ORIGINAL ARTICLE

Sorption of methylene blue on treated agricultural adsorbents: equilibrium and kinetic studies

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Abstract Agricultural adsorbents are reported to have a remarkable performance for adsorption of dyes. In the present study, formaldehyde and sulphuric acid treated two agricultural adsorbents; potato peel and neem bark are used to adsorb methylene blue. On the whole, the acid-treated adsorbents are investigated to have high sorption efficiency compared to HCHO treated adsorbents. The percentage removal efficiency of H₂SO₄ treated potato peel (APP) increases considerably high from 75 to 100 % with increase in adsorbent dose, whereas the removal efficiency of H₂SO₄ treated neem bark (ANB) is found to be 98 % after adding the first dose only. The monolayer sorption behaviour of HCHO treated potato peel (PP) and APP is well defined by Langmuir, whereas the chemisorptions behaviour of HCHO treated neem bark (NB) and ANB is suggested by Temkin's isotherm model. The maximum adsorption capacity measured is highest in ANB followed by NB, PP and APP with the values of 1000, 90, 47.62 and 40.0 mg/g, respectively. The pseudo-second-order kinetic model fitted well with the observed data of all the four adsorbents. The results obtained reveal that NB and ANB both are good adsorbents compared to PP and APP.

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Bhagwan Mahavir Institute of Engineering and Technology, Sonepat 131001, Haryana, India e-mail: sharma.neetu80@gmail.com $\begin{tabular}{ll} \textbf{Keywords} & HCHO & treated potato & peel & (PP) \cdot HCHO \\ treated & neem & bark & (NB) \cdot H_2SO_4 & treated & potato & peel & (APP) \cdot H_2SO_4 & treated & neem & bark & (ANB) \cdot Methylene & blue & (MB) \cdot Percentage & removal \\ \end{tabular}$

Introduction

The demands of synthetic dyes have experienced significant growth in the past decades. Recent reports highlighted that more than 100,000 of various dyes are commercially available and annual worldwide production of dyestuffs surpassed to 7×10^5 metric tons (Pearce et al. 2003; Gong et al. 2005; Robinson et al. 2001). Extensive use of dyes and inefficiency in dying process leads to great loss of dyes to the main stream. The effluent from dying industry comprises moderate concentration (10–200 mg ml⁻¹) of dyestuffs which contribute significant contamination of aquatic ecosystem (Neill et al. 1999). These stable compounds do not dissociate in environment and get accumulate in biota which further leave severe toxic and carcinogenic effects (Hu et al. 2006). Numerous physical, chemical and biological techniques are applied in past to attain maximum colour removal. Among them, adsorption technique is well accepted and considered to be an effective method for decreasing concentration of dissolved dyes in waste water (Reife et al. 1996). The adsorption process is fulfilled with little or no toxic pollutants and requires low initial capital and operating costs (Crini 2006).

Agricultural by-products have received significant interest in recent past by the researchers due to their abundance, non-hazardous nature and simple process of dye removal. Plentiful agricultural waste is used in recent past such as neem leaf powder (Bhattacharyya and Sharma 2004), tamarind fruit shell (Somasekhara Reddy 2006),



jack fruit peel (Jayarajan et al. 2011), grapefruit peel (Abassi and Razzaghi 2009), rice husk (Abdelwahab et al. 2005), bark (McKay et al. 1999; Patil et al. 2011), sunflower seed husk (Ong et al. 2010) wheat straw (Hassanein Taha and Koumanova 2010) and saw dust (Shukla et al. 2002). These wastes have negligible economic value and often pose a disposal problem, so trying them as suitable adsorbent is the best alternative to utilize them.

In the present study, the removal efficiency of chemically treated potato peel and neem bark is observed on methylene blue (MB). The adsorption of MB by raw potato peel and neem bark has already been reported earlier as well by Öktem et al. 2012 and Srivastva and Rupainwar 2010, respectively. The novelty of the present research is to examine the efficiency of these adsorbents by modifying them with chemicals. One part of both the adsorbents is treated with HCHO, and other part is treated with H₂SO₄. The purpose of pre-treatment of raw adsorbent with formaldehyde is to remove the colour of raw adsorbents, which can interfere with the dye studied in order to achieve maximum colour removal with accuracy, whereas the acid treatment of both the adsorbents is done due to significant performance recorded by acid-treated adsorbents in past (Garg et al. 2003; Raghuvanshi et al. 2004; Parvathi et al. 2010). Adsorption isotherm and chemical kinetics are considered to find out the nature and mode of adsorption process. The present research will help in selecting the proficiently and economically suitable adsorbent for treatment of textile industry waste.

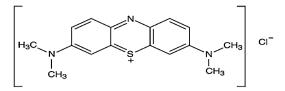
Materials and methods

Selection and preparation of sorbents

Two adsorbents potato peel and neem bark are selected as suitable adsorbents. Potato peel is obtained from mess of BMIET, while neem bark is collected from surrounding area of the same institute. Both the adsorbents are first washed with water and air dried in oven at 65 °C for 24 h. After drying, they are grinded and further treated with formaldehyde to restrict the integration of adsorbent's own colour with the dye tint.

Formaldehyde treatment of raw adsorbents

For removing the colour of raw adsorbents, the grinded potato peel is dipped in 6 % HCHO overnight and washed further with distil water until the decanted water became colourless and pH reached at neutral. The colour-abated potato peel is then dried in oven at 70 °C and further sieved to obtain the particles of 80–150 µm mesh size. For removing the colour of neem bark, the grinded mixture is



 $C_{16}H_{18}CIN_3S$ MW = 319.86

Fig. 1 Structure of methylene blue

dipped in a solution of 37 % HCHO and 0.2 N H₂SO₄ overnight followed by soaking in NaHCO₃ overnight to remove acid (Garg et al. 2003). After that, the same procedure of washing and drying is followed like potato peel. Finally the light brown coloured HCHO treated potato peel (PP) and dark brown coloured HCHO treated neem bark (NB) powders are obtained.

Acid treatment of the adsorbents

Both the adsorbents are treated with $\rm H_2SO_4$ in 1:1 ratio and left overnight followed by washing with distil water many times until the pH reached at neutral. The black coloured powders of both the adsorbents are obtained after drying in oven at 105 °C and sieving through the mesh size of 80–150 μ m mesh size.

The morphological and structural properties of all the four adsorbents are examined with the help of Hitachi, model S-3700, Scanning Electron Micrograph and Thermo Scientific Nicolet-380, FTIR.

Sorbet

The dye, MB, chemical formula = $C_{16}H_{18}ClN_3S$, MW = 319.86, λ_{max} = 662 is selected as sorbet. The structure of dye is given below in Fig. 1.

Analytical procedure

Experiments are conducted by preparing 1,000 ppm of stock solution of MB. For determining the concentration of dye, Lambert–Beer's method is used. Analysis is done on double-beam Chemito UV–Visible Spectrophotometer having the wavelength range of 190–1,100 nm and bandwidth of 1.6/1.0 nm. All the samples are examined on the recorded wavelength of 662 nm. Standard concentrations of MB (1–10 ppm) are prepared to obtain the calibration curve, and graph factor is calculated to get accurate results with minimum error. A comparative study of removal of dye on all the four adsorbents is studied by considering various parameters like adsorbent dose, concentration of dye, pH of the solution and time. The experiments are performed in batches at room temperature by shaking



50 ml of 100 ppm (except the concentration effects) dye solution with 0.250 g (except the dose effects) of each adsorbent taken in 100 ml of Erlenmeyer conical flask at the speed of 125 rpm.

Sorption of dye on various adsorbents is calculated by the mass balance equation given below where q denotes the amount of dye adsorbed on the adsorbent (mg/g); V denotes the volume of the solution (1); m denotes the amount of adsorbent taken in g; C_i and C_f denotes the dye concentration in mg/l before and after adsorption, respectively.

$$q = \frac{C_{\rm i} - C_{\rm f}}{m} \times V \tag{1}$$

whereas percentage removal of dye is calculated by the given formula:

$$\% \text{ removal } = \frac{C_{i} - C_{f}}{C_{i}} \times 100$$
 (2)

Experimental validation

Three different isotherm models, Langmuir, Freundlich and Temkin, are fitted to analyse the isotherm data. The initial concentrations for analysing the suitable isotherm model followed by all the four adsorbents are selected as 20, 50, 100, 150 and 250 ppm.

Langmuir's and Freundlich's equations are commonly used to express the isotherms at constant temperature for waste water treatment operations (Eren and Acar 2006). The linear form of Langmuir's model supports the monolayer sorption hypothesis which occurs on the outer surface having infinite number of active sites. The empirical equation of Freundlich isotherm model is based on the sorption of dye over heterogeneous surface. Temkin's isotherm model usually applied to observe the chemisorptions behaviour of sorbent and sorbet. The linear form of Langmuir's, and empirical equation of Freundlich and Temkin's isotherm models are, respectively, explained by the following expressions:

$$\frac{C_{\rm e}}{q_{\rm e}} = \frac{1}{Q_0 b} + \frac{C_{\rm e}}{Q_0} \tag{3}$$

$$\log q_{\rm e} = \log K_{\rm F} + \frac{1}{n} \log C_e \tag{4}$$

$$q_{\rm e} = B \ln A + B \ln C_{\rm e} \tag{5}$$

where in Langmuir's equation, C_e is the equilibrium concentration (mg/l); q_e is the amount of dye adsorbed (mg/g); Q_0 is the maximum dye per gram of adsorbent (mg/g); and b is Langmuir's constant related to energy of adsorption (l/mg). In Freundlich's equation, K_F is the constant related to sorption capacity. 1/n is an empirical parameter related to the sorption intensity of the adsorbent. A is Temkin's constant representing sorbet—sorbent interactions, and B is another constant related to the heat of adsorption.

One more essential feature of Langmuir's isotherm is determined which is a dimensionless constant separation factor (RL), expressed by the following equation given by Hall et al. (1966)

$$RL = 1/(1 + bC_0) \tag{6}$$

where C_0 is initial concentration of dye (mg/l), and b is Langmuir's constant (l/mg). The value of RL defines the nature of adsorption as below

RL > 1 = unfavourable, RL = 1 linear, RL = 0 irreversible, 0 < RL > 1 = favourable

The experimental data are analysed to observe the appropriate kinetic model followed by adsorption process including mass transfer and chemical reactions. Pseudo-first-order and pseudo-second-order kinetic models are used to describe the kinetics of adsorption. The integrated form of Lagergren first-order kinetic model is represented by the equation given below:

$$\log(q_{\rm e} - q_{\rm t}) = \log q_{\rm e} - (k_1/2.303)t \tag{7}$$

where k_1 is the rate constant of first-order adsorption (1/min); q_e is the amount of dye adsorbed in mg/g at equilibrium; and q_t is the amount of dye adsorbed in mg/g at any time t. The linear form of pseudo-second-order kinetic model rate equation is expressed as:

$$t/q_{\rm t} = 1/K_2 q_{\rm e}^2 + (1/q_{\rm e})t \tag{8}$$

where k_2 is the rate constant of second-order adsorption (1/min) depends on the sorption capacity of solid phase. The initial sorption rate, h (mg/g min), is also calculated with the help of second-order kinetic model by the following expression:

$$h = k_2 q_e^2 \tag{9}$$

Results and discussion

Morphological and structural study of adsorbents

All the four adsorbents obtained are analysed on SEM and FTIR to know about their morphological and structural properties. The SEM images of all the four adsorbents given in Fig. 2 show significant particle and pore size for the dye to be adsorb. From the images, it is evident that PP reflects the presence of smooth particles of size ranges up to 22–30 μ m while NB shows enough rough shape particles of average size from 8.38 to 10.6 μ m. The SEM images confirmed that both the adsorbents have sufficient pore size and surface area for the sorption of dye.

The comparative chart of FTIR spectra of all the four adsorbents represents clearly that almost all the adsorbents show more or less similar characteristics adsorption spectra. From the chart, it is well depicted that % transmittance



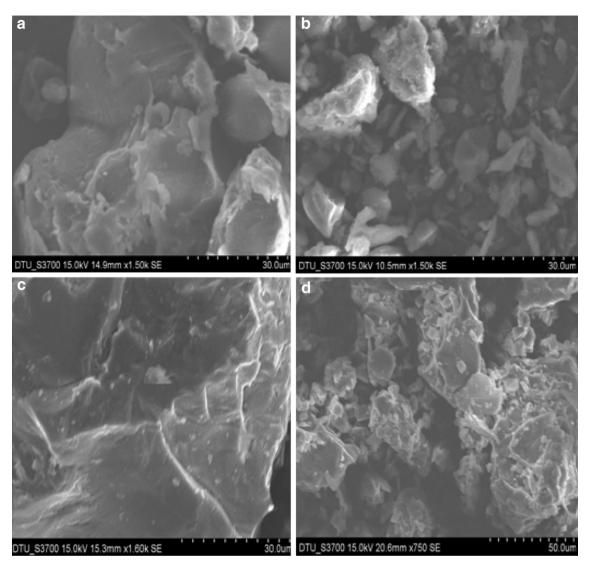


Fig. 2 SEM images of a PP, b NB, c APP, d ANB

Table 1 Different functional group recognized on all the four adsorbents through FTIR spectra

Group	Wavelength range (cm ⁻¹)	PP	APP	NB	ANB	MB-loaded PP
O-H stretch	3,400–3,300 (broad)	3,403	3,429	3,413	3,402	3,441
C-H stretch	2,950-2,800	2,920	_	2,922	_	2,920
C=C stretch	1,690-1,630	1,648				1,652
Carboxylic group	1,500–1,640	1,517	1,629	1,618	1,616	1,517
Esters (C-O-C)	1,260–1,170	1,244, 1,158	1,170		1,206	1,159
Amines (C–N stretch)	1,200–1,025	1,024				1,024
Sulphoxides (S=O)	~1,050		1,035	1,035		

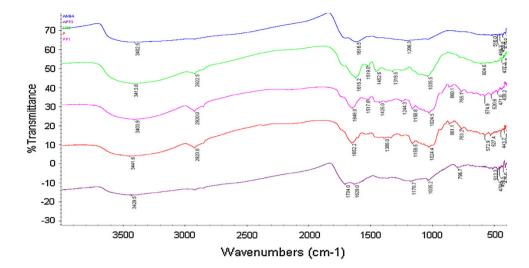
is relatively greater in case of NB and $\rm H_2SO_4$ treated neem bark (ANB) compared to PP and $\rm H_2SO_4$ treated potato peel (APP). Table 1 elucidates various functional groups present on the surface of all the four adsorbents studied. These functional groups play major role in attracting the dye molecule from aqueous solution. It is well defined from the table that both potato peel and neem bark contain carboxylic group (hemicelluloses, pectin and lignin), phenolic (lignin, lipids and waxes), amines and sulphoxides group (Fig. 3).

Effect of dose

The effect of dose is studied by varying the amount of dose from 0.1 to 1.0 g of each adsorbent in 50 ml of 100 ppm of



Fig. 3 Comparative chart showing FTIR spectra (starting from below—a APP, b methylene blue adsorbed on PP, c PP, d NB, e ANB)



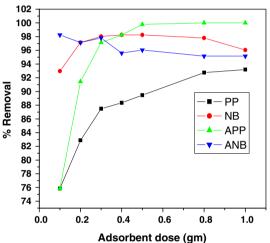


Fig. 4 Effect of adsorbent dosage in removal of MB

MB dye solution. Shaking is done at 125 rpm for 30 min at room temperature. It is clear from Fig. 4 that with increase in dose, the % removal of PP, NB and APP also increases from 75 to 93, 92 to 96 and 75 to 100 %, respectively. Maximum removal is achieved by APP. It may be due to presence of more active sites on the surface. Initial increase in adsorption is due to availability of more surface area on the adsorbents (Namasivayam et al. 1996; Garg et al. 2003), but further increase in dose saturates the adsorbents and steady state is observed. It is as a result of aggregation of adsorption sites by too much adsorbent which reduces the adsorption efficiency (Araujo and Teixeira 1997). The % removal of ANB decreases with increase in dose due to maximum absorbance (98 %) achieved with in short span of time, and further addition of adsorbent results in overcrowding which in turn decreases the efficiency. Unit adsorption of dye is decreased in all cases after increasing dose. This can be due to overlapping of adsorption sites as

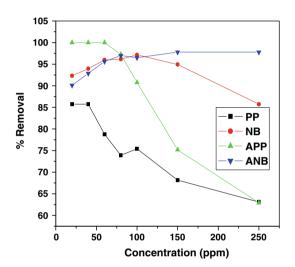


Fig. 5 Effect of initial dye concentration in removal of MB

a result of overcrowding of adsorbent particles (Namasi-vayam et al. 1998).

Effect of concentration

Effect of dye concentration is studied by keeping adsorbent dose constant at 0.250 g in 50 ml of different concentration of dye varying from 20, 50, 60, 80, 100, 150 and 250. Shaking is done for 30 min at room temperature. Decolourization is observed in first 15 min at lower concentrations. The % removal of dye in case of PP, APP and NB decreases significantly from 85 to 63, 100 to 62 and 92 to 85 %, whereas in ANB, the % removal increases from 90 to 97 % as depicted by the Fig. 5. The decrease in removal can be attributed to saturation of sorption sites on the adsorbent as the concentration of dye increases (Tan et al. 2010), while initial increase in removal up to 100 mg/l and further saturation of ANB may be due to sufficient sorbent accessibility to adhere with the sorbet.



Effect of pH

Adsorption of dye on all the four adsorbents showed significant removal with change in pH. The optimum pH for removal of MB by PP, NB, APP and ANB is observed to be 8, 4, 12 and 2, respectively, as shown in Fig. 6. HCHO treated potato peel and APP showed significant variation in percent removal from 63 to 80 and 76 to 94 %, respectively, on changing pH from 2 to 14, while NB and ANB showed less variation in per cent removal from 93 to 83 and 98 to 90 %, respectively, by varying the same pH. HCHO treated neem bark and ANB showed good removal in acidic condition, while PP and APP showed significant removal in alkaline condition. Increase in negative charged ions on PP and APP favoured the adsorption of cationic dye in alkaline condition due to pectin present on surface of the two adsorbents (Flavio et al. 2008). Maximum removal of MB on NB and ANB in acidic medium may be due to the cellulosic compounds (carboxylic and phenolic groups) present on the adsorbent surface (Ferrero 2007).

Adsorption isotherm

The various isotherm (Langmuir, Freundlich and Temkin) parameters specified in Table 2 confirmed different type of

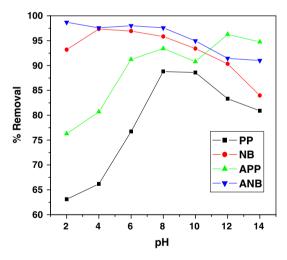


Fig. 6 Effect of pH in removal of MB

sorption processes followed by the four adsorbents studied. From Table 2, it is clear that PP and APP responded good behaviour with all three types of isotherm models but Langmuir's isotherm fitted quite well with them which is also well explained by the good regression coefficient of 0.99 and 0.96, respectively. The Langmuir's model described the monolayer sorption nature of PP and APP. The maximum adsorption capacity of PP and APP obtained is 47.62 and 41.60 mg/g, respectively, which are higher than 33.55 mg/g measured by Öktem et al. 2012 on raw potato peel.

It is well defined from Table 2 that Temkin's isotherm is best fitted with the adsorption behaviour of NB and ANB, showing good regression coefficient of 0.85 and 0.99, respectively, compared to other two isotherms studied. Temkin's isotherm model reflects chemical adsorption mechanism between the dye and sorbent which takes into account the acquisition of the more energetic adsorption sites at first (Abdel-Ghani et al. 2007). The maximum adsorption capacity of neem bark powder for MB estimated by Srivastva and Rupainwar 2010 is 3.11×10^3 mol g⁻¹(994 mg/g) which is higher than the NB efficiency of 90 mg/g but lower than the ANB efficiency of 1,000 mg/g measured.

In present study, the value of RL lies between 0.11 and 0.625 for PP and 0.03 and 0.31 for APP which are in the range of 0–1, indicating that the adsorption process is favourable for both the adsorbents. The value of n estimated is >1 for all the four adsorbents as depicted in Table 2. This represents that adsorption is favourable in nature.

Chemical kinetics

The kinetic plots of Eq. (8) and (9) are made at the initial concentration of 100 ppm for all the four adsorbents. From Table 3, it is concluded that the second-order kinetic model fitted well with the observed data of all the four adsorbents. Similar results are reported for sorption of MB on potato peel (Öktem et al. 2012) and neem bark (Srivastva and Rupainwar 2010). The initial adsorption rate h is higher in case of both NB and ANB as compared to PP and APP. The calculated values of q_e for all the four adsorbents from

Table 2 Comparison of adsorption isotherm of different adsorbents studied

Isotherm models→	Langmuir's isotherm model			Freundli	Freundlich's isotherm model			Temkin's isotherm model		
Adsorbent ↓	$K_{\rm L}$ (b)	Q_0	R^2	$\overline{K_{ m F}}$	N	R^2	\overline{B}	A	R^2	
PP	0.03	47.62	0.99	1.87	1.46	0.95	8.89	0.39	0.98	
NB	0.03	90.91	0.21	3.63	1.35	0.56	0.06	43,477	0.85	
APP	0.107	41.60	0.96	7.46	2.80	0.64	0.116	0.056	0.86	
ANB	0.006	1,000.0	0.005	6.51	1.10	0.84	19.60	1.35	0.99	



Table 3 Kinetics of pseudo-first and pseudo-second order of different adsorbents studied

Adsorbents	Pseudo-first order			Pseudo-second order				q _e (exp)
	$q_{\rm e}$ (cal)	K_1	R^2	$q_{\rm e}$ (cal)	K_2	R^2	Н	
PP	1.5	0.009	0.14	17.5	0.29	0.99	2.7	17.6
NB	0.71	0.016	0.29	19.2	0.016	0.99	22.1	19.2
APP	5.04	0.034	0.70	20.0	0.015	0.99	0.4	20.0
ANB	0.52	0.006	0.10	19.6	2.60	0.99	42.2	19.6

second-order kinetic equation are found to be agreed well with the experimental values of $q_{\rm e}$.

Conclusion

The efficiency of four adsorbents PP, APP, NB and ANB for removing MB is evaluated by varying dose, pH and concentration. On varying dose, the % removal of PP, NB and APP also increases from 75 to 93, 92 to 96 and 75 to 100 %, respectively, due to availability of more surface area on the adsorbents, while the % removal of ANB decreases with increase in dose due to overcrowding of the adsorbent which in turn decreases the efficiency. On changing concentration, the % removal of dye in case of PP and APP decreases significantly from 85 to 63 and 100 to 62 % while little decrease is observed in case of NB and ANB from 92 to 85 and 90 to 97 %, respectively. The decrease in removal may be attributed due to saturation of sorption sites on the adsorbent as the concentration of dye increases (Tan et al. 2010). The optimum pH for removal of MB on PP, NB, APP and ANB are observed to be 8, 4, 12 and 2, respectively.

The maximum dye adsorption capacity, Q_0 observed for NB and ANB, is 90.91 and 1,000 mg/g, respectively, which is >76.9 mg/g for the removal of MB on hazelnut shell as reported by Ferrero 2007. The Q_0 estimated for NB in present study is lower than the value 3.11×10^3 mol/g estimated by Srivastva and Rupainwar 2010, but the later reported is less than the ANB sorption capacity of present study. The Q_0 observed for PP and APP is 47.62 and 41.6 mg/g, respectively, which are higher than the efficiency measured for MB on cherry saw dust, Oak saw dust and pitch pine saw dust reported by Ferrero 2007.

The physical adsorption behaviour of PP and APP is well supported by Langmuir's adsorption isotherm. HCHO treated neem bark and ANB followed chemisorptions nature which is acknowledged by the good correlation of Temkin's isotherm. SEM and comparative chart of FTIR spectra of all the four adsorbents are in favour of the results obtained. The second-order kinetic model fitted well with the observed data of all the four adsorbents.

From the present research, it is estimated that NB and ANB are better adsorbent than PP and APP but neem bark performed efficiently in acid treated nature whereas potato peel worked better in HCHO treated form.

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