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Equilibrium and kinetics of color adsorption on agriculture by-products/wastes (sugarcane bagasse, corncob, sawdust)

Tayyaba Aftab[a], Farzana Bashir[a,*], Bushra Khan[b], Javed Iqbal[a], Rauf Ahmad Khan[a]

[a]*Centre for Environmental Protection Studies, Pakistan Council of Scientific and Industrial Research Laboratories Complex Lahore, Lahore, Pakistan, Tel. 0300-4245230; email: beefarzana@yahoo.com (F. Bashir), Tel. +924299230710; emails: tayabaa14@hotmail.com (T. Aftab), javedchemist13@gmail.com (J. Iqbal), dr.rauf.pcsir@gmail.com (R.A. Khan)*
[b]*College of Earth and Environmental Sciences, University of the Punjab, Lahore, Pakistan, email: busheekhan86@gmail.com*

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ABSTRACT

In this study, sawdust, formaldehyde treated sugarcane bagasse and thermally activated corncob were used as biosorbents for the removal of reactive Congo red dye color in wastewater of an industrial unit, throwing effluent into a small stream located in the vicinity of Lahore city. The batch experiments were conducted to study the influence of pH, contact time and adsorbent dose on the removal efficiency of dye color. The equilibrium was attained after 45, 75 and 90 min for sawdust, corncob and bagasse, respectively. The adsorption efficiency was found to be in order of 78.8%, 55.1% and 45.6% for formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust, respectively. Different models were used to fit experimental data and to understand the possible sorption mechanism. The results depicted that Langmuir isotherm was more significant than Freundlich isotherm, which indicated homogenous and monolayer surface of three adsorbents. In isotherm studies, the dimensionless factor R^2 revealed that the adsorption processes more favorable for formaldehyde treated sugarcane bagasse than thermally activated corncob than sawdust. The first-order and second-order kinetic measurement were performed for dye adsorption onto three adsorbents. The $q_{e,\mathrm{exp}}$ and the $q_{e,\mathrm{cal}}$ values from the pseudo-second-order kinetic model were very close to each other in case of formaldehyde treated sugarcane bagasse and sawdust, while these values were in good agreement from pseudo-first-order for thermally treated corncob.

Keywords: Langmuir; Freundlich; Adsorption; Sawdust; Formaldehyde treated sugarcane bagasse; Thermally activated corncob

1. Introduction

Industrial wastewater pollution is one of the major environmental problems presently faced by Pakistan. Several efforts are being vigorously pursued to control it in various industries [1]. The textile industry wastewater is rated as the most polluting among all industries in terms of both volume and composition of the effluents [2,3]. Textile processing employs a variety of chemicals, enzymes, detergents, dyes, acids, sodas and salts depending on the nature of the raw material and products in a process [4].

Growing concern about environmental issues has prompted the textile industry to investigate appropriate and environment-friendly treatment technologies. During last three decades, several physical, chemical and biological treatment methods have been reported [5]. Adsorption on activated carbon has been found to be an effective process for dye removal, but it is too expensive [6]. The use of cheap and eco-friendly adsorbents have been studied as an alternative substitution of activated carbon for the removal of dyes from wastewater. Among various water and wastewater treatment technologies, adsorption process is considered to be easy, more convenient and simple to design. A number of materials have been extensively investigated as low-cost adsorbents in water

* Corresponding author.

and wastewater pollution control. Some of the important ones include wood [7]; natural coal [8]; peat [9]; chitin and chitosan [10]; corncob waste [11]; sawdust [6,12]; maize cob [13]; bagasse [14] and also bagasse pith [15]. Different types of synthetic resins like alizarin red–S-loaded amberlite IRA-400 resin (ARSA) [16], crystal violet modified amberlite IR-120 resin [17], nanostructures like SPION/b-cyclodextrin core–shell nanostructure [18], polyaniline Zr(IV) seleno tungsten phosphate nanocomposite [19], a novel bio-based adsorbent curcumin formaldehyde resin (CFR) [20] and ligand anchored nanomaterial based facial adsorbents [21] were used to remove color, phenols, metals and other pollutants in aqueous medium.

In this study, the main focus was aimed to develop a cheap and suitable technique for the removal of pollutants specially dyes color from a textile-processing unit namely N.T. Industries (Pvt) Ltd (Lahore, Pakistan). The industry was throwing its effluent directly in nearby water stream. The dyes color interferes with the adsorption of sunlight, which reduces the natural process of photochemical reactions for self-purification of surface waters to evaluate the potential of agricultural wastes to be used as adsorbents sugarcane bagasse, corncob and sawdust were selected to remove the dyes color. These agro wastes were given some treatment and then used for the treatment of N.T. effluent. The equilibrium sorption and kinetic data were analyzed through different models to understand the possible sorption mechanism of the dyes molecules onto these three adsorbents.

2. Results and discussion

2.1. Elemental analysis of adsorbents

The elemental analysis of sugarcane bagasse, corncob and sawdust was determined using CHNS analyzer (Elementar, Germany), and the results are reported in Table 1.

2.2. Infrared study of reactive Congo red dye adsorbed on sugarcane bagasse, corncob, sawdust

The Fourier transform infrared (FTIR) spectra of sugarcane bagasse, corncob and sawdust before and after dye adsorption are presented in Figs. 1(a)–(c). The characteristic absorption peaks of –OH group adsorbent cellulose are observed at 3,384, 3,328 and 3,334 cm^{-1} before dye adsorption for corncob, sugarcane bagasse and sawdust, respectively. This has been observed that after dye adsorption there is no prominent change in intensity and peak position, which clearly indicate that the adsorption of reactive Congo red dye on three adsorbents was by physical forces instead of chemical forces.

Table 1
Elemental analysis of three adsorbents, formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust used in color removal of N.T. industrial wastewater

Parameters	Formaldehyde treated sugarcane bagasse, %	Thermally activated corncob, %	Sawdust, %
Carbon	45.99	46.98	49.96
Hydrogen	5.55	6.57	5.65
Nitrogen	42.79	45.68	43.69
Sulfur	0.83	0.45	0.58
Oxygen	0.04	0.02	0.01

2.3. Optimization of pH

The pH affects the nature of the surface charge of the adsorbent used, the extent of ionization, speciation of the aqueous adsorbate species in effluent, and the rate of adsorption and dissociation of functional groups present in adsorbate and adsorbent molecules [22].

The effect of pH on formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust exhibited in Table 2. Minimum removal at low pH 2.0 can be justified as the H^+ ions compete with the dye cation for the adsorption sites in adsorption system, which in turn leads to partial release of the cations. As the pH of the solution decreased, the number of negatively charged adsorbent sites decreased, and the number of positively charged surface sites increased, which did not favor the adsorption of positively charged dye cations [23]. The surface of cellulose in contact with water is negatively charged. Basic dyes, which ionize to give the colored cationic dye base, undergo attraction, approaching the anionic structure of the adsorbents, so the results were confirmed by the study of McKay et al. [15].

Maximum adsorption of 78% and 78.8% was observed for formaldehyde treated sugarcane bagasse at pH 10 and 12, respectively, with a difference of 0.8% (Table 2). The percentage removal by formaldehyde treated sugarcane bagasse was in the range of 34.3–78.8. The increase in removal efficiency was fast from pH 2 to 6 while it was not so fast from pH 10 to 12 as mentioned in Table 2 [24].

For thermally activated corncob, the maximum adsorption was 64.8% at pH 12 (Table 2), which was 14% less than formaldehyde treated sugarcane bagasse. The removal efficiency increased from 11.8% to 64.8% in case of thermally activated corncob at the same adsorbent dose pattern while using thermally activated corncob, in the beginning, the removal efficiency was low, as the pH increased the removal efficiency increased and the adsorption followed the behavior of a natural low-cost adsorbent containing lignocellulose material [25].

For sawdust, the removal efficiency of 58.2% reduced more to 6.6% than thermally treated corncob and 20.6% than formaldehyde treated sugarcane bagasse at pH 12 (Table 2). From the results, it was concluded that in alkaline media, the negatively charged species starts dominating and the surface tends to acquire a negative charge in an adsorbent, thus increasing electrostatic attraction between positively charged adsorbate species and negatively charged adsorbent particles increased the adsorption of the dye. It was concluded that formaldehyde treated sugarcane bagasse showed good adsorption behavior than thermally activated corncob, which in turn was better than sawdust.

2.4. Effect of operating time

Treatment of dyes in the effluent N.T. industrial by adding three adsorbents, formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust, was studied at 15 min time intervals for a period of 120 min, keeping

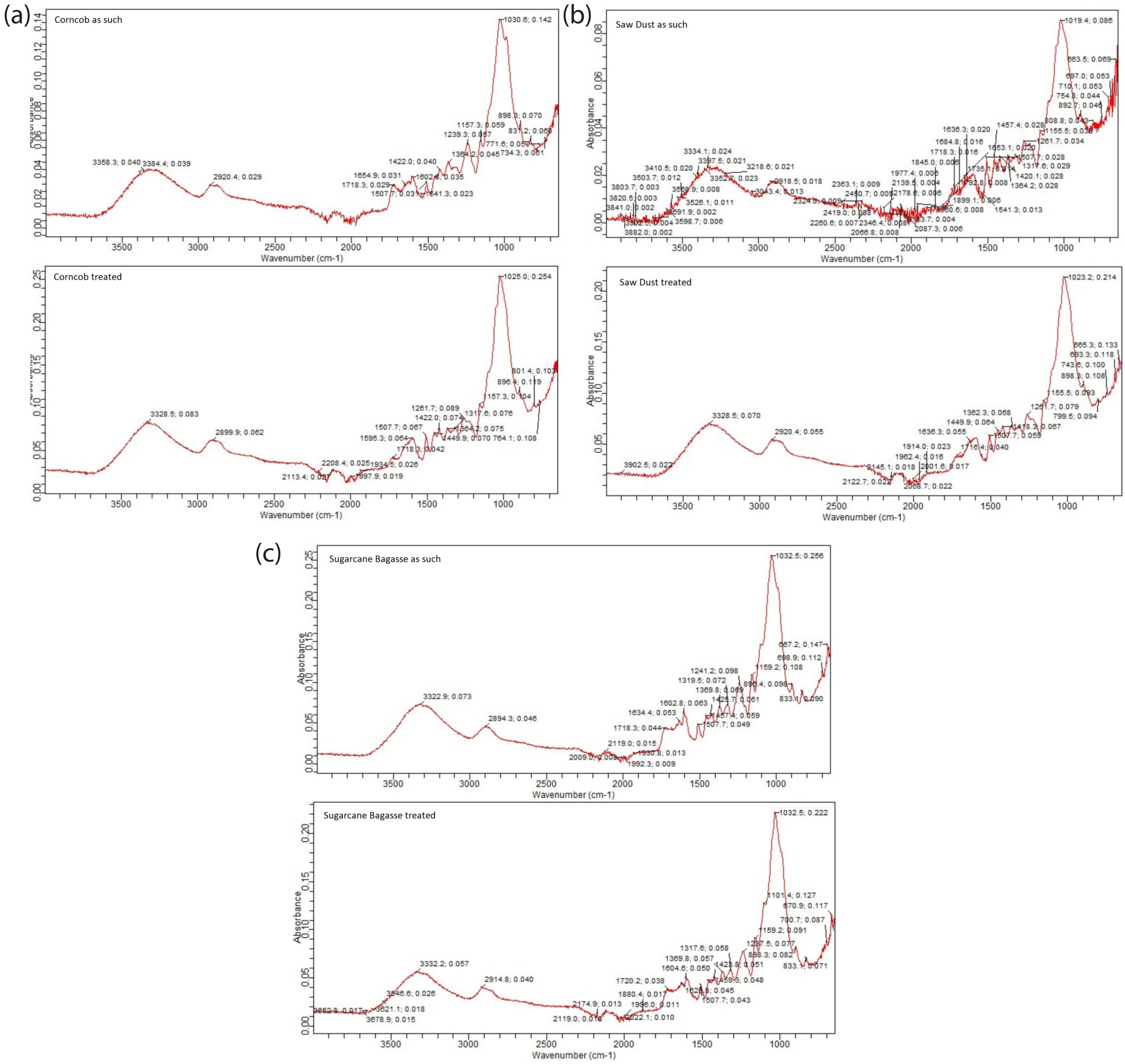

Fig. 1. (a) FTIR spectra of corncob as such and corncob treated (after dye adsorption), (b) FTIR spectra of sawdust as such and sawdust treated (after dye adsorption), (c) FTIR spectra of sugarcane bagasse as such and sugarcane bagasse treated (after dye adsorption).

pH constant. Fig. 2 represents 78.8% color removal in N.T. effluent by adding 1.0 g formaldehyde treated sugarcane bagasse after 25 min of contact time. A slow removal was seen after 25 min up to 90 min, where equilibrium was maintained during study period of 120 min. The adsorption was fast at the beginning because more adsorption sites were available. With the laps of time, the sites were difficult to occupy due to repulsion forces between solute and the bulk phases. Here the equilibrium adsorption capacity of formaldehyde treated sugarcane bagasse for dye removal in wastewater was measured, which followed Langmuir isotherm [26].

The adsorbent thermally activated corncob attained equilibrium within 40 min of contact time having 54.8% dye removal efficiency as seen in Fig. 3. The time of equilibrium state prevailed was same 40 min but the adsorption of dye color in effluent was lower than formaldehyde treated sugarcane bagasse. Same study was conducted by using thermally activated corncob as cellulose material for removal of reactive dye Orange 16 in aqueous solution. The study conducted on the adsorbent sawdust revealed (Fig. 4) that maximum adsorption was 45.2% with 0.6 g adsorbent used and the equilibrium was set after 24 min of contact time. The removal efficiency was lower as compared with other two adsorbents used. The study results showed that formaldehyde treated sugarcane bagasse has better adsorption capacity than thermally activated corncob, which in turn than sawdust [27].

Table 2
Effect of pH on color removal of N.T. industrial wastewater by three adsorbents, formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust

pH	Color removal by formaldehyde treated sugarcane bagasse, %	Color removal by corncob, %	Color removal by sawdust, %
2	34.3	11.8	24.8
4	58.0	24.7	30.2
6	73.1	30.0	33.9
8	74.6	42.2	36.0
10	78.0	53.4	44.6
12	78.8	64.8	58.2

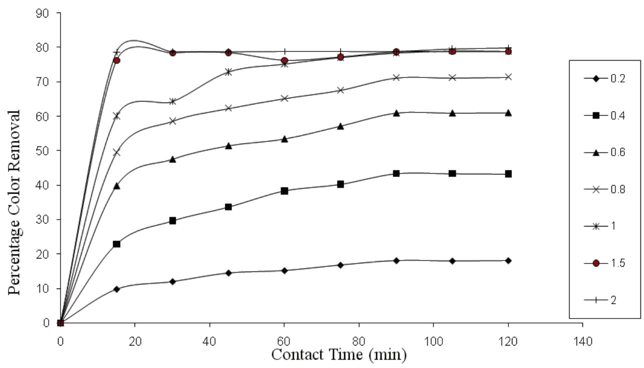

Fig. 2. Effect of formaldehyde treated sugarcane bagasse dose with time on color removal of N.T. industrial wastewater.

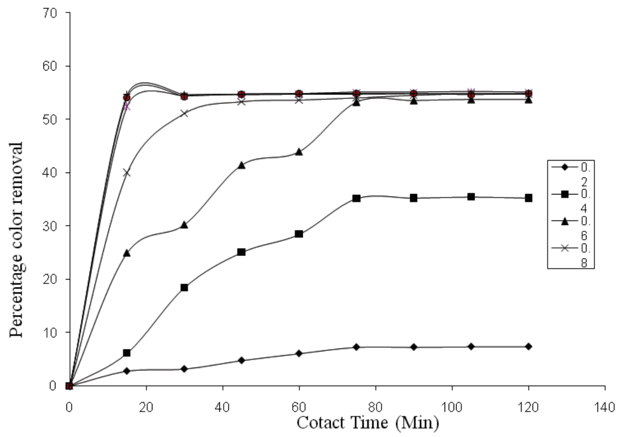

Fig. 3. Effect of thermally activated corncob dose with time on color removal of N.T. industrial wastewater.

2.5. Optimization of adsorbent dose

The adsorption behavior of formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust was studied in N.T. effluent, by varying the dose from 0.2, 0.4, 0.6, 0.8 and 1.0 g up to 2.0 g/100 ml effluent as shown in Figs. 2–4 while keeping pH constant and total time of 120 min. The

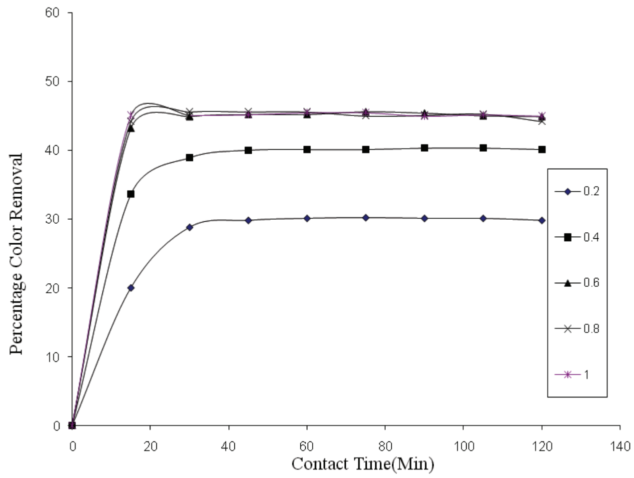

Fig. 4. Effect of sawdust dose with time on color removal of N.T. industrial wastewater.

percentage removal of color increases with increasing adsorbents doses from 0.2 g to 1.0 g/100 ml. No significant increase in removal was noted with all three adsorbents dose from 1.0 g to 2.0 g/100 ml. The maximum dose for formaldehyde treated sugarcane bagasse was 0.8–1.0 g, for thermally activated corncob 0.6–0.8 g and for sawdust 0.6 g to attain maximum removal efficiency and equilibrium state with a minimum difference. The data fit well in Langmuir and Freundlich isotherm. It was concluded that at certain dose, the amount of ions bound to the adsorbent and free ions remain constant, so maximum adsorption attained. The increase in color removal depends upon greater availability of the exchangeable sites or surface area at higher concentration of the adsorbent [28].

2.6. Sort of the adsorbent

All three adsorbents showed different adsorption efficiency for N.T. effluent (Figs. 2–4). It mainly depends upon the activation process, composition of the adsorbent and surface area available for adsorption. It has been observed that formaldehyde treated sugarcane bagasse gave the maximum removal of color 78.8%. Thermally activated corncob worked up to 54.8% and untreated sawdust, 45.2% for original N.T. industrial effluent.

2.7. Adsorption isotherms

The data obtained from isotherm models provide important information about adsorption mechanism and also the surface properties and affinities of the adsorbent. The most widely used models for single solute systems are Langmuir and Freundlich models. These models are applied in the present study to obtain different parameters.

2.8. Freundlich isotherm

The Freundlich isotherm is the earliest known adsorption equation (Eq. (1)). The Freundlich isotherm is valid for a heterogeneous adsorbent surface with a non-uniform distribution of heat of adsorption over the surface [27].

$$\left(\log q_e\right) = \log K_F + \frac{1}{n}\left(\log C_{eq}\right) \quad (1)$$

In Freundlich plots, the constants K_F and n are calculated from the intercepts and slopes of the plots of log(q_e) vs. log(C_e), respectively, and the values are given in Table 3. If $n = 1$, adsorption is homogeneous, and there is no interaction between the adsorbed species. If $n < 1$, the adsorption is unfavorable, and if $n > 1$, the adsorption is favorable. In present studies, we have found that n is greater than unity for two adsorbents formaldehyde treated sugarcane bagasse and thermally treated corncob, which indicate that adsorption is favorable but unfavorable for the third adsorbent used sawdust where the value of $n < 1$ and the values of correlation coefficient (R) is closer to unity for all three adsorbents [27]. It has also been reported that the value of K_F for Freundlich model gives rough estimation about the degree of adsorption. In the present studies, it was observed that the value of K_F (17.18 mg/g) for the adsorption of dye onto formaldehyde treated sugarcane bagasse already reported in a study for methyl red [24] is greater than thermally treated corncob of K_F value 8.084 mg/g, which in turn is greater than sawdust having K_F value 2.49 mg/g (Table 3).

2.9. Langmuir isotherm

The Langmuir equation (Eq. (2)) in its linear form is described as follows:

$$\frac{C_e}{q_e} = \frac{1}{Q_b} + \left(\frac{1}{Q}\right)C_e \quad (2)$$

The Langmuir isotherm, however, assumes that the sorption takes place at specific homogeneous sites within the adsorbent. A plot of C_e/Q_o vs. C_e should indicate a straight line of slope $1/Q_o$ and an intercept of $1/(K_L Q_o)$, where C_e is the equilibrium concentration (mg/dm^3), Q_o is the amount of dye sorbed (mg/g), Q_o for a complete monolayer (mg/g), and K_L is the sorption equilibrium constant (dm^3/mg). These parameters are described in Table 3.

In our study, Q_o is 77.52 mg/g for formaldehyde treated sugarcane bagasse. The value is much greater when compared with reported values such as untreated sugarcane bagasse for Congo red dye having 4.43 mg/g, Q_o value, which showed that formaldehyde treated bagasse have better performance as compared with untreated sugarcane bagasse, which in turn is greater than thermally treated corncob having value of Q_o, 74.62 mg/g. Both are very near to each other but far away as compared with sawdust having very low value of Q_o, 0.921 mg/g (Table 3).

The correlation coefficient (R^2) value for the equilibrium curve is the most significant parameter to optimize the design of an adsorption system to remove color from wastewater. In the present study, the correlation coefficient values of Langmuir isotherm (Eq. (2)) are more nearer to one than Freundlich isotherm (Eq. (1)), so Langmuir isotherm (Eq. (2)) is more significant than Freundlich isotherm (Eq. (1)), which indicating homogenous and monolayer surface of adsorbent.

2.10. Kinetic studies

First- and second-order kinetic models (Eqs. (3) and (4)) were applied for the adsorption studies of color onto sawdust, formaldehyde treated sugarcane bagasse and thermally treated corncob. The obtained kinetic parameters are presented in Table 4, which are helpful for the prediction of adsorption rate and also provide important information for designing and modeling adsorption processes. The conformity between experimental data and the model predicted values was expressed by the correlation coefficients (R^2).

First-order kinetic equation:

$$\log(q_e - q_t) = \log(q_e) - k_t / 2.303\, t \quad (3)$$

Second-order kinetic equation:

$$t / q_t = 1/h + 1/q_e\, t \quad (4)$$

In the first-order kinetic model (Eq. (3)), the plot of the experimental adsorption capacity log($q_e - q_t$) vs. t gives a linear relationship from which K_1 and predicted q_e (Fig. 5) can be determined from the slope and intercept of the plot, respectively.

When the second-order kinetic model (Eq. (4)) is applied, then the plot of t/q_t vs. t shows a linear relationship, and the values of K_2 and equilibrium adsorption capacity q_e were calculated from the intercept and slope (Fig. 6), respectively.

The $q_{e,\text{exp}}$ and the $q_{e,\text{cal}}$ values for the pseudo-first-order and pseudo-second-order models are shown in Table 4. The correlation coefficients (R^2) result in the range of 0.855–0.975 for the first-order model and fall within the range of 0.898–0.999 for second-order model.

The $q_{e,\text{exp}}$ and the $q_{e,\text{cal}}$ values from the pseudo-second-order kinetic model (Eq. (4)) are very close to each other in case of formaldehyde treated sugarcane bagasse and sawdust, while these values are in good agreement from pseudo-first-order

Table 3
Freundlich and Langmuir isotherm parameters for dye adsorption in N.T. industrial wastewater on formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust at 32°C

Material	Freundlich isotherm (32°C)			Langmuir isotherm (32°C)		
	K_F	n	R^2	K_L	Q_o (mg/g)	R^2
Formaldehyde treated sugarcane bagasse	17.18	2.83	0.9787	10.66	77.52	0.9846
Thermally activated corncob	8.084	2.13	0.9612	26.26	74.62	0.9603
Sawdust	2.49	0.238	0.9354	3.09	0.921	0.8627

Table 4
Kinetic parameters for the adsorption of dye adsorption onto formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust at 32°C

Material	First-order kinetics				Second-order kinetics		
	$q_{e,cal}$, mg/g	$q_{e,exp}$, mg/g	K_1	R^2	q_e, mg/g	K_2	R^2
Formaldehyde treated sugarcane bagasse	35.09	45.25	0.029	0.963	51.28	2.73	0.990
Thermally treated corncob	44.48	46.35	0.00207	0.975	400	3.23	0.898
Sawdust	11.90	50.375	0.059	0.8551	52.08	19.68	0.999

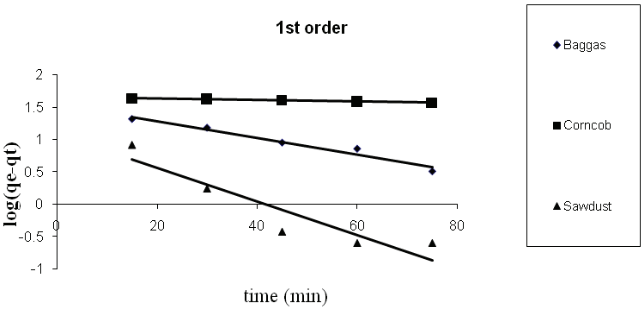

Fig. 5. Pseudo-first-order kinetic plot for the adsorption of dye onto formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust at 32°C.

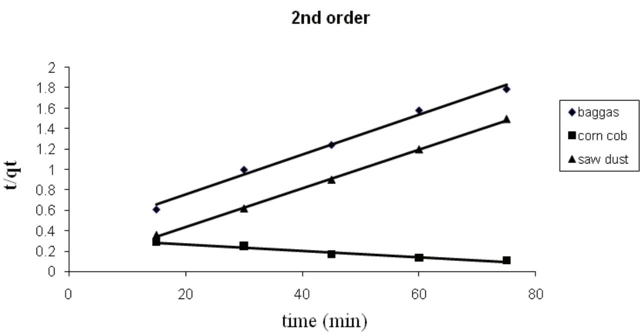

Fig. 6. Pseudo-second-order kinetic plot for the adsorption of dye onto formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust at 32°C.

(Eq. (3)) for thermally treated corncob (Table 4). The calculated correlation coefficients (R^2) are also closer to unity for pseudo-second-order kinetics than that for the pseudo-first-order kinetic model. Therefore, the sorption can be approximated more appropriately by the pseudo-second-order kinetic model for the adsorption of formaldehyde treated bagasse and sawdust and pseudo-first-order kinetic model for thermally treated corncob.

3. Experimental

The N.T. Industries (Pvt) Ltd. is cotton dyeing and finishing textile unit located in the vicinity of Lahore was chosen for the study of effluent color removal efficiency by adsorption process using three locally available low-cost agricultural wastes. The industry used different dyes in combination, and after dyeing and finishing, red color effluent was seen to throw away in nearby stream. Grab sampling was carried out weekly from industrial outlet. Effluent was collected in dry, polypropylene bottles, which were kept in ice during transportation to preserve the characteristics of wastewater. Sample was stored in the refrigerator at 4°C.

3.1. Adsorbents

The adsorbent samples of sugarcane bagasse, corncob and sawdust were prepared in the solid waste laboratory at College of Earth and Environmental Sciences, University of the Punjab, Lahore, Pakistan. For the grinding of the adsorbents, shredder (national) was used to obtain uniform size of material. The samples were sieved by using sieve set (Habib test sieve) to mesh size 200 micron. The specific surface area of the three adsorbents was 145 cm/gm as calculated by particle distribution curve [29].

3.2. Formaldehyde treated sugarcane bagasse

The sugarcane bagasse was obtained from local sugarcane juice shop, thoroughly washed with distilled water and then dried in sunlight to evaporate all moisture. The dry bagasse was ground to a fine powder and sieved to 200 micron mesh size. To polymerize and immobilize the color and other impurities in wastewater soluble substances, the ground bagasse was treated with 1% formaldehyde in the weight to volume ratio, then oven dried at 50°C for 4 h. The bagasse was washed with distilled water by using Buchner funnel to remove free formaldehyde and activated at 80°C in the air oven for 24 h [6]. The material was kept in air tight container for further experimental study use.

3.3. Thermally activated corncob

Corn is a significant crop all around the world. The annual production worldwide is about 520 × 10^9 kg [7]. Asia is the second major production region of corn. The corncob is the waste generated during processing corn. Since the ratio between corn grain and corncob may reach 100:18, therefore, a large quantity of corncob is generated. In present study, corncob was collected from the corn street seller. The adsorbent material was washed with distilled water, cut into small pieces, then oven dried at 80°C for 72 h [30] and finally grinded to 200 micron mesh size. The material was stored in an airtight jar for further experiments.

3.4. Sawdust

The sawdust was collected from a local carpenter workshop at Lahore. Without applying any treatment, the sawdust was sieved by using sieve set to 200 micron mesh size and used for experimental purposes.

3.5. FTIR spectrum of the adsorbents

The FTIR spectra of sugarcane bagasse, corncob and sawdust, before and after dye adsorption, were drawn by using Cary 360 FTIR of Agilent Technologies (USA).

3.6. Batch adsorption experiments

For batch studies, each adsorbent was accurately weighed and transferred into 250 ml conical flasks, 100 ml of wastewater was added in three replicates, and one blank sample was run at 250 rpm at room temperature. At time interval of 15 min, the supernatant was drawn, and the absorption was measured by UV-VIS spectrophotometer (Specord-200, Analytik Jena, Germany) to calculate the concentration at λ_{max} at 500 nm. The removal efficiency was calculated from mathematical equation (Eq. (5)) [31]:

$$\text{Decolorization} = \frac{(\text{Absorbance})_0 - (\text{Absorbance})_t}{(\text{Absorbance})_0} \times 100 \quad (5)$$

where $(\text{Absorbance})_0$ is the absorbance at 500 nm before the experiments, and $(\text{Absorbance})_t$ is the absorbance at time t.

The experiments were individually set for each adsorbent with blank. The effect of contact time was determined by comparing the removal efficiency of color during 0–120 min. The absorption was measured every 15 min interval for pH range 2–12 and adsorbents dose from 0.2, 0.4, 0.6, 0.8 and 1.0 g up to 2.0 g (200 micron mesh size) for each 100 ml dye effluent followed by 5 min of magnetic agitation. The mixtures were filtered, and the supernatants were analyzed in UV/Vis spectrophotometer. Earlier data was used to build the Langmuir and Freundlich isotherm.

3.7. Kinetic studies

To determine the necessary time for adsorption, 100 ml of wastewater was taken in a series of 250 ml conical flasks. Known amount of adsorbent was added to different flasks. The flasks were kept in shaker and stirred at constant speed. At the end of predetermined time (t) the intensity of color was measured.

4. Conclusion

In this study, the efficiency and comparison of three adsorbents, formaldehyde treated sugarcane bagasse, thermally activated corncob and sawdust for removal of color from textile effluent of the N.T. Industries (Pvt) Ltd. have been made. The parameters varied were pH, contact time, initial dye concentration and adsorbent dose. Initial pH range 2–10, was favorable for all three adsorbents but the optimum pH for removal was alkaline. Formaldehyde treated sugarcane bagasse was the most effective 78.8% for removal of dye color in industrial effluent at 32°C temperature. It is concluded that the modification of adsorbents increased the removal efficiency of dye color. Low cost with higher removal efficiency of adsorbents would make the process economical and efficient. In the present study, the correlation coefficient values of Langmuir isotherm (Eq. (2)) were more nearer to one than Freundlich isotherm (Eq. (1)), so Langmuir isotherm (Eq. (2)) is more significant than Freundlich isotherm (Eq. (1)), which indicating homogenous and monolayer surface of adsorbent.

It was observed that the adsorption process followed pseudo-second-order kinetic model (Eq. (4)) for the adsorption of formaldehyde treated bagasse and sawdust and pseudo-first-order kinetic model (Eq. (3)) for thermally treated corncob. These results will be the starting point for further research, on other types of textile dyes, usually present in the industrial wastewater from textile chemical finishing processes. The method may be helpful for designing and fabricating a dye rich effluent treatment plant in industry. After applying the simple method of adsorption, the pollution load of textile wastewater can be reduced to prescribed limits of National Environmental Quality Standards.

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