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Biosorption of Congo Red from Aqueous Solution using Wheat Bran and Rice Bran: Batch Studies

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Biosorption of Congo Red from Aqueous Solution using Wheat Bran and Rice Bran: Batch Studies

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Abstract: The batch adsorption experiments were carried out using low-cost agricultural by-products, wheat bran and rice bran, for the removal of Congo red from aqueous solution at pH 8.0 and temperature of 25°C. Effects of important parameters such as contact time, adsorbent concentration, adsorbent modification and ion strength were investigated. The raw biomass and loaded Congo red biomass were characterized by FT-IR. The pseudo-first order equation and pseudo-second order equation were tested and the results showed that adsorption of Congo red followed the pseudo-second order very well. The Langmuir and Freundlich equations were applied to the data related to the adsorption isotherms and the observed maximum adsorption capacities (q_m) were 22.73 and 14.63 mg g^{-1} for wheat bran and rice bran, respectively. The effects of adsorbent concentration and ionic strength on the Congo red adsorption were marked. The adsorption performance has been significantly improved using rice bran modified by $Cu(NH_3)_4^{2+}$.

Keywords: Biosorption, Congo red, isotherm, kinetics, low-cost adsorbent

INTRODUCTION

The release of azo dyes into the environment is a concern due to coloration of natural waters and the toxicity, mutagenicity, and carcinogenicity of the dyes and their biotransformation products (1).

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Accordingly, dye removal has been targeted as one of the important issues in textile wastewater treatment (2). Various techniques have been employed for the removal of dyes from wastewaters. These methods principally include adsorption (2–5) photocatalytic degradation (6), electrokinetic coagulation (7), advanced chemical oxidation (8–9), ozonation (10), liquid-liquid extraction (11), supported liquid membrane (11), and biological process (12–13).

The adsorption process is one of the efficient methods to remove dyes from effluent due to its low initial cost, simplicity of design, ease of operation and insensitivity to toxic substances. Activated carbon is the most widely used adsorbent with great success due to its large surface area, micro-porous structure, high adsorption capacity, etc. However, its use is limited due to its high cost. Therefore, the use of low-cost, easily obtained, high efficiency, and eco-friendly adsorbents has been investigated as an ideal alternative to the current expensive methods of removing dyes from wastewater (14). Some adsorbents including activated carbon (15), red mud (16), banana pith (17), coir pith (18), dead fungus (*Aspergillus niger*) (19), and biomass of *Trametes versicolor* (20) have used to remove Congo red from aqueous solution and promising results obtained.

In recent years, agricultural by-products being economic and eco-friendly due to their unique chemical composition, available in abundance, renewable, low in cost and more efficient have been used as potential adsorbents to sequester heavy metal ions from aqueous solution (21–22). However, the related reports on utilizing agricultural by-products to remove azo dye Congo red from aqueous solution are relatively scarce. The aim of this study was to investigate the potential of using agricultural by-products, wheat bran and rice bran, as low-cost adsorbents to sequester azo dye Congo red from aqueous solution. The effects of important factors, such as contact time, adsorbent concentration, adsorbent modification, and ionic strength on Congo red adsorption were studied. The Langmuir and Freundlich adsorption isotherms were employed to quantify the adsorption equilibrium. Two kinetic equations (i.e., pseudo-first-order equation and pseudo-second-order equation) were applied to investigate the adsorption mechanisms.

MATERIALS AND METHODS

Adsorbent

The untreated adsorbents, wheat bran and rice bran, were dried in an oven at 105°C for a period of 24 h, and then ground and sieved to get size fraction of <150 μm.

Five grams of the raw biomass were mixed with 100 ml of 0.02 mol dm^{-3} Cu $(NH_3)_4SO_4$ (2% ammonium and 0.02 mol dm^{-3} $CuSO_4$) and the reaction mixture was shaken for 24 h, after which the supernatant was decanted. The same procedure was repeated four times. Finally, the treated samples were rinsed with deionized water five times, freeze-dried (−40°C), and ground to get size fraction of <150 µm.

Adsorbate

The adsorbate Congo red also called Direct red, Direct red 28, or Cotton red, utilized was obtained from M/s Merck. It has molecular formula $C_{32}H_{22}N_6O_6S_2Na_2$ (mol. Wt. 696.7 g mol^{-1}) with Color Index Number 22120 and CAS Number 573-58-0. The structure is as illustrated in Fig. 1. The color of Congo red in aqueous solution is strongly pH-dependent due to its structure transformation. Its color in aqueous solution is blue at pH <3, but red at pH >5. In real Congo red-contaminated wastewaters, the solution pH value ranges from 8.0 to 10.0. Thus, in the present investigation, the initial pH was adjusted to be 8.0 with a pH meter (PHS-3C).

Analytical Technique

The dye was made up in stock solution of concentration 1000 mg dm^{-3} and was subsequently diluted to the required concentrations. Calibration curve for this dye was prepared by recording the adsorbance values for a range of known concentrations of dye solution at the wavelength to

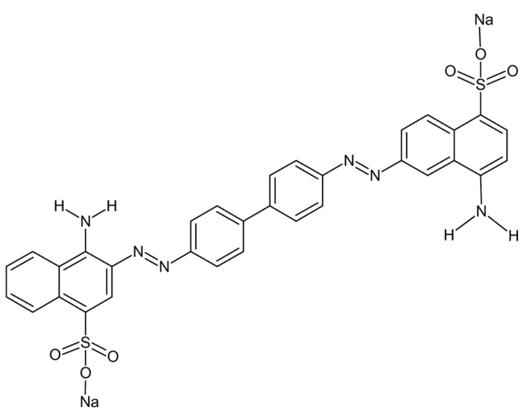

Figure 1. The structure of Congo red molecule.

maximum adsorbance of the dye. The value of λ_{max} (488 nm) was used in all subsequent investigations. All measurements were made on an UV/Vis spectrophotometer (UNICO-7200).

Pretreatment of Glassware

Dyes are strongly adsorbed by glass. To minimize this effect, all glassware to be used in contact with dye solutions was steeped before use for at least 24 h in a solution (1000 mg dm^{-3}) of a cationic surface-active agent, cetyl trimethylammonium bromide, which is preferentially adsorbed. The glass surfaces were then thoroughly rinsed with water before use (23).

Batch Experiments

Batch kinetic experiments were carried out at constant pH 8.0 with initial concentration of 80 mg dm^{-3} and adsorbent concentration of 5 g dm^{-3} at a temperature of 25°C. After shaking, the solution samples were withdrawn at pre-determined time intervals.

Batch equilibrium experiments were conducted using 125-ml conical flask at a total sample volume of 50 ml for each adsorption run. The samples were agitated in a reciprocating shaker to reach equilibrium. At the end of the reaction time, a known volume of the solution was removed and centrifuged for analysis.

The impacts of adsorbent concentrations and ion strength (sodium chloride (NaCl) as background electrolyte) on uptake were also investigated. Temperature control was provided by the water bath shaker units.

Fourier Transform Infrared Analysis (FTIR)

FT-IR analysis of the adsorbents used in this study was performed using a Fourier transform infrared spectrometer (FT/IR-5300). The adsorbent powders were blended with IR-grade KBr in an agate mortar and pressed into tablet. The spectra of adsorbents were recorded.

Calculation

The amount of dye adsorbed at time *t*, q_t, was calculated from the mass balance equation

$$q_t = \frac{(C_0 - C_t)V}{m} \tag{1}$$

when time t (min) is equal to the equilibrium contact time, $C_t = C_e$, $q_t = q_e$, then the amount of dye ions adsorbed at equilibrium, q_e, was calculated using equation (1). Where q_t and q_e, are the amount of solute adsorbed onto the unit mass of the adsorbent at time t (min) and at equilibrium (mg g^{-1}), respectively; C_0, C_t and C_e the concentration of the solute in the initial solution and in the aqueous phase at time t, and at equilibrium (mg dm^{-3}), respectively; V the solution volume of the aqueous phase (dm^3); and m the amount of adsorbent used (g).

RESULTS AND DISCUSSION

FTIR Analysis

The infrared spectra for each raw biomass are shown in Fig. 2. Wave number of 3420 cm^{-1} indicates the possible presence of –OH, –NH groups on the adsorbent surface. The bands that are observed at 2930

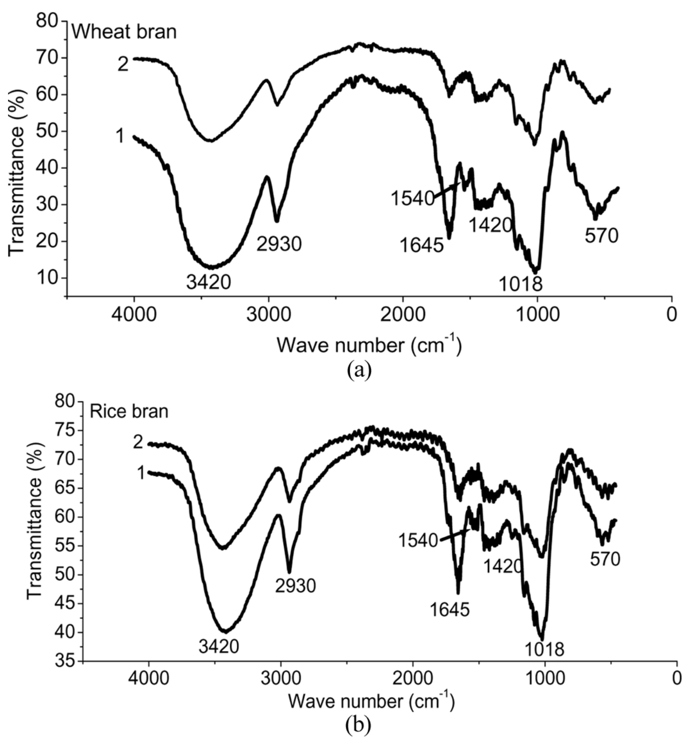

Figure 2. The plots of FTIR (1: raw biomass; 2: Congo red-loaded biomass).

and 1420 cm^{-1} suggest the presence of C−H group. The peak at 1645 cm^{-1} band is caused by the C=O stretching band of the carboxyl group. The absorption peak at 1540 cm^{-1} is ascribed to the C−O bending vibration of the carboxylate ions. The absorption peaks at 1160 and 1050 cm^{-1} are assigned to the –CN stretching vibration of the protein fractions.

To elucidate the Congo red adsorption, the infrared spectra of Congo red-loaded biomass are compared with that of the raw biomass and also illustrated in Fig. 2. The adsorption of Congo red induced some modifications of the infrared spectra. The disappearance of the peak located at 1540 cm^{-1} was observed and this suggests the C−O on the surface of the adsorbents might be involved in Congo red adsorption. Although slight changes of the other absorption frequencies were also observed, it was difficult to interpret how these absorption peaks were related with Congo red adsorption.

Effect of Contact Time

The variations of Congo red adsorption as a function of time for each biomass are shown in Fig. 3. Inspection of these data suggests that the adsorption of Congo red onto wheat bran was very fast and attained equilibrium at nearly 90 min. In comparison, the uptake of Congo red onto rice bran was relatively slower and reached equilibrium at 400 min. In the following experiments, shaking time of 500 min was chosen to ensure to reach adsorption equilibrium.

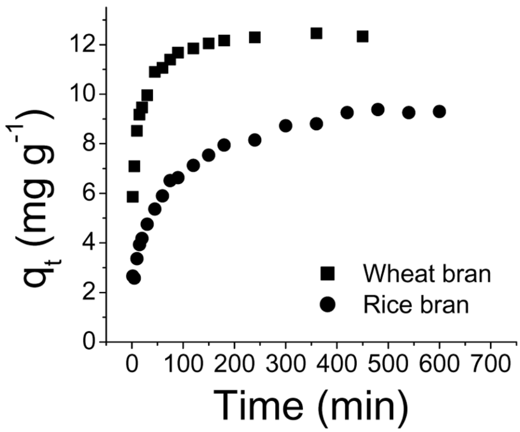

Figure 3. The Congo red amount adsorbed as a function of reaction time (initial concentration 80 mg dm^{-3}; initial solution pH 8.0; adsorbent concentration 5 g dm^{-3}; temperature 25°C).

Kinetic Modeling

In order to examine the mechanism of adsorption processes, two commonly used kinetic models i.e., pseudo first order equation (24) and pseudo second order equation (25) were applied to fit the data in the present study.

Pseudo-first-order Equation

$$\ln(q_{e,1} - q_t) = \ln q_{e,1} - k_1 t \tag{2}$$

where k_1 (min^{-1}) is the rate constant of pseudo first order equation. $q_{e,1}$ (mg g^{-1}) and q_t (mg g^{-1}) are the amount sorbed at equilibrium and at time t (min), respectively. A straight line of ln ($q_{e,1}-q_t$) versus t suggests the applicability of this kinetic model.

The plots of ln ($q_{e,1}-q_t$) versus t for both biomass are presented in Fig. 4 and the related kinetic parameters obtained from linear fitting are shown in Table 1. The values of $q_{e,1}$ deviate significantly from the experimental values $q_{e,exp}$, indicating that the Congo red adsorption kinetic was not a pseudo first order reaction.

Pseudo-second-order equation

$$\frac{t}{q_t} = \frac{1}{k_2 q_{e,2}^2} + \frac{t}{q_{e,2}} \tag{3}$$

where k_2 (min g mg^{-1}) is the rate constant of pseudo second order equation. $q_{e,2}$ (mg g^{-1}) is the amount sorbed at equilibrium. If the pseudo second order model is applicable, the plot of t/q_t versus t should be linear, from which $q_{e,2}$ and k_2 can be obtained.

The fitting results are also illustrated in Fig. 4 and the kinetic parameters are given in Table 1 as well. Based on the obtained correlation coefficients (R), the experimental data conformed better to the pseudo-second-order equation, evidencing chemical sorption as rate-limiting step of adsorption mechanism (25). The experimental q_e values are in agreement with the ones obtained from the fitting of pseudo-second-order equation, which also suggests the Congo red adsorption onto each biomass was closer to chemisorption.

Adsorption Isotherm

The adsorption isotherm represents the relation between the concentrations in the liquid phase at equilibrium and the concentrations of

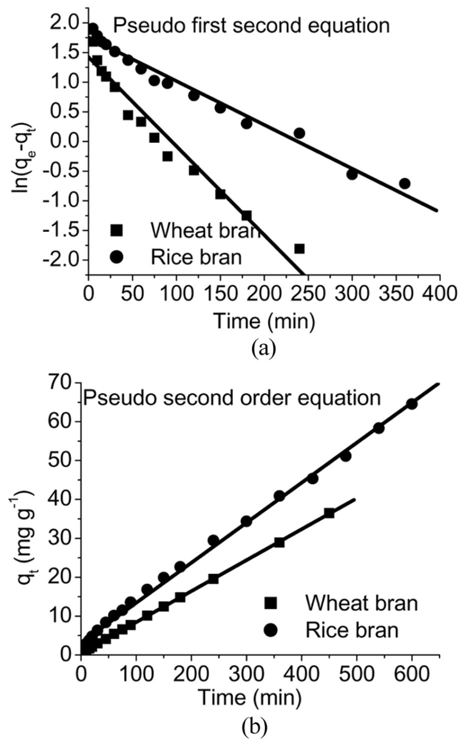

Figure 4. Plots of pseudo first order equation and pseudo second order equation for Congo red adsorption onto both biomass (initial concentration 80 mg dm^{-3}; initial solution pH 8.0; adsorbent concentration 5 g dm^{-3}; temperature 25°C).

adsorbate in the solid adsorbent phase for a constant temperature, and is very important in the design of adsorption system. In the present study, the most commonly used two isotherms, i.e., Freundlich and Langmuir isotherms, were applied to correlate the equilibrium data.

Table 1. Kinetic parameters for the removal of Congo red by different biomass

	Pseudo first order equation			Pseudo second order equation			Experimental value
Adsorbents	$q_{e,1}$ (mg g^{-1})	k_1 (min^{-1})	R	$q_{e,2}$ (mg g^{-1})	k_2 (min g mg^{-1})	R	q_{exp} (mg g^{-1})
Wheat bran	4.14	0.01499	0.9753	12.54	0.01355	0.9999	12.45
Rice bran	5.78	0.00737	0.9890	9.72	0.00338	0.9983	9.38

The Freundlich Isotherm (26)

$$q_e = k_f C_e^{1/n} \tag{4}$$

where k_f [(mg g^{-1}) (mg $dm^{-3})^{-1/n}$] and n (dimensionless) are the Freundlich constants, indicating adsorption capacity and adsorption intensity, respectively.

The Langmuir Isotherm (27)

$$q_e = \frac{q_m b C_e}{1 + bC_e} \tag{5}$$

where q_m (mg g^{-1}) and b (dm^3 mg^{-1}) are Langmuir constants which are indicators of the maximum adsorption capacity and the affinity of the binding sites, respectively.

Congo red adsorption isotherms for each biomass and the results are shown in Fig. 5 and Table 2. Values of k_f and n were obtained using nonlinear regressions and found to be 6.21 (mg g^{-1}) (mg $dm^{-3})^{-1/n}$ and 4.13, respectively for wheat bran and 4.94 (mg g^{-1}) (mg $dm^{-3})^{-1/n}$ and 5.16, respectively for rice bran. The values of the Freundlich parameter, n, are larger than unity, indicating favorable adsorption of Congo red onto both adsorbents.

The values of the Langmuir constants, q_m and b for each adsorbent were found to 22.73 mg g^{-1} and 0.06978 dm^3 mg^{-1}, respectively for wheat bran and 14.63 mg g^{-1} and 0.06934 dm^3 mg^{-1} respectively for rice bran. The difference in Congo red adsorption onto both adsorbents is principally ascribed to the different chemical compositions of each adsorbent (Table 3).

The Freundlich isotherm is an indication of surface heterogeneity of the adsorbent while the Langmuir isotherm corresponds to surface homogeneity of the adsorbent. The high correlation coefficients for Langmuir isotherm (Table 2) for each adsorbent implied that the surface of both adsorbents investigated is made up of homogeneous adsorption patches.

Effect of Chemical Treatment of Adsorbent

The effect of chemical treatment of both adsorbents on Congo red adsorption is illustrated in Fig. 6. The small influence on the Congo red adsorption is attributed to the low contents of cellulose and lignin in wheat bran (Table 3). However, for rice bran, the chemical treatment

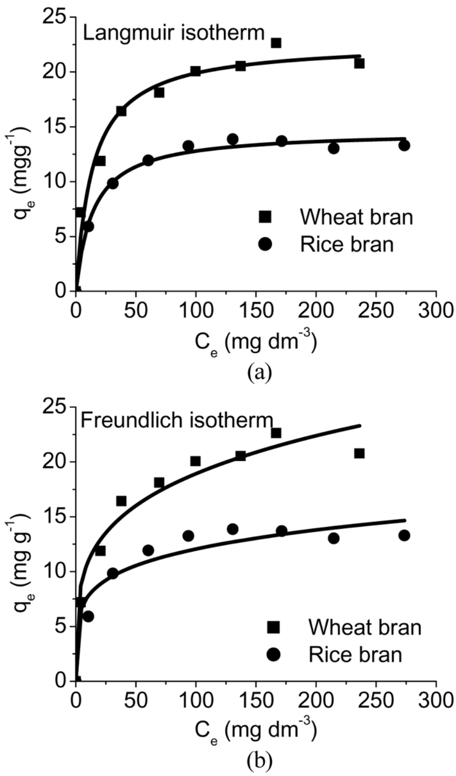

Figure 5. Adsorption isotherms on each adsorbent with the results fitted to the two equations (contact time 500 min; initial solution pH 8.0; adsorbent concentration 5 g dm^{-3}; temperature 25°C).

markedly affected Congo red adsorption of rice bran. This can be explained as follows: when the rice bran was mixed with $Cu(NH_3)_4^{2+}$, the cellulose was dissolved into solution and more O-containing

Table 2. Isothermal parameters for the removal of Congo red by both biomass

	Freundlich			Langmuir		
Adsorbents	k_f[(mg g^{-1}) (mg dm^{-3})$^{-1/n}$]	n	R	q_m (mg g^{-1})	b (dm^3 mg^{-1})	R
Wheat bran	6.21	4.13	0.9831	22.73	0.06978	0.9875
Rice bran	4.94	5.16	0.9694	14.63	0.06934	0.9949

Table 3. Typical compositions of wheat bran (28) and rice bran (22)

Composition	Wheat bran (%)	Rice bran (%)
Cellulose	8.69	32.24
Hemicellulose	32.9	21.34
Lignin	4.81	21.44
Water	13.6	8.11
Mineral ash	5.6	15.15
Protein	15.9	–
Lipid	4.0	–
Others	14.5	1.82

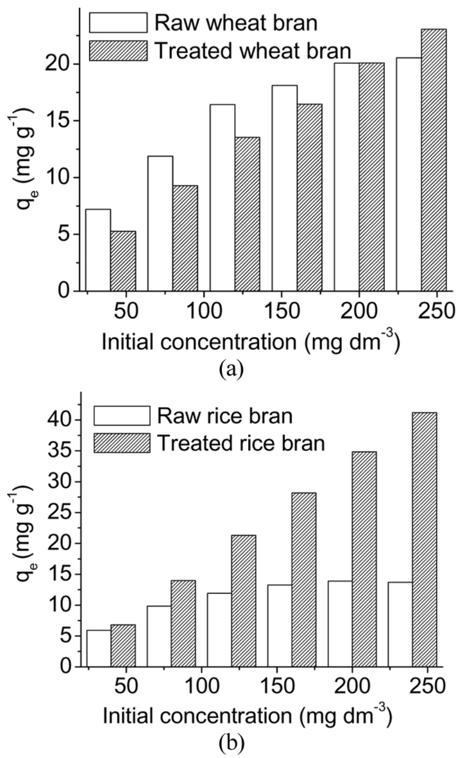

Figure 6. Effect of chemical treatment of adsorbents on Congo red adsorption (contact time 500 min; initial solution pH 8.0; adsorbent concentration 5 g dm^{-3}; temperature 25°C).

functional groups in lignin were exposed on the surface (29) and as a result, led to higher Congo red adsorption.

Effect of Adsorbent Concentration

Figure 7 shows the Congo red adsorption as a function of adsorbent concentrations in a solution at pH 8.0 and temperature of 25°C. It is found that by increasing the adsorbent concentrations the removal efficiency increased but adsorption density (i.e. adsorption amount per unit mass) decreased. Increase in removal efficiency at higher adsorbent concentration was attributed to the availability of more adsorption sites at those concentrations while the decrease in adsorption density with an increase in the adsorbent concentration could be ascribed to the fact that some of the adsorption sites remained unsaturated during the adsorption process.

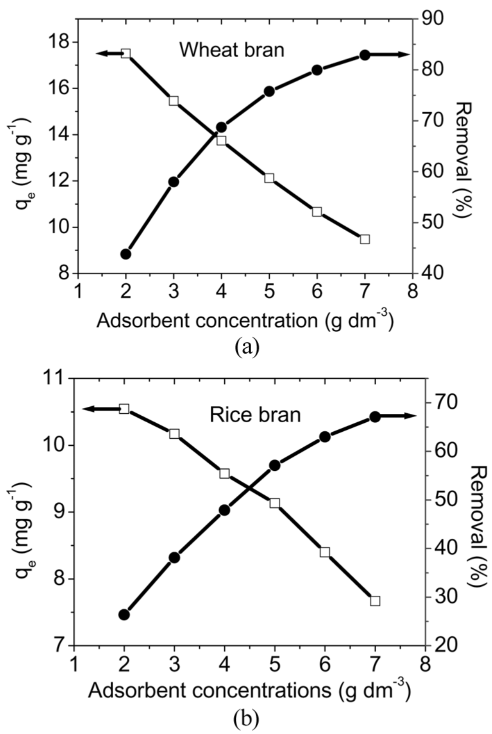

Figure 7. Effect of adsorbent concentration on Congo red adsorption (contact time 500 min; initial solution pH 8.0; initial concentration 80 mg dm^{-3}; temperature 25°C).

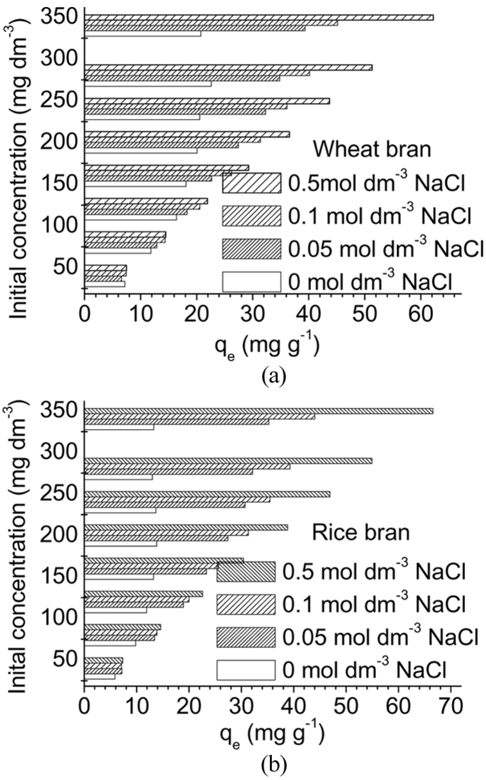

Figure 8. Effect of ion strength on Congo red adsorption (contact time 500 min; initial solution pH 8.0; adsorbent concentration 5 g dm^{-3}; temperature 25°C).

Effect of Ion Strength

The effects of ionic strength on Congo red adsorption for different adsorbents are shown in Fig. 8. It is found in Fig. 8 that an increase in solution ionic strength (NaCl) would be helpful in the Congo red adsorption onto different adsorbents, which is likely due to the compression of the electrostatic double layer (30).

CONCLUSIONS

The present study shows that the wheat bran and rice bran are effective adsorbents for the removal of Congo red from aqueous solution. The adsorption kinetics could be explained adequately by the pseudo second order equation. The equilibrium data conformed better to Langmuir equation based on the comparing the correlation coefficients determined for Langmuir and Freundlich equations, respectively. The removal efficiency increased with a rise in adsorbent concentrations. The Congo red adsorption increased markedly with an increase in ionic strength.

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