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Treatment of color through the adsorption efficiency of waste tire-derived char using response surface methodology

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ABSTRACT

Waste tire-derived char prepared from hazardous waste scrap tires was physicochemically characterized and used for the removal of reactive red textile dye color on fixed bed column. The color removal efficiency was carried out using response surface methodology based on three levels, three factorial Box–Behnken design. The significance of independent variables and their interactions were tested by means of the analysis of variance with 95% confidence limits. High regression coefficient between the variable and response (R^2 = 99.9%) showed good evaluation of experimental data by polynomial regression model. The color removal conditions were optimized for flow rate (5–15 mL/min), concentration (0.2–0.4 mg/L) of dye, and pH (2–6). The optimum conditions suggested by the model for the three variables such as flow rate, pH, and initial color concentration were 10 mL/min 2.5 and 0.15 mg/L, respectively, with maximum removal of 97.9%. The experimental value was 98.6%, very close to predicted value in model.

Keywords: Waste tire char; Adsorption; Color removal; RSM; Reactive dye

1. Introduction

The motor vehicles on the road have been increasing annually. Consequently, the consumption of tires has also been on the rise and the disposal of scrap tires is a serious environmental problem. Waste tire is almost immune to biological degradation. The disposal of waste tire is a major environmental issue throughout the world. The worldwide disposed of waste tire is also 1 billion units per year with an accepted increase of around 2% each year. Less than 7% of produced waste tires are recycled, excluding reuse, retreading, or incineration. A solution to the disposal of waste tires is the sequential, pyrolysis/combustion process that produces liquid, gaseous hydrocarbons, and char which is a solid particle material and is more economical as compared to carbon black produced from petroleum [1,2]. Tire char has two uses as reinforcing filler and an adsorbent [3]. This process creates economically valuable product out of waste. Raw material is cheap and easy to process. In addition, carbon material is resistant to corrosive and toxic environment [4].

Various kinds of synthetic dyes appear in the effluent of industries such as textile, leather, paper, and plastic. The textile dyeing industries consume large quantities of water at its different steps of dyeing and finishing among other processes. Because color is the

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most obvious indicator of water pollution so the discharge of colored wastes into receiving streams not only affects the esthetic nature but also interferes with transmission of sunlight into streams and therefore reduces photosynthetic activity [5]. Since many dyes are very visible in water at very low concentration and can be toxic to aquatic life. Dyes can be classified as anionic (direct, acid, and reactive dyes), cationic (basic dyes), and nonionic (disperse dyes) [6]. Wastewaters offer considerable resistance for their biodegradation due to the presence of these heat and light stable dyes, thus upsetting aquatic life [7]. Some dyes are reported to cause allergy, dermatitis, skin irritation, cancer, and mutations in humans [8]. Thus, the removal of dyes from effluents before mixing with unpolluted natural water bodies is very important. Congo red is azoic, anionic reactive red water soluble dye. Growing concern about environmental issues has forced the industries to investigate appropriate and environment friendly treatment technologies.

Various techniques like adsorption, chemical coagulation, and biochemical have been employed for the removal of dyes from wastewaters [9–14]. The process of adsorption has an advantage over the other methods due to its sludge free, clean operation, and complete removal of dyes, in terms of initial cost, simplicity of design, ease of operation, and insensitivity to toxic substances. Many types of adsorbent of agricultural based, biomass, etc. were used for the purpose [15–17]. The main adsorbent most widely used for dye removal is activated carbon because it has excellent adsorption efficiency for the organic compound but the cost of activated carbon is very high. This had led to further studies to find more economical, easily available, and highly effective substitute to be used as adsorbent for the removal of dyes.

At present, pyrolytic tire char [18–20] was successfully used for methylene blue and other class of dyes reported in literature [21,15,16]. The characteristics, abundance, availability, and low-cost material of waste, scrap tire-derived char illustrate that it can be used as an adsorbent for the removal of pollutants from the environment [22,23].

Azo dye is a class of dyes that cause special environmental concern due to their degradation products such as amines which are considered highly carcinogenic [2]. Consequently, dyes have to be removed in textile wastewater before discharge into the environment.

The conventional method of treatment by changing one factor of the process at one time is laborious and time consuming. Statistical methods like response surface methodology (RSM) are used commonly nowadays for the optimization of processes in which all factors are varied over a set of experimental runs [24]. In this case, RSM makes treatment process modeling simple and efficient in light of time and resource utilization. RSM has been widely used for optimization of the process variables of adsorption [25,26]. Nevertheless, in these publications, the optimization process is generally conducted to investigate the effects of several process parameters such as initial pH, initial concentration, temperature, and adsorbent dose on the adsorption in batch process. To the best of our knowledge, little information about applying RSM to optimize the process parameters for obtaining an adsorbent in column studies with desired properties was reported. Hence, the objectives of the present investigation are to quantify the adsorptive removal of reactive red dye on activated carbon derived from laboratory produced tire char and to apply the RSM in treatment studies to improve adsorption process. In this study, three levels, three factorial Box–Behnken experimental designs were applied to analyze and investigate process parameters affecting the adsorption of textile reactive red dye on fixed bed column studies by tire-derived char after activation.

2. Materials and methods

2.1. Preparation of waste derived tire chars (WDTC)

Waste tires were obtained from one of the local auto workshops, shredded to reduce into small pieces and pyrolyzed for destructive carbonization at 450–600℃ in a specially designed, closed, and sealed steel container with a narrow outlet for the escape of gases. The prepared char was leached with solution of HCl and HNO_3 in 1:1 ratio for the removal of inorganic substances. The char was extracted with n-hexane, dried at 90℃ in an oven for 50 min. Then, sieved (AS 200 Retsch, Germany) to mesh size of 200 μ and activated in an alumina crucible and heated to the preset temperature of 900℃ in a muffle furnace. The system was allowed to cool, and the crucibles were then covered with lids, removed from the furnace, and further cooled in desiccators and used for further studies [27]. The activation process usually follows hydrocarbon pyrolysis performed in an inert environment, but it is possible to accomplish pyrolysis and activation in one stage by pyrolyzing under mildly oxidizing conditions [28,29] showing linear relationships between activation time and burn-off, burn-off and surface area, and burn-off and methylene blue value (an adsorption test).

2.2. Adsorbent characterization

The WDTC was physicochemically characterized (Table 1) for pH, bulk density, carbon, hydrogen,

Table 1
Main characteristic of the waste tire-derived char (WTDC)

Parameter	Value	Parameter	Value
pH	6.45	BET surface area (m^2/g)	172.6
Conductivity (μS/cm)	2,032	BJT surface area (m^2/g)	137.4
Bulk density (g/cm^3)	0.749	Pore volume (cm^3/g)	0.54
C (%)	81.60	The size of used WDTC (um)	<63
H (%)	0.906	Pore size (nm)	49
N (%)	0.706	Ash (%)	10.63
S (%)	3.518	Zinc (%)	2.7

Table 2
Physicochemical properties of Congo red dye

Molecular formula	$C_{32}H_{24}N_6O_6S_2Na_2$
Color Index	22,120
Synonyms	Congo red
IUPAC name	Disodium 4-amino-3-[4-[4-(1-amino-4-sulfonato-naphthalen-2-yl) diazenylphenyl] phenyl] diazenyl-naphthalene-1-sulfonate
Molar weight ($g\,mol^{-1}$)	696.67
λ max (nm)	497
Purity (%)	85

nitrogen, sulfur, zinc, and ash contents. The BET surface area, BJT-specific surface area, pore volume, pore size, and particle size were also studied according to the recommended method [22]. To study the chemical structure of carbon particles as well as the presence of any functional group in carbon particles, the FTIR spectra of the char were recorded before and after the dye adsorption.

A stock solution of Congo red dye, having characteristics mentioned in Table 2 was prepared (1,000 mg/L) by dissolving a required amount of dye powder in deionized water. The stock solution was diluted to obtain the desired concentration ranging from 0.2 to 0.4 g/L.

2.3. Fixed bed column studies

Fixed bed column studies were conducted in glass column of 3.5 cm diameter and bed height of 10.0 cm. The experimental setup consisted of three parallel glass columns set. The glass column was filled with adsorbent material on gauze and cotton wool support. One set was used as reference, second set for blank, and third set for experimental study.

The effect of dye concentration was investigated by varying concentrations from 0.2 to 0.4 g/L, at room temperature (32 ± 2˚C) with varying pH 2.0, 4.0, and 6.0. The pH of the solution was adjusted to acidic and basic by the use of 1.0 N HCl and 1.0 N NaOH solutions. The dye solution was introduced to the column and allows to flow by gravity at flow rate of 5, 10, and 15 mL/min which was maintained and controlled at the bottom neck with the use of stopper. For column study, the treated sample was collected manually and analyzed.

2.4. Experimental design

The three input parameters; initial dye concentration, pH, and flow rate were varied and the three factor level were studied by Box–Behnken design (BBD) model as given in Table 3. The actual experimental design matrix which is a total of 16 experiments has been designed in duplicate by BBD as given in Table 4.

The main effects and contour plots of the factor were plotted in response surface and determined by

Table 3
Experimental factors and levels of BBD

		Level		
S. No	Factors	−1	0	+1
1	Flow rate (mL/min)	5	10	15
2	Concentration (mg/L)	0.2	0.3	0.4
3	pH	2	4	6

Table 4
BBD for efficiency of color removal by waste derived tire char (WDTC)

Experiment number	Flow rate (mL/min)	Concentration (mg/L)	pH	Actual color removal (%)	Predicted color removal (%)
1	15	0.3	2	94	94.8, 95
2	15	0.3	6	90.1	90.1
3	15	0.4	4	89.9	89.9
4	15	0.2	4	93.9	93.9
5	5	0.3	2	96.5	96.6
6	5	0.3	6	91.9	91.8
7	5	0.4	4	91.7	91.7
8	5	0.2	4	95.5	95.5
9	10	0.2	2	97.8	97.7
10	10	0.2	6	92.9	93.0
11	10	0.4	2	93.8	93.7
12	10	0.4	6	88.9	89.0
13	10	0.3	4	92.4	92.4
14	10	0.3	4	92.4	92.4
15	10	0.3	4	92.4	92.4
16	10	0.3	4	92.4	92.4

fitting a second-order polynomial equation developed to fit the experimental data and determine the relevant model terms can be written as

$$y = \beta_o + \sum \beta_{ii} X + \sum \beta_{ii} X_{ii}{}^2 + \sum \beta_{ij} X_i X_j \quad (1)$$

where y represents a response variable, β_o is the coefficient of interception, β_i is the coefficient of linear effect, β_{ii} is the coefficient of quadratic effect, and β_{ij} is the coefficient of interaction effect.

2.5. Statistical analysis

The statistical analysis of data and plots was performed using STATISTICA software version 7, response surface regression analysis was conducted.

F- and *t*-tests were employed to determine the significance of model parameters. The coefficient of correlation determination (R^2) was calculated to evaluate the performance of the regression equation. The optimum levels of the selected variables were obtained from the desirability charts. The results are analyzed by analysis of variance (ANOVA). A variable was considered significant if the calculated probability value (p) was smaller than the significance level (0.05).

2.6. Analytical procedure

The elemental analysis was carried out by Vario MACRO Elemental Analyzer. Ash contents by ASTM method-830. The pH was measured by JENCO, 6173 pH meter and conductivity by JENWAY 4010 conductivity meter, zinc by ICP-OES (Optima DV 5300, Perkin Elmer). FTIR spectra of WDTC were taken by Thermo Nicolet IR-200. Concentration of dye was determined by UV–visible Spectrophotometer (SPECORD—200, Analytik Jena) at 536 λ max.

The percent of sorbed dye was calculated from the following equation:

$$\text{Percentage sorption} = C_i - C_e/C_i \times 100 \quad (2)$$

where C_i is the adsorbance of sample before addition of the sorbent, C_e is the absorbance of sample after treatment with the adsorbents.

3. Results and discussion

3.1. Characterization of WDTC

The physicochemical characteristics of WDTC are listed in Table 1, which shows that it contains 81.6 wt.% of C, 0.706 wt.% of N which is similar to the results (0.7% N) reported by [30] 3.5 wt.% of S, 0.90 wt.% of H, and 2.7% of Zn. These findings were comparable with previous data of carbon, nitrogen, and sulfur contents as reported by [31] (81–82.17 wt.% C, 0.45–0.61 wt.% N, and 2.28–2.53 wt.% S), (2) (71 wt.% of C, 13.3 wt.% of O, 2.8 wt.% of S, and 2.3 wt.% of Zn), and [32] (2–2.5 wt.% S, 0.70–1.04 wt.% N).

The ash contents of WDTC was 10.63% when compared with carbon derived from other sources like biowaste and commercial activated carbon which have less amount of ash, i.e., 6.99% and 2.91%, respectively, as reported by Ahamed and Aa [33]. High ash content for tire-derived char was also reported by Li et al. [31] 12.32–14.58%, Roy et al. [32] 10.6–12.2 wt.%.

The pH, bulk density, and conductivity were 6.45, 0.749 g/cm^3, and 2,032 μs/cm, respectively. Li reported 6.7 pH of activated pyrolytic tire char; thus, the surface charge of WDTC is positive when solution pH is lower than 6.45. Bulk density was also comparable with reported values of 0.64 and 0.603 08 [33,34].

Fig. 1 shows the FTIR spectrum of WDTC particles, which indicated the presence of some functional groups of hydrocarbon, N–H, and oxygen. In both ((a) without dye, (b) after dye adsorption) FTIR spectrum, peak at 3,673 cm^{-1} for O–H stretch which is due to the contamination of moisture, peak at 3,165.65 and 2,938.04 cm^{-1} are for =C–H and C–H stretch in alkane. The peak at 1,537.02 is for N–H bending and at 1,090.33 cm^{-1} is for C–O stretch. A very strong and sharp peak at 2,352.80 cm^{-1} for N–H stretching is observed in char spectra after dye adsorption (b).

3.2. RSM for dye removal by WDTC

The BBD with 16 experiments in duplicate was performed to study the effects of flow rate, initial dye concentration, and pH on color removal efficiencies of WDTC. Observed and predicted values of responses are presented in Table 3. The Fisher *F*-test (Table 5) with a very low probability value (P model > F = 0.0001) demonstrates a very high significance for the regression model. The fitness of the model is checked by the determination coefficient (R^2). The coefficient of determination (R^2) was calculated to be 0.9991. This implies that more than 99.91% of experimental data was compatible with the data predicted by the model (Table 5). The R^2 value is always between 0 and 1, and a value > 0.75 indicates aptness of the model [35]. For a good statistical model, R^2 value should be close to 1.0. The adjusted R^2 value corrects the R^2 value for the sample size and for the number of terms in the model. The value of the Adj. R^2 (0.9977) is also high to advocate for a high significance of the model. The experimental results are analyzed through RSM to obtain in empirical model for the best response.

The significance of each coefficient was determined by Student's *t*-test and *p*-values, which are listed in Table 5. The larger the magnitude of the *t*-value and

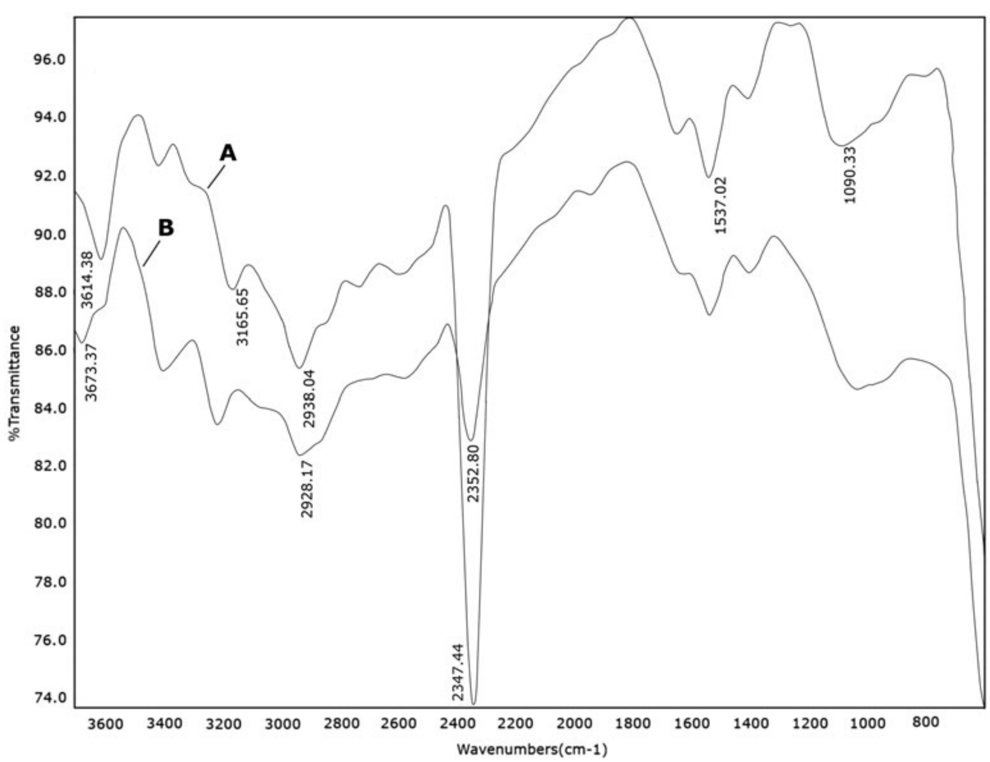

Fig. 1. FTIR Scan of waste derived tire char: (A) without dye adsorption and (B) with dye adsorption.

Table 5
ANOVA for efficiency of color removal using Box–Behnken model

	Analysis of variance				Parameter estimates		
Variables	SS	Degree of freedom	MS	F	Coef.	t-values	p-values
Intercept	227.87	1	227.8775	16,879.82	109.962	129.9223	0.000000*
Flow rate (mL/min)	0.2588	1	0.2588	19.17	−0.2825	−4.3787	0.007164*
Concentration (mg/L)	0.7579	1	0.7579	56.14	−30.00	−7.4927	0.000669*
pH	3.8710	1	3.8710	286.74	−2.7312	−16.9334	0.000013*
Flow rate × flow rate	0.0975	1	0.0975	7.22	0.0065	2.6874	0.043433*
Conc. × Conc.	0.1298	1	0.1298	9.62	18.7500	3.1009	0.026827*
pH × pH	3.8710	1	2.1467	159.02	0.1906	12.6102	0.000056*
Flow rate × Conc.	0.0100	1	0.0100	0.74	−0.1000	−0.8607	0.428754
Flow rate × pH	0.0025	1	0.0025	0.19	0.0025	0.4303	0.684872
Conc. × pH	0.0000	1	0.0000	0.00	−0.0000	−0.0000	1.000000
Error	0.0675	5	0.0135				

*Significant values.

smaller the p-value, the more significant is the corresponding coefficient. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case, the linear and square effects are significant for color removal, while interactive effect of flow rate, initial concentration, and pH is not significant because their p-values are greater than 0.05.

3.3. Optimization by response surface modeling

From the second-order regression Eq. (3), the optimum treatment conditions for flow rate, initial dye concentration, and pH were determined as flow rate—10 mL/min, dye concentration—0.15 g/L, and pH—2.5, and the percentage removal is 97.89, under which the WDTC was expected to have maximum removal percentage. These optimized conditions of all the factors were verified by conducting experiments. The experimental value was 98.63%, very close to predicted value in model.

$$y = 227.87 + 0.2588X_1 + 0.7579X_2 + 3.8710X_3 + 0.0975X_1^2 + 0.1298X_2^2 + 3.8710X_3^2 + 0.0100X_1X_2 + 0.0025X_1X_3 + 0.000X_2X_3 \quad (3)$$

3.4. Effect of flow rate

Effect of flow rate on adsorption of dye at WDTC was studied for 5.0, 10.0, and 15.0 mL/min. Table 4 shows that at 0.3 g/L dye concentration, pH 2, and flow rate of 5 mL/min, the removal of Congo red dye was 96.5%. When the flow rate increased to 15 mL/min at same concentration and pH, the dye removal was decreased to 94.2%. As the results show, breakpoint (Fig. 2) occurred faster at higher flow rates but breakpoint reaching saturation was increased with decreasing of influent flow rate since dye molecules have more time to contact with adsorbent particles. It is obvious that an increase in flow rate decreases the color removal efficiency of adsorbent [36]. At higher flow rate, the contact time between adsorbate and adsorbent is minimized leading to an early break through. Effect of flow rate is helpful for large scale treatment systems in order to utilize fixed bed for its maximum capacity with minimum flow rate.

3.5. Effect of dye concentration

The initial concentration of dye is important and limiting factor, since a given mass of adsorbent can only adsorb a fixed mass of dye and also the rate of adsorption decreased with the increase in dye concentration. The adsorption performance curve was obtained for waste tire-derived char (WTDC) at varying concentrations of adsorbents 0.2–0.4 g/L. At lower influent Congo red dye concentrations, breakthrough curves were dispersed and breakthrough occurred slower as shown in Fig. 3. As influent concentration increased, sharper breakthrough curves were obtained. These results illustrate that the change of concentration gradient affects the saturation rate and breakpoint of the curve. This can be explained by the fact that more adsorption sites were being covered as the dye concentration is increased. The larger the influent concentration, the steeper is the slope of the breakthrough curve and the smaller is the breakpoint of the curve. As the influent concentration increases, the dye loading rate increases, so does the driving force increase for mass transfer [37]. Similar trends were obtained

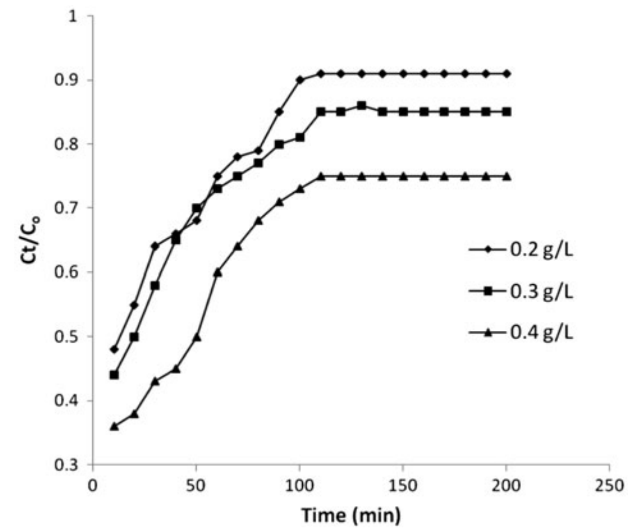

Fig. 2. Effect of initial dye concentration on breakthrough curve for dye adsorption on WTDC (bed height 10 inches, pH 2, flow rate 5 mL/min).

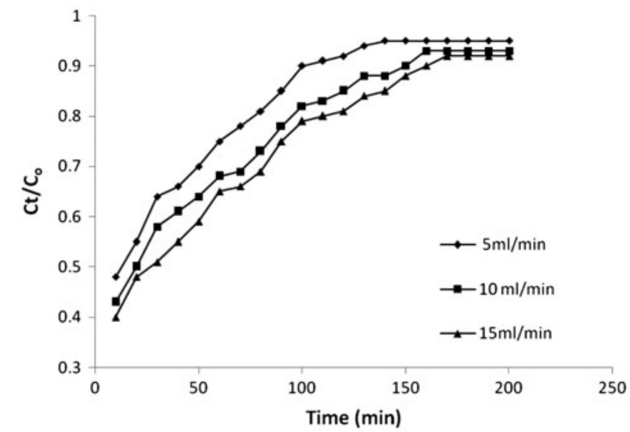

Fig. 3. Effect of flow rate on breakthrough curve for dye adsorption on WTDC (dye conc. 0.3 g/L, pH 2, bed height 10 inches).

for the adsorption of the reactive azo dye onto the granular activated carbon [38]. At higher initial concentration, binding sites were quickly filled, resulting a decrease in equilibrium time. It has also been observed that adsorbent saturation is fast at higher concentration. The percentage of removal decreased with increase in initial concentration of dye while the amount of dye adsorption increases with increase in initial concentration because initial concentration provides an important driving force to overcome mass transfer resistance of dye anions between aqueous and solid phases [39,40]. When dye is at lower concentration, i.e., 2 g/L and other two variables of flow rate and pH were 15 mL/min and 4 (Table 4), respectively, then the dye removal was 93%. Under the same conditions of flow rate and pH, while dye concentration increases to 0.4 g/L, 89.90% of dye removal was achieved. As for the given quantity of WTDC, the available surface area is limited; increasing the concentration of dye does not increase the percentage removal.

3.6. Effect of pH

Adsorption of dye on to WTDC was studied at varying pH range from 2 to 6. The optimum pH for dye adsorption by WTDC was found to be 2.0, while as the pH increases adsorption decreases. At pH 2 (Table 4), dye concentration 0.3 g/L and flow rate 15 mL/min, the dye removal was 94.2%. Under the same conditions of dye concentration and flow rate, when pH increased to 6, the dye removal is reduced to 90.2%. The pH of the aqueous solution is an important factor during dye separation, as it affects the surface charge of the adsorbent material as well as the degree of ionization of the dye molecule. At low pH, most of the basic group on WDTC is protonated which is necessary for the attraction of anionic group of the dye molecule. The similar result was also reported by [41,42]. The adsorption was decreased after pH reached at 6.0. This could be explained by the fact that at high pH, more hydroxyl groups (OH^-) are available in solution to compete with the anionic groups of dye for the adsorption sites, leading to a decrease in the number of adsorption sites for anionic dye molecule. Similar results were reported in the literature for the adsorption of acid black 172 onto nonviable *Penicillium* [43].

4. Conclusion

The WTDC was prepared, characterized, and used for color removal studies. It is concluded that WTDC is a good adsorbent for the removal of reactive red azo Congo red textile dye from water. The effect of independent variables of pH, initial dye concentration, and flow rate on color removal efficiency in aqueous solution was monitored. The RSM model chosen for present study shows high correlation coefficient (0.9991). The F-test with a very low probability value (p), also describes a very high significance for the second-order equation. The optimum treatment conditions for flow rate, initial dye concentration, and pH were determined as 10 mL/min, 0.15 g/L, and 2.5, respectively, and the percentage removal was 97.9. The experimental value was 98.63%, very close to predicted value in model.

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