

COMPARATIVE STUDIES OF PHYSICAL CHARACTERISTICS OF RAW AND MODIFIED SAWDUST FOR THEIR USE AS ADSORBENTS FOR REMOVAL OF ACID DYE

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The present paper aims to investigate the physical characteristics of sawdust relative to its use as an adsorbent for removal of an acid dye (Orange G) from aqueous solutions. The raw sawdust was sieved to have a uniform size and was activated by sulphuric acid by refluxing the content at 60 °C for 4 h. Surface morphology and surface functional groups of both raw and modified sawdust samples were investigated by Scanning Electron Microscope (SEM), Energy Dispersive X-ray Analysis (EDX), Fourier Transformation Infrared (FTIR), and elemental analysis. All these analyses displayed significant change in the structure of the sawdust. The data obtained from batch adsorption experiments for the removal of the selected dye confirmed that adsorption characteristics of the modified sawdust were better than those of raw sawdust.

Keywords: Characterization; FTIR; pH_{zpc} ; Sawdust; SEM

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INTRODUCTION

Sawdust is a by-product of saw mills, where it is produced from the cutting of wood. The residual wooden powder is known as sawdust. Sawdust is a minor fuel and is used to warm houses. Use of sawdust as an adsorbent for the removal of dyes from industrial effluents has also been reported (Ansari and Mosayebzadeh 2010). Dyes are used in a number of industries. Synthetic dyes are used in the textile, paper, carpet, plastic, petroleum, food, paint, cosmetics, pharmaceutical, rubber, ceramic, and leather industries (Chandra et al. 2007; Tahir et al. 2009; Gong et al. 1998). Direct, reactive, acidic disperse, vat, and basic dyes account for about 85 percent of the dyes used in these industries (Liew Abdullah et al. 2005). The textile industry constitutes one of the most important industrial branches in India. The total dye consumption in the textile industry alone is in excess of 10^7 kg/year, and an estimated 90 percent of this is consumed in dyeing fabric (Sharma and Uma 2010). Textile industries consume large volumes of water and chemicals for the wet processing of textiles, and approximately 10^6 kg of dyes are discharged by textile industries in the waste water stream annually (Won et al. 2006). The presence of dyes even at very low concentration in effluents discharged from these industries is highly visible. Human eyes can clearly detect a concentration of 0.005 mg/L of reactive dyes in water (Geethakarathi and Phanikumar 2011). Therefore, the presence of dye exceeding this limit would not be permitted aesthetically.

Low-cost alternatives such as agricultural waste rice husk, wheat bran, maize cob, industrial waste, red mud, benotite, kaolite, indigenous materials, clay, soil, and sawdust (Acemioglu 2005; Özcan et al. 2004; Ozdemir et al. 2004; Ghosh and Bhattacharyya 2002; McKay et al. 1999; Mohan and Singh 2002; Amphol et al. 2009; Kalderis et al. 2008; Kavitha and Navasivayam 2008; Chiou and Li 2002; Sharma 2008; Ho and McKay 2003) for the treatment of the effluents containing these dyes have been proposed, but the main drawback with most of these materials is their low efficiency of dye removal.

The present communication deals with preparation of modified sawdust and then applying it for the removal of a toxic dye, Orange G, from aqueous solutions. Characterization of modified sawdust, the adsorbent used in this study, was carried out by suitable techniques such as FTIR, EDS, and SEM. Elemental analysis of the adsorbents was also carried out. Additionally, the pH_{zpc} of the adsorbents was determined. The effect of initial concentration of the dye on its removal by selected adsorbents was investigated. Equilibrium studies were also performed.

MATERIALS AND METHODS

Materials

The sawdust was collected from a local furniture factory. It was washed several times with distilled water to remove impurities. The cleaned sawdust was oven dried completely at 110°C , cooled, and sieved to 45 to $150\ \mu\text{m}$. Then it was used without further treatment.

Another part of same size fraction of sawdust was exposed to activation using H_2SO_4 (Hameed et al. 2007). 100 g of sawdust was activated by refluxing it with 625 mL of 40 wt% H_2SO_4 at 60°C for 4 h in a rounded-bottom flask. The suspension was cooled in air and then washed with double-distilled water and soaked in 1% sodium bicarbonate solution to remove residual acid. The material was dried in an oven at 110°C for 24 h and used for adsorption studies.

Adsorbate

Orange G dye was purchased from Merck Speciation Private Limited, Mumbai. The chemical structure of Orange G dye is shown in Fig. 1.

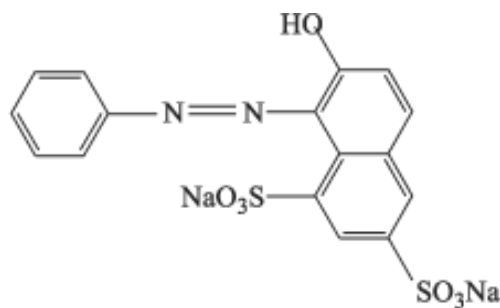


Figure 1. The molecular structure of orange G.

Characterization of Adsorbent

EDS and SEM

The chemical compositions are analyzed by Energy-dispersive X-ray spectroscopy (EDS). Before analysis, the samples were coated with platinum to render them electrically conductive. The scanning electron microscopy (SEM) observations were carried out with a Jeol instrument, Japan, JSM 6390LV microscope equipped with an Oxford Links-Isis energy dispersive X-ray analyzer (EDS). The SEM measures the surface morphology of conducting and non-conducting materials by analyzing back-scattered electrons (BSE) and secondary electrons (SE). Scanning electron microscopy (SEM) technique was employed to observe the surface physical morphology of raw and modified sawdust.

Point of Zero Charge

Determination of pH_{zpc} was done to investigate how the surface charge of adsorbent material depended on pH. Raw and modified sawdust for the determination of pH_{zpc} , 0.01 M NaCl was prepared, and its initial pH was adjusted between 2.0 to 12.0 by using NaOH/HCl in each batch. Then, 50 mL of 0.01 M NaCl was taken in 250 mL Erlenmeyer flasks and 0.20 g of adsorbent was added to each solution. These flasks were kept for 48 h, and the final pH of the solutions was measured by using a pH meter. Graphs were plotted between “ pH_{final} vs. $pH_{initial}$ ” (Fig. 4a and 4b). The point of intersection of the curves of pH_{final} vs. $pH_{initial}$ was recorded as pH_{zpc} .

FTIR

Infra-red spectra of raw and modified sawdust were measured with a Fourier transform infrared spectrophotometer (Varian 1600 FT-IR Scimitar Series) to elucidate the functional group presenting in sawdust before and after activation. For measuring IR spectra, 5 mg of powdered raw sawdust or modified sawdust was encapsulated in 400 mg of KBr. A translucent disk was made by pressing the ground mixed material with a hydraulic pallet press (PCI services, Bhandup, Mumbai) for 1 min. The spectra were recorded in a FTIR within the range of 500 to 6000 cm^{-1} (Soom et al. 2009).

Analytical Procedures

Batch adsorption experiments were carried out by adding a fixed amount of adsorbent (1.0 g) to a series of 250 mL conical flasks filled with 50 mL diluted solutions (2.5 to 10 mg/L). The conical flasks were then sealed and placed in a water bath shaker and shaken at 120 rpm with a required adsorbent time at 298, 308, and 318 K. The flasks were taken from the shaker at regular time intervals. At the adsorption period, the supernatant was centrifuged for 10 minutes at 10,000 rpm. Unless specified, all the experiments were carried out at pH 2 for the sorption of Orange G on the raw sawdust and modified sawdust. The pH values were adjusted by adding a few drops of dilute NaOH or HCl, and were measured by a pH meter, which was calibrated using pH = 4.0 and pH = 7.0 buffer solutions. Blank experiments were carried out with dye solution and without adsorbent to ensure that no dye was adsorbed onto the walls of the conical flasks. After centrifugation the concentration of supernatant solutions was determined using a UV spectrophotometer at 480 nm.

The amount of adsorption at equilibrium, q_e (mg/g) and % of color removal were calculated using Equations (1) and (2).

$$q_e = (C_o - C_e) V/W \quad (1)$$

$$\% \text{ removal} = 100 \times (C_o - C_e) / C_o \quad (2)$$

RESULTS AND DISCUSSION

Surface Characterization of Adsorbent

The chemical composition of the adsorbent was analyzed by EDS, and results are shown in Table 1.

Results of the SEM study showed that sulphuric-acid-activated sawdust was porous with well-developed pores. Raw sawdust was characterized by a highly oriented structure in the form of filaments filled with material, conferring an anisotropic character to the sawdust. This isotropy was eliminated by treatment with sulphuric acid. Sulphuric acid was selective in its effects. Firstly, it cleans the filaments, thereby eliminating sawdust anisotropy and leaving empty channels. Then, it reacts with the sawdust components, thereby preserving a honeycomb structure (Alvarez et al. 2004; Camacho et al. 1996).

Table 1. Chemical Composition of Raw and Modified Sawdust

Elements	Raw sawdust	Modified sawdust
	(Weight %)	
C	45.52	46.98
O	30.65	34.06
Ca	0.61	0.50
Cu	2.39	1.36
Zn	1.80	1.34
Au	19.04	15.24
S	0	0.52
Total	100.00	100.00

FTIR Analysis

The IR spectra (Fig. 3a and 3b) of sawdust exhibited broad bands at 3395.60 and 3359.29 cm^{-1} , which can be attributed to the stretching of OH groups. These broad bands indicate high concentrations of phenol and alcohol. The intermolecular O-H stretching vibration band of raw sawdust spectrum in the range of 3200 to 3400 cm^{-1} appeared broader compared to the modified sawdust.

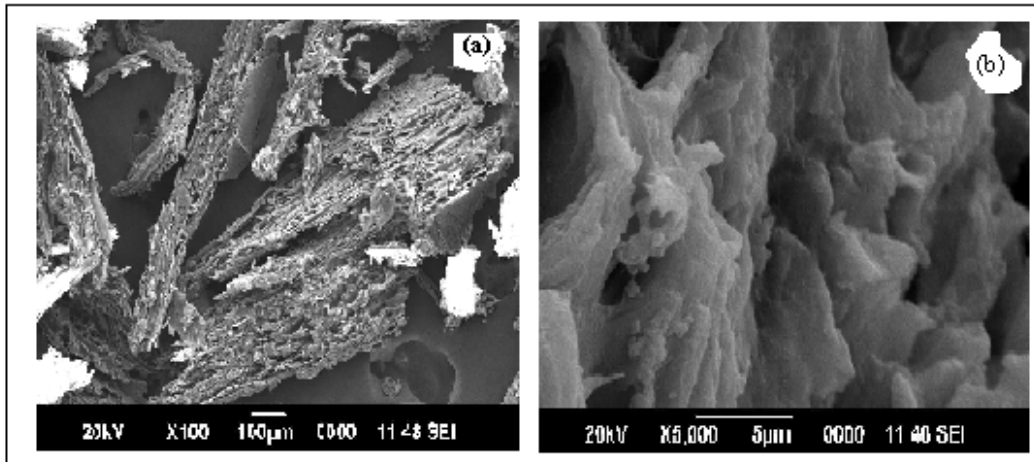


Fig. 2. SEM micrograph of the sawdust: 2a raw and 2b modified sawdust

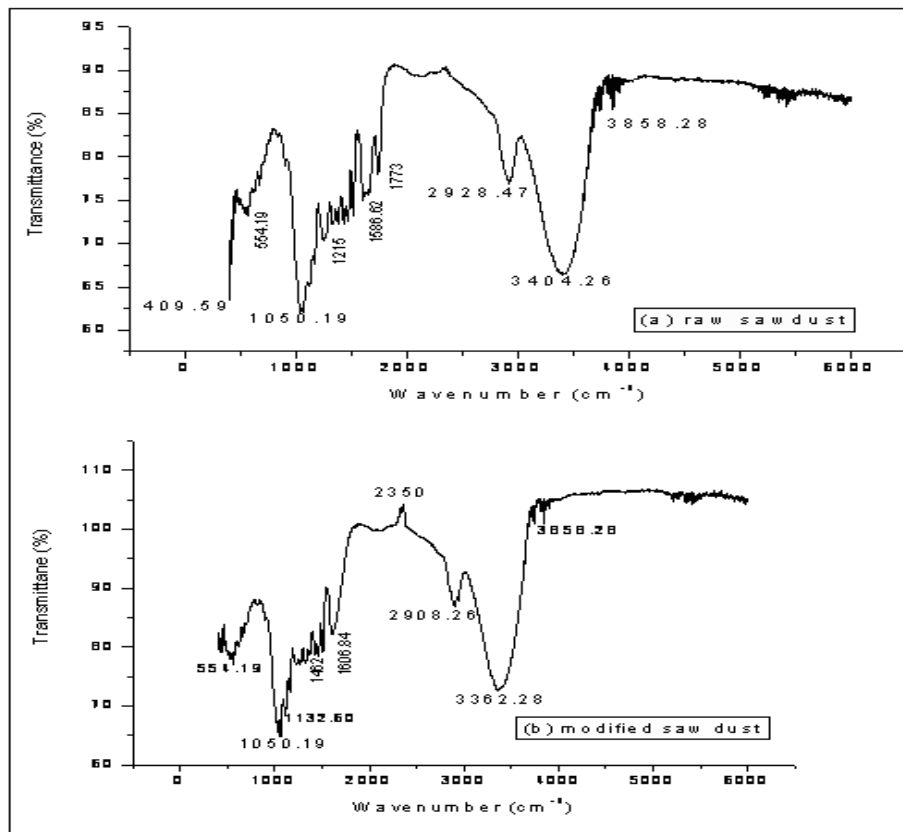


Fig. 3a. FTIR of raw sawdust Fig. 3b. FTIR of modified sawdust

A greater number of OH groups on the glucose units of the cellulose polymers broadened the peak. The peak at 1550 to 1610 cm^{-1} was assigned to the C=O bonds. The absorption bands at 2939.54 and 2880.23 cm^{-1} are due to a contribution from C-H stretching. These medium stretching bands show the presence of vinyl and methyl groups. The strong band observed at 1053.57 and 1046.32 cm^{-1} indicated the stretching of many C-OH and C-O-C bonds. The strong band between 500 and 600 shows the presence of the C-X group.

Point of Zero Charge (ZPC)

In the present work, the pH_{zpc} of raw sawdust and modified sawdust were found to be 7.29 and 6.68 respectively (Figs. 4a and 4b). At a pH of solution below the pH_{zpc} of the adsorbent, the surface of sawdust is negatively charged and can attract cations from the solution; and when solution $\text{pH} < \text{pH}_{\text{zpc}}$, the surface of sawdust is positively charged and attractive to anions (Sharma et al. 2009; Sun et al. 2007). In the present studies, a low pH was favorable for adsorption of the anionic dye (Orange G) by sawdust. As the pH of the system was decreased, the number of negatively charged adsorbent sites decreased and the number of positively charged surface sites increased. These effects favor the adsorption of negatively charged dye anions due to electrostatic attraction. Also, lower adsorption of Orange G at basic pH can be ascribed to the presence of excess OH^- ions competing with dye for adsorption sites.

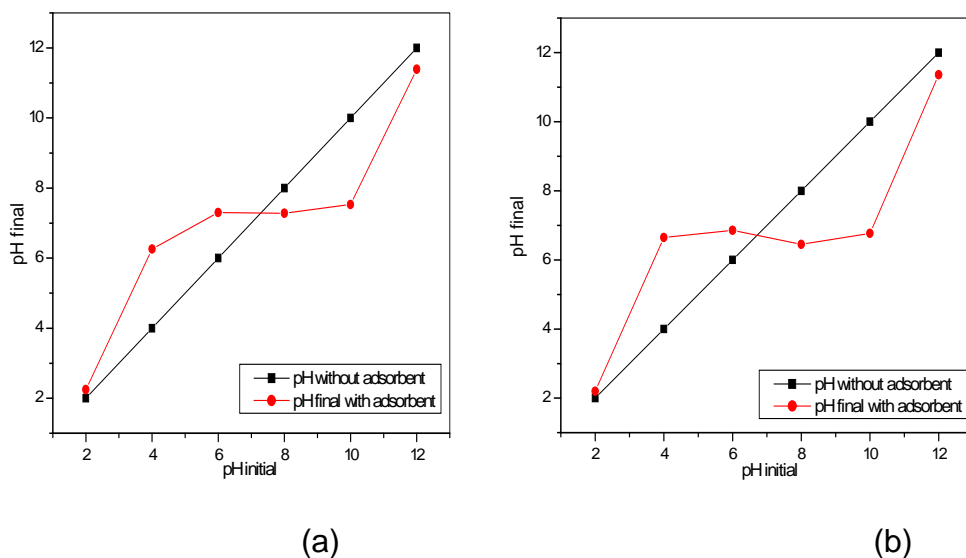


Fig 4. pH_{zpc} 4(a) raw sawdust 4(b) modified sawdust

Effect of Initial Concentration and Contact Time on Raw Sawdust and Modified Sawdust

The adsorbate concentration and contact time between adsorbent and adsorbate species were found to play significant roles in the process of removal of dyes from water and wastewater by adsorption at a particular temperature and pH (Fig. 5). Concentrations from 2.5 to 10 mg/L at 25°C and pH 2 were considered. A rapid uptake of the dye species

and establishment of equilibrium in a short period indicates the efficiency of the adsorbent for its use in wastewater treatment.

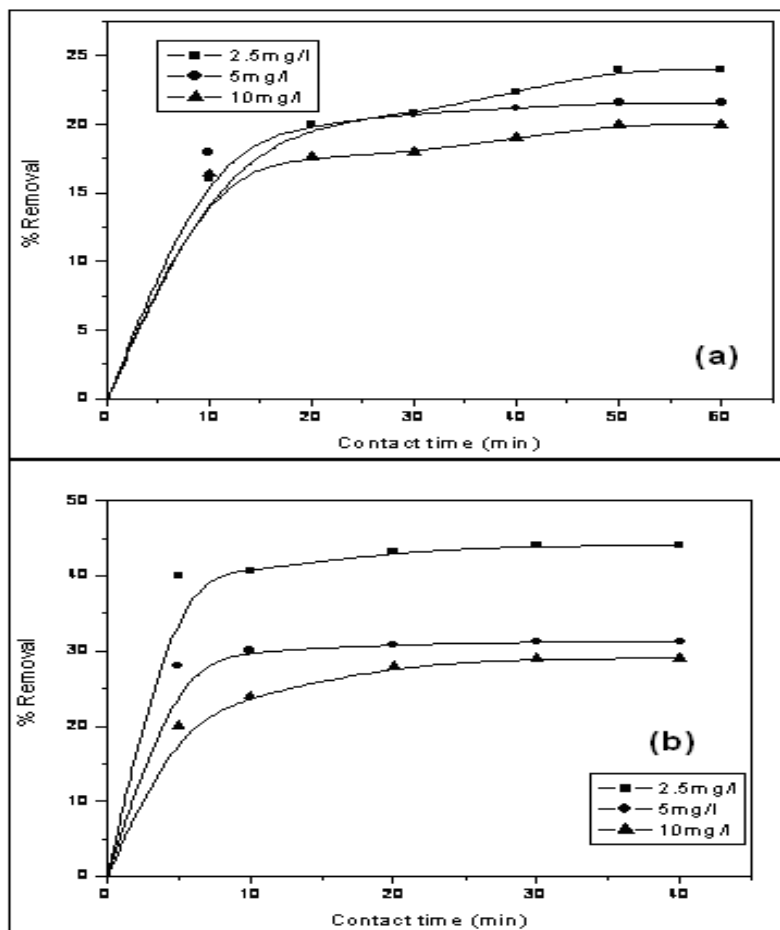


Fig. 5a. Effect of contact time and concentration on removal of Orange G dye by adsorption on raw sawdust, and **Fig.5b.** by modified sawdust

In addition to this, the contact time is one of the factors affecting the development of surface charges at the solid solution interface. The initial concentration of adsorbate also plays an important role, as a given mass of the adsorbent can adsorb only a certain amount of the solute. The more concentrated the solution or effluent, the smaller is the volume of effluent that a given mass of adsorbent can purify. In high concentration range, the fractional adsorption is low (Khan et al. 2009; Özacar et al. 2005). The adsorption capacity at equilibrium (q_e) increased from 0.03 to 0.10 mg/g by raw sawdust up to 0.05 to 0.15 mg/g by modified sawdust with increase in initial adsorbed proportions decreasing from 24 to 20% and 44 to 29% by raw sawdust and modified sawdust, respectively, by increasing concentration of dye from 2.5 to 10 mg/L. The effect of initial concentration of Orange G dye on removal by raw sawdust and modified sawdust are shown in Figs. 5a and 5b, respectively. At lower concentration, the ratio of initial number of dye (Orange G) molecules to the available surface area is low; consequently, the fractional adsorption became independent of the initial concentration. However, at higher

concentration, the available sites of the adsorption become fewer and hence percentage removal of Orange G was dependent upon initial concentration. It is apparent from the figure that the removal of Orange G acquires a maximum at the time of equilibrium (20 minutes) and becomes gradual thereafter. This indicates that the rate of adsorption is very fast. After that, no significant change in the extent of adsorption was observed.

Effect of pH

The pH of an aqueous solution is an important parameter for removal of pollutants from water and wastewater by adsorption processes. The pH also controls the degree of ionization and speciation of the adsorbate. In the present studies the removal of Orange G by sawdust was studied at pH values of 1.0, 2.0, and 4.0. Maximum removal was 63.2% at pH 1.0, and it decreased with increase in the pH value (figure not shown). With a decrease of pH from 4.0 to 1.0, the uptake of Orange G increased from 8 to 36% for raw sawdust and 28 to 63.2% for modified sawdust; these values are close to the satisfactory level by sawdust at 25°C, agitation speed 120 rpm, and the initial dye concentration 2.5mg/L.

Adsorption Isotherms

Langmuir isotherm model

For the equilibrium studies, the experiments were carried out at the three selected concentrations, viz. 2.5 mg/L, 5 mg/L, and 10 mg/L of the dye at three temperatures: 298, 308, and 318 K. The Langmuir adsorption isotherm model assumes a mono-layered coverage of the adsorbent surface by the solute. The Langmuir isotherm is based on the assumption that maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface and that the energy of adsorption is constant. The linearized expression of Langmuir model can be expressed as follows (Langmuir 1918; Won et al. 2006):

$$\frac{C_e}{q_e} = \frac{1}{Q^0 b} + \frac{C_e}{Q^0} \quad (3)$$

where C_e (mgL^{-1}) and q_e (mgg^{-1}) are the concentrations of adsorbate and amount of adsorbate adsorbed at equilibrium, respectively. Q^0 (mgg^{-1}) and b (Lmg^{-1}) are the terms related to capacity and energy of adsorption, respectively, and are known as Langmuir's constants. The equilibrium data was plotted for ' C_e/q_e vs C_e .' For the Langmuir isotherm, a dimensionless separation factor can be expressed by the following equation (Weber and Chakkravorti 1974; Sharma et al. 2009),

$$R_L = \frac{1}{(1 + bC_0)} \quad (4)$$

where C_0 is the initial solute concentration (mgL^{-1}) and b is the Langmuir adsorption equilibrium constant (Lmg^{-1}). The dimensionless constant separation factor, R_L is used to test whether the adsorption is favorable or not. The value of R_L indicates the type of the

isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$). Langmuir capacities were calculated by using Equation 4.

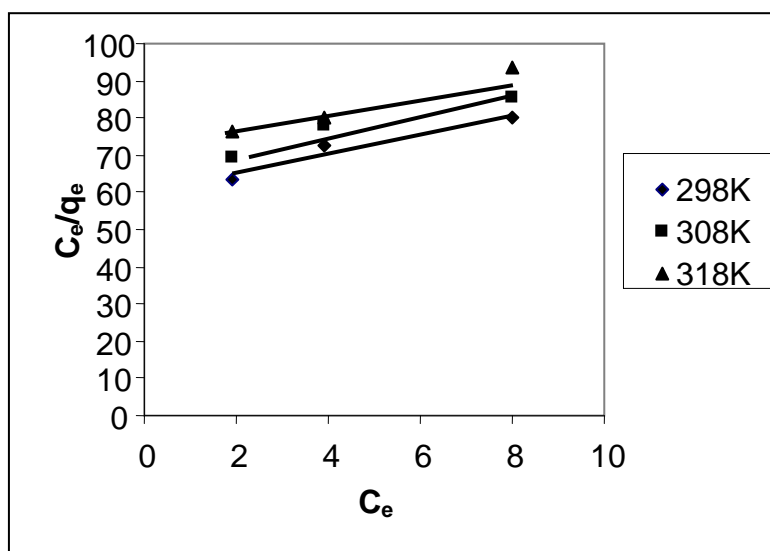


Fig. 6. (a) Langmuir isotherm for adsorption of Orange G onto raw sawdust at different temperatures (298, 308, and 318 K; $C_o = 2.5$ – 10 mg/l; sawdust dosage = 1g/l; initial pH, 2.0)

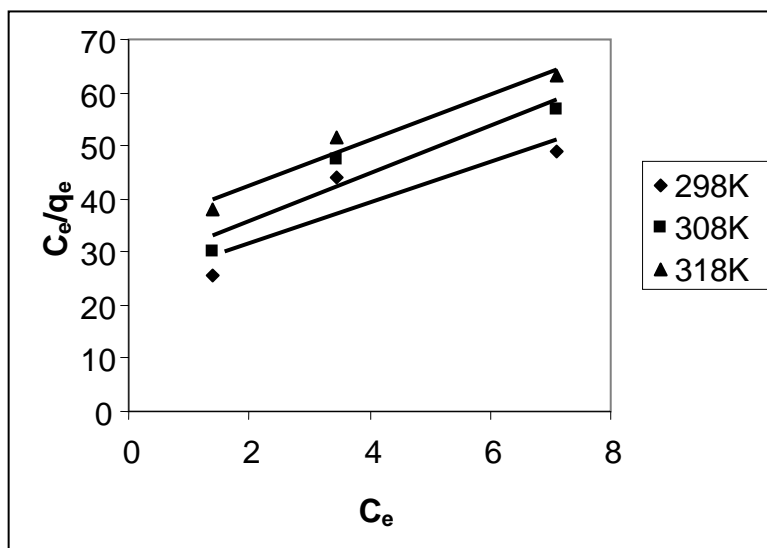


Fig. 6 (b) Langmuir isotherm for adsorption of Orange G onto modified sawdust at different temperatures (298, 308, and 318 K; $C_o = 2.5$ – 10 mg/l; sawdust dosage = 1g/l; initial pH, 2.0)

The values of Langmuir constants Q_o (mg/g) and b (L/mg) were calculated from intercepts and slopes of ' C_e/q_e vs. C_e ' (Figs. 6a and 6b). The linear plots of the figure confirm the validity of the above model for the studies undertaken (Wahab et al. 2005). The values of R_L , a dimensionless parameter, were obtained from Equation 2. The values of R_L were less than 1, indicating that adsorption of Orange G onto sawdust is favorable. The values of Langmuir's constants obtained from the plots are tabulated in Table 2.

Table 2. Values of Langmuir's Constants for Adsorption of Orange G onto Sawdust at Different Temperatures

Adsorbents	Temperature (K)	Q_o (mg/g)	b (l/mg)	K (L/g)	R_L	R^2
Modified sawdust	298	0.3846	0.0433	0.01578	0.697	0.9355
	308	0.4045	0.0375	0.01443	0.727	0.9402
	318	0.3500	0.0409	0.01313	0.707	0.9878
Raw sawdust	298	0.2636	0.1550	0.03928	0.392	0.7792
	308	0.2330	0.1750	0.03333	0.363	0.8797
	318	0.2476	0.1190	0.02662	0.456	0.9511

CONCLUSIONS

1. The results of this study indicate that sawdust, either raw or modified, can be successfully used for the removal of the dye Orange G from aqueous solutions.
2. The treatment of raw sawdust with sulphuric acid modified its composition, properties, and behavior. The modified sawdust resulted in higher uptake of dye as compared with the raw sawdust.
3. The FTIR study showed that the main functional groups present in the sawdust were phenol and alcohol.
4. The changes in the characteristics of the modified sawdust were verified by EDS and SEM.
5. Isotherm studies also confirmed that modified sawdust is a suitable adsorbent and can be used for treatment of effluents containing Orange G and similar dyes.

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