n < 1$  indicating favorable dye decolorization [21].

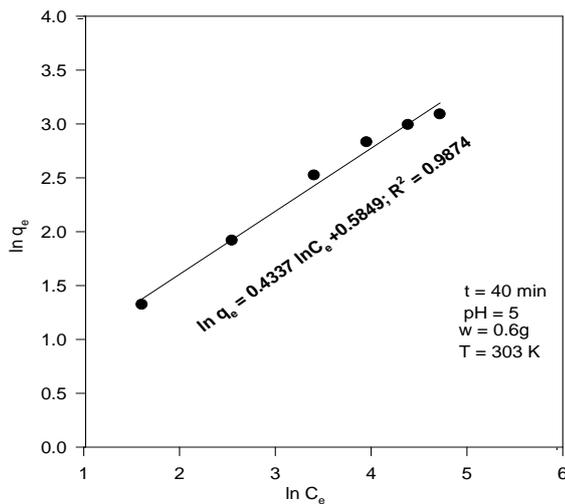


Fig. 5.2 Freundlich isotherm solo chromo black (SCB) dye

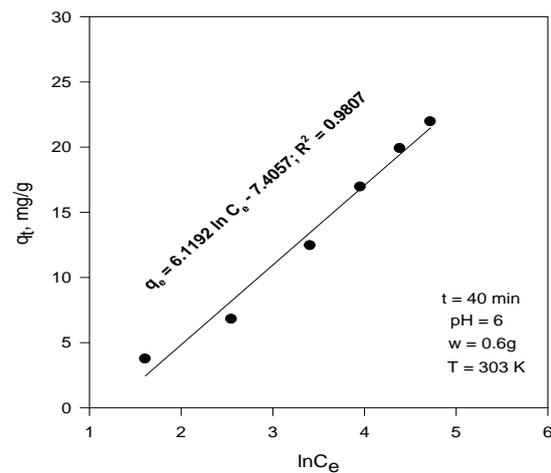


Fig. 5.3 Temkin isotherm solo chromo black (SCB) dye

5.3. Temkin isotherm:

Temkin and Pyzhev isotherm equation describes the behavior of many dye decolorization systems on the heterogeneous surface and it is based on the following equation [21, 28]

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

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  $A_T = \exp [b(0) \times b(1) / RT]$   
 $b(1) = RT / b_T$  is the slope  
 $b(0) = (RT / b_T) \ln(A_T)$  is the intercept and  
 $b = RT / b(1)$

The present data are analyzed according to the linear form of Temkin isotherm and the linear plot is shown in Fig.6.3. The equation obtained for solo chromo black (SCB) dye decolorization is  $q_e = 6.1192 \ln C_e - 7.4057$  with a correlation coefficient 0.9807. 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 decolorization data are well represented by Freundlich isotherm with a higher correlation coefficient of 0.9951, followed by Langmuir and Temkin isotherms with correlation coefficients of 0.9874 and 0.9807 respectively [21-27].

Table - 1 Isotherm values		
Langmuir isotherm	Freundlich isotherm	Temkin isotherm
$q_m = 29.5858 \text{ mg/g}$	$K_f = 1.5429 \text{ mg/g}$	$A_T = 0.2981 \text{ L/mg}$
$R_L = 0.02549$	$n = 0.600508$	$b_T = 411.6783$
$R^2 = 0.9951$	$R^2 = 0.9874$	$R^2 = 0.9807$

6. Kinetics

The order of adsorbate-adsorbent interactions has been described using kinetic model. Traditionally, the first order model of Lagergren finds wide application. In the case of dye decolorization preceded by diffusion through a boundary, the kinetics in most cases follows the first order rate equation of Lagergren: [28-31]

$$(dq_t/dt) = K_{ad} (q_e - q_t)$$

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 decolorization.

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

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

$$\log (q_e - q_t) = 0.4686 - 0.0261t$$

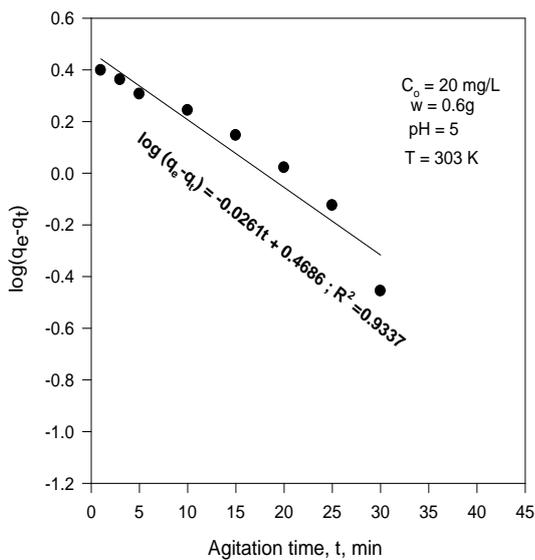


Fig. 6.1 Lagergren first-order kinetics for solo chromo black (SCB)

The plot of  $\log (q_e - q_t)$  vs '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: [32–35]

$K_{ad} (q_e - q_t)$  does not represent the number of available dye decolorization sites and

1.  $\log q_e$  is not equal to the intercept.
2. 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:

$$(dq_t / (q_e - q_t)^2) = K dt$$

$$\text{let } q_e - q_t = x$$

$$dq_t = dx$$

$$1/x = Kx + C$$

$$C = 1/q_e \text{ at } t = 0 \text{ and } x = q_e$$

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:

$$(t/q_t) = (1/Kq_e^2) + (1/q_e) t$$

$$(t/q_t) = 0.3075t + 3.4938$$

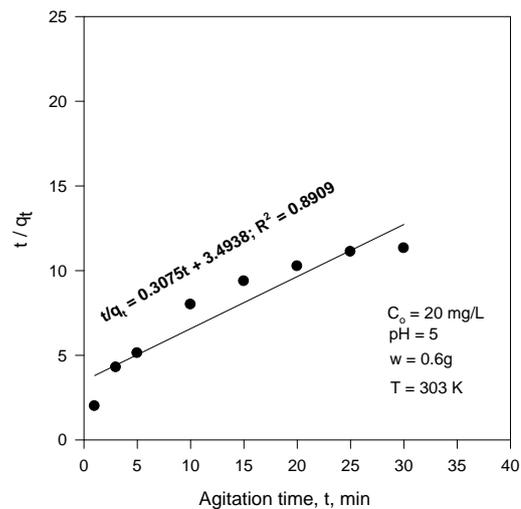


Fig. 6.2 Pseudo second order kinetics for solo chromo black

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  $(t/q_t)$  vs '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. Lagergren plots of  $\log (q_e - q_t)$  versus contact time (t) for dye decolorization of solo chromo black (SCB) the interaction time intervals of 1 to 180 min are drawn in figs. 6.1 & 6.2.

Table - 2 Equations and rate constants			
Order	Equation	Rate Constant	R <sup>2</sup>
Lagergren first order	$\log (q_e - q_t) = 0.4686 - 0.0261t$	$0.06010 \text{ min}^{-1}$	0.9337
Pseudo second order	$t/q_t = 3.4938 + 0.3075t$	$4g / (mg \cdot \text{min})$	0.8909

**Conclusion:**

In this study, the green synthesis of iron nanoparticles for solo chromo black (SCB) dye decolorization was examined. In the early stages the rate of adsorption capacity was gradually enhanced till 40 minutes (60%) and then onwards sustained a static position. % removal SCB dye gradually increased from 55% to 74% with pH 2 to 5 and the optimum adsorption capacity of the iron nanoparticle solution was attained at pH 5 (Acidic). Furthermore, as pH of the aqueous solution increases the % removal SCB dye decreases gradually. The optimum % removal of dye attained at a concentration of 20 ppm (75%), because, the sunrays can easily penetrate into

nanoparticle solution and enhance a photo catalytic activity at a dosage of 0.6 g (90%). Hence with the above conclusions it can be confirmed that Datura leaves extract with iron nano particles are capable of removing SCB dye.

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