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RESEARCH ARTICLE

EVALUATING THE EFFICIENCIES OF NATURALLY AVAILABLE LOW COST ADSORBENTS FOR REMOVAL OF METHYL RED DYE

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ABSTRACT

Colour is the common notable pollutant in wastewater and is caused by variety of dyes, which are stable and less biodegradable. The elimination of the azo dye, methyl red, from aqueous solution by low cost naturally available adsorbents like neem leaves and papaya seeds were studied in a batch process. The experiments were conducted with both adsorbents to study the effect of different parameters like initial dye concentration, adsorbent dose, time and pH on dye removal efficiencies. Further the suitability of the adsorbents was tested by fitting the adsorption data with Freundlich and Langmuir adsorption isotherms. At a dye concentration of 500 mg/L with adsorbent concentration of 0.5 g, dye removal efficiency with neem leaves was 62.5% and papaya seeds was 87.5 %. The adsorption isotherm data for methyl red was best fitted to Freundlich isotherm using neem leaves adsorbent with R^2 = 0.865 and Langmiur isotherm using papaya seed adsorbent with monolayer adsorption capacity of 17.241 mg/g. The dimensionless constant separation factor R_L indicates that adsorption is more favourable with papaya seeds adsorbent than neem leaves adsorbent.

INTRODUCTION

Wastewater effluents from different industries such as textiles, rubber, paper and plastics, contain several kinds of synthetic dyestuffs. Discharging even a small amount of dye into water bodies can affect aquatic life and food webs due to the carcinogenic and mutagenic effects of synthetic dyes (Sharma *et al.*, 2005; Tahir *et al.*, 2008; Vinoth *et al.*, 2010; Abbas *et al.*, 2011; Karthik *et al*, 2012). A majority of the used dyes are azo dyes which are bright in colour due to the presence of one or several azo (–N=N–) groups associated with substituted aromatic structures (Vinoth *et al.*, 2010). Also these dyes are difficult to degrade due to their complex aromatic structures and tend to persist in the environment (Tahir *et al.*, 2008; Vinoth *et al.*, 2010). Various physico – chemical and biological methods used for dye removal include chemical oxidation, reverse osmosis, ion exchange, ozonation, membrane filtration, coagulation, adsorption and microbial degradation (Voudrias *et al.*, 2002; Tahir *et al.*, 2008; Abbas *et al.*, 2011; Fathi *et al.*, 2011). Among these, adsorption is quite popular due to simplicity, cost effectiveness, ease of operation, insensitivity towards toxic substances and high efficiency, as well as the availability of a wide range of adsorbents. It has proved to be an effective method for removal of dye from wastewater (Sharma *et al.*, 2005; Karthik *et al*, 2012). Activated Carbon is the most commonly used adsorbent for its efficiency in adsorbing a wide variety of adsorbates, but its large scale use is limited owing to high cost, at least in developing countries (Voudrias *et al.*, 2002; Sharma *et al.*, 2005; Santhi *et al.*, 2010; Fathi *et al.*, 2011; Karthik *et al*, 2012). Thus the need of the hour is to test the use of low cost naturally available agro based waste as adsorbents for successfully treating colour from dyes. Several researchers have used different low cost adsorbents for removing dyes like neem leaves (Bhattacharya *et al.*, 2003; Sharma *et al.*, 2005; Tahir *et al.*, 2008; Karthik *et al*, 2012; Pandhare *et al.*, 2013), papaya seeds (Nasuha *et al.*, 2011), *Annona squmosa* seed from a hedge plant (Santhi *et al.*, 2010), Banana trunk fibres (Rosemal *et al.*, 2010),Yam leaf fibres (Vinoth *et al.*, 2010), Cassave peel waste (Adowei *et al.*, 2012), Hazelnut seeds (Fathi *et al.*, 2011). This study is done with a well known azo dye, Methyl red (2-(N,N-Dimethyl-4-aminophenyl) azobenzenecarboxylic acid) due to intense colour and carcinogenic effects. This research is carried out to study the comparative efficiencies of adsorbents made from two naturally available low cost materials, neem leaves and papaya seeds in removing colour of methyl red dye due to paucity of work done by other researchers with methyl red using these adsorbents.

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MATERIALS AND METHODS

Preparation of Adsorbent

Initially Neem leaves are collected from the Sir M Visvesvaraya Institute of Technology college campus. These are washed with distilled water for 3-4 times to remove dirt and other contaminants. Then the leaves are kept inside the hot air oven for drying at 80^oC until the leaves are turned pale yellow. Leaves are then grinded and the powder obtained was washed with distilled water several times to remove excess color and turbidity. These were dried again in hot air oven till completely dry. Then the leaf powder was screened through 25 mesh size sieve to get uniform particle size distribution of raw neem leaf adsorbent. The prepared adsorbent is stored in bottles for further use. Papaya seeds are collected from fresh papaya fruit and the seeds are washed with distilled water for 3-4 times to remove other contaminants. Seeds are then boiled at 100^{o}C for 30 min and then dried in a hot air oven at 80^{o}C for 48 hours. Seeds are crushed and passed through 25 mesh size sieve to get uniform particle size distribution of raw papaya seed adsorbent. The prepared adsorbent is stored in bottles for further use.

Plate 1: Papaya seed adsorbent

Plate 2: Neem leaves adsorbent

Preparation of Adsorbate

A stock solution of methyl red was prepared by dissolving 1 g of methyl red dye in 1 L of distilled water by constant stirring in a magnetic stirrer at 400 rpm and 80^{o}C for 1 hour for complete mixing. The solution is then cooled and filtered to get the clear stock solution and this is stored for further use.

Batch Adsorption Studies

The effect of initial dye concentration, adsorbent dose, contact time of agitation and pH of adsorbate were studied using 100 mL of methyl red dye solution in 250 mL standard conical flasks and required amount of adsorbents were added to each flask. The solutions were agitated at a constant speed and temperature using Secor India Griffin Flask Shaker. After agitation the solutions were centrifuged at 7000 rpm for 15 minutes to remove colloidal materials. Then the absorbance of the supernatant solution was found out using Systronics Photoelectric Colorimeter 114, to estimate the final dye concentration at 315 nm and 515 nm since methyl red absorbs light of two different wavelengths (George *et al.*, 2007). The percentage removal of adsorbate adsorbed on the adsorbent is given as

$$\%Dye\ Removal = \frac{C_o - C_f}{C_o} * 100 ------------- (1)$$

Where C_o = Initial concentration of dye (mg/L)
C_f = Final concentration of dye (mg/L)

Effect of Initial Dye Concentration

For this aliquots of stock methyl red solution (100 mg/L – 500 mg/L) were taken in 5 conical flasks and 0.5 g of adsorbent was added to each flask. These were kept in the shaker for 100 minutes. After agitation the solutions were centrifuged at 7000 rpm for 15 minutes to remove colloidal materials. Then the absorbance of the supernatant solution was found to estimate the final dye concentration. The amount of methyl red adsorbed per unit weight of adsorbent was calculated as

$$Q = \frac{(C_o - C_f)*V}{W} mg/g --------------- (2)$$

Where C_o = Initial concentration of dye (mg/L)
C_f = Final concentration of dye (mg/L)
V = Volume of final solution (L)
W = Quantity of adsorbent (g)

Effect of Adsorbent

For this 300 mg/L of stock methyl red solution were taken in 5 conical flasks and 0.1, 0.2, 0.3, 0.4, 0.5 g of adsorbent was added to each flask. These were kept in the shaker for 100 minutes. After agitation the solutions were centrifuged at 7000 rpm for 15 minutes to remove colloidal materials. Then the absorbance of the supernatant solution was found to estimate the final dye concentration.

Effect of Time of Agitation

For this 300 mg/L of stock methyl red solution were taken in 5 conical flasks and 0.5 g of adsorbent was added to each flask. These were kept in the shaker for different time intervals of 20, 40, 60, 80 and 100 minutes. After agitation the solutions were centrifuged at 7000 rpm for 15 minutes to remove colloidal materials. Then the absorbance of the supernatant solution was found to estimate the final dye concentration.

Effect of pH

For this 300 mg/L of stock methyl red solution were taken in 20 conical flasks, 10 each for neem leaves and papaya seeds adsorbents. The pH of 5 flasks was adjusted to acidic range (4.5 to 6.5) by adding 0.1N HCl and 0.5 g of adsorbent was added to each flask. Similarly, the pH of another set of 5 flasks was adjusted to alkaline range (7.5 to 9.5) by adding 0.1N NaOH and 0.5 g of adsorbent was added to each flask. These were kept in the shaker for fixed time intervals of 100 minutes. After agitation the solutions were centrifuged at 7000 rpm for 15 minutes to remove colloidal materials. Then the absorbance of the supernatant solution was found to estimate the final dye concentration.

RESULTS AND DISCUSSIONS

Effect of Initial Dye Concentration

As seen in Figure 1, for a fixed adsorbent dose of 0.5 g, the percentage dye removal decreased for neem leaves and increased with papaya seeds with increase in initial concentration of methyl red dye (100 – 500 mg/L). The rapid initial stage of dye removal for neem leaves is attributed to the abundant availability of active sites on adsorbent and with gradual occupancy of these sites, the sorption becomes less efficient. The results are in accordance with previous studies (Bhattacharya *et al.*, 2003; Rosemal *et al.*, 2010; Karthik *et al.*, 2012). However in case of papaya seeds, the initial stage of dye removal is slow (Figure 1). This might be due to the fact that the dye molecules have to encounter the boundary layer effect before diffusing from boundary layer film onto adsorbent surface and then its diffusion into porous structure of adsorbent. The percentage dye removal for neem leaves is 62.5 % and papaya seeds is 87.5 % at a dye concentration of 500 mg/L. Figure 2 indicates an increase in adsorption capacity for both papaya seeds and neem leaves with increase in dye concentration. This is because increasing the initial dye concentration enhances the number of collisions between dye molecules and adsorbent, which increases the adsorption process and this finding, is in line with research done by (Bhattacharya *et al.*, 2003 and Vinoth *et al.*, 2010).

the dye removal increased as the adsorbent dose increased for both the adsorbents. This is attributed to the fact that increase in adsorbent dose provides increase in pores available for adsorption and also the surface area gets increased. Similar findings have been observed by researchers Rosemal *et al.*, 2010; Vinoth *et al.*, 2010; Abbas *et al.*, 2011. In this study, the maximum dye removal of 80% was obtained with 0.5 g of papaya seeds and 0.4 g of neem leaves adsorbents.

Effect of Time

The effect of contact time was investigated with both papaya seeds and neem leaves adsorbents to determine the time taken

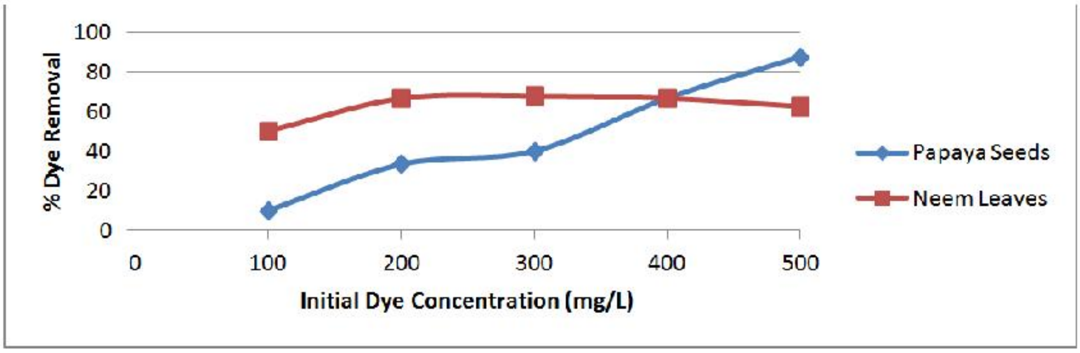

Figure 1. Effect of Initial Dye Concentration on Adsorption with at a Constant Adsorbent Dose of 0.5 g

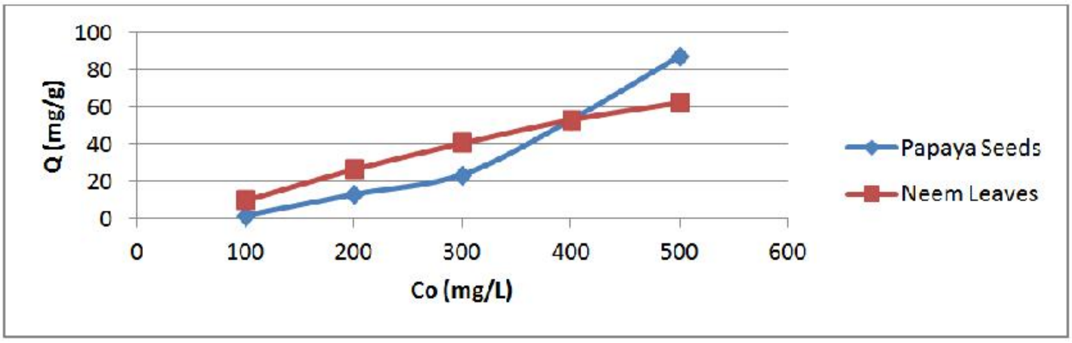

Figure 2. Effect of Initial Dye Concentration on Adsorption Capacity with at a Constant Adsorbent Dose of 0.5 g

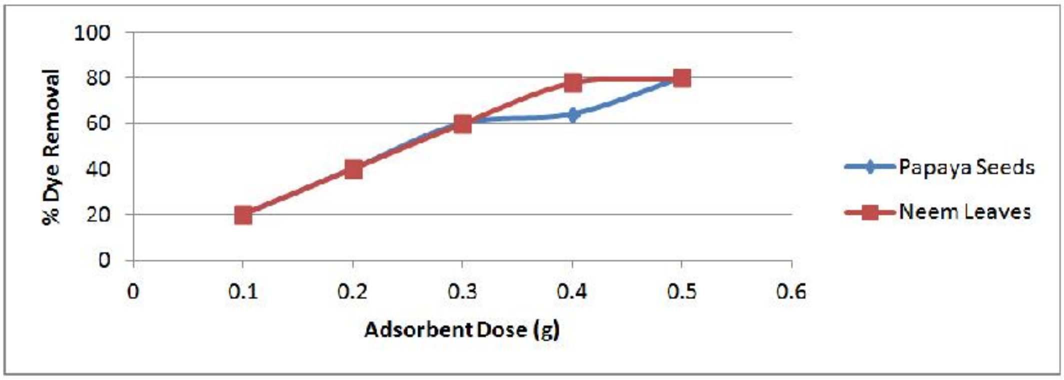

Figure 3. Effect of Adsorbent Dose on Initial Dye Concentration of 300 mg/L on Adsorption

Effect of Adsorbent Dose

Removal of colour directly depends on the mass of adsorbent. The study of adsorbent dosages for removal of methyl red dye from aqueous solution was carried out using papaya seeds and neem leaves adsorbents ranging from 0.1 to 0.5 g and fixing the dye concentration at 300 mg/L. It is seen in Figure 3 that

by methyl red dye at concentration of 300 mg/L to reach equilibrium. The equilibrium time is the time after which no significant adsorption takes place. Figure 4 shows a rapid dye removal percentage at initial time. Increase in removal efficiencies with increase in time of contact can be attributed to the fact that more time becomes available for the dye to make an attraction complex with the adsorbents. This is similar to the findings of researchers Abbas *et al.*, 2011 and Karthik *et al.*, 2012. The equilibrium time obtained for 80 % dye removal was achieved with papaya seeds at 100 minutes and neem leaves at 80 minutes.

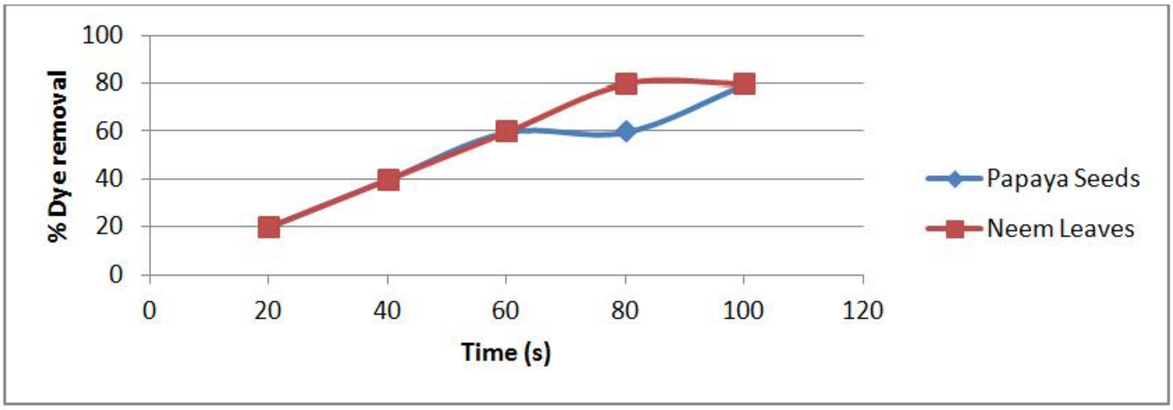

Figure 4. Effect of Time on Initial Dye Concentration of 300 mg/L on Adsorption with at a Constant Adsorbent Dose of 0.5 g

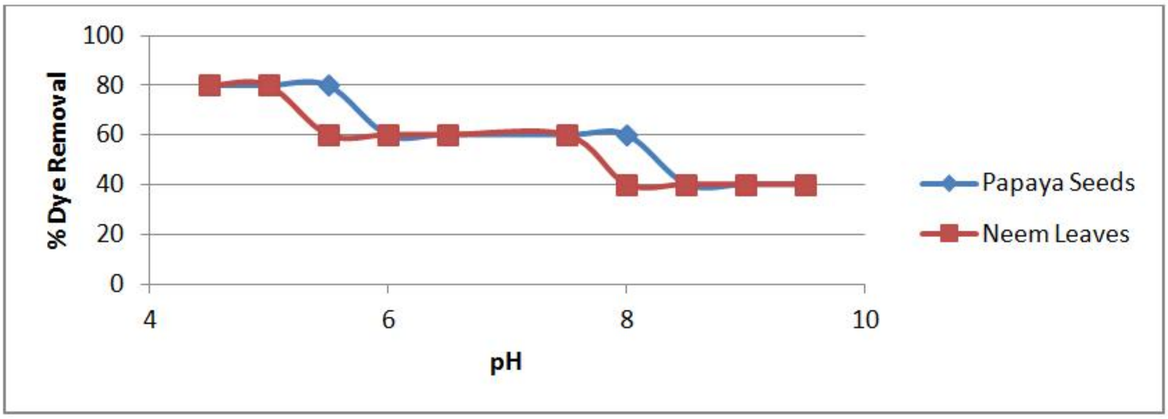

Figure 5. Effect of pH on Initial Dye Concentration of 300 mg/L on Adsorption with at a Constant Adsorbent Dose of 0.5 g

Effect of PH

The efficiency of adsorption is dependent on the pH of solution since variation in pH leads to the variation in the surface properties of the adsorbent and the degree of ionization (Vinoth *et al.*, 2010). Figure 5 shows the pH variation over the pH range of 4.5 to 9.5 for both papaya seeds and neem leaves adsorbents. It is observed that the percentage removal decreases with increase in pH for both the adsorbents. The maximum dye removal was 80 % with neem leaves for the dye pH upto 5.0 and pH 5.5 for papaya seeds. Hence it can be concluded that the acidic range of dye is favourable with both the adsorbents. This is in accordance with the studies carried out by Karthik *et al.*, 2012. The lower percentage removal in alkaline range may be due to competition from hydroxide ions with the anionic dye molecules for the adsorption sites and this is also in line with the studies done by Vinoth *et al.*, 2010.

Adsorption Isotherms

These are prerequisites to understand the nature of the interaction between adsorption and the adsorbent used for the removal of pollutants. An adsorption isotherm describes the relationship between the amount of adsorbate taken up by the adsorbent and the adsorbate concentration remaining in solution (Fathi *et al.*, 2011). The parameters obtained from the different isotherm equations provide important information on the surface properties of the adsorbent and its affinity of adsorbent to the adsorbate (Fathi *et al.*, 2011). The important isotherms applied in this study are the Freundlich and Langmuir isotherms. The Freundlich Isotherm Equation is given as

$$Q = KC_f^{1/n} \quad ------------------(3)$$

The logarithmic form of Freundlich equation can be written as

$$\log Q = \log K + \frac{1}{n}\log C_f \quad ------------(4)$$

The plot of log Q versus log C_f gives a straight line with slope of 1/n and intercept of log K. K denotes the adsorption capacity of the adsorbent. It is defined as the adsorption or distribution coefficient and represents the quantity of dye adsorbed onto the adsorbents for unit equilibrium concentration. 1/n indicates the adsorption intensity of the dye onto the adsorbent or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. A value for 1/n below 1 indicates a normal Langmuir isotherm while 1/n above 1 is indicative of cooperative adsorption (Santhi *et al.*, 2010, Fathi *et al.*, 2011). Figures 6 and 7 show the Freundlich isotherm plots of methyl red dye using papaya seeds and neem leaves adsorbents and it helped in deciding whether the isotherm fits the adsorption experimental data. It is seen that

Freundlich isotherm plot is better fitted with neem leaves adsorbent than papaya seeds adsorbent.

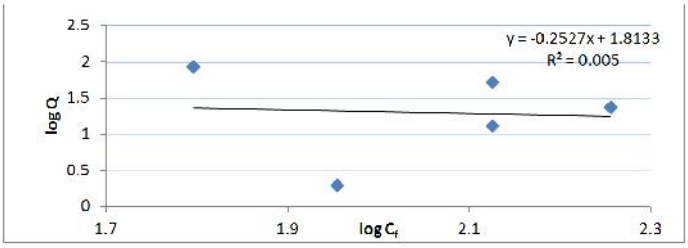

Figure 6. Freundlich Isotherm Plot for the Adsorption of Methyl Red Dye with concentrations 100 – 500 mg/L with a constant Papaya Seeds Adsorbent Dose of 0.5 g

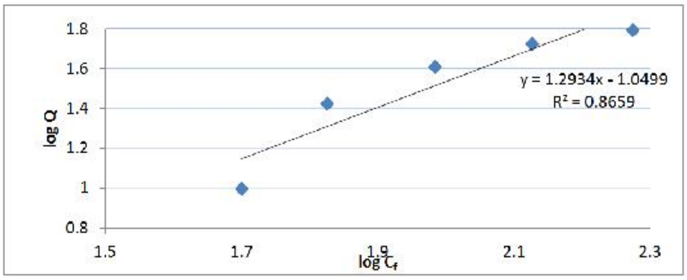

Figure 7. Freundlich Isotherm Plot for the Adsorption of Methyl Red Dye with concentrations 100 – 500 mg/L with a constant Neem Leaves Adsorbent Dose of 0.5 g

The goodness of fit criterion (R^2 value) was computed by linear regression for both adsorbents and is presented in table 1. It is observed from table 1 that 1/n value of papaya seed adsorbent is less than one, indicating it follows the normal Langmuir isotherm. This is further validated from a very low R^2 value. Similarly, neem leaves adsorbent has a high R^2 value and 1/n value is more than 1, indicating the existence of cooperative adsorption, which means that the already adsorbed molecules tend to enhance the adsorption of other molecules. This implies that the adsorbate-adsorbent interactions are of less importance than the adsorbate-adsorbate interactions.

Table 1. Freundlich coefficients for the Adsorption of Methyl Red Dye with concentrations 100 – 500 mg/L

Adsorbents	1/n	n	K	R^2
Papaya Seeds	-0.252	-3.968	65.013	0.005
Neem Leaves	1.293	0.773	0.89	0.865

The Langmuir isotherm is based on the assumption that the adsorption process takes place at specific homogeneous sites within the adsorbent surface and that once a dye molecule occupies a site, no further adsorption can take place at that site. In other words, the adsorption process is monolayer in nature (Voudrias *et al.*, 2002; Fathi *et al.*, 2011). The Langmuir equation is represented in the linear form as follows

$$1/Q = 1/b + 1/KbC_f \text{ -------------- } (5)$$

The constants K and b can be found by plotting a graph of 1/Q vs. $1/C_f$. The slope of the graph gives 1/Kb and intercept is 1/b. K is a direct measure for the intensity of the sorption process (L/mg) and b is a constant related to the area occupied by monolayer of adsorbate (mg/g), reflecting the sorption capacity (Voudrias *et al.*, 2002). Figures 8 and 9 show the Langmuir isotherm plots of methyl red dye using papaya seeds and neem leaves adsorbents and it helped in deciding whether the isotherm fits the adsorption experimental data.

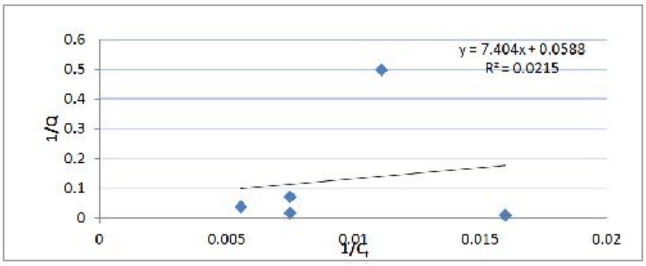

Figure 8. Langmuir Isotherm Plot for the Adsorption of Methyl Red Dye with concentrations 100 – 500 mg/L with a constant Papaya Seeds Adsorbent Dose of 0.5 g

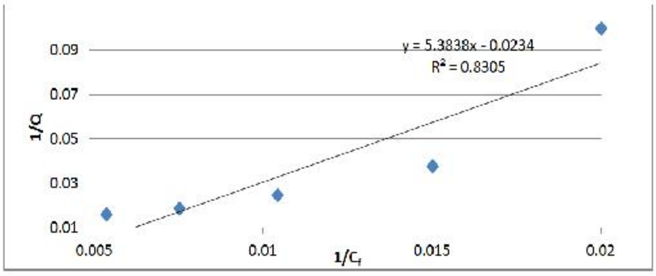

Figure 9: Langmuir Isotherm Plot for the Adsorption of Methyl Red Dye with concentrations 100 – 500 mg/L with a constant Neem Leaves Adsorbent Dose of 0.5 g

The goodness of fit criterion (R^2 value) was computed by linear regression for both adsorbents and is presented in table 2. It is observed that Langmuir isotherm is appropriate for the adsorption of dye by both papaya seeds and neem leaves adsorbents. However negative values for Langmuir isotherm constants are obtained with neem leaves adsorbent and it indicates the inadequacy of the isotherm to explain the adsorption process since these constants are indicative of the surface binding energy and monolayer coverage. This finding is similar to the research work of Maroof *et al.*, 2003.

Table 2. Langmuir coefficients for the Adsorption of Methyl Red Dye with concentrations 100 – 500 mg/L

Adsorbents	1/b	b	K	R^2
Papaya Seeds	0.058	17.241	0.00783	0.021
Neem Leaves	-0.023	-43.478	-0.00427	0.830

The essential features of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor R_L given by the equation

$$R_L = \frac{1}{1 + KC_0} \text{ ---------------- } (6)$$

The parameter R_L indicates the shape of the isotherm (He *et al.*, 2010; Rosemal *et al.*, 2010; Sharma *et al.*, 2005). The effect of the shape can be used to predict whether an adsorption system is favourable or unfavourable given in Table 3.

Table 3. Relationship of R_L with type of isotherm

Values of R_L	Type of Isotherm
$R_L > 1$	Unfavourable
$R_L = 1$	Linear
$0 < R_L < 1$	Favourable
$R_L = 0$	Irreversible

Source: He *et al.*, 2010

papaya seed adsorbent with monolayer adsorption capacity of 17.241 mg/g. The dimensionless constant separation factor R_L indicates that adsorption is more favourable with papaya seeds adsorbent than neem leaves adsorbent. It is thus concluded that at higher concentration of methyl red dye, papaya seeds are more efficient than neem leaves as adsorbents for removal of colour.

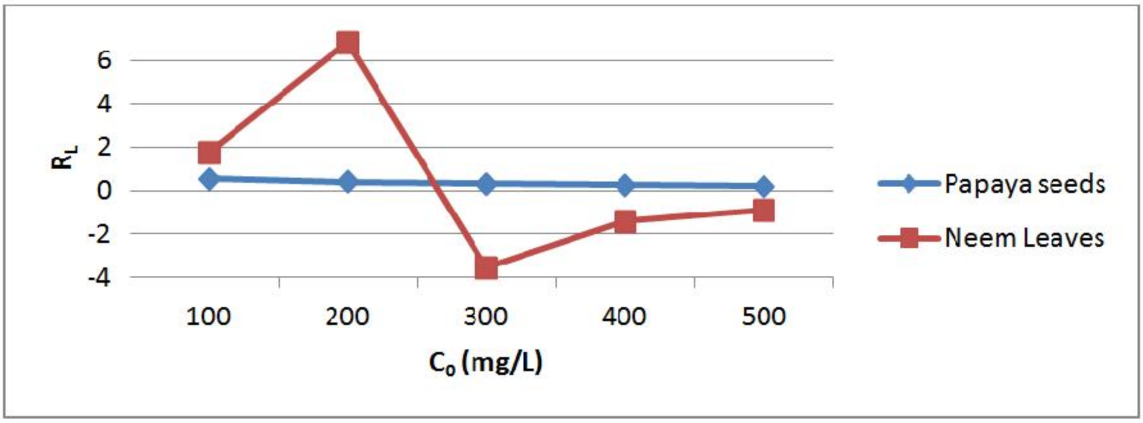

Figure 10. Plot of R_L against initial Methyl

Red Dye concentrations

Figure 10 indicates that adsorption is more favourable with papaya seeds adsorbent than neem leaves adsorbent as the values of R_L lie between 0 and 1 as shown in Table 3. The R_L values for papaya seeds are close to 1, indicating that the adsorption of methyl red dye on papaya seeds adsorbent is linear in nature.

Conclusion

In the present research, an attempt is made to study the effectiveness of two low cost naturally available adsorbents prepared from neem leaves and papaya seeds for removal of colour of methyl red dye. From the experimental data it is found that dye removal efficiency with neem leaves is 62.5% and papaya seeds is 87.5 % at a dye concentration of 500 mg/L with adsorbent dose of 0.5 g. This study also revealed that maximum dye removal of 80% was obtained with 0.5 g of papaya seeds and 0.4 g of neem leaves adsorbents at a fixed dye concentration of 300 mg/L. Experiments with 300 mg/L dye at adsorbent dose of 0.5 g showed equilibrium time obtained for 80 % dye removal was achieved with papaya seeds at 100 minutes and neem leaves at 80 minutes. The efficiency of adsorption is dependent on the pH of solution since variation in pH leads to the variation in the surface properties of the adsorbent and the degree of ionization. From this study, it is found that maximum dye removal was 80 % with neem leaves for the dye pH upto 5.0 and pH 5.5 for papaya seeds. An adsorption isotherm describes the relationship between the amount of adsorbate taken up by the adsorbent and the adsorbate concentration remaining in solution. In this study two popular adsorption isotherms studied are Freundlich and Langmuir isotherms. This study showed that the adsorption isotherm data for methyl red dye was best fitted to Freundlich isotherm using neem leaves adsorbent with R^2 = 0.865 and Langmiur isotherm using

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