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A COMPARISON STUDY OF REMOVAL OF METHYLENE BLUE DYE BY ADSORPTION ON NEEM LEAF POWDER (NLP) AND ACTIVATED NLP

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Abstract. The adsorption of basic dye, Methylene Blue Dye (MBD) onto neem (*Azadirachta indica*) leaf powder (NLP) and activated NLP with sulphuric acid was compared in this investigation. The surface analyses of NLP and activated NLP (particle size distribution, porosity, pore volume, pore diameter and BET surface area) were conducted using sophisticated instrument. The process parameters like adsorbent dose, pH, temperature and contact duration have been studied. The experimental data were analyzed using Langmuir and Freundlich isotherm, in which adsorption capacities and intensities were evaluated. Three kinetic models, pseudo first-, second-order and Elovic equations were applied to describe the adsorption process, in which correlation coefficient for each kinetic equation were calculated and evaluated.

Keywords: Adsorption, methylene blue dye, neem leaf powder, isotherm models, kinetic equation.

Introduction

Synthetic dyes have a wide application in the food, pharmaceutical, textile, leather, cosmetics and paper industries due to their ease of production, fastness, and variety in color compared to natural dyes (Adedayo *et al*. 2004). Dyes usually have a synthetic origin and complex aromatic molecular structures which make them more stable and more difficult to biodegrade. Degradation of dyes is typically a slow process (Carlos *et al*. 2009). The removal of color is needed to be considered in the disposal of textile wastewater due to aesthetic deterioration as well as the obstruction of penetration of dissolved oxygen and sunlight into water bodies, which seriously affects aquatic life. Besides, the dye precursors and degradation products are proven carcinogenic and mutagenic in nature (Somasiri *et al.* 2006). Consumption of dye-polluted water can cause allergy reactions, dermatitis, skin irritation, cancer and mutation both in babies and matures. (Kalyuzhnyi, Sklyar 2000).

Many techniques have been used to remove harmful dyes from colored wastewater, including chemical coagulation/ flocculation, precipitation, ozonation, adsorption, oxidation, ion exchange and photo degradation. Some of these techniques have shown to be effective, although they have limitations. Among these are: excess amount of chemical usage, or accumulation of concentrated sludge with disposal problems; expensive plant requirements and operational costs; lack of effective color reduction; and sensitivity to a variable wastewater input (Jaikumar, Ramamurthi 2008). One of the powerful and convenient treatment processes is adsorption. Some natural adsorbents such as fly ash (Lin *et al*. 2008), zeolite (Alpat *et al.* 2008; Mazeikiene *et al*. 2010), gulmohar (*Delonix regia*) plant leaf powder (Ponnusami *et al*. 2009), orange peel (Khaled *et al*. 2009), walnut shell (Nazari-Moghaddam *et al*. 2010) etc. are exploited for removal of various types of dyes. Raising Neem Juss seedling (Singh *et al.* 2009) and Neem leaf powder (Bhattacharyya, Sharma 2005) was also utilized for treatment of textile industrial effluent. Some acids were also utilized for activation of adsorbents (Rozic *et al*. 2008, Bhattacharyya, Gupta 2008).

This investigation is indented to prepare NLP and activated NLP using sulphuric acid and compare for the adsorption of MBD. The process parameters like adsorption dose, pH, temperature and contact duration were explored. The data were analyzed using adsorption isotherms (Freundlich and Langmuir isotherm) and kinetic models (pseudo first-, second-order and Elovic equation) models.

1. Material and methods

1.1. Adsorbent

The neem (scientific name: *Azadirachta indica*) belongs to the meliaceae family and is native to Indian subcontinent. Its seeds and leaves have been in use since ancient times to treat a number of human ailments and also as a household pesticide. The structure and ingredient presented in *Azadirachta indica* was mentioned by Sethu (Sethu, Andersen 2009). The mature leaf of plant was washed thrice with de-ionized water to remove dust and water soluble impurities and was dried until the leaves become crisp. The dried leaves were crushed and powdered and further washed with de-ionized water till the washings were free of color and turbidity. Then this powder was dried in an oven at 60 ± 2 °C and placed in desiccator for the adsorption studies, thus NLP prepared.

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For activated NLP, it was stirred with 0.1 N sulphuric acid for 30 min. Then after, it washed thrice with 1 liter de-ionized water to remove untreated acid and dried in an oven at 60 ± 2 °C. The surface characteristics of NLP and activated NLP, such as particle size distribution was investigated using Laser Particle Size Analyzer, Sympatec, Germany (Model No.: Helos-BF). The porosity, pore diameter and pore volume of adsorbents were determined by Mercury Porosimeter, Thermo Quest (Model No.: PASCAL – 140). The surface area was calculated using the multipoint Brunauer-Emmett-Teller (BET) model.

1.2. Adsorbate

Methylene blue (CI No. 52015) is a heterocyclic aromatic chemical compound with molecular weight 319.85l. The dye was purchased from Sigma Aldrich, India and structure of same was mentioned in Fig. 1. It has many uses in a range of different fields, such as biology, chemistry, medicine, etc. The concentration of MB in each aqueous solution was measured on an UV–visible spectrophotometer (ELICO SL 164 Double Beam UV-VIS Spectrophotometer) at λ_{max} = 665 nm (Wang *et al.* 2005).

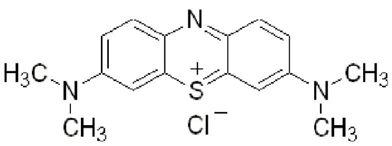

Fig. 1. Structure of Methylene Blue dye

1.3. Experimental details

The experiments were carried out as shown in Table 1 and maintaining process parameters of dye initial concentration of 200 mg/L and agitation speed of 200 rpm. The pH of system was maintained by 1.0 N HCl or 1.0 N NaOH during experiment. All other chemicals used were of analytical reagent grade.

Table 1. Experimental details for comparison of NLP and activated NLP for adsorption of MBD

Effect of System	Adsorption Dose (g/L)	pH	Temperature (K)	Contact Duration (min)
Effect of Adsorption dose	0.5, 1.0, 1.5, 2.0, 2.5 and 3.0	7	300	60
Effect of pH	1.0	1, 3, 5, 7, 9 and 11	300	60
Effect of Temperature	1.0	7	310, 320, 330, 340, 350 and 360	60
Effect of Contact duration	1.0	7	300	30, 45, 60, 75, 90 and 105

The percentage of removal and quantity of dye adsorbed, q_e (mg/g) was calculated using the following formula:

$$\% \text{ Removal} = (C_o - C_e) \text{ X } 100 / C_o, \quad (1)$$

$$q_e = (C_o - C_e) \text{ X } V / W, \quad (2)$$

were, C_o and C_e are initial and equilibrium concentration of dye respectively. V the volume of the solution and W the weight of the adsorbent used.

1.4. Equilibrium isotherm

The Freundlich isotherm is an empirical equation employed to describe heterogeneous systems and is expressed by the following equation.

$$q_e = K_F C_e^{1/n} \text{ OR } \log q_e = \log K_F + 1/n \log C_e, \quad (3)$$

where K_F (L/g) and n are the Freundlich constants reflecting the adsorption capacity and intensity respectively which are calculated from the intercept and slope of the plots of log C_e *vs.* log q_e.

The Langmuir equation is widely used for adsorption equilibria because of its thermodynamic basis. The Langmuir isotherm model assumes monolayer coverage of adsorbate over a homogeneous adsorbent surface, and at equilibrium, a saturation point is reached where no further adsorption can occur. The Langmuir isotherm model is expressed as

$$1/q_e = 1/q_{max} + (1/q_{max} K_L)(1/C_e), \quad (4)$$

where C_e is the equilibrium concentration (mg/L); q_e is the quantity of dye adsorbed onto adsorbents (mg/g); q_{max} is q_e for a complete monolayer (mg/g), a constant related to sorption capacity; and K_L is the Langmuir constant related to the affinity of the binding sites and energy of adsorption (L/mg) (Venkateswarlu *et al.* 2007).

1.5. Kinetic isotherm

There are different variables that depend upon rate of sorption such as surface area, porosity, particle size, etc. of adsorbent and properties of adsorbate. In order to investigate the mechanism of adsorption and potential rate controlling steps such as chemical reaction, diffusion control and mass transport processes, kinetic models have been used to test experimental data. These kinetic models included the pseudo first-order equation, the pseudo second-order equation and the Elovich equation.

The pseudo first-order equation of Lagergren is generally expressed as follows

$$\ln (q_e - q_t) = \ln q_e - k_1 t, \quad (5)$$

where q_t and q_e are the amounts of MBD adsorbed at time t and equilibrium (mg/g), respectively, and k_1 is the pseudo first-order rate constant for the adsorption process (1/min). The linear graph of ln (q_e − q_t) *vs.* t shows the applicability of first order kinetic. Also, the pseudo second-order chemisorption kinetic rate equation is expressed

$$t / q_t = 1/ k_2 q_e^2 + (1/q_e)t, \quad (6)$$

where k_2 is the equilibrium rate constant of pseudo second order equation (g/mg min). The linearity of t / q_t *vs* t suggests the best fit with pseudo second order kinetic (Paliulis 2006).

The Elovic equation was firstly used in the kinetics of chemisorption of gases on solids, it has been successfully applied for the adsorption of solutes from a liquid solution. The Elovic equation is given as follows:

$$q_t = 1/\beta \ \mathrm{In}\ (\alpha\beta) + 1/\beta \ \ln t, \quad (7)$$

where α is the initial sorption rate (mg/g min) and the parameter β is related to the extent of surface coverage and activation energy for chemisorption (g/mg). The linear graph of q_t *vs.* ln t shows the applicability of Elovic kinetic (Bulut, Tez 2007).

2. Results and discussion

2.1. Surface analysis of adsorbents

Table 2 compares the surface analysis of NLP and activated NLP using H_2SO_4, in which particle size, porosity, pore diameter, pore volume and surface area of activated NLP were moderately increased than those of NLP. While in comparison to NLP, pore diameter of activated NLP was slightly increased. This shows that H_2SO_4 was effective in creating well-developed pores on the surface of NLP with large surface area and porous structure.

Table 2. Surface analysis of Adsorbents

Analysis	NLP	Activated NLP
Particle Size (mesh)	122	184
Porosity (%)	24	37
Pore Volume (cm^3/g)	0.052	0.087
Pore Diameter (nm)	8.7–9.5	9.0–9.7
BET surface Area (m^2/g)	412	524

2.2. Effect of adsorbent dose

Fig. 2 depicted effect of adsorption doses of activated NLP and NLP (0.5 to 3.0 gm/L) for adsorption MBD at temperature of 300 K and contact duration of 60 min at neutral pH, in which constant increment in removal of MBD was found. The value of percentage removals were found to be 41.1 to 82.9% using activated NLP and also, 37.1 to 72.2% of removal using NLP at adsorbent dosage of 0.5 to 3.0 g/L respectively. This may be due to the increase in availability of surface active sites resulting from the increased dose and conglomeration of the adsorbents (D'Ilario *et al*. 2008). Also, the removal of MBD by activated NLP was found higher than NLP. The pore sizes after acid treatment of NLP are bigger and also, possibility that the small increase in the pore sizes of activated NLP as compared to normal NLP was due to formation of small new pores during acid activation at the higher acid concentration (Brezovska *et al*. 2005).

2.3. Effect of pH

The pH exploits an important part on the quantity of the dye fixed. The choice of the pH values was carried out in order to examine the evolution of the adsorption of the dye associated with the various chemical forms present. The influence of pH for percentage adsorption of MBD onto activated NLP and NLP was represented in Fig. 3, in which maximum MBD removal was found to be 72.3 and 79.9% using NLP and activated NLP respectively. The adsorption of MBD on activated NLP and NLP shows that the best adsorption may be obtained at basic pH which shows there is protons release from the surface of the carbon due to cation exchange between the sorbent and the dye solution. This reveals that the increase of pH deprotonate the acidic groups on the surface of the carbon and provides more negative sites for the sorption of cationic form of the dye molecules (Malarvizhi, Sulochana 2008). It is observed that percentage removal of activated NLP is higher than that of NLP.

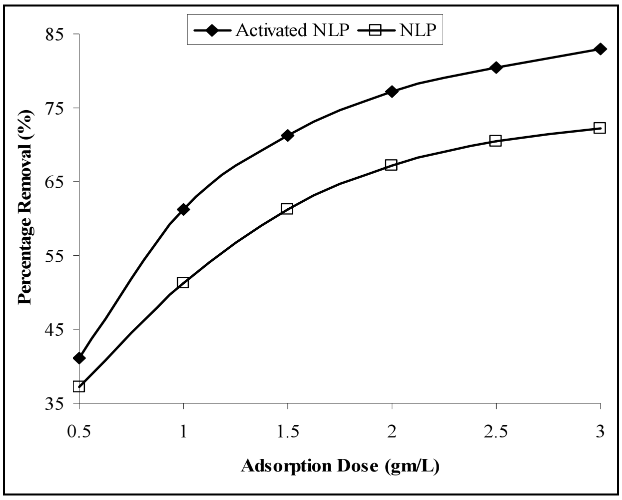

Fig. 2. Effect of adsorption dose on adsorption of MBD by activated NLP and NLP

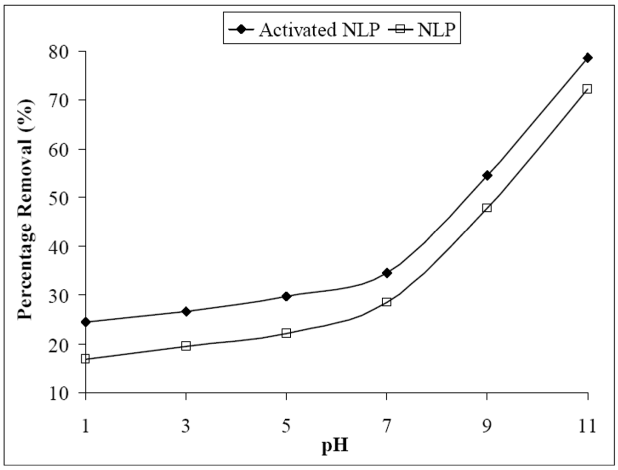

Fig. 3. Influence of pH on adsorption of MBD by activated NLP and NLP respectively at pH 11

2.4. Effect of temperature

To study the effect of temperature on the color removal, experiment was performed at temperature of 310, 320, 330, 340, 350 and 360 K (Fig. 4). The nature of curves of graph is considerable increase as increasing temperature and highest removal was found to be 44.8 and 49.8% using NLP and activated NLP respectively at temperature of 360 K. The adsorbed amounts of dyes decrease with the rise in temperature, which indicate that the adsorption process is of exothermic nature. This may be due to a tendency for the dye molecules to escape from the solid phase to be bulk phase with the increasing temperature of the solution (Ozacar, Sengil 2004). It is verified that removal of MBD by activated NLP was higher than the NLP.

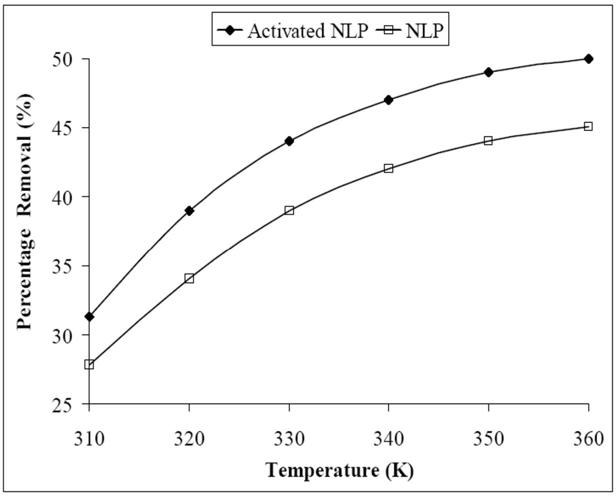

Fig. 4. Effect of temperature on adsorption of MBD by activated NLP and NLP

2.5. Effect of contact duration

The influence of contact duration on adsorption of MBD was presented in Fig. 5, in which amount of adsorption of dye are continuously increasing with increasing contact durations and The value of percentage removals were found to be 37.2 to 63.7% using activated NLP and also, 32.1 to 56.2% of removal using NLP at contact duration of 30 to 105 min respectively. That is probably due to the larger surface area of natural materials at the beginning for the adsorption of color. As the surface adsorption sites become exhausted, the uptake rate is controlled by the rate at which the adsorbate is transported from the exterior to the interior sites of the adsorbent particles (Bulut, Tez 2007). Also, it is confirm that adsorption of MBD on activated NLP is higher than NLP.

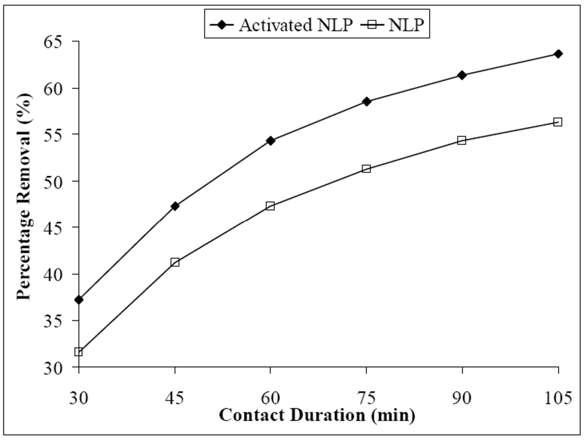

Fig. 5. Influence of contact duration on adsorption of MBD by activated NLP and NLP

2.6. Adsorption isotherm:

The isotherms parameters and correlation coefficient values were mentioned in Table 3, in which maximum adsorption capacity (Q_{max}) of activated NLP (401.6 mg/g) was found higher than NLP (352.6 mg/g). So, the activated NLP is more suitable adsorbent than NLP. Many studies are conducted for adsorption of Methylene Blue removal using adsorbent, rice husk, cotton waste, hair, coal, GLP and MLP and their Q_{max} values are 312, 277, 158, 250, 315.6 and 304.6 mg/g respectively (McKay *et al.* 1999; Patel, Vashi 2009). The coefficient values (r^2) of Freundlich and Langmuir models represented that the data were fitted to both isotherm. Also, we observed that r^2 values of Freundlich isotherm were found higher than Langmuir isotherm for both adsorbents, so, adsorption process is well fitted to Freundlich isotherm, confirming multilayer coverage of adsorbate having heterogenic adsorbent surface. The same pattern was observed for absorption of various types of dye onto adsorbents (Malarvizhi, Sulochana 2008; Inbaraj, Sulochana 2002; Prahas *et al.* 2008).

Table 3. Isothermal and kinetic parameters for adsorption of MBD using activated NLP and NLP

Model	Parameters	Adsorbent	
		Activated NLP	NLP
Freundlich	K_F (L/g)	4.99	4.84
	n	1.43	1.46
	r^2	0.994	0.997
Langmuir	K_L (L/g)	0.032	0.039
	Q_{max} (mg/g)	402	353
	r^2	0.998	0.993
Pseudo first-order	$K_1 \times 10^{-2}$ (1/min)	1.97	1.73
	r^2	0.985	0.954
Pseudo second-order	$K_2 \times 10^{-2}$ (g/mg min)	1.42	1.13
	r^2	0.996	0.991
Elovich equation	α (mg/g min)	30.1	25.5
	β (g/mg)	0.12	0.11
	r^2	0.995	0.967

2.7. Kinetic studies

Values of kinetic parameters (derived from slope and intercept) and the coefficients for these equations determined by non-linear regression for all kinetic models, Pseudo first-, Pseudo-second order and Elovich equation for adsorption of MBD onto activated NLP and NLP are listed in Table 3. High r^2 (> 0.99) implies that the correlations fit well with the experimental data. It can be observed that correlation coefficient values, 0.9960 and 0.9909 of Pseudo-second order equation, for activated NLP and NLP respectively, were higher than other kinetic isotherms. So, Pseudo-second order equation is most fitted than other kinetic isotherm. Similar views have been expressed by earlier scientists (Lin *et al.* 2008; Alpat *et al.* 2008; Nagda, Ghole 2008).

Conclusions

1. According to the surface analysis of activated NLP and NLP, the surface of activated NLP is more porous than NLP.

2. The comparison study of adsorption of MBD by activated NLP and NLP was conducted, in which effect of adsorption dose, pH, temperature and contact duration was exploited, and concluded that percentage removal of MBD by activated NLP was found higher than NLP. Also, parameter of adsorption dose is found to be more effective for removal of MBD onto activated NLP and NLP. The highest removal of MBD (82.9%) was achieved using 3.0 gm/L of activated charcoal at pH 7 and contact duration of 60 min.

3. The Freundlich and Langmuir isotherms were utilized, in which maximum adsorption capacity (Q_{max}) of activated NLP and NLP was found to be 401.6 and 352.6 mg/g respectively, indicating activated NLP is more efficient than NLP. Also, Freundlich isotherm was more application than Langmuir isotherm for MBD adsorption.

4. The data were analyzed using kinetic models, pseudo first- and second-order and Elovich equation, in which pseudo second-order model is more applicable among others investigated models, derived from correlation coefficient.

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