Environmental Health Engineering and Management Journal 2016, 3(3), 123–129
http://ehemj.com
doi 10.15171/EHEM.2016.10

Environmental Health Engineering and Management Journal

Original Article

Environmental Health Engineering and Management

Open Access Publish Free

Biosorption of acid dyes from aqueous solution using *Curcuma angustifolia* scales

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Abstract

Background: The presence of even a very small quantity of dye in water bodies is undesirable and affects the water bodies. Dye removal from industrial waste water is significant; hence in this study, a material that is an economical waste product was employed to test its acid dye removing capacity from aqueous solution.

Methods: In this study, batch mode experiments were performed in the sorption process of Acid Red 97 (AR 97), Acid Red 114 (AR 114) and Acid Red 151 (AR 151) onto *Curcuma angustifolia* scales (CS). Also, the effect of process parameters like pH and adsorbent dosage was studied. The experimental data of AR 97, AR 114 and AR 151 sorption was fitted to Langmuir, Freundlich and Temkin isotherm models. Kinetic results in AR 97, AR 114 and AR 151 sorption were fitted at various concentrations to pseudo-first order, pseudo-second order, Elovich and Intra-particle diffusion model.

Results: The monolayer sorption capacity of the acid dyes was found to be AR 97 (350.87 mg/g), AR 114 (202.42 mg/g) and AR 151 (168.91 mg/g). The Pseudo-second order model proved to be the best fit for the acid dyes. Boyd plot, confirms film diffusion in all acid dye sorption processes.

Conclusion: The results showed higher dye removal for acid dyes at pH 2. The isotherm data, demonstrated good sorption capacity with AR 97>AR 114>AR 151. Employing the CS material in this study proves to be a potential alternative to costlier adsorbents, utilized for the treatment of dye containing industrial waste water.

Keywords: Biosorption, Acid dyes, Dye removal, Isotherm, Kinetics

Citation: Suresh S. Biosorption of acid dyes from aqueous solution using Curcuma angustifolia scales. Environmental Health Engineering and Management Journal 2016; 3(3): 123–129. doi: 10.15171/EHEM.2016.10.

Article History:
Received: 17 May 2016
Accepted: 25 July 2016
ePublished: 27 August 2016

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Introduction

The presence of dyes in textile industry effluents are usually non biodegradable, and leads to a serious threat to human beings and the environment. The highly coloured dyes in effluent, affect sunlight penetration, and retard the photosynthetic activity, leading to reduction in the growth of aquatic biota in water bodies (1). The disposal of dye into water resources, damage the aquatic life and causes carcinogenic and mutagenic effects (2). Hence, colour removal from dye containing effluents by different treatment techniques becomes very important.

Since dyes are difficult to decolorize, there is need to obtain a technology that works well and cost effective for the treatment of effluents. Conventional treatment processes found to be effective in reducing the concentration of dye includes, photo degradation (3), coagulation (4), electrochemical oxidation (5), ozonation and coagulation, chemical oxidation (6,7). The most commonly used method that is known to be efficient for the treatment of a wide range of dye in wastewater, is adsorption (8).

Adsorption using activated carbon is one of the commonly utilized methods for the removal of dye in wastewaters. The high cost of activated carbon and the loss involved in regeneration, prompted the search for effective and inexpensive biosorbents (9) for wastewater treatment.

In developing countries, the high cost of activated carbon helped the search for low cost biosorbents for the treatment of colored effluents. Researchers have focused on the use of biosorbents in the treatment of colored water with variation in the degrees of success (10). Some of the biosorbents used are, sugarcane bagasse (11), sawmill waste (12), jackfruit leaf powder (13), waste pea shells (14), jute stick powder (15), soybeans meal hull (16), mansonia wood sawdust (17), etc. Still, it is necessary to search for effective biosorbents for the removal of dyes from wastewater.

The biosorbent employed in this study is a cheap material available free of charge in India; hence, it was employed in the present method of dye removal. In this work, the paper discusses the use of an effective and inexpensive natural raw *Curcuma angustifolia* Scales as biosorbent, for the removal of acid dyes from aqueous solution.

Methods

Materials

In this study, *C. angustifolia* (CS) tubers were collected from Nagercoil, Tamil Nadu. The scales were removed from the tubers and used as biosorbent. The acid dyes employed in this study were Acid Red 97 (AR 97), Acid Red 114 (AR 114) obtained from Sigma-Aldrich (Bangalore, India), and Acid Red 151 (AR 151) obtained from SHIDIMO, Interaux Pvt. Ltd. India. The characteristics of the anionic dyes are as follows: AR 97 (CI: 22890; molecular weight: 698.32 g mol^{-1}; molecular formula: $C_{32}H_{20}N_4Na_2O_8S_2$); AR 114 (CI: 23635; molecular weight: 830.81 g mol^{-1}; molecular formula: $C_{37}H_{28}N_4Na_2O_{10}S_3$). AR 151 (CI: 26900; molecular weight: 454.43 g mol^{-1}; molecular formula: $C_{22}H_{15}N_4NaO_4S$). The dyes are utilized as coloring agent for fabrics in textile industries. The pH was adjusted using HCl solution and NaOH pellets, obtained from Merck, India. The molecular structure of AR 97, AR 114 and AR 151 is illustrated in Figure 1.

Preparation of biosorbent

The raw material CS scales were collected and washed with distilled water repeatedly, till the wash water is free from any color, to ensure the removal of dirt, dust and surface impurities. The material was kept in a hot air oven at 60°C for 24 hours. The dried biomass was grinded, using a domestic mixer. The grinded material was sieved to uniform particle size, in the range of 80-100 mesh (BSS). The biosorbent thus obtained, is labeled as CS (18).

Batch sorption experiments

Sorption studies, using fixed biosorbent mass at varying dye concentrations, were studied for the removal of acid dyes at pH 2. To investigate the efficiency of dye removal and to study the isotherm constants, experiments were carried out with 50 mL of different dye concentration with 50 mg of the biosorbent in a series of conical flasks. This mixture was kept on a mechanical shaker for a period of 4 hours at constant speed. The dye concentration at equilibrium was measured spectrophotometrically at the wavelength of maximum absorbance AR 97 (498 nm), AR 114 (522 nm) and AR 151 (549 nm). The percentage dye removal by the biosorbent was calculated using the formula:

$$\% \text{ Dye Removal} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

The amount of dye sorbed at equilibrium q_e (mg/g) was calculated by:

$$q_e = C_i - C_f \times \frac{V}{M} \quad (2)$$

Where C_i and C_f are the initial and final liquid phase concentrations (mg/L). The volume of acid dye solution (V) in mg/L and the biosorbent mass (g) is given by M.

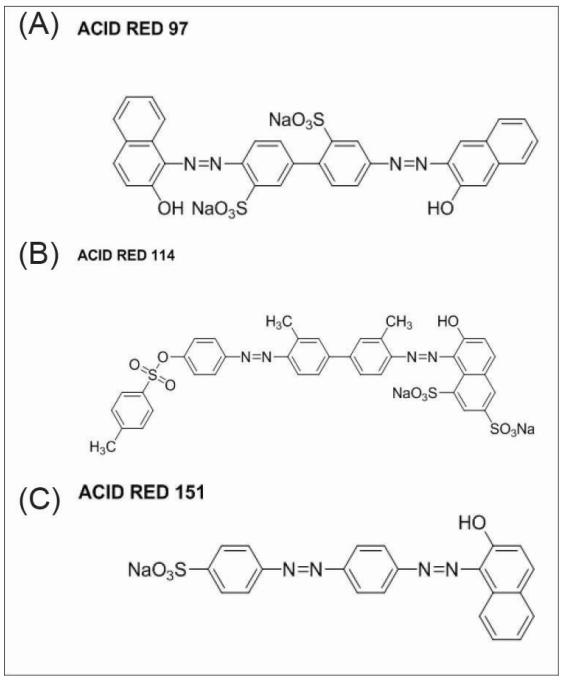

Figure 1. Molecular structure of acid dyes (A) AR 97 (B) Acid Red 114 (C) Acid Red 151.

Results

Studies on pH and biosorbent dosage

In the present study of pH, the percentage dye removal for 50 mL of 100 mg/L of dyes was mixed with 0.1 g of biosorbent, and was observed in the pH range of 2 to 12 for all three acid dyes (Figure 2). To investigate the effect of sorbent dosage onto AR 97, AR 114 and AR 151, a dye concentration of 100 mg/L in a series of standard 50 mL dye volume was taken at pH 2, with all other experimental conditions constant. The graphical results are shown in Figure 3.

Isotherm studies

The isotherm data for the biosorption of AR 97, AR 114 and AR 151 onto the biosorbent was analyzed by means of Langmuir, Freundlich and Temkin (19) model. The obtained isotherm parameters, using the linear form of the isotherm equations are given in Table 1. Moreover, the R_L value observed ($0 < R_L < 1$), linear ($R_L = 1$), unfavorable ($R_L > 1$) or irreversible $R_L = 0$, indicates that in CS, a favorable

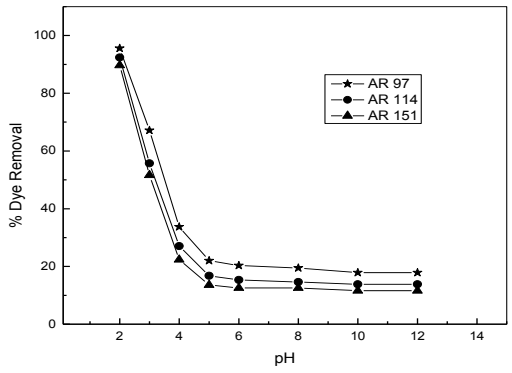

Figure 2. Effect of pH in sorption of acid dyes onto *Curcuma angustifolia* Scales (C_0 = 100 mg/L; m=100 mg; V=0.05 L).

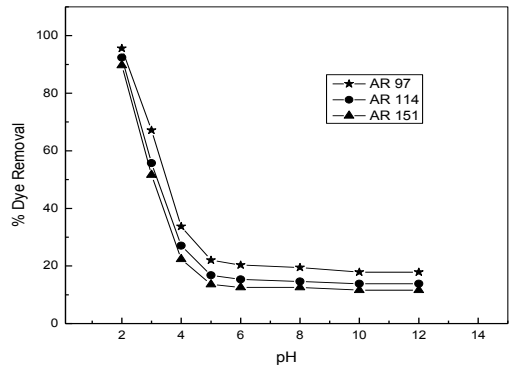

Figure 3. Effect of biosorbent dosage in the sorption of acid dyes onto *Curcuma angustifolia* scales (C0 = 100 mg/L; V = 0.05 L).

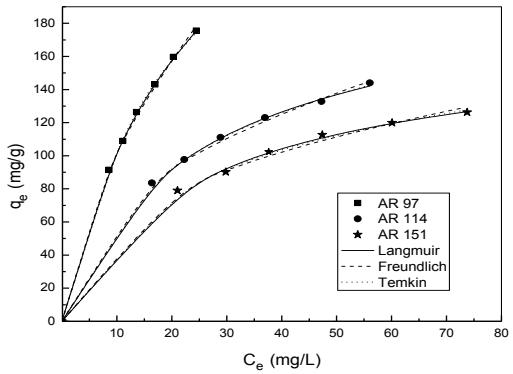

Figure 4. The fit of experimental data for the sorption of acid dyes onto *Curcuma angustifolia* scales (t = 4 hours; m = 50 mg/0.05L)

Table 1. Langmuir, Freundlich and Temkin constants for the biosorption of acid dyes onto *Curcuma angustifolia*

Langmuir constants	AR 97	AR 114	AR 151
Q_m (mg/g)	350.87	202.42	168.91
K_L (L/mg)	0.0409	0.0421	0.0407
R^2	0.9936	0.9985	0.9911
R_L	0.1965-0.1089	0.1919-0.1061	0.1972-0.1094
Freundlich constants			
K_F (mg/g)	24.27	25.26	24.71
n	1.6023	2.3041	2.5953
R^2	0.9955	0.9923	9809
Temkin constants			
KT (L/mg)	0.353	0.3423	0.3573
B1	80.84	48.34	39.03
R2	0.9976	0.9975	0.9903

sorption process occurs for the acid dyes. The Freundlich constants were determined from the linear form of the model, and the value of K_F, n and the correlation coefficients are given in Table 1.

The Temkin isotherm constants for biosorption of AR 97, AR 114 and AR 151 onto CS calculated from the plot of qe vs. lnCe are given in Table 1. The comparative fit of experimental data for sorption of AR 97, AR 114 and AR 151 onto CS is shown in Figure 4.

Studies on sorption kinetics and mechanism

In order to analyze the biosorption kinetics, pseudo-first order, pseudo-second order, Elovich and intraparticle diffusion equations (19) were utilized. A plot of log (q_e-q_t) vs. time for CS to enable the calculation of the rate constant K_1 and the results are tabulated in Table 2. The kinetic data was tested for designing and modeling the sorption process, employing the pseudo-second order model and the best fit was investigated (Figure 5). Table 2 presents the various kinetic constants and $q_{e(calc)}$ in the sorption of AR 97, AR 114 and AR 151 onto the biosorbent. The Elovich constants α and β calculated for the acid dye sorption is shown in Table 3. The intraparticle diffusion rate constant (K_{id}) and the intercept (C) was obtained for AR 97, AR 114 and AR 151 from the slope and intercept of the plot of q_t vs. $t^{1/2}$, the results are presented in Table 3. The Boyd model is essential in predicting the rate limiting step, in

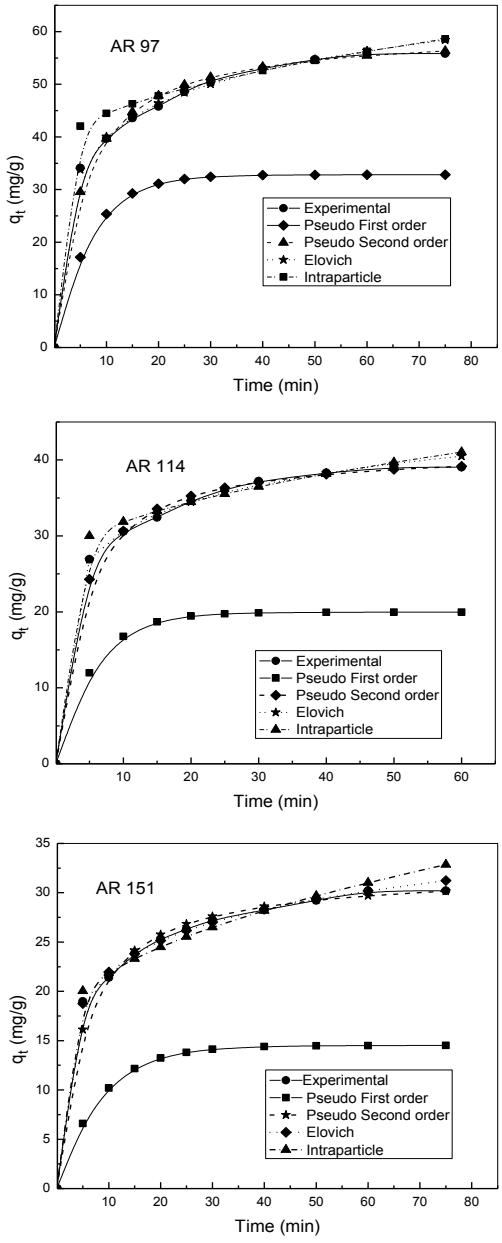

Figure 5. Comparison of various kinetic models with experimental values for the sorption of acid dyes onto *Curcuma angustifolia* scales (C0 =140 ppm; m=1 g/0.5L)

Table 2. Kinetic values for the biosorption of acid dyes on to CS.

Adsorbent	Concentration mg/L	$q_{e(exp)}$ (mg/g)	Pseudo first order			Pseudo second order			
			$q_{e(Calc)}$ (mg/g)	K_1 (min^{-1})	R^2	$q_{e(Calc)}$ (mg g^{-1})	K_2 (g mg^{-1} min^{-1})	h (mg $g^{-1}min^{-1}$)	R^2
AR 97	100	48.77	32.68	6.43×10^{-2}	0.9860	52.91	3.32×10^{-3}	9.26	0.9987
	140	55.83	32.82	6.31×10^{-2}	0.9870	60.24	3.19×10^{-3}	11.58	0.9989
	180	58.53	34.29	6.26×10^{-2}	0.9818	62.89	3.16×10^{-3}	12.50	0.9989
	220	64.31	35.75	6.11×10^{-2}	0.9841	68.95	2.95×10^{-3}	14.02	0.9988
AR 114	100	31.26	19.45	8.79×10^{-2}	0.9857	33.55	7.47×10^{-3}	8.41	0.9992
	140	39.24	19.95	7.97×10^{-2}	0.9897	41.49	6.85×10^{-3}	11.01	0.9993
	180	40.33	22.51	7.91×10^{-2}	0.9661	43.10	5.93×10^{-3}	11.81	0.9988
	220	44.37	23.29	7.28×10^{-2}	0.9574	47.61	5.14×10^{-3}	11.65	0.9982
AR 151	100	20.10	13.57	5.87×10^{-2}	0.9804	22.17	6.26×10^{-3}	3.05	0.9976
	140	30.19	14.51	5.85×10^{-2}	0.9853	32.15	6.20×10^{-3}	6.47	0.9989
	180	34.11	18.41	5.83×10^{-2}	0.9827	36.13	5.17×10^{-3}	6.93	0.9986
	220	38.66	20.12	5.27×10^{-2}	0.9894	41.32	4.80×10^{-3}	8.30	0.9988

Table 3. Intraparticle and Elovich values for the biosorption of acid dyes on to *Curcuma angustifolia*

Adsorbent	Concentration mg/L	Intraparticle diffusion			Elovich		
		K_{ip} (mg/g $min^{1/2}$)	C (mg/g)	R^2	α (mg/g min)	β (g/mg)	R^2
AR 97	100	2.33	31.36	0.9885	40.94	0.1132	0.9907
	140	2.52	36.31	0.9635	74.94	0.1101	0.9955
	180	2.58	39.34	0.9741	91.62	0.1099	0.9905
	220	2.70	43.73	0.9815	127.79	0.1090	0.9902
AR 114	100	1.15	23.25	0.9115	59.35	0.2004	0.9833
	140	1.38	28.33	0.9768	126.25	0.1823	0.9915
	180	1.73	29.42	0.9855	147.40	0.1723	0.9906
	220	2.12	29.61	0.9765	162.33	0.1624	0.9825
AR 151	100	1.19	10.99	0.9873	11.83	0.2598	0.9890
	140	1.37	19.58	0.9922	48.57	0.2171	0.9950
	180	1.46	22.87	0.9766	54.06	0.1835	0.9818
	220	1.59	26.36	0.9837	66.39	0.1675	0.9847

the sorption of AR 97, AR 114 and AR 151 onto CS. Bt vs. time plot (Figure 6) can be used to test the linearity of the experimental values. The Di values were obtained for all acid dyes, using the calculated B values and the calculated Di values for AR 97, AR 114 and AR 151 as shown in Table 5.

Discussion

Effect of pH

From Figure 2, it is obvious that the solution pH affects the amount of dye, adsorbed with a high percentage dye removal for AR 97, AR 114 and AR 151 at pH 2. The dye removal increases drastically below pH 4 to pH 2 and higher dye removal of acid dyes was observed at pH 2 for all acid dyes, as a result, further adsorption work was carried out in the optimum pH 2. At lower pH, the surface of the biosorbent will react as positively charged, and hence, effectively attracts the negatively charged anionic dye, leading to an increased sorption of acid dyes. At higher pH, the surface of the biosorbent becomes negatively charged, which reduces the interaction of the anionic dye. Literature also reveals a similar phenomenon for the removal of acid dyes using soybeans meal hull (16).

Effect of biosorbent dosage

It was observed that the percentage dye removal increases for the dyes AR 97, AR 114 and AR 151 with an increase in the amount of biosorbent, owing to the availability of more sorption sites with increased biosorbent dosage. As observed from Figure 3, the dye removal increases from 20 to 120 mg for all acid dyes with more dye uptake in between 40 to 80 mg for all acid dyes, and reaches a limit for dye sorption of 120 mg, reaching saturation of the active sites in the biosorbent (16).

Biosorption isotherms

The study on sorption isotherm of CS onto AR 97, AR 114 and AR 151 is necessary from theoretical and practical point of view. The high R^2 value implies the applicability of the Langmuir isotherm in all three acid dye sorption processes. The experimental data for all dyes provide the best correlation to Langmuir isotherm, indicating the homogenous nature of the biosorbent surface and hence, showing the monolayer coverage of the all dyes onto the

Table 4. Biosorbents reported for the removal of acid dye from aqueous solution.

Biosorbent	Dyes	q_m (mg/g)	Reference
Bagasse pith	Acid Red 114	101	(26)
Orange peel	Acid violet 17	19.88	(27)
Coir pith	Acid brilliant Blue	16.67	(28)
Lady's Finger	Acid Blue 113	169.9	(29)
Soy meal hull	Acid Blue 92	114.94	(16)
	Acid Red 14	109.84	(16)
Hazelnut shell	Acid Blue 25	60.2	(30)
Wood sawdust	Acid Blue 25	5.92	(31)
Curcuma angustifolia Scales	Acid Red 97	350.87	This work
	Acid Red 114	202.42	This work
	Acid Red 151	168.91	This work

Table. 5. Diffusion coefficient (D_i) for biosorption of acid dyes onto *Curcuma angustifolia*

Adsorbent	Concentration (mg/L)	D_i (cm^2/s)
AR 97	100	8.52×10^{-11}
	140	7.52×10^{-11}
	180	7.34×10^{-11}
	220	7.41×10^{-11}
AR 114	100	1.03×10^{-10}
	140	9.35×10^{-11}
	180	9.34×10^{-11}
	220	8.54×10^{-11}
AR 151	100	6.67×10^{-11}
	140	6.18×10^{-11}
	180	6.83×10^{-11}
	220	6.84×10^{-11}

biosorbent surface (20). The R_L calculated from the results using Eq. 3 were found to be in the range of 0.1965 to 0.1089 for AR 97, 0.1919 to 0.1061 for AR 114 and 0.1972 to 0.1094 for AR 151.

$$R_L = 1/(1+K_L C_0) \quad (3)$$

Since the biosorbent has good adsorption for AR 97, AR 114 and AR 151; the material can be a good alternative to commercial activated carbon in the removal of dye from aqueous solution.

From Table 1, the maximum monolayer sorption capacity (Q_m) for the acid dyes was found to be 350.87 for AR 97, 202.42 for AR 114 and 168.91 mg/g for AR 151. The higher sorption of the dyes AR 97, AR 114 and AR 151 on the biosorbent could be due to the surface charge present on the dye surface, interacting effectively with the biosorbent surface. The applicability of the Langmuir model to all three dyes is proved by the high correlation coefficients R^2 > 0.99, suggesting that the Langmuir isotherm provides a good model of the sorption system (21).

In Freundlich isotherm, the biosorption capacity is related to the constant K_F and the magnitude of the component 'n' indicates the favorability of sorption process. According to Treybal (22), it has been demonstrated that n > 1, represents favorable sorption. The *n* values of the acid dyes indicate favorable sorption onto the biosorbent. The *n* value was found to be 1.60 for AR 97, 2.30 for AR 114

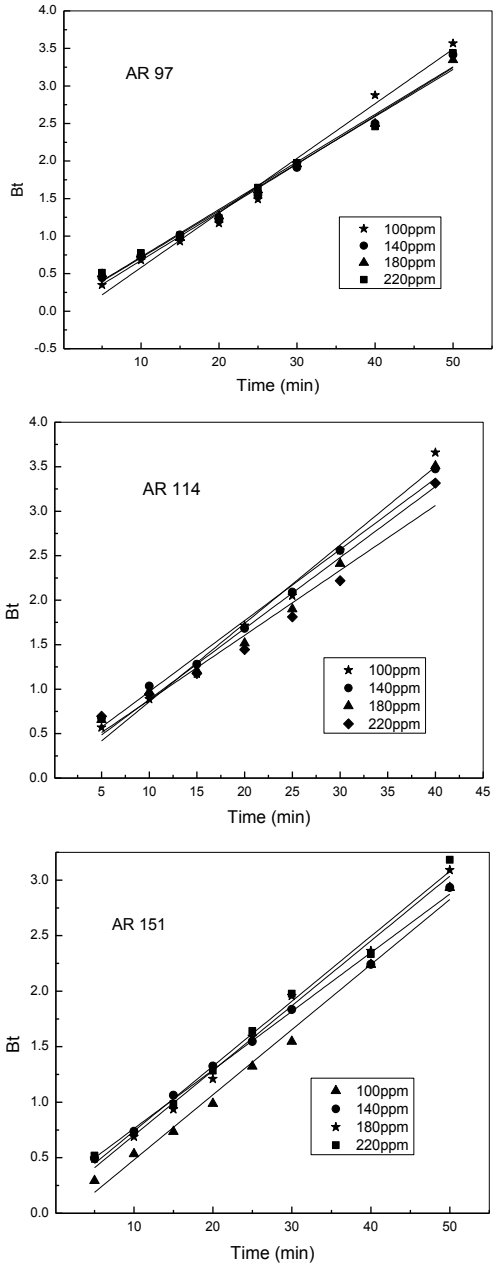

Figure 6. Boyd plot for the sorption of acid dyes onto *Curcuma angustifolia* scales.

and 2.59 for AR 151, which indicates favorable sorption. Temkin considered the sorbate-biosorbent interactions, and the heat of sorption for the molecules in the layer, reducing linearly with coverage (18). The sorption data were analyzed by means of the linear form of Temkin equation, and it was observed that this isotherm fitted well for all three acid dyes with R^2>0.99. From Figure 4, it was observed that a better fit for the experimental q_e value is indicated by the Langmuir model for all three acid dyes, and proves the monolayer distribution of the dyes active site on the biosorbent surface, followed by Temkin and Freuendlich isotherm. The Freuendlich isotherm represents the poor fit for the experimental data, compared to the other isotherms. It is reasonably concluded that the sorption of acid dye is appreciably better in the biosorbent

CS with sorption capacity in the order of AR 97 > AR 114 > AR 151.

Biosorption kinetics

The kinetic study for the removal of AR 97, AR 114 and AR 151 onto CS is a time dependent sorption process, hence it is essential to know the sorption rate of acid dyes by CS in order to evaluate the efficiency of CS. The dye sorption process seems to proceed by a rapid binding of the sorbate onto the CS surface and reaches saturation in 75 minutes, for AR 97 and AR 151 and 60 minutes in AR 114.

The low correlation coefficient (R^2) values in CS for all acid dyes indicate that, pseudo first order is not applicable to the system. The kinetic data was tested for designing and modeling the sorption process, employing the pseudo first order model for dyes AR 97, AR 114 and AR 151, while the best fit was studied for the kinetic results with an initial dye concentration of 140 mg/L (Figure 5). The experimental q_e value and the calculated q_e values do not coincide for pseudo-first order model in all three acid dyes; and this confirms that the sorption of acid dyes onto the biosorbent does not provide the best fit for the Pseudo-first order model.

On plotting t/q_t vs. t, we can find k_2 and hence the initial sorption rate h, can be determined utilizing the equation:

$h = k_2 q_e^2$ (4)

Presently, the initial sorption rate h, rate constant k_2 and $q_{e(calc)}$ for AR 97, AR 114 and AR 151 can be obtained from the plot of t/q_t vs. t. In CS, the correlation coefficient was >0.99 for pseudo-second order model in AR 97, AR 114 and AR 151 and therefore proves to be the well suited model for the sorption of acid dyes onto the biosorbent. The second order rate constant k_2 was observed to decrease with increase in acid dye concentration onto the biosorbent. A similar trend was observed for decrease in k_2 with increase in solute concentration (23). It was observed that the calculated q_e values agree well with the experimental q_e values for the three acid dyes. This confirms that the sorption of acid dyes onto the absorbent, fitted well in the Pseudo second order model. This demonstrates that the biosorption of the acid dyes onto CS, follows the pseudo second order model and a chemisorption process follows in the present work.

The Elovich model assumes that the solid surface is energetically heterogeneous (24). The initial sorption rate α for the acid dyes increases from 40.94 to 127.79 for AR 97, 59.35 to 162.33 for AR 114 and 11.83 to 66.39 for AR 151. This suggests that the sorption rate is good for all three dyes on the biosorbent. From Table 3, the extent of surface coverage (β) decreases for all acid dyes with an increase in acid dye concentration, indicating that the availability of the biosorbent surface in CS decreases with an increase in AR 97, AR 114 and AR 151 dye concentration.

The intercept C value for the acid dyes indicates the thickness of the boundary layer. The larger the intercept C, the more the contribution of surface sorption at the rate controlling step (25). The increase in C with an increase in dye concentration for AR 97, AR 114 and AR 151 indicates the increase in the thickness of the boundary layer and internal mass transfer. The comparative fit for the sorption of AR 97, AR 114 and AR 151 onto the biosorbent, is shown in Figure 5. It was also observed from the results that the calculated q_e value and the experimental q_e values coincide with the intraparticle model for all three acid dyes.

Table 4 presents some of the low cost biosorbents employed for the sorption of acid dyes, alongside CS. In this study, it was observed that the sorption of the acid dyes by the biosorbent CS is good when compared with the low cost biosorbents reported in literature.

Boyd model

To predict if film diffusion or particle diffusion controls the rate of sorption onto the biosorbent, the Boyd kinetic model (18) was employed in the linear plots (Bt vs. time) of the three acid dyes. Hence, if the line passes through the origin, then particle diffusion is the slowest step, and if the linear plots were not passing through the origin in acid dye sorption onto CS, then film diffusion controls the sorption process. In this study, the Bt versus time plot (Figure 6) for AR 97, AR 114 and AR 151 at various concentrations were found to be linear, and does not pass through the origin. This confirms film diffusion in the sorption of AR 97, AR 114 and AR 151 onto CS.

Conclusion

The study revealed that CS scales can be utilized as an effective material in the sorption of acid dyes from aqueous solution. The sorption of acid dyes (AR 97, AR 114 and AR 151) onto CS was found to increase with the decrease in pH, and increases with increase in biosorbent dosage. The isotherm data prove monolayer sorption for all acid dyes, and the adsorption capacity was found to be 350.87, 202.42 and 168.91 mg/g for AR 97, AR114 and AR 151 respectively. The kinetic study results prove that the pseudo-second order model fits well for all the acid dye sorption onto the biosorbent. Boyd plot results confirmed film diffusion process as the slowest step involved in all acid dye sorption process. Hence, it can be concluded that CS is an easily available low cost biosorbent having a good sorption capacity and can be utilized as an alternative material to costly adsorbents for the removal of dye in textile wastewater treatment process.

Ethical issues

The author declares that all the data collected during the present study are published in this work and there is no ethical issue in this work.

Competing interests

The Author declares that there is no competing interest in this biosorption work.

Author's contribution

The author declares that he performed all the experiments

and drafted the manuscript.

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