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Adsorption of Dyes from Aqueous Solutions onto Tur Dal Husk: Characterisation, Equilibrium and Kinetic Studies.

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ABSTRACT

In the present study, agricultural waste tur dal husk was used for the adsorption of the dyes amaranth and methylene blue. The operating variables studied were initial concentration, initial solution pH, adsorbent dosage and contact time. Experimental equilibrium data were fitted to Freundlich and Langmuir isotherms. The kinetics of adsorption of methylene blue and amaranth onto tur dal husk was found to follow a pseudo first order kinetics. The maximum adsorption of amaranth and methylene blue was 45.09 and 216.68 mg/g of the adsorbent respectively. The Fourier transformed infrared spectroscopy reveals that –OH, C=O and C-O groups present in the tur dal husk is involved in the adsorption process. The optimum pH for the adsorption of methylene blue and amaranth was 6 and 2 respectively. Characterization of the tur dal husk showed that the relative percentage of protein is very less making it an excellent adsorbent for the removal of dyes from wastewater effluents.

Keywords: Agriculture waste, tur dal husk, amaranth, methylene blue, FTIR, Isotherms

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INTRODUCTION

Dyes are widely used in industries such as textiles, rubber, plastics, printing, leather, cosmetics, etc., to colour their products. As a result, they generate a considerable amount of coloured wastewater. There are more than 10,000 commercially available dyes with over 7 x 9 10^5 tonnes of dye stuff produced annually. It is estimated that 2 % of dyes produced annually is discharged in effluents from associated industries (Allen and Koumanova 2003). Among various industries, textile industry ranks first in usage of dyes for coloration of fiber. The total dye consumption of textile industry worldwide is in excess of 107 kg/year and an estimated 90 % of this ends up on fabrics. Consequently, 1,000 tonnes/year or more of dyes are discharged into waste streams by the textile industry worldwide (Marc 1996). Discharge of dye-bearing wastewater into natural streams and rivers poses severe problems to the aquatic life, food web and causes damage to the aesthetic nature of the environment.

Dyes can have acute and/or chronic effects on exposed organisms depending on the exposure time and dye concentration. Dyes can cause allergic dermatitis, skin irritation, cancer, mutation, etc. Dyes can be classified as (Mishra and Tripathy 1993): anionic (direct, acid and reactive dyes), cationic (basic dyes) and non-ionic (dispersive dyes). Many treatment processes have been applied for the removal of dye from wastewater such as: Fenton process (Behnajady et al. 2007), photo/ferrioxalate system (Huang et al. 2007), photo-catalytic and electrochemical combined treatments (Neelavannan et al. 2007), photo-catalytic degradation using UV/TiO2 (Sohrabi and Ghavami 2008), sono-chemical degradation (Abbasi and Asi 2008), Fenton biological treatment (Lodha and Chaudhari 2008), biodegradation (Daneshvar et al. 2007), photo-Fenton processes (Garcia-Montano et al. 2007), integrated chemical–biological degradation (Sundarjanto et al. 2006), electrochemical degradation (Fan et al. 2008), adsorption process (Tan et al. 2007; Hameed et al. 2007a, b), chemical coagulation/flocculation, ozonation, cloud point extraction, oxidation, nano-filtration, chemical precipitation, ion exchange, reverse osmosis and ultra-filtration (Lorenc-Grabowsk and Gryglewic 2007; Malik and Saha 2003;Malik and Sanyal 2004; Banat et al. 1996). Among treatment technologies, adsorption is rapidly gaining prominence as a method of treating aqueous effluent. Some of the advantages of adsorption process are possible regeneration at low cost, availability of known process equipment, sludge-free operation and recovery of the sorbate (Kapdan and Kargi 2002).

Agricultural waste materials have little or no economic value and often pose a disposal problem. The utilization of agricultural waste is of great significance (Geopaul 1980). A number of agricultural waste materials are being studied for the removal of different dyes from aqueous solutions at different operating conditions. The agricultural waste that have been studied include coir pith (Namasivayam and Kavitha 2002), orange peel (Rajeswari et al. 2001), banana peel (Annadurai et al. 2002), rice husk (Malik 2003), straw (Kannan and Sundaram 2001), date pit (Banat et al. 2003a), oil palm trunk fiber (Hameed and El-Khaiary 2008a), durian (Durio zibethinus Murray) peel (Hameed and Hakimi 2008), guava (*Psidium guajava*) leaf powder (Ponnusami et al. 2009), almond shell (Ardejani et al. 2008), pomelo (*Citrus grandis*) peel (Hameed et al. 2008a), broad bean peel (Hameed and El-Khaiary 2008b), peanut hull (Tanyildizi 2011), *Citrullus lanatus* rind (Bharathi and Ramesh 2012).

The agricultural industry creates huge amount of waste and is matter of concern due their potential of creating environmental pollution. Under the provision of various environmental acts the agricultural waste needs to be disposed safely and require significant investment on such waste disposal. The cost on such treatments and disposal however can be reduced if the agricultural byproducts is utilized for some useful purpose instead of throwing (Tumin et al., 2008). The agro food industry has the tremendous potential to remove pollutants from effluent by adsorption. The adsorption properties of agro waste is natural and is due to the presence of various functional groups cellulose, hemicelluloses, lignin, starches, sugar, protein, lipid present in agro based waste. These functional groups have the capability of complexing organic and inorganic molecules (Sud et al, 2008) and remove them by process of adsorption.

India is the largest producer of pulses (edible legume) in the world. Pigeon pea or Tur (*Cajanus cajan*) is the second most major pulse produced in India and is mainly processed to convert into product known as “Tur dal”. The manufacturing industries of Tur dal generate considerable amount of byproduct/waste in the form of husk. Tur dal husk, which is the low cost by product of this industry, is generally considered for cattle feed only. During processing many times due to use of lime water, alkali and other chemicals the husk become unfit even for cattle feed. In the present study the possibility of using Tur dal husk for removing dyes – methylene blue and amaranth is explored.

MATERIALS AND METHODS

Biosorbent and Dye solution

The tur dal husk was collected from dehulling unit and was washed extensively in running tap water to remove dirt and other particulate matter. This was later subjected to colour removal through washing and boiling in distilled water repeatedly. Subsequently the husk was oven dried at 105°C for 24 hours, stored in a desiccator and used for biosorption studies in the original piece size. Amaranth and Methylene blue has been used as the representative anionic and cationic dye in the present study. Stock solutions were prepared by dissolving accurately weighed samples of dye in distilled water to give a concentration of 1000 mg/L and diluting when necessary. Initial pH was adjusted by adding dilute solutions of HCl or NaOH.

Table 1: The general data of two dyes used in this study

Commercial name	Classification	λ_{max} (nm)
Amaranth	Anionic Monoazo	520
Methylene Blue	Cationic Thiazine	665

Batch adsorption experiments

Batch adsorption studies were conducted in a routine manner. The 250 ml flasks containing 100 ml of the dye solution was contacted with the predetermined amount of the biosorbent at equilibrium time. The flasks were agitated at a 120 rpm constant shaking rate to ensure that equilibrium is achieved. The dye solution was separated from the biosorbent using Whatman No.1 filter paper. Adsorption uptake values were determined as the difference between the initial dye concentration and the one in the supernatant. All the experiments were carried out in duplicates and the average values were used for further calculations.

RESULTS

Characterisation of tur dal husk:

The carbon, hydrogen and nitrogen content of tur dal husk showed very low percentage of nitrogen (1.13%) in comparison to the carbon quantities (40.6%). This indicates that few nitrogen containing compounds are involved in the adsorption of dyes. A relatively larger percentage of hydrogen (6.35%) in comparison to nitrogen compounds indicates that carbon-hydrogen groups might be available for adsorption of dyes. The relatively low percentage of nitrogen shows that very less percentage of protein might be present in the husks. This is advantageous over protein rich adsorbents since proteinious materials are likely to putrefy under moist conditions.

The FT-IR spectra of the tur dal husk in the range of 400-4000 cm^{-1}were taken in order to obtain information on the nature of functional groups on the husk and dye interaction. The absorption spectra of TDH display a broad, intense --OH stretching absorption trough at 3431 cm^{-1}, although the bands are dominated by the -OH stretch due to bonded water. The strong peak at 1733 cm^{-1} is caused by the C=O stretching band of the carboxyl group. The peak at approximately 1100 cm^{-1} is due to either the C-O stretch of the -OH bend. However, the N-H stretch (3300 cm^{-1}) and the C-N stretch (1000 cm^{-1}) are not seen in this spectra due to the dominance of the ---OH stretch.

Effect of adsorbent dosage

The biosorption of methylene blue and amaranth was studied at various tur dal husk concentrations ranging from 0.5 to 5 mg/L. The percentage removal of the dye increased with increase in the sorbent dosage (Fig 1 a and b). The results revealed that the colour removal increased with adsorbent dosage of 1 g l^{-1} and 3 g l^{-1} for methylene blue and amaranth respectively and then it remained almost constant. Increase in the percentage of dye removal with adsorbent dosage could be attributed to an increased in the adsorbent surface area, which increased the availability of more adsorption sites. In the further experiments the adsorbent dosage was fixed at 0.5 g l^{-1} for adsorption of methylene blue and at 1 g l^{-1} for amaranth.

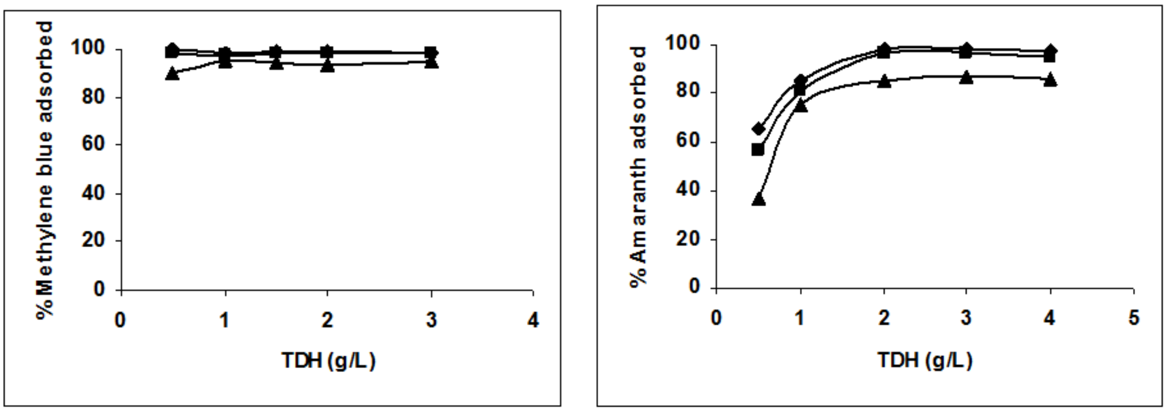

Fig 1 a & b: Effect of adsorbent dosage on Methylene blue and Amaranth biosorption by Tur Dal Husk (TDH)
(♦10 mg/L ■ 20 mg/L ▲50 mg/L)

Effect of contact time

The uptake of methylene blue and amaranth by tamarind husk increased with the increase in contact time and it remained constant after an equilibrium time as shown in Figure 2 a and b.

The percent dye removal at equilibrium time decreased with increase in dye concentration, although the amount of dye removed increased with increase in initial dye concentration. It is clear that the percentage removal of dyes depends on the initial concentration of the dye. The time taken to attain equilibrium for 10, 20, 50 and 100 mg/l amaranth solutions was 40-50 minutes and the time required for methylene blue adsorption is 60 minutes for 10 and 20 ppm solutions and for 50 and 100 ppm solutions, the equilibrium time increased to 70 and 80 minutes respectively.

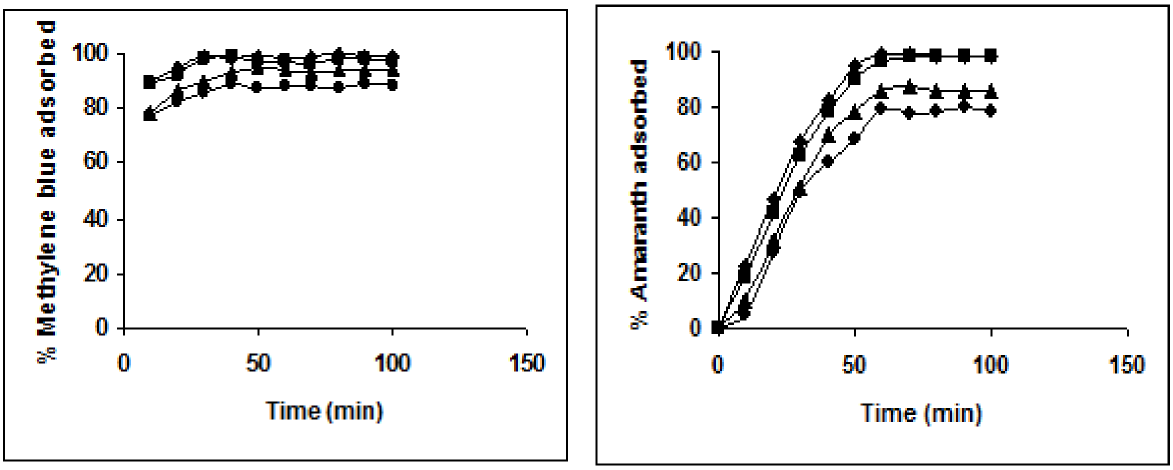

Fig 2 a & b: Effect of agitation time on Methylene blue and Amaranth biosorption by Tur Dal Husk (TDH)
(♦10 mg/L ■ 20 mg/L ▲50 mg/L ●100mg/L)

Mameri *et al* (1999) reported that the available adsorption sites on the biosorbent are the limiting factor for metal uptake. The equilibrium time required by the adsorbents used in the present study is less, compared to others reported in literature. This is significant as equilibrium time is one of the important considerations for economical water and wastewater applications. In process application, this rapid (or instantaneous) biosorption phenomenon is advantageous since the shorter contact time effectively allows for a smaller size of the contact equipment, which in turn directly affects both the capacity and operation cost of the process.

Effect of initial pH

The pH of the solution is a crucial controlling parameter in the biosorption (Javadian et al 2014, Crini et al., 2007). This is possibly due to its impact on both the surface binding sites of the biosorbent and ionization status of the dye molecule in water. Effect of pH on methylene blue removal for different concentrations (10, 20, 50 and 100 mg/L) by tur dal husk show that the percent removal was very efficient in the wide pH range of 6 to 11 (Figure 3 a). However the percent removal decreased when the pH was reduced below 6. The decreased adsorption of methylene blue, a cationic dye at highly acidic pH is probably due to the presence of excess H+ ions competing with dye cations for the adsorption sites. The negatively charged surface sites on the adsorbent favours the adsorption of dye cation due to electrostatic attraction (Saaed et al., 2010; Han et al., 2006).

Figure 3b show that the maximum removal of anionic dye, amaranth occurred at pH 2.0 for the different dye concentrations of 10, 20 and 50 mg/L and the percent removal decreased with increase in initial pH. The surface is positive at low pH where reaction 1 predominates, and is negative at higher pH when reaction (2) takes over.

$$M–OH + H^+ = MOH^{2+}\text{--------------------}(1)$$

$$M–OH + OH^- = MO^- + H_2O \text{ -----------}(2)$$

As shown in the Figure 3a, it is found that the amount of amaranth adsorbed decreased with increasing solution pH.

At low pH, the following equation predominates:

$$MOH^{2+} + Dye^- = MOH^{2+}Dye^- \qquad (3)$$

With an increase in pH, positive charge at the oxide/solution interface decreases hence the adsorbent surface becomes negatively charged and will be associated with positively charged ions of the solution in the following manner:

$$MO^- + K^+ = MO^-K^+ \qquad (4)$$

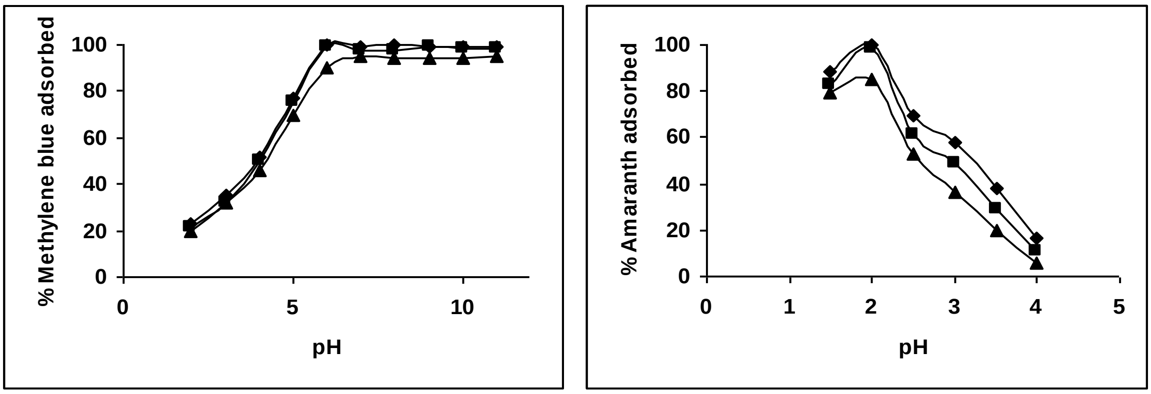

Fig 3a & b: Effect of pH on Methylene blue and Amaranth biosorption by Tur Dal Husk (TDH) (♦10 mg/L ■ 20 mg/L ▲50 mg/L)

Thus, there are no exchangeable anions on the outer surface of the adsorbent at higher pHs and consequently the adsorption decreases. Similar trends were observed in the adsorption of Congo red on red mud (Namasivayam and Arasi, 1997) and wollastonite (Singh, *et al* , 1994) and waste Fe (III)/Cr (III) hydroxide (Namasivayam *et al* , 1994).

Adsorption kinetics

Adsorption kinetics depends on the adsorbate–adsorbent interaction and system condition and has been investigated for their suitability for application in water pollution control.

The kinetic data were treated with the following Lagergren's pseudo-first-order rate equation:

$$\log_{10} (qe\text{-}q) = \log_{10} q_e\text{-} Kad\ t / 2.303 \ldots\ldots\ldots\ldots\ldots(1)$$

where q and qe are amounts of adsorbate adsorbed (mg/g) at time, t (min) and at equilibrium, respectively, K_{ad} is the rate constant of adsorption (l/min). The linear plots of log10 (qe-q) vs t for the dyes were studied at different concentration shows the applicability of the above equation. Values of K_{ad} were calculated from the slope of the linear plots. The rate constant for the dyes generally decreased with increase in adsorbate concentration. The high values of correlation coefficient showed that the data conformed well to the pseudo-first-order rate kinetic model (Tables 2a&b).

Table 2a: Effect of initial Methylene Blue concentration on Lagergren rate constant by Tur dal husk (Adsorbent dose-0.10 g/100 mL; pH – 9.0)

Initial dye concentration	Kad (l/min)	R^2
10	$1.17 X 10^{-1}$	1
20	$1.33 X10^{-1}$	1
50	$6.5 X 10^{-1}$	0.9498
100	$8.9 X 10^{-1}$	0.9862

Table 2b: Effect of initial Amaranth concentration on Lagergren rate constant by Tur dal husk (Adsorbent dose-0.3g/100 mL; pH – 2.0)

Initial dye concentration	Kad (l/min)	R^2
10	$7.1 X 10^{-2}$	0.927
20	$5.45 X10^{-2}$	0.9717
50	$5.7 X 10^{-2}$	0.9709
100	$4.7 X 10^{-2}$	0.9962

Isotherm Modeling

Equilibrium isotherm is described by a sorption isotherm, characterized by certain constants whose values express the surface properties and affinity of the sorbent sorption equilibrium is established when the concentration of sorbate in the bulk solution is in dynamic balance with that at the sorbent interface (Oladoja et al. 2008). The analysis of equilibrium adsorption data by fitting them to different isotherm models is an important step to find the suitable model that can be used for design purposes (Haghseresht and Lu 1998). In the present study, the isotherm data were analyzed using two of the most commonly used equilibrium models, Langmuir (Langmuir, 1918) and Freundlich (Freundlich, 1906) isotherm models. The mathematical expressions are given by Equations 2 and 3, respectively, as follows:

$$q = q_{max}\, b\, C_{eq} / 1 + b\, C_{eq} \quad \text{……………………… (2)}$$

$$q = Kf\, C_{eq}^{\;1/n} \quad \text{…………………………………… (3)}$$

The calculated isotherm constants given in Table 3 were evaluated from the linear plots represented by Equations 4 and 5, respectively for Langmuir and Freundlich isotherms.

$$C_{eq}/q = 1/q_{max}.b + C_{eq}/ q_{max} \quad \text{……………………….. (4)}$$

$$\ln q = \ln K_f + 1/n \ln C_{eq} \quad \text{………………………………….. (5)}$$

The best-fit equilibrium model was determined based on the linear squared regression correlation coefficient R^2. From Table 3, it was observed that the equilibrium sorption data were very well represented by Langmuir isotherms followed by the Freundlich model with high correlation coefficients. Hence, the best fit of equilibrium data in Langmuir isotherm expressions confirm the monolayer coverage process of methylene blue and amaranth by tamarind husk. Furthermore, the value of Freundlich exponent n in the range of 1–10, indicates a favourable adsorption (Ho and McKay, 1998). Also, high adsorption capacity indicates the strong electrostatic force of attraction between dye molecules and biosorbent binding-sites. The comparison of results of this work with the others found in the literature showed that tur dal husk has a significantly high adsorption capacity for binding the dyes.

Table: 3 Sorption isotherm constants and coefficients of determination for adsorption of dyes by TDH

	Langmuir equation			Freundlich equation		
	Q_{max} (mg/g)	b (l/mg)	R^2	K_F (mg/g)	n	R^2
Amaranth	45.08	0.026	0.99	6.61	3.09	0.93
Methylene Blue	216.68	0.011	0.98	6.79	1.72	0.98

Desorption studies

Both incineration and land disposal represent possible options for final disposition of spent adsorbent material. However, both methods directly or indirectly pollute the environment. If regeneration of dyes from the spent adsorbent were possible then it would not only protect the environment but also help recycle the adsorbate and adsorbent and hence contribute to the economy of wastewater treatment.

Desorption experiments were carried out at different pH values. Desorption of the dyes with water was not significant. If the adsorption is by physical bonding then the loosely bound solute can be easily desorbed with distilled water in most of the cases (Agarwal *et al.*, 2006). Hence, physical adsorption is ruled out. Among the dyes, the percentage of amaranth desorbed was the highest with increase in pH. About 12.32% of the dye was desorbed from tur dal husk. The percentage of methylene blue desorbed did not exceed 2.45%. (Figure 4a &b).

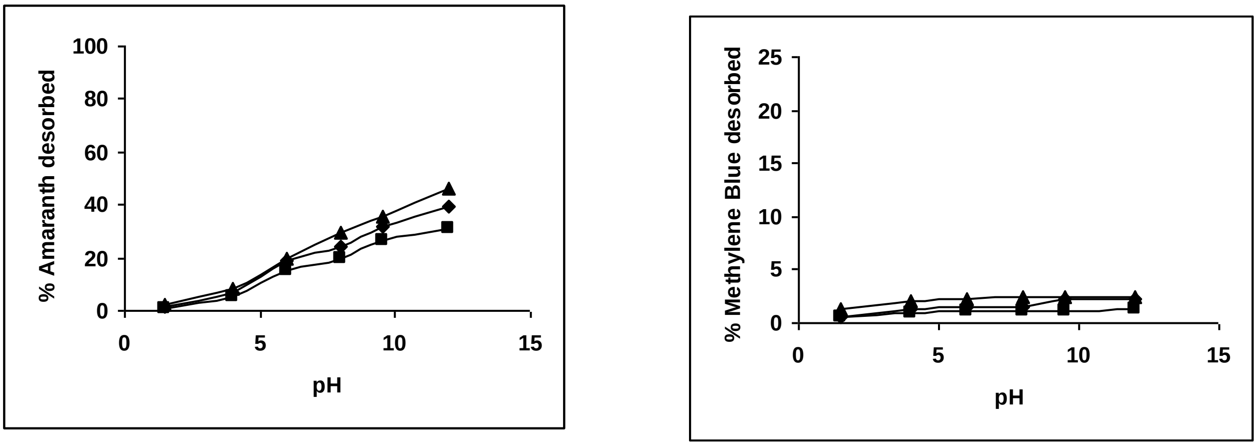

Fig 4 a & b: Effect of pH on the desorption of Amaranth and Methylene Blue by Tur Dal Husk (TDH) (♦10 mg/L ■ 20 mg/L ▲ 50 mg/L)

CONCLUSION

The results of the present investigation show that tur dal husk is an effective adsorbent for the removal of methylene blue and amaranth dyes from aqueous solutions, and its adsorption capacity is quite comparable to the other adsorbents reported in literature. Tur dal husk was selected for studying adsorption due to its availability in India, as well as to assess the possibility of utilising an agricultural waste for dye removal. The results show that initial dye concentrations, pH and adsorbent dose highly affect the dye uptake capacity of adsorbent. The equilibrium has been analyzed using Freundlich and Langmuir adsorption isotherms. The kinetics of adsorption followed a pseudo first order rate reaction. The calculated isotherm constants were used to compare the adsorptive capacities of adsorbents for dye removal. Thus the use of tur dal husk as an adsorbent can be viewed as an effective waste management strategy.

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