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Removal of Torque Blue Dye from Aqueous Solution by Kail Sawdust

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Abstract: Kail sawdust (*Pinus wallichiana*) was used for the removal of Torque Blue dye (textile dye) from water using a batch adsorption technique. The effect of initial dye concentration, adsorbent size (particle size) and dosage, pH and temperature on the removal of dye from water was studied to understand the process of adsorption and the kinetics of adsorption process. It was found that maximum (98.7%) adsorption occurred at an adsorbent dosage of 0.5 gm/100 ml dye solution with a particle size of 75 µm in 15 mg/L dye concentration at 37ºC temperature and 7.5 pH. The Langmuir adsorption isotherm model fitted the data well. The rate of adsorption was better fitted by pseudo first order kinetics. The values of thermodynamic parameters (ΔGº, ΔHº and ΔSº) indicated that adsorption was spontaneous and exothermic. The surface morphology and the functional groups on the adsorbent surface were characterised by SEM and FTIR spectroscopic techniques.

Key words: Adsorption, Kail sawdust, Langmuir, Freundlich, Temkin isotherm model.

Introduction

Fresh water is an essential and important component of the earth's ecosystem and plays a vital role in our daily lives. The shortage in the supply of fresh water is becoming a serious problem worldwide. Synthetic dyestuffs existed in the effluents of wastewater from different industries such as textiles, paper, leather, and plastics (Li et al., 2015). These coloured effluents can be mixed in surface water and groundwater systems and then they may also transfer to drinking water. The treatment and disposal of dye-contaminated wastewater is one of the most serious environmental problems. The dyes have complex chemical structures and therefore do not degrade easily. Dyes are coloured and impede light penetration, retards photosynthetic activity, inhibit the growth of biota and also have a tendency to chelate metal ions which produce micro-toxicity to organisms (Zhang et al., 2015). There are about more than 100,000 commercial dyes with a production of $>7\times10^5$ tonnes/year all over the world.

The discharge of these dyes from different industries into the hydrosphere poses a significant threat to the environment and human health even at very low concentrations (Liu et al., 2015). The dye contaminated water needs to be treated before discharging into the environment (Kumar et al., 2008; Bhatti et al., 2015). There are a number of methods such as coagulation, flocculation, biological oxidation, and sedimentation, photo-Fenton treatment, oxidation with chemical oxidants (ozone or hydrogen peroxide etc.), advanced oxidation processes (AOPs), photocatalytic oxidation/

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degradation, membrane processes, electrochemical oxidation/degradation, adsorption and combined methods for the removal of dyes from water solutions. Different methods and their disadvantages are given in Figure 1.

In the last years, adsorption technologies have become a very important technique for removing dyes from industrial wastewater, attracting much attention due to their low cost, high efficiency and simple operation (Zhou et al., 2015). Therefore, exploring some novel adsorption materials with high adsorption capacity and even high selectivity towards specific dyes has drawn great attention. So far, many kinds of adsorption materials reported have been used for the treatment of wastewater. During recent years attempts are being made to use engineered nanomaterials (ENMs) as adsorbents for water treatment but the data are limited (Adeleye et al., 2016). There are many low cost agricultural adsorbents such as orange peel wastes, olive stones, coconut shell, cocoa shell, apricot stone, palmyra palm, peach stone, pecan nuts, hazelnut husk, almond, beech and poplar woods, rice husk, rice husk ash, plant maize, corn, fly ash, bagasse, leaves, sago waste, sawdust, seed hull, tea waste, coffee, leaf powder, sunflower waste, sunflower seed peel pistachio, palm kernel fibre, wheat bran, peat, palm shell, oil palm tree, onion skins, belpatra bark etc. which have been used for purification of water (Saka et al., 2012; Gupta et al., 2015). Torque blue dye is frequently used in dyeing textiles in many textile industries located in and nearby Ghaziabad (UP), India. The effluent containing Torque blue dye is highly poisonous and health hazard.

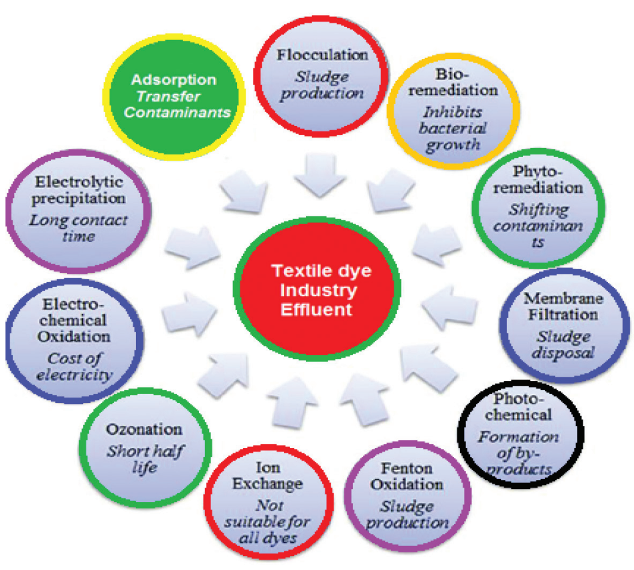

Figure 1: Different methods of dye removal from effluent of textile industry and their drawback.

In this paper the removal of Torque blue dye from water solution has been studied by using Kail sawdust for the first time. Preliminary experiments have shown that Kail sawdust (waste wood coming out of carpentry work), locally available can serve as an adsorbent for the removal of Torque blue dye from water solution. Effect of contact time, initial dye concentration, particle size of adsorbent, adsorbent dosage, pH of the solution and temperature on the removal of Torque blue dye by Kail sawdust as an adsorbent has been investigated. Different adsorption isotherm and kinetic models were tested. Thermodynamic parameters have also been evaluated.

Materials and Methods

Materials

The sawdust of Kail wood (Figure 2) used as adsorbent was collected from the furniture manufacturing shop of Ghaziabad. Torque blue (TB) dye (Figure 3) used as an adsorbate for the study was supplied by Rituraj Textile Industry, Mohan Nagar, Ghaziabad (UP), India.

Figure 2: Sawdust of Kail wood.

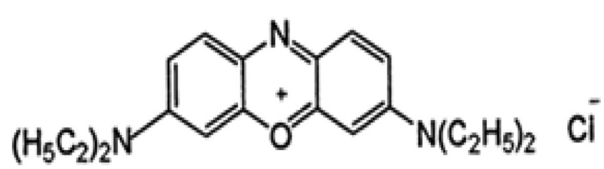

Figure 3: Structure of Torque blue (TB) dye.

Methods

Preparation of Adsorbent

Kail sawdust was washed many times with distilled water to remove surface adhered impurities. It was then dipped in distilled water for two days to remove all the colouring materials present at the surface of sawdust. After removing from the water, it was kept in dilute HCl for 30 minutes to remove all the remaining impurities and colour. The sawdust was filtered and dried in sunlight for 5-6 days followed by oven drying for 15 hours at 120ºC. The dried sawdust was crushed into powder and passed through different sieves to have a particle size of 75 µm, 150 µm, and 300 µm. The sawdust of different particle sizes was stored in air tight closed bottles for use as an adsorbent.

Preparation of Adsorbate

A stock solution of Torque blue dye (TB) was prepared by dissolving 50 mg of dye in 100 ml of double distilled water. This stock solution was diluted to desired concentrations as per experimental requirements.

SEM Studies of Kail Sawdust

The surface morphology of Kail sawdust was examined with SEM model no. Zeiss 18 EVO.

FTIR Spectral Studies

FTIR spectra of Kail sawdust before and after adsorption of TB dye were recorded in KBr phase with FTIR spectrophotometer.

Removal of TB Dye and Concentration Determinations

UV–visible spectra of TB dye of different concentrations in water were recorded with the help of Shimadzu UV-1800 spectrophotometer and λ_{max} = 665 nm was found out. Calibration curve was plotted. Adsorption studies were carried out by shaking Kail sawdust (0.3-0.7 gm) of different particle size (75-300 µm) with a 100 ml dye solution having concentration 10-30 mg/L at different temperatures (27-47ºC) and pH (4.5-9.0). The desired pH of the solution was maintained by adding an appropriate amount of 0.1N solution of HCl or NaOH. Kail sawdust was dispersed in TB solution and the assembly was kept in a water thermostat. After every 15 minutes, the solutions were filtered through Whatman filter paper no. 41 and concentrations of TB dye in the solutions were determined by measuring the absorbance. The process was continued till equilibrium was attained.

Result and Discussion

Adsorption Capacity of Kail Sawdust and Removal of TB Dye

The following two equations were used to determine the adsorption capacity and percentage removal of TB dye by Kail sawdust (Agrawal et al., 2015; Nagpal et al., 2014).

$$q_e = [(C_o - C_e)/M] \times V \quad (1)$$

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

where q_e is the amount of dye adsorbed at equilibrium, C_o is the initial concentration of dye, C_e is the concentration of dye at equilibrium, M is the mass of adsorbent (g) and V is the volume of solution (l).

All experiments were carried out at the wavelength corresponding to the maximum absorbance, λ_{max} = 665 nm. Concentrations were calculated from the calibration curve. The effect of various parameters on the adsorption of TB dye was investigated.

Effect of Contact Time and Initial Dye Concentration

Adsorption of TB dye on Kail sawdust (particle size 150 µm) was studied at five different initial dye concentrations (10, 15, 20, 25 and 30 mg/L) at an adsorbent dosage of 0.5 gm/100 ml, 7.5 pH and 37ºC temperature. The percent dye removal increased with the time and an equilibrium state was established in about 60 minutes (Figure 4). It was found that the removal of the dye was rapid for the first stage from 0 to 20 minutes. The second stage could be described at a lower rate from 20 to 40 minutes and then the process reached a saturated state say third stage. Since adsorbent was very porous having large internal surface area, three consecutive mass transport steps might be involved in the process of adsorption. In the initial stage molecular diffusion took place where dye molecules might have moved from the solution to the exterior surface of the adsorbent. In the second stage pore diffusion occurred, where dye molecules entered from exterior surface into interior site of adsorbents and finally in the third step, dye molecules were adsorbed on the active sites of the adsorbents. In the initial stages there were more available vacant sites on the surface of Kail sawdust and hence adsorption of dye molecules was much faster. As the process of adsorption continued the number of active sites decreased and the process of adsorption slowed down and reached an equilibrium stage and finally became almost constant (Malik, 2004; Seow et al., 2016).

It was seen that the percentage removal of dye decreased from 97.5% to 70.0% with increasing dye concentration from 10 to 30 mg/L. Adsorption process was very much dependent on initial dye concentration. This is due to the fact that at lower concentrations of dye, the adsorbate/adsorbent ratio was low so that a large number of active adsorption sites were free but at higher concentration of dye, this ratio became quite high and the availability of active site was low. As a result, the adsorption of dye at higher concentration was low (Srivastava et al., 2011; Hilal et al., 2012).

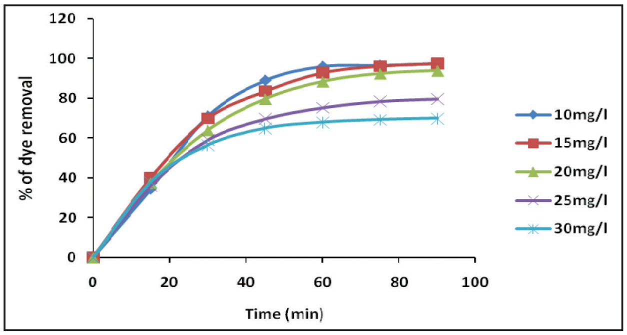

Figure 4: Effect of contact time and initial dye concentration on adsorption of TB dye by Kail sawdust (Temp. = 37°C, pH = 7.5, adsorbent dosage = 0.5 gm/100 ml, particle size = 150 µm).

Effect of Particle Size

The effect of particle size of the adsorbent on the dye removal at pH = 7.5, temp. = 37°C, adsorbent dosage = 0.5 gm/100 ml and initial dye concentration = 15 mg/L is shown in Figure 5. It was observed that percentage removal of dye increased from 89.7% to 98.7% (maximum adsorption) with time and the values were lower at higher particle size at all the times. Smaller particles have greater surface area and hence higher adsorption capacity. As a result the percentage removal of dye by Kail sawdust having a particle size of 75 µm was highest.

Effect of Adsorbent Dosage

The percentage removal of TB dye was increased from 65.5% to 95.3% with an increase of adsorbent dosages from 0.3 gm to 0.7 gm/100 ml solution, keeping other parameters constant (Figure 6). Because of an increase in the adsorbent dosage, available surface area and adsorption sites increased and hence adsorption increased (Dubey et al., 2012). As a result of this, the percentage removal of dye increased with increase of adsorbent dosage. The percentage removal of dye after a certain amount of dosage became constant because concentration of dye molecules reached equilibrium state between solid and solution phase (EI-Bindary et al., 2015).

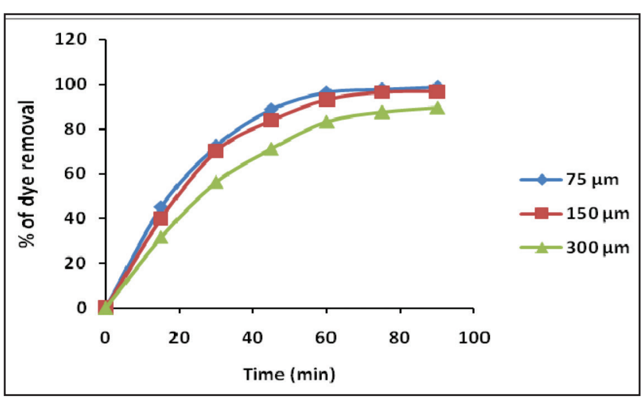

Figure 5: Effect of particle size on adsorption of TB dye by Kail sawdust (Temp. = 37°C, pH = 7.5, adsorbent dosage = 0.5 gm/100 ml, dye conc. = 15 mg/L).

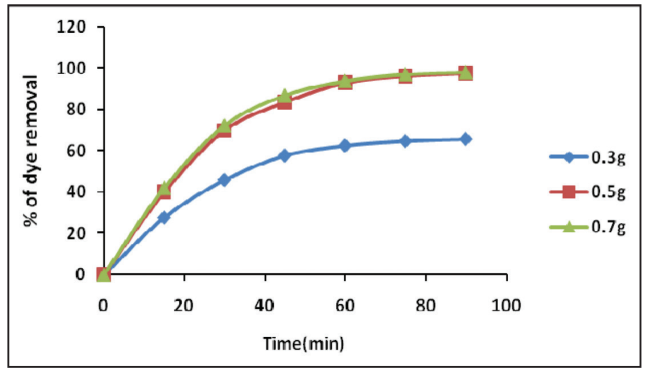

Figure 6: Effect of adsorbent dosage on adsorption of TB dye by Kail sawdust (Temp. = 37°C, pH = 7.5, dye conc. = 15 gm/L, particle size = 150 µm).

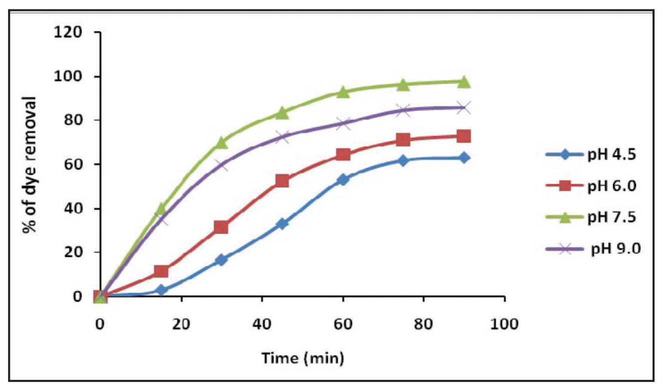

Figure 7: Effect of pH on adsorption of TB dye by Kail sawdust (Temp. = 37°C, adsorbent dosage = 0.5 gm/100 ml, dye conc. = 15 mg/L, particle size = 150 µm).

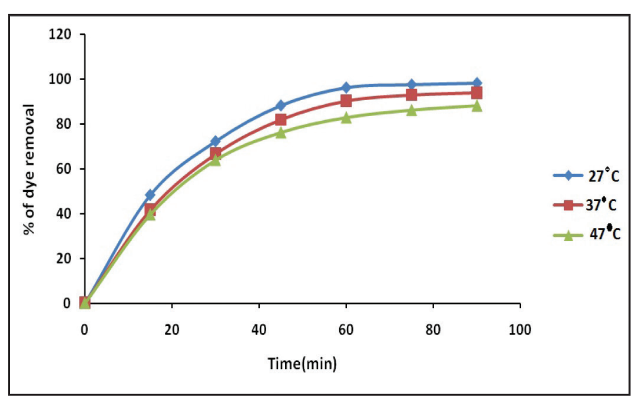

Figure 8: Effect of temperature on adsorption of TB dye by Kail sawdust (pH = 7.5, adsorbent dosage = 0.5 gm/100 ml, dye conc. = 15 mg/L, particle size = 150 µm).

Effect of pH

The effect of pH (4.5–9.0) on the removal of TB dye at an adsorbent dosage of 0.5 gm/100 ml, initial dye concentration = 15 mg/L, temperature = 37°C and particle size = 150 µm is shown in Figure 7. It was seen that percentage dye removal was lowest (63.0%) at pH 4.5.

This is due to the fact that in the acidic medium, the adsorbent surface is positively charged so there is a repulsion between protonated dye molecules and positively charged adsorbent surface. Hence the rate of adsorption decreased. In the basic medium (pH = 7.5), the adsorbent surface was negatively charged so that electrostatic attraction between positively charged dye molecules occurred which increased the adsorption and as a result, percentage removal of dye increased: at higher pH (more than 7.5) it decreases in adsorption because the hydroxide ions started forming soluble complexes between the dye and the adsorbent (Khattri et al., 1999). Therefore, Kail sawdust proved to be a good adsorbent at pH 7.5 with dye removal of 97.7%.

Immobilized halophilic bacterial strain (AMETH148) collected from Kelambakkam and Marakkanam salterns, nearby East Coast of Tamil Nadu, India was also used for the removal of Torque Blue dye and 72.84% could be removed. (Jayaprakashvel et al., 2014). Beside this Belpatra (Aegel marmelos) bark powder has also been used for colour removal of Torque Blue dye. Its colour removal efficiency was 98% at an adsorbent dosage of 0.7 g/100 ml dye solution having 15 mg/L dye concentration at 310 K temperature and 7.5 pH (Gupta et al., 2015).

Effect of Temperature

The effect of temperature on percentage dye removal by Kail sawdust is shown in Figure 8. As the temperature increased the removal efficiency decreased. Percent removal of dye decreased from 98.3% to 88.3% when the temperature increased from 27ºC to 47ºC. This specifies an exothermic nature of the existing process. This is because on increasing the temperature, there is a decrease in residual forces on the surface of adsorbent and hence decrease in surface energy of adsorbent, resulting in lower adsorption.

Thermodynamic Parameters

The various thermodynamic parameters, e.g. the changes in standard free energy ($\Delta G°$), enthalpy ($\Delta H°$) and entropy ($\Delta S°$) were estimated by equations (3), (4) and (5) respectively.

$$\Delta H° = R\left[\frac{T_2 T_1}{T_2 - T_1}\right] \ln\left(\frac{K_2}{K_1}\right) \quad (3)$$

$$\Delta G° = -RT \ln K \quad (4)$$

$$\Delta S° = \left(\frac{\Delta H° - \Delta G°}{T}\right) \quad (5)$$

where R is the gas constant and K, K_1, K_2 are equilibrium constants at temperature T (27°C), T_1 (37°C) and T_2 (47°C) respectively. The equilibrium constant (K) can be calculated by equation (6).

$$K = \frac{\text{Concentration of dye present on the adsorbent surface}}{\text{Remaining concentration of dye in solution}} \quad (6)$$

The values of thermodynamic parameters ($\Delta G°$, $\Delta H°$ and $\Delta S°$) for TB dye at different temperatures are given in Table 1.

Table 1: Thermodynamic parameters of TB dye

Temp. (°C)	*Thermodynamic Parameters*		
	–ΔG° [KJ/mol]	*–ΔH° [KJ/mol]*	*– ΔS° [J/(Kmol)]*
27	10.17	102.53	307.85
37	7.09	59.97	170.58
47	5.39	–	–

The negative values of ($\Delta G°$), suggest that adsorption process was spontaneous. The $\Delta S°$ values (Table 1) are also negative corresponding to a decrease in adsorbed species' degree of freedom at the solid/solution interface of the whole adsorption process. Negative values of enthalpy ($\Delta H°$) confirmed an exothermic adsorption in accordance with the decreasing adsorption capacity with an increasing adsorption temperature (Jiang et al., 2012).

Adsorption Kinetics

Pseudo first order and pseudo second order models were used to know the adsorption kinetics (Shi et al., 2014).

Pseudo First Order Kinetics

The Lagergren pseudo first order rate equation explains the adsorption of solid-liquid phases and is expressed by equation (7).

$$\log (q_e - q) = \log q_e - \frac{k_1}{2.303} t \quad (7)$$

where q_e and q are the amounts of adsorbate (mg/g) at equilibrium and at any time t respectively and k_1 is the pseudo first order rate constant (L min^{-1}). A plot of log ($q_e - q$) versus t gave straight line showing the validity of equation (7) (Figure 9). The correlation coefficient (R^2 = 0.992) is very near to one, indicating a good fit for pseudo-first order rate. The values of k_1 and q_e calculated from the line are given in Table 2.

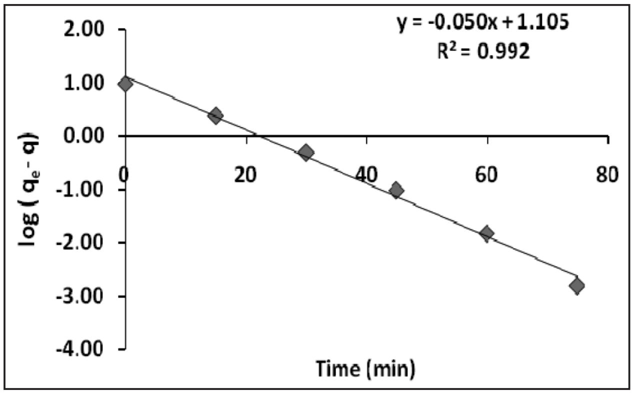

Figure 9: The pseudo first order plot for the adsorption of TB dye on to Kail sawdust.

Table 2: Kinetic parameters for adsorption of TB dye onto Kail sawdust

Pseudo first order model			q_e (experimental)
k_1 (L min^{-1})	q_e (calculated)	R^2	
0.115	2.75	0.992	
Pseudo Second Order Model			2.65
k_2 (g/mg/min)	q_e (calculated)	R^2	
0.020	2.93	0.959	

Pseudo Second Order Kinetics

The pseudo second order rate is expressed by equation (8).

$$t/q = 1/(k_2\, q_e^2) + t/q_e \tag{8}$$

where k_2 is the pseudo second order rate constant (g/mg/min). The plot of t/q versus t also gave straight line (Figure 10) but the correlation coefficient ($R^2 = 0.959$) was found lower than that of pseudo first order kinetic equation. Therefore, adsorption process was better fitted by pseudo first order rate equation. The values of R^2 and k_2 are also given in Table 2.

Adsorption Isotherms

The adsorption capacity of the adsorbent can be determined by the study of adsorption isotherms. It is an equation between the amount of dye adsorbed onto the adsorbent and the equilibrium concentration of the dye in solution at a given temperature. The analysis was done by using Langmuir, Freundlich and Temkin isotherm models.

Langmuir Adsorption Isotherm

The Langmuir isotherm model is given by the following equation (Srivastava et al., 2011; Foo et al., 2010).

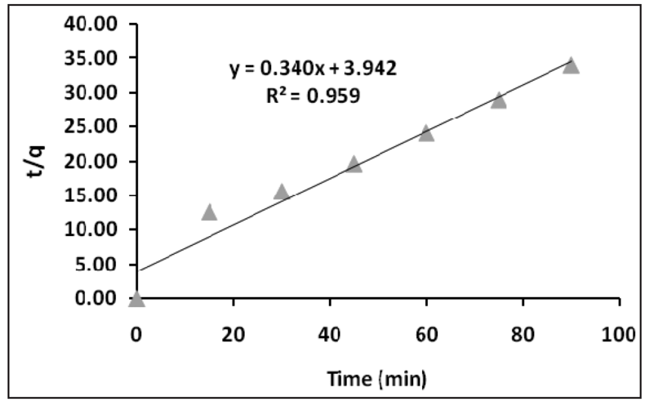

Figure 10. Pseudo second order plot for the adsorption of TB dye on to Kail sawdust.

$$\frac{C_e}{q_e} = \frac{1}{Q^o b} + \frac{C_e}{Q^o} \tag{9}$$

where C_e is the equilibrium concentration of dye (mg/L), q_e is the amount of dye adsorbed at equilibrium (mg/g), Q^o (mg/g) and b (L/mg) are the Langmuir constants. The plot of C_e/q_e versus C_e (Figure 11) is almost linear ($R^2 = 0.999$), which shows the good fit of Langmuir isotherm. The values of Q^o (mg/g) and b are calculated from the slope and intercept of the linear plots and are given in Table 3.

The suitability of Langmuir isotherm can also be proved by calculating the separation factor or equilibrium parameter (R_L), which is determined by using the following equation:

$$R_L = \frac{1}{1 + bC_o} \tag{10}$$

where C_o is the initial concentration of dye (mg/L) and b is the Langmuir constant (L/mg) discussed above. The value of R_L is 0.015, which shows that adsorption of dye onto Kail sawdust is a favourable process since the R_L value lie between 0 and 1.

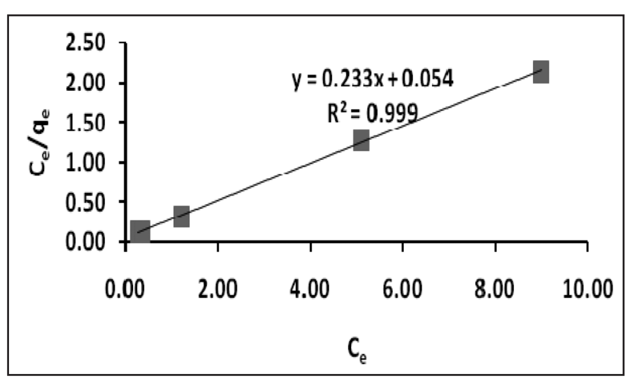

Figure 11: Langmuir adsorption isotherm for TB dye.

Table 3: Langmuir, Freundlich and Temkin adsorption constants of TB dye onto Kail sawdust

Langmuir adsorption isotherm		
Q^o	B	R^2
4.275	4.307	0.999
Freundlich adsorption isotherm		
n	K_f	R^2
5.780	3.069	0.754
Temkin adsorption Isotherm		
B	A	R^2
0.530	176	0.825

Freundlich Adsorption Isotherm

The Freundlich model is based on adsorption phenomenon on the heterogeneous surface. Linearized equation of Freundlich isotherm model can be represented as:

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \qquad (11)$$

where K_f and n are Freundlich constants related to adsorption capacity (L/mg) and adsorption intensity respectively. A plot of log q_e versus log C_e (Figure 12) was used to determine the Freundlich constants. The values of n and K_f calculated from the slope and intercept are given in Table 3. A straight line was not found, so Freundlich isotherm was not followed. Regression coefficient ($R^2 = 0.754$) shows that data does not fit Freundlich adsorption isotherm.

Temkin Isotherm

The Temkin isotherm model is based on adsorbent-adsorbate interactions and is expressed by the following equation (12):

$$q_e = B \ln A + B \ln C_e \qquad (12)$$

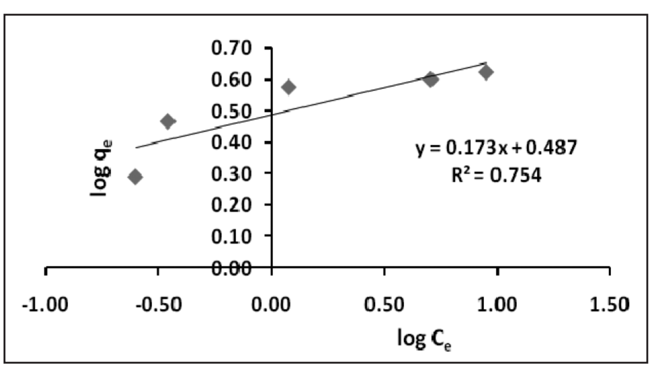

Figure 12: Freundlich adsorption isotherm for TB dye.

where $B = \frac{RT}{b}$, A (L/g) and B are Temkin constants, R is the gas constant, T is the absolute temperature, and b (J/Mol) is a constant related to the heat of sorption. Applicability of Temkin isotherm was checked by plotting a graph between q_e and ln C_e. It was observed that points did not lie on a straight line (Figure 13). R^2 value is also not near one (Table 3). Hence, Temkin model also does not fit the data.

All the parameters of the above three models are given in Table 3. R^2 value clearly indicated that the Langmuir adsorption isotherm model fits the data well.

SEM Studies

SEM picture of Kail sawdust (Figure 14) shows that the adsorbent surface has a large number of pores making it more effective for adsorption. Because of the pores dye molecules enter into the pores also and get adsorbed there.

Fourier Transform Infra-Red (FTIR) Studies

FTIR spectra of sawdust (Figure 15a) show a characteristic of a generic oxygenated hydrocarbon, as this sample is dominated by cellulosic biomass. FTIR

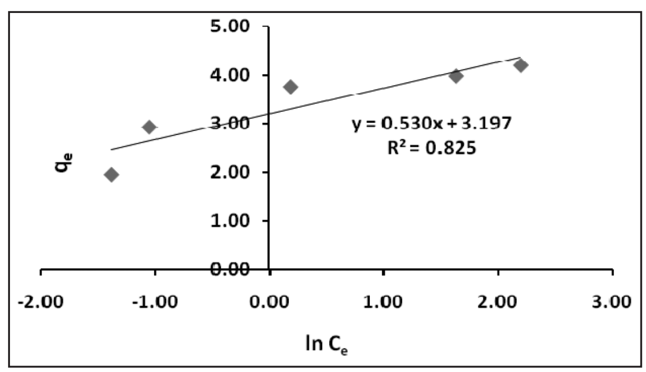

Figure 13: Temkin adsorption isotherm for TB dye.

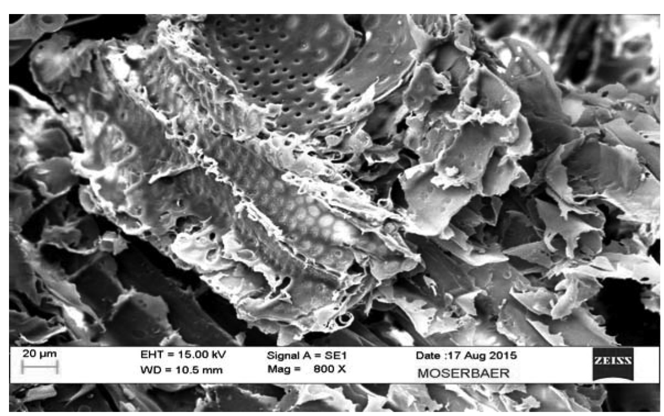

Figure 14: Scanning electron micrograph of Kail sawdust.

spectra show a number of bands in the frequency range of 4000 to 650 cm^{-1}. The band centred at 3350 cm^{-1} is attributed to the presence of –OH functional groups, while the band at around 2970 cm^{-1} is likely due to alkyl C–H stretches. There is an intense band occurring at 1040 cm^{-1} which is characteristic of a C–C–O (or may be C–O–C) asymmetric stretch. There are also a number of bands between 1200 and 1800 cm^{-1}, which can be attributed to –OH in-plane bending modes, the presence of water and carbonyl (C=O) and other common alkane and oxygenated hydrocarbon functional groups. These bands are in the same range as reported earlier (Azlina et al., 2014).

Figure 15b shows the FTIR spectrum of Kail sawdust after dye adsorption. This spectrum revealed a slight lowering of the position of –OH and –CH peaks and two new bands are appearing at the wave numbers 1650 and 1420 cm^{-1}. These bands may arise due to some interaction of adsorbent with dye molecules on the surface of Kail sawdust, through weak interaction. It is suggested that adsorption of dye on Kail sawdust is a physical adsorption.

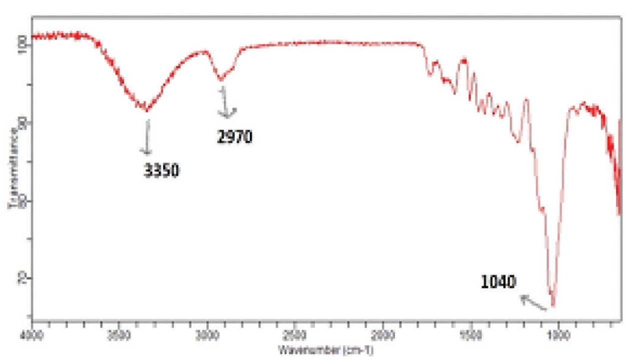

Figure 15a: FTIR spectrum of Kail sawdust before adsorption.

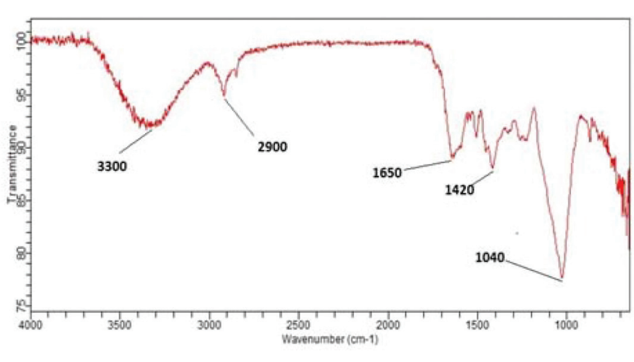

Figure 15b: FTIR spectrum of Kail sawdust after adsorption.

Conclusions

Kail sawdust was used for the first time as an adsorbent for the removal of Torque blue dye from aqueous solution and 98.7% dye was removed in one hour. The percent removal of the dye increased with dosage of sawdust and pH of dye solution. The phenomenon of adsorption was found to be exothermic. Langmuir adsorption isotherm model and pseudo first order kinetic model fitted the data. Porosity of Kail sawdust helped in accelerating the process of adsorption and removal of dye from water solution.

Kail sawdust is a waste material abundant in nature, low cost, biodegradable, easily available and eco-friendly adsorbent. Hence, use of Kail sawdust for removal of TB dye could be proved as effective, economical and green technology for reuse of waste water.

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