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"Removal of Alizarin Red-S Dye from Aqueous Solution by Sorption on Coconut Shell Activated Carbon"

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

Original Research Article

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ABSTRACT

The removal kinetics and mechanism of alizarin red-s dye, adsorptions on coconut shell activated carbon [CSAC] is studied. The use of low cost ecofriendly adsorbent has been investigated as an ideal alternative to the current expensive methods of removing the dye from aqueous solution. The adsorption removal of Alizarin Red-S dye on CSAC was investigated using batch adsorption technique. The different variables such as initial dye concentration, adsorbent dose, contact time, pH and temperature were determined to optimize the method. The percentage removal of dye was observed to be most effective at pH8 and at contact time 120 min and at an adsorbent dose 4 g/L. The study shows that a CSAC can be an alternative to other expensive adsorbent used for the removal of dyes from aqueous solution. The study indicates that the percentage removal of the dye increases with increase in initial dye concentration; adsorption dose and contact time and attain equilibrium at optimum conditions. The adsorbent was also characterized by FTIR, SEM, EDAX studies. This results shows that, the CSAC is suitable for the removal of alizarin red-s dye from aqueous solution.

Keywords: Equilibrium adsorption; low cost adsorbent; alizarin red-s; coconut shell activated carbon; FTIR; SEM; EDAX.

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1. INTRODUCTION

The dyes are widely used in many industries, mainly in textile industries. The effluents they generate are highly colored and their discharge into rivers makes water unfit for domestic, agricultural and industrial purposes. Many dyes and their breakdown product may be toxic for living organism. Dyes even in low concentration are visually detected and affect the aquatic life. The dyes affect the photosynthetic activity in aquatic life by reducing light penetration. The dyes have a synthetic origin and have a complex aromatic structure and hence are difficult to biodegrade when discharged in to wastewater streams [1]. The dyes are carcinogenic and toxic [2] mutagenic and teratogenic and they may cause serious effects on flora and fauna [3-5]. Therefore, the removal of dye is important aspect of wastewater treatment before discharge.

The method of color removal from industrial effluent includes biological treatment [3], coagulation [4], flotation [5], adsorption oxidation [6] and ultra filtration [7]. The adsorption appears to be having considerable potential for the removal of color from the industrial dyeing effluents. A survey reported that an adsorption of dyes using carbons is a important process for removal of color from dyeing industries [8-9]. Commercially available activated carbon is very expensive and its regeneration is difficult. So the economical removal of dyes from wastewater still remains a problem. This has led to further studies for cheaper substitution for this reason, recent research has been focused on low cost and easily available and naturally abundant adsorbent material. Adsorption can be effective for the treatment for dyes at the Intermediary concentrations.

The agricultural by-products are considered to be low value products because of their low utilization. Most of these materials get discharge or set on fire. The disposal of these material causes' resource losses and creates environmental pollution. The exploitation and uses of these material bring obviously economic and social benefits to mankind [10-11]. Presently attention is focused on the utilization of the native agriculture by-product as an adsorbent. The objective of the present work is to investigate the feasibility of CSAC for the removal of alizarin red-s dye from aqueous solution.

2. MATERIALS AND METHODS

2.1 Adsorbent

The coconut shells were collected from the local sources. The collected material was dried in sunlight for 2 days and then cutted into small pieces using a cutter . The most of the volatile organic compound in the raw material are removed. The material is converted into char by pyrolysis in a horizontal muffle furnace at a temp 300ºC to 500ºC for 2 hours. The obtained material was activated at a constant temperature between 800ºC to 1000ºC furnace in inert gas medium. The physical characterization of the resulting activated carbon was then analyzed by SEM analysis. The obtained activated carbon was used without further purification as an adsorbent.

2.2 Adsorbate

The basic dye alizarin red-s (CI=58005) used in the study has the chemical formula $(C_{14}H_7NaO_7S)$, molecular weight of 240 g/mol and wavelength of maximum absorbance of 480 nm and was supplied by S.D. fine chemical Mumbai. The dye has the following structure (Fig. 1).

Fig. 1. Alizarin red-s dye

An accurately weighed quantity of dye was dissolved in double distilled water to prepare a stock solution (1000mg/L) for the experimental study.

2.3 Experimental Method and Measurement

The adsorption experiments were carried in a batch process at room temperature by using an aqueous solution of alizarin red-s dye. In each experiment an accurately weighed amount of CSAC was added in 50ml of the dye solution in 100ml Stoppard conical flask and then mixture was agitated in the mechanical shaker for a definite time at a room temperature. The adsorbent was separated from the solution by centrifugation. The absorbance of the supernatant solution was estimated to determine the residual dye concentration. The residual dye concentration was determined before and after treatment at 480 nm wave length with systronics-118 spectrophotometer using a quartz's cell of path length 10 cm. The experiments were carried out at initial pH values ranging from 2 to 8.The values of pH were measured by using a pH meter equiptronics model EQ 607.The initial pH was controlled by adding 0.1NHCl and 0.1N NaOH solution. The kinetic of adsorption was determined by analyzing adsorptive uptake of dye from aqueous solution at different time intervals [12]. The FTIR, SEM, EDAX elemental analysis of adsorbent was also carried out. Energy dispersive X ray spectroscopy is a chemical microanalysis techniques used in conjunctions with SEM.EDAX analysis was used to characterize the elemental composition of CSAC.

3. RESULTS AND DISCUSSION

3.1 Adsorbent Characterization

For structural and morphological characteristic FTIR, SEM and EDAX of adsorbent CSAC were carried out.

3.1.1 SEM and EDAX analyses

The scanning electron microscope (SEM) is widely used to study the morphological features and surface characteristics of adsorbent material. It is useful to determine particle shape, porosity and appropriate size distribution of the adsorbent material. The CSAC was analyzed by SEM before and after adsorption of alizarin Red-s (Fig. 2). The figure shows surface texture and porosity of the material analysed. The examination of SEM micrograph of CSAC shows rough area of the surface and microspores were identifiable. The comparisons before and after adsorption dyes shows that the alizarin Red-S dye is adsorbed on the surface of CSAC.

Before Adsorption **Before Adsorption**

Fig. 2. The SEM micrograph of CSAC before and after adsorption of alizarin red-s dye. 3.1.2. EDAX

The results obtained from the elemental analysis shows that CSAC contained 28.51% of C; 28.21% O; 2.48%Mg; 1.09%Al; 6.87%S; 11.54% Ca; 8.87% Br and 2.01%Fe before adsorption and 88.40% C ; 9.18% O; 0.99 % K; 0.55% Ca after adsorption.

3.1.2 FTIR

The FTIR spectra were obtained for CSAC before and after adsorption process shown in (Fig. 3a). The broad intense adsorption peak around 3834-3726 are indicative of the existing of OH stretching frequency (H-Bonded), the peak observed at 3045 cm⁻¹ is for aromatic C-H stretching frequency, the peak around 2801cm⁻¹ is for aliphatic C-H stretching frequency, and 1704 cm^{-1} is for C=O stretching frequency and 1521 cm^{-1} is for C=C stretching frequency. The intense band at 1218 cm^{-1} is for C-O stretching frequency and 956 cm^{-1} is for O-H out of plane bending frequency. Some of these peaks in sample shifted to lower frequency after adsorption as shown in Fig. 3b, it suggest the participation of the functional group in adsorption of alizarin red-s by CSAC.

3.2 Optimization of Condition for Maximum Adsorption Capacity

3.2.1 Effect of pH

The pH of a solution is an important factor in the adsorption of dye on to adsorbent. The pH of the system affects the nature of surface charges of the adsorbent, effect of ionization and the extent of rate of adsorption. The initial pH values of the solution has more influence than the final pH value [13-14]. To study the effect of pH on alizarin red-s adsorption on to CSAC, the experiments were carried out at 60 mg /L of initial dye concentration with 4 g/L adsorbent dose for 120 min equilibrium time in the range of pH change from 2 to 14. Intialy when pH increases from 2 to 7 the percentage removal increases from 29% to 80% and it slightly increases up to 83.34% for pH 8 and then percentage removal decreases to 80% (Fig. 4)

and hence the pH8 is selected for the removal of alizarin red-s by CSAC. This may be due to greater association of dye cation with adsorbent.

3.2.2 Effect of contact time

The effect of contact time and dye concentration on percentage removal of dye by CSAC is presented in Figs. 5 and 6.

The amount of dye adsorbed qt (mg/g) increased with increasing in agitation time and reached to the equilibrium after 120 min, for the dye concentration 60, 80,100,120 mg/L (Fig. 5). Thus, the time required to achieve a definite fraction of equilibrium adsorption was found to be 120 min and it is independent of dye concentration, similar observation are also indicated by other workers [15]. The adsorption density qt (mg/g) also increases with dye concentration.

The percentage of dye removal at equilibrium decreases from 94.33% to 72% as dye concentration increased from 60 to 120 mg/L for 4 g/L of adsorbent does at pH8. This shows that removal of Alizarin red-s depends on concentration of dye. The removal curves are single, smooth and continuous to saturation suggesting possible monolayer coverage of dye on surface of the adsorbent [16].

Fig. 4. Effect of pH on percentage removal of alizarin red -s dye by CSAC, adsorbent dose 4 g/L, contact time 120 min; dye concentration 60 mg/L

3.2.3 Effect of adsorbent dose

Adsorbent dose is an important parameter because this factor determines the capacity of an adsorbent for a given initial concentration of adsorbate. The effect of adsorbent dose was studied on the removal of alizarin red-s by keeping all other experimental condition constant. The removal of alizarin red-s by CSAC at different doses (0.5 g/L) to 5 g/L) for the dye concentration 60, 80, 100, 120mg/L is shown in (Fig. 7). When adsorbent dose was increased from 0.5 to 5 g/L, the amount of dye absorbed was observed to be increased from 55% to 88.9% mg/L for 60mg/L dye concentration, contact time 120 min and pH8 (Fig. 7). When the concentration of dye was increased to 120mg/L the amount of dye adsorbed was from 23% to 92%.

The result shows that as adsorbent concentration increased, the percentage removal of dye is also increased, but the amount adsorbed per unit mass of adsorbent decreased considerably (Fig. 8). The decrease in unit adsorption with increasing dose of adsorbent is basically due to adsorption sites remaining unsaturated during the adsorption reaction. The percentage removal of alizarin Red-s increased with increasing dose of adsorbent and that is due to the increase in availability of surface active sites resulting from increase of dose and agglomeration of adsorbent [17]. The increase in the extent of removal of alizarin Red-s is found to be significant at the dose 4g/L and hence it is fixed as the optimum dose for

adsorbent (Fig. 7). The result shows that as adsorbent concentration is increased, the percent removal of dye also increased [18].

Fig. 5. Effect of contact time and concentration of alizarin red-s dye on percentage removal adsorbent dose 4g/L and pH8

Fig. 6. Amount of dye adsorbed qt (mg/g) with time for different initial dye concentration, adsorbent dose 4 g/L, pH8

Fig. 7. Effect of adsorbent dose on % removal of alizarin red-s by coconut shell activated carbon for different initial dye concentration, contact time 120min, pH8

3.2.4 Effect of initial dye concentration

The adsorbent and dye solution of different initial dye concentration 60mg to 120mg/L was contacted for 120 min at pH 8 and at a constant adsorbent dose 4g/L. The result shows that the percentage removal of dye decreases from 87.1% to 84.4%-when initial dye concentration increases from 60 to 120 mg/L .The amount of dye adsorbed decreases from 87.4%-to 87.1% (Fig. 9). This shows that the alizarin red-s removal by adsorption on CSAC is concentration dependent [19].

3.3 Adsorption kinetics

The adsorption kinetic study is quite significant as it describes the solute uptake rate which in turn controls the residence time of adsorbate uptake at the solid- solution interface. The dye concentration and adsorbent dose are the important parameter because they determine capacity of adsorbent for a given initial concentration of dye solution. The study includes adsorption rate study and intrapartical diffusion study .The kinetics of adsorption of alizarin Red-s on CSAC were analyzed using pseudo first order (Lagergren), Pseudo 2^{nq} order, Elovich and Intrapartical diffusion kinetic models. The conformity between experimental data and the model predicted values were expressed by correlation coefficient (r^2) . A relatively high $r²$ value indicates that the model successfully describes the kinetics of alizarin Red-s adsorption. The dye removal from the solution is through the mechanism of adsorption [20].

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Fig. 8. Amount of dye adsorbed (mg/g) with adsorbent dose for different initial dye concentration contact time 120 min, pH 8

Fig. 9. Effect of initial dye concentration on percentage removal of alizarin red-s by CSAC, adsorbent dose 4 g/L and pH8

3.4 Pseudo First Order Equation

The pseudo first order kinetic model has been widely used to predict dye adsorption kinetics.

The linear form of Pseudo first -order model was given by (Lagergren) equation [21] as

$$
Log (qe-qt) = log qe-(K1/2.303) t
$$
 (1)

Where qe and q_t are the amount of dye adsorbed (mg/g) at equilibrium and at time 't, respectively, K_1 is a rate constant of pseudo first- order adsorption (min⁻¹). The rate constant K**¹** and qe for first- order equation were determined from the slope and intercept of the plot of log (qe-qt) vs. time respectively (Fig. 10).

Fig. 10. Lagergren 1st order plot for adsorption of alizarin red-s by coconut shell activated Carbon for different initial dye concentration, adsorbent dose 4gm/L and pH8

The linear relationship of the plot for 60, 80, 100, and 120 mg/L dye concentration indicates the validity of equation [21]. Even though the correlation coefficient r^2 of the first order equation is considerably high (0.9230 and 0.9750) .The values of qe calculated from first order kinetic plot were small as compared to the experimental values and does not shows good agreement between the experimental and calculated qe values Table 1. It shows that the rate constant K_1 decreases with increase in initial dye concentration and adsorbent dose. It shows the inapplicability of Lagergren equation to describe the kinetic of alizarin red -s.

3.5 The Pseudo Second Order Equation

The kinetic data were further analyzed using Pseudo second order kinetic model which is expressed as [22].

$$
t' qt = 1/h + t/qe and h = K_2qe^2
$$
 (2)

Here K_2 is rate constant of second order adsorption (mg/min), h-the initial adsorption rate (mg/ (g min)).The plot of t/qt vs. t. (Fig. 11)

Fig. 11. Plot of the pseudo 2nd order model at different initial dye Concentration, adsorbent dose 4gm/lit and at Ph=8

Of the above equation gives linear relationship from which qe and $K₂$ can be determined from the slope and intercept of the plot respectively. The linear plot of t/qt vs. t shows good agreement of experimental data with second order kinetic model for different initial dye concentration 60, 80, 100, 120 mg/L and for different adsorbent dose. The calculated qe, K_2 and corresponding linear regression correlations coefficients r^2 value are sumrised in a Table 1. The values are greater than 0.99 which indicate the applicability of kinetic equation and Pseudo second order nature of adsorption process of alizarin red-s on CSAC. The qe value increases with increase in initial dye concentration and adsorbent dose. (18) The calculated qe values agree well with the experimental values. Similar phenomena are observed in the desorption of dyes [21-23]

3.6 The Elovich Equation

The simplified Elovich equation is

$$
qt=1/\beta \ln (a\beta) + 1/\beta \ln t \tag{3}
$$

Where α is the initial adsorption rate (mg/mg/gm/min), ß is the desorption constant (g/mg). The adsorption of alizarin Red-s fits the Elovich model; a plot of qt vs. lnt is a linear with a slope 1/ß and intercept 1/ß ln (αß) (Fig. 12). As concentration increases from 60 to120 mg/L, the value of ß decreases from 0.242 to 0.126 for 4g/L of adsorbent dose (Table 2).

Table 1. Comparison of Adsorption rate constant, Calculated and experimental qe values for different initial dye concentration and adsorbent dose for different kinetic model

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Fig. 12. Test of Elovich model for adsorption of Alizarin red-s on CSAC at different initial concentration adsorbent dose 4g/L, contact time 120 min and pH8

3.7 Intrapartical Diffusion Model

The adsorbate species are most probably transported from the bulk of the solution in to the solid phase through and intrapartical diffusion processes, which is often the rate limiting step in many adsorption processes. The possibility of intrapartical diffusion was explored by using intrapartical diffusion model. The kinetic data were well represented by Weber and Morris plot of qt vs. square root of time (Fig. 13). It was observed that there were two linear portions. The first linear portion ended with smooth curve followed by the second linear portion. The double nature of the curve reflects the two stages external mass transfer followed by intrapartical diffusion [23] and is described by the equation.

$$
qt= kpt^{1/2} \tag{4}
$$

Where K_P is intrapartical diffusion rate constant (mg/gm/min). The slope of the second linear portion characterizes the rate parameter K_P corresponding to the intrapartical diffusion where as the intercept of this second linear is proportional to the boundary layer thickness. The intrapartical diffusion kp at different initial concentration and adsorbent dose are represented in Table 2.The values of kp were found to be in the range of 3.580 – 6.852 for dye concentration 60, 80, 100, 120 mg/L of dye and adsorbent dose 4g /L of CSAC.

3.8 Adsorption Isotherms

The distribution of the dye between the liquid phase and the adsorbent is a measure of the position of equilibrium in adsorption process. The adsorption isotherms indicate how the adsorbed molecules distribute between the liquid phase and solid phase when the adsorption process researched to an equilibrium stage. The analysis of the isotherm data by

fitting them to different isotherm models is an important stage to find the suitable model that can be used for design purpose. Several equilibrium models have been developed to describe adsorption isotherm relationships. Two isotherm equations were tested in this work; theye were Langmuir and Freundlich isotherm. The applicability of the isotherm models was evaluated by the correlation coefficients, R^2 value of each plot. Higher the R^2 value, the better fit to the model.

Fig. 13. Test of Intrapartical diffusion for adsorption of alizarin red-s on CSAC different initial dye concentration; adsorbent dose 4g/L, pH8

3.9 Freundlich Isotherms

This empirical isotherm can be used for non ideal sorption that involves heterogeneous surface energy system. The linear plot of log qe vs. log Ce confirms the applicability of the model. The logarithmic form of Freundlich isotherms equation is

 $Log qe = log k f + 1/n log Ce$ (3)

Where qe is the amount of dye adsorbed per unit mass of the adsorbent (mg/g), Ce is the equilibrium liquid-phase concentration of the dye (mg/L), the k_f represent quantity of dye adsorbed in mg/g for unit concentration of dye and 1/n is measure of adsorption density. Values of n>1 represent the favorable adsorption condition. The plot of log qe vs. log Ce will obtain a straight line with slope of 1/n and intercept of log Kf. For 100 mg/L dye concentration dye solution (Fig. 14). The k_f and n values were calculated from the intercept and slope of the plot are presented in Table 2. The value of r^2 for Freundlich adsorption isotherm is 0.787 which more than Langmuir adsorption isotherm and system is favorable. For Freundlich isotherms.

Fig. 14. Freundlich plot for adsorption of alizarin red-s by CSAC, pH 8 and adsorption dose 4g/L

3.10 Langmuir Isotherm

Langmuir isotherms equation has been widely applied to describe experimental adsorption data based on the assumption that adsorption energy is constant and independent of surface coverage; that adsorption occurs on localized sites with no interaction between adsorbate molecule, and the maximum adsorption occurs when the surface is covered by a monolayer adsorbate. The Langmuir isotherm equation is represented by the following equation [24-25].

$$
Ce/qe = 1/ab + ce/b
$$
 (4)

Where Ce is the concentration of the dye solution (mg/L) at equilibrium. The constant 'a' signifies the adsorption capacity (mg/g) and 'b' is related to energy and adsorption (L/mg). The linear plot of Ce/qe vs. Ce shows that the adsorption fallows the Langmuir isotherms (Fig. 15). The values of 'a and' b 'were calculated from the slope and intercept of the linear plot and represented in Table 3. The applicability of the Langmuir isotherms suggest the

monolayer coverage of the dye on the surface of CSAC. The essentials characteristics of the Langmuir isotherms can be expressed by the dimensional constant called equilibrium parameter RL [26-30] and defined by the equation.

$$
RL=1/(1+bci)
$$
 (5)

Where b is the Langmuir constant and C_i is the initial dye concentration (mg/L). According to the values of the RL, The isotherms shape may be interpreted as if RL is>1, unfavorable. RL=1 Linear, 1>RL>0 favorable and RL=0

Irreversible. The r^2 value for langmuir adsorption is 0.756.

Fig. 15. Langmuir plot for adsorption of alizarin red-s by CSAC, for dye con.100 ppm, at pH8 and adsorbent dose 4g/L, contact time 120 min

Table 3. Freundlich and Langmuir coefficient for adsorption of alizarin red-s of CSAC for 100g/L dye concentration and adsorbent dose 4g/L at pH8 and contact time 120 min

4. CONCLUSION

The adsorption study indicates that, the agro based material coconut shell activated carbon can be used as an effective adsorbent for the removal of alizarin red-s dye from aqueous solution. The adsorption experiments were conducted in a batch mode for the concentration range 60 to120mg/L at pH8 and for an adsorbent dose 0.5g/L to 5g/L and up to contact time of 120 min. The equilibrium was achieved in an about 120 min. and maximum adsorption capacity were observed at $pH8$ and adsorbent dose $4q/L$. The amount of dye uptake (mg/L) was found to be increases with increase in contact time, initial dye concentration and it decreases with an increase in adsorbent dose. The adsorption rate was found to conform to pseudo second order kinetics with good co relation. The equilibrium data fit very well in Freundlich isotherms equation. Thus the CSAC is an inexpensive and easily available material and can be an alternative for the most costly adsorbent used for removal of dyes in the wastewater treatment processes.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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