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Valorization of a natural residue (sawdust) as adsorbent to remove the acetic acid in aqueous solutions

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ABSTRACT

The objective of this work seeks a double environmental aspect. On the one hand, a valorization of a natural residue (sawdust) as adsorbent; and, on the other hand, the study of its ability to remove the acetic acid in aqueous phase. The raw sawdust was prepared by alkaline treatment and calcination at 250°C. The physicochemical characterization was carried out by thermogravimetry, Fourier transform infrared spectroscopy and analysis of the specific surface area by BET method. The effect of the initial concentration, contact time, stirring speed, adsorbent mass, solution temperature and ionic strength was studied in batch mode. The adsorption kinetics of acetic acid follows a pattern of pseudo-second-order with the contribution of the intraparticle diffusion. The results of the adsorption equilibrium were analyzed by three models (Langmuir, Freundlich and Temkin). The results are of interest and encourage us to test other potential chemical pollutants of water.

Keywords: Adsorption; Sawdust; Acetic acid; Kinetic; Equilibrium

1. Introduction

In many cases, industrial wastewater effluents contain organic pollutants which must be imperatively eliminated. Their removal has attracted much public and academic interest, owing to increased concern with their environmental impact.

One of the organic pollutants is acetic acid. This is produced in large quantities by many petrochemical processes while fine chemical industries are using acetic acid as a chemical reagent for the production of many chemical compounds (pharmaceuticals, dyes, pesticides, plastics, etc). Treatment of wastewater containing significant amount of acetic acid to meet future water quality standards has been recognized as a significant expense to the industry and the environment [1–3].

Several separation techniques which exhibit relatively high efficiencies include reduction precipitation, ion exchange, electrochemical reduction, evaporation, reverse osmosis and direct precipitation; however, they necessitate a large exposed liquid surface area and long detention periods [4–6]. In turn, some methods such as ion exchange and reverse osmosis are costly while others such as precipitation techniques have problems for disposal of metal-containing sludge. Alternatively, adsorption methods have been widely

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proposed, on the premises of a high efficiency and economical process. Activated carbon is the commonly adopted adsorbent for removing metals and dyes from wastewater, offering high efficiency at disproportionately high cost.

The use of low-cost adsorbents has been investigated as a replacement for costly current methods. Some agro waste materials studied for their capability to remove heavy metals, organics and dyes from aqueous solutions have been cited [7–9]. Sawdust is one of the most attractive materials used for removing pollutants from water/wastewater [10]. The material contains primarily lignin and cellulose. Interest in the use of sawdust as adsorbent has been stimulated by the good results that have been obtained in previous work [11,12]. For the increase of the adsorption capacities of these adsorbents, alkaline modification has been shown to be useful as pretreatment [13,14]. The authors that explained the sawdust was treated with a strong alkaline solution (NaOH) to remove pectin, lignin and hemicelluloses and liberate new adsorption sites on the sawdust surface. This type of treatment was effective for removing toxic heavy metal ions.

The aim of the present study is to investigate the feasibility of alternative sawdust as an adsorbent for the removal of acetic acid from wastewaters. Batch experiments are carried out for kinetic and equilibrium studies. The influence of various contributing parameters such as contact time, initial acetic acid concentration, adsorbent dosage, stirring speed, temperature and ionic strength is investigated for the sorption process. Three adsorption isotherm models (Langmuir, Freundlich and Temkin equation) are used to study the fit of the experimental equilibrium data obtained in the study, and several kinetic models are used to evaluate the adsorption mechanism.

2. Materials and methods

2.1. Chemical modification of sawdust

The sawdust was washed several times with distilled water to remove surface adhered particles and soluble materials. The oven dried sawdust was sieved, and the fraction with particle size between 0.3 and 0.5 mm was selected. To prepare the sawdust modified with NaOH, the sawdust fraction (9 g) was agitated in the presence of NaOH (5 M) for 24 h, then rinsed with deionized water and dried in oven (105˚C) overnight. The product obtained was treated in a muffle furnace at the temperature of 250˚C for 3 h.

2.2. Characterization of the adsorbent

Thermogravimetric analysis and differential thermal analysis (DTA) of the precursor decomposition were performed on a Perkin-Elmer TGA7 and Perkin-Elmer DTA7 devices, respectively, from 25 to 900˚C at a heating rate of 10˚C min^{-1} and under an air flow of ca. 60 mL min^{-1}.

Fourier transform infrared (FTIR) spectra were recorded in the 4,000–400 cm^{-1} range with a Perkin-Elmer 1,730 FTIR, using the KBr pellet technique (about 1 mg of sample and 300 mg of KBr were used for the preparation of the pellets).

The specific surface area of the samples was determined by applying the BET method to nitrogen adsorption isotherms recorded at −196˚C, using a Micromeritics apparatus model ASAP-2000. Prior to adsorption, the samples were degassed during 2 h at 180˚C.

2.3. Batch adsorption studies

Adsorption studies of acetic acid from aqueous solutions onto sawdust were performed using the batch equilibrium method at ambient temperature. A standard technique is used to determine the dye concentration using UV–vis spectrophotometer. Kinetic studies were carried out for an initial acetic acid concentration range of 60–120 mg L^{-1}, and the contact time was varied between 0 and 90 min. To observe the effect of adsorbent dose on dye adsorption, different amounts of adsorbent (varying from 0.04 to 1.6 g L^{-1}) are used with an initial dye concentration of 60 mg L^{-1}. To observe the effect of temperature on dye adsorption, experiments are carried out at four different temperatures (20, 30, 40 and 50˚C), where all other variables remain unchanged. A common adsorbent dose of 1 g L^{-1} and stirring speed of 800 rpm is used for all the above experiments. Different agitation speeds of 200–800 rpm are used for observing the effect of turbulence on the dye adsorption.

The amount of dye adsorbed per unit weight of adsorbent; q_t (mg g^{-1}) was calculated using the mass balance equation given by:

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

where C_0 and C_t (mg L^{-1}) are the liquid-phase concentrations of the dye initially and at any time (t), respectively.

The dye removal percentage can be calculated as follows:

$$R = \frac{C_0 - C_t}{C_0} \times 100 \qquad (2)$$

Equilibrium isotherms were determined by varying the initial dye concentration in the range of 30–900 mg L^{-1}. Dye solutions (20 mL) of a known initial concentration were agitated with 30 mg of the adsorbent on a shaker at 200 rpm for 5 h, followed by centrifugation.

3. Results and discussion

3.1. Characterisation of the adsorbent

Fig. 1 displays the TG curve performed over the raw sawdust. A relatively small weight loss (of ca. 5%) is observed between 25 and ca. 180°C. This mainly corresponds most likely to desorption of chemisorbed or hydration water that may remain in the raw sawdust. A second appreciable weight loss (64%) is produced between ca. 180 and 360°C. A final apparent weight loss (ca. 28.4%) is observed between ca. 360 and 520°C. It was reported from the literature [15,16] that the lignocellulosic biomass is thought to be stable up to 200°C, with minor mass losses associated with the removal of moisture and the hydrolysis of some extractives. About 95% of the weight loss occurs in the temperature range of 200–400°C.

The FTIR spectrums of raw sawdust and alkaline treated sawdust are shown in Fig. 2. The comparison between the two spectra reveals several changes. The intense bands about 1,628 cm^{-1} and 3,400 cm^{-1} characteristic of adsorbed H_2O molecules, disappear with the alkaline treatment [16]. A band at 1,730 cm^{-1} assigned to C=O in the carboxylic groups appears [17]. The band at 3,600–3,200 cm^{-1} indicates the possible presence of –OH on the surface. The bands that were observed at 660 and 605 cm^{-1} suggest the presence of C–H groups. This indicates that functional groups including hydroxyl and carboxylic groups can contribute to the adsorption of acetic acid.

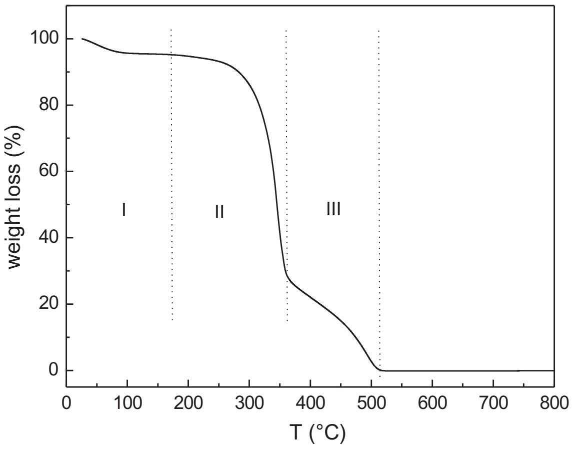

Fig. 1. Thermogravimetrical analysis of the raw sawdust.

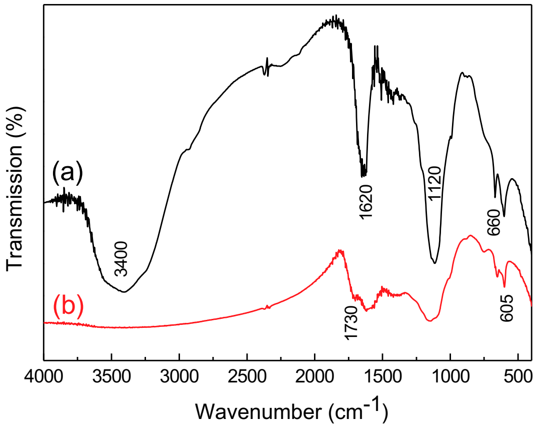

Fig. 2. FTIR spectrum (a) raw sawdust and (b) alkaline treated sawdust.

The results obtained for the specific surface area show a slight improvement after chemical treatment (64 $m^2 g^{-1}$ from raw sawdust to 70 $m^2 g^{-1}$ for the alkaline treated sawdust). The enhanced surface properties of sawdust on alkaline treatment can favour the increased adsorption of acetic acid in solution [18].

3.2. Adsorption studies

3.2.1. Effect of contact time and initial dye concentration

Equilibration time is an important parameter for economical wastewater treatment. The effects of contact

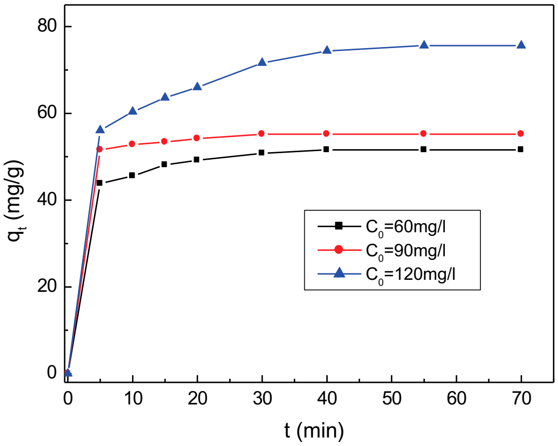

Fig. 3. Effect of contact time and initial concentration of acetic acid on the adsorption.

Table 1
Effect of stirring speed

Stirring speed (rpm)	Removal (%)
200	78.18
350	79.09
500	81.82
800	80.76

Table 2
Effect of temperature

Temperature (°C)	Removal (%)
20	79.09
30	78.18
40	80.02
50	78.46

time and initial dye concentration on removal are shown in Fig. 3.

Equilibrium adsorption was established within 30 min for 60 and 90 mg L^{-1}, and 50 min for 120 mg L^{-1} acetic acid, respectively. These results show that the contact time required for maximum uptake of the acetic acid by sawdust was strongly dependent on initial concentration. The results also show that the amount of adsorbed acetic acid increased with an increase in initial dye concentration [19,20].

3.2.2. Effect of adsorbent dosage

The effects of adsorbent dosage on acetic acid removal are presented in Fig. 4. The removal efficiency increased from 38% to 89% with an increase in the dosage from 0.01 to 0.1 g, and then remained almost constant. This was caused by the fact that, with increasing adsorbent dosage, more adsorption sites are available. This is a common observation whenever either the number of adsorption sites or the active surface area is increased [20–22]. However, increasing the sites had little effect on the removal efficiency at high adsorbent dosage because of the establishment of equilibrium at an extremely low adsorbate concentration in the solution before reaching saturation.

3.2.3. Effect of stirring speed

The effect of stirring speed on the removal of acetic acid on sawdust was investigated at different stirring speeds such as 200–800 rpm at 23°C as seen from Table 1. The difference of the adsorption rate was insignificant as the stirring speed increased; the dye removal does not exceed ca. 80% in any case [23,24].

3.2.4. Effect of temperature

The effect of temperature on the removal of acetic acid on sawdust was investigated at different temperature such as 20–50°C, as collected by Table 2. We observed that the temperature has practically no effect on the elimination rate of acetic acid within the examined temperature range.

3.2.5. Effect of ionic strength

The effect of salt concentration (ionic strength) on the removal percentage of acetic acid is analyzed along the KCl amount range from 1 to 40 mg. Sorption kinetics are carried out for acetic acid initial concentration of 60 mg L^{-1}, solution volume of 1 L and a sorbent mass of 1 g. The obtained results are shown in

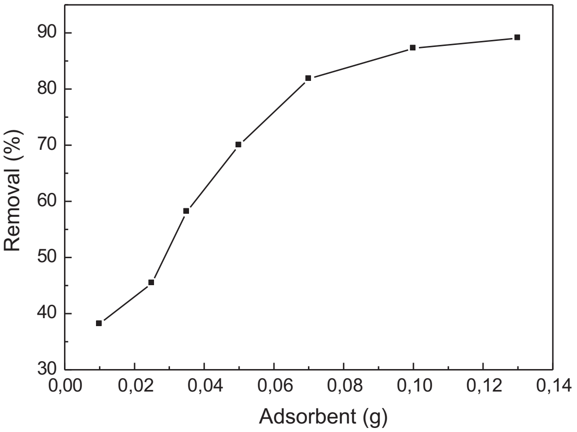

Fig. 4. Effect of adsorbent dosage on the adsorption of acetic acid onto sawdust.

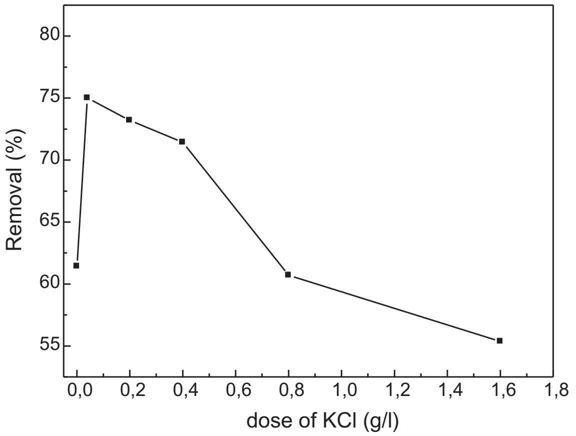

Fig. 5. Effect of KCl concentration on the removal capability.

Fig. 5. The addition of KCl shows an optimum adsorption level of 70% for an amount of KCl 0.1 $\mathrm{g\,L^{-1}}$, and a remarkable decrease in the adsorbed amount at high KCl concentration. Such a behaviour is anticipated due to an interaction between the surface and added solutes which may block some of the adsorption active sites for the acetic molecules [23].

3.3. Kinetic studies

In any adsorption process, determination of the rate of adsorption of an adsorbate as well as the rate-determining step is crucial, especially for designing the adsorption system of a full scale batch process. Adsorption kinetics is controlled by different kinds of mechanisms, such as film diffusion, pore diffusion or chemical reaction (complexation or ion exchange) [25]. Three kinds of kinetic models were used to test the experimental data [26]. These are Lagergren-first-order equation (Eq. (3)), second-order equation (Eq. (4)) and intraparticle diffusion model (Eq. (5)).

$$\ln\,(q_e - q_t) = \ln q_e - k_1 t \tag{3}$$

$$1/q_t = 1/k_2 q_e^2 + t/q_e \tag{4}$$

$$q_t = k_{id} t^{0.5} + I \tag{5}$$

where q_t ($\mathrm{mg\,L^{-1}}$) is the amount of adsorption at time t (min), q_e is the amount of adsorption at equilibrium ($\mathrm{mg\,g^{-1}}$), k_1 is the rate constant of the first-order equation ($\mathrm{min^{-1}}$), k_2 ($\mathrm{g\,mg^{-1}\,min^{-1}}$) is the rate constant of the second-order equation and k_{id} ($\mathrm{mg\,g^{-1}\,min^{-1}}$) is the rate constant of intraparticle diffusion. Values of I give information about the thickness of the boundary layer, i.e. the larger the intercept the greater is the boundary layer effect.

Table 3 lists the results of the kinetic parameters calculated using the pseudo-first-order and pseudo-second-order models. The curve fitting plots of ln (q_e–q_t) vs. t do not show good results for the sorption (figure not shown) because the coefficient of determination for this model is low (R^2 = 0.78–0.94).

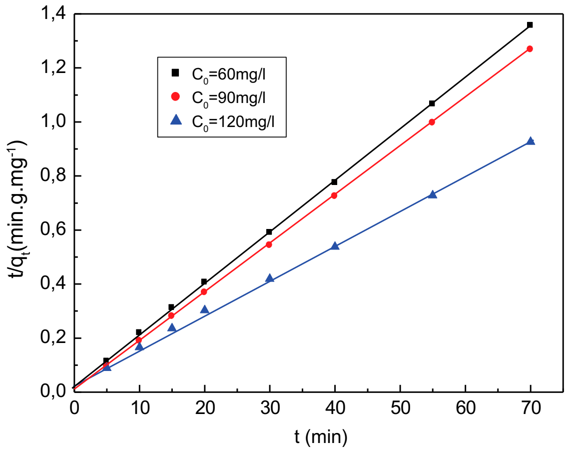

Fig. 6. Pseudo-second-order kinetic plots for the adsorption of acetic acid on sawdust.

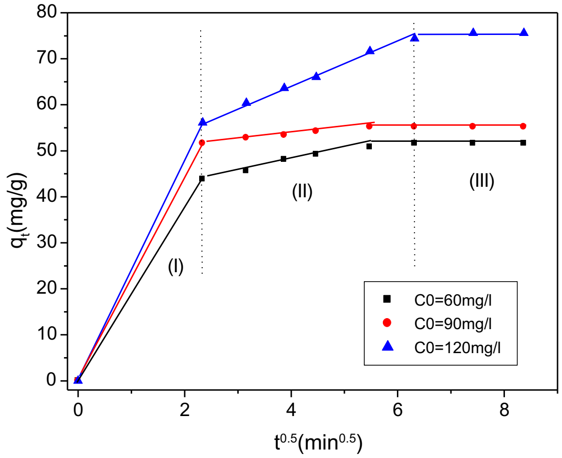

Fig. 7. Intraparticle plots for the adsorption of acetic acid on sawdust.

Table 3
Kinetic parameters for adsorption of acetic acid on sawdust

		Pseudo-first-order			Pseudo-second-order			Intraparticle diffusion		
C_0 (mg/l)	$q_{e,exp}$ (mg/g)	k_1 (min^{-1})	$q_{e.cal}$ (mg/g)	R^2	k_2 (g.mg^{-1}.min^{-1})	$q_{e.cal}$ (mg/g)	R^2	k_{id} (mg./g.min^{-1})	I (mg/g)	R^2
60	51.6	0.123	25.77	0.902	0.015	52.71	0.999	2.302	38.60	0.977
90	55.2	0.174	22.07	0.782	0.039	55.65	0.999	1.132	49.07	0.993
120	75.6	0.089	47.66	0.944	0.004	79.18	0.999	4.646	45.49	0.996

The plots of t/q_t vs. t give a straight line as seen from Fig. 6. The correlation coefficients of pseudo-second-order were close to unity and calculated q_e values computed from pseudo-second-order equation showed good agreement with experimental values [17,19]. This supports the assumption of the model that the adsorption is due to chemisorption [27].

To identify the importance of diffusion in the adsorption process, the parameters calculated using the plots of q_t vs. $t^{0.5}$ are presented in Table 3. Fig. 7 shows the involvement of two steps in the adsorption process, the first one representing adsorption of dyes on the surface of adsorbent and the second one describing the diffusion of dyes to the adsorption site. A surface adsorption mechanism was dominant in the first 5 min of contact time thereafter diffusion became a rate-limiting process. The values of $I \neq 0$, indicates that the intraparticle diffusion was involved in the adsorption process, but was not the only rate controlling step [28].

3.4. Equilibrium isotherms

The adsorption isotherm of acetic acid onto sawdust is presented in Fig. 8. The isotherm showed the shape of type "L" according to the classification of Giles et al. [29]. The L or Langmuir shape of the isotherms means that there is no strong competition between the solvent and the adsorbate to occupy the adsorbent sites. In this case, the longitudinal axes of the adsorbed molecules are parallel to the adsorbent surface [29]. For the determination of equilibrium isotherms Langmuir [Eq. (6)], Freundlich [Eq. (7)] and Temkin [Eq. (8)], corresponding models were applied [30].

$$\frac{q_e}{q_m} = \frac{K_L \cdot C_e}{(1 + K_L \cdot C_e)} \quad \text{or} \quad \frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{K_L \cdot q_m} \tag{6}$$

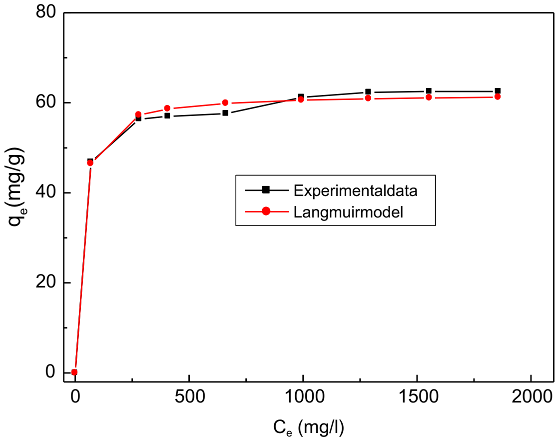

Fig. 8. Equilibrium isotherm of acetic acid onto sawdust.

$$q_e = K_F C_e^{1/n} \text{ or } \ln q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{7}$$

$$q_e = \frac{RT}{b_T} \ln (K_T \cdot C_e) \text{ or } q_e = B_T \ln K_T + B_T \ln C_e \tag{8}$$

It is very difficult to select the best-fit adsorption isotherm. Therefore, the correlation coefficient, R^2 and q_m were employed to find out the best-fit isotherm model for the experimental equilibrium data. The values of the correlation coefficient R^2 and q_m are presented in Table 4. By comparing these results it was found that the Langmuir isotherm fitted well to the observed data for acetic acid adsorption onto sawdust. Similar results were obtained during the adsorption of acetic acid on other different adsorbents [1,31,32].

Table 4
Correlation isotherm parameters and correlation coefficients for the adsorption of acetic acid onto sawdust

Isotherm models	Correlation parameter		Correlation coefficient R^2
	Name	Value	
Langmuir isotherm	q_m (mg g^{-1})	62.00	0.995
	K_L (L mg^{-1})	0.043	
Freundlich isotherm	K_F (L mg^{-1})	33.32	0.948
	$1/n_F$	0.086	
Temkin isotherm	K_T (L g^{-1})	345.27	0.960
	B_T (mg/l)	4.754	
	b_T	512.41	

4. Conclusion

The present study shows that sawdust is an effective adsorbent for the removal of acetic acid from an aqueous solution. The adsorption kinetics could be explained adequately by pseudo-second-order kinetics, suggesting that the rate-limiting step may be chemical adsorption. The multi-linearity curve indicates that intraparticle diffusion is not a fully operative mechanism in the sorption of acetic acid on sawdust. The equilibrium is described best by Langmuir model; maximum sorption capacity at a temperature of 20°C for sawdust was $62.0\,\mathrm{mg\,g^{-1}}$ for acetic acid. The removal efficiency increased with increasing adsorbent concentrations. We conclude that sawdust has considerable potential as an adsorbent for acetic acid from an aqueous solution.

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