Methyl Red Dye Removal From Aqueous Solution by Adsorption on Rice Hulls

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Abstract

In this study agricultural waste, grounded rice hulls was examined as sorbent material to remove methyl red dye from aqueous solutions. Batch studies were performed to evaluate the effects of various parameters:(contact time, pH, initial dye concentration, adsorbent dosage and particle size of adsorbent), on adsorption capacity of methyl red dye.

Rice hulls was found to be effective in removing methyl red dye and reached equilibrium in (100 min). The adsorption capacity was found to be pH dependant and it increases with the increasing of the adsorbent dosage and initial concentration of methyl red dye. The adsorption capacity decreases with increased particle size of rice hulls.

Equilibrium isotherms were analyzed by freundlich and Langmuir isotherm equations using correlation coefficients. Adsorption data were well described by the Langmuir and freundlich models. The results indicated that the rice hulls could be an alternative material in place of more costly adsorbents used for dye removal.

Keywords: Rice Hulls, Methyl red, Isotherm models.

الخلاصة:

في هذه الدراسة استخدمت الفضلات الزراعية وهي قشور الرز المطحونة كمادة مازة (مزيلة) في ازالة صبغة المثيل الاحمر من المحاليل المائية. تم دراسة نظام الدفعات في تخمين تأثير معاملات مختلفة(:وقت التلامس, قيم pH للمحاليل، التركيز الاولي للصبغة، كمية المادة المازة وحجم حبيبات المادة المازة) على قابلية امتزاز صبغة المثيل الاحمر.

وجد ان قشور الرز فعالة وتصل حد التوازن بعد (١٠٠) دقيقة. ان سعة الامتزاز تعتمد على pH للمحلول وتزداد هذه السعة بزيادة كمية المادة المازة والتركيز الاولي للصبغة، وتقل قابلية الامتزاز بزيادة حجم حبيبات قشور الرز.

تم مقارنة معامل التوازن مع معادلتي فرندليش ولانكمير بالاعتماد على معامل الترابط (R^2). بيانات التوازن والامتزاز مترابطة مع معادلتي فرندليش ولانكمير. اوضحت النتائج ان قشور الرز ممكن ان تكون مادة بديلة لكثير من المواد المازة (المزيلة) ذات الكلفة العالية في ازالة الصبغة.

الكلمات المفتاحية: قشور الرز، صبغة المثيل الاحمر، موديلات التوازن.

1. Introduction

Many materials can cause pollution like inorganic salts, acids, alkaline, organic matters, suspended solids, floating (solid or liquid), heat, color, toxic materials,microorganisms, radioactive materials and foam-reducing matters (Nemerow, 1971).

Pure water is colorless, but water in nature often colored by foreign substances. Color contributed by dissolved solid that remain after removal of suspended matter is known as true color. Highly colored water is unsuitable for laundry, dying, paper making, dairy production and other industries. Thus the color in water effect its marketability for both domestic and industrial use (Peavy *et al.*, 1986).

Water is one of the most important commodities which man has exploited than other resources for sustenance of his life. Water can be regarded polluted when it gets changed in its quality or composition either naturally or as a result of human activities (Goel, 2006).

Most dyes are inert and non-toxic at the concentration discharged into the receiving water; however, they impart color undesirable to the water user. The release of these colored wastewaters in the ecosystem is a dramatic source of esthetic pollution, of eutrophication and of perturbations in the aquatic life (Mane *et al.*, 2007).

Colored waste water can be treated by several methods, like coagulation, membrane separation, biological treatment, adsorption, chemical oxidation (using chlorine, Fenton's reagent and ozone) and photo-oxidation (using UV light, TiO2 in presence of visible light). Adsorption among these methods is included in almost all waste water treatment facilities as a polishing stage for meeting the effluent discharge standards (Sarkar and Bandyopadhyay, 2010).

Colour is a characteristic of wastewater, and can easily be detected. Elimination of colour from dyebearing wastewater is one of the major environmental problems, because of difficulties in treating such wastewaters by conventional methods, as most of the dyes are stable to heat and oxidizing agents. Colour pollution may cause potential toxicity and turbidity problems, thus contributing significantly to the pollution of aquatic ecosystems (Adowei *et al*., 2012).

Recently, use of naturally occurring low cost and harmless material for removing of trace contaminants from colored wastewater has attracted extensive attention (Noroozi *et al*., 2004). Raw materials, which are either abundant or wastes from other industrial and agricultural operations, can be used as biosorbents. Consequently, various potential adsorbents such as silkworm pupa, orange peel, rice husk ash, sugarcane bagasse and peel waste have been implemented for the removal of dyes and specific types of pollutants from water (Noroozi *et al*., 2004; Velmurugan *et al*., 2011; Gullpalli *et al*., 2011; Azhar *et al*., 2005 and Adowei *et al,* 2012).

Rice hulls is considered an agricultural waste. In this study, it was used as low-cost adsorbent to remove methyl red from aqueous solution without any additives.

Rice hulls (husks) are the hard protecting coverings of grains of rice an agricultural waste material (Morcali *et al*.,2013; Noor and Rohasliney,2012).It is made of hard materials, including opaline silica and lignin in order to protect the seed during the growing season.The husk is mostly indigestible to humans. During the milling process, the husks are removed from the grain to create brown rice; the brown rice is then milled further to remove the bran layer to become white rice (Noor and Rohasliney, 2012).

Batch experiments were carried out to determine adsorption capacity of adsorbent and the behaviors at different conditions such as pH, initial concentration of adsorbate, contact time, adsorbent dose, and particle size of adsorbent. The equilibrium of adsorption was modeled. Results of this study will be useful for future in using this material as a low-cost adsorbent for the removal of similar dyes.

El-Maghraby and EL Deeb., (2011) described the use of grounded rice hulls as adsorbent material. Aqueous solutions of various methylene blue concentrations (5-25 mg/l) were shaken with certain amount of adsorbents to determine the adsorption capacity. Both treated and untreated rice hulls were used for methylene blue adsorption. The effects of adsorbent dose, initial PH, initial dye concentration and contact time on dye removal have been studied. Pretreatment of rice hulls with citric acid did not reveal any beneficial effect. The results showed that ground rice hulls can be considered as potential adsorbent for methylene blue removal from dilute aqueous solutions.

Morcali *et al*.,(2013) investigated Rice hulls,a biomass waste product, and Lewatit TP 214,a thiosemicarbazide sorbent, as adsorbents for the adsorption of platinum(IV)ions from synthetically prepared dilute chloroplatinic acid solutions. The effects of the different adsorption parameters on the percent adsorption were studied in detail for batch sorption. The adsorption equilibrium data were best fitted with the Langmuir isotherm model. The maximum monolayer adsorption capacities, Qmax, at 25 °C were found to be 42.02 and 33.22 mg/g for the rice hulls and Lewatit TP 214, respectively. The results indicated that the rice hulls could be effectively used for the removal of platinum from aqueous solution.

Gulipalli *et al.*,(2011) involves the study of Se(IV) adsorption onto rice husk ash (RHA). The adsorbents were coated with the ferric chloride solution for the effective removal of selenium Se(IV). Batch experiments were carried out to determine the effect of various factors such as adsorbent dose (w), initial pH, contact time (t), and temperature (T) on adsorption process. Se(IV) adsorption is high at low pH values, and decreased with the rise in initial pH. Temperature study shows that the uptake of Se(IV) is more at 293 K within the temperature range studied. Equilibrium isotherms have been analyzed using Langmuir isotherm, Freundlich isotherm and Temkin isotherm.

Taha *et al.*, (2012) investigated the use of activated rice husk as adsorbent for the removal of Janus Green B dye from industrial wastewater. Adsorption studies were carried out in a batch process with adsorbent dose , contact time , pH , initial dye concentration and adsorbents' particle size at ambient temperature .The well Known Langmuir and Freundlich isotherm models were applied for the equilibrium adsorption data and the various isotherm parameters were evaluated .

2. Aim of study

The main aims of this work can be summarized by:

-Identify the capability of using rice hulls as adsorbent for the removal of methyl red dye from aqueous solution.

-Determination the effect of different adsorption parameters like: adsorbent dose, contact time, pH solution, initial dye concentration, and particle size on the adsorption capacity of the adsorbent used.

-Calculation of the adsorption capacity using Langmuir and Freundlich isotherm models for the adsorbent.

3. Materials and methods:

3.1. Adsorbent (Rice Hulls):

Rice hulls is a widely available waste , obtained from the rice mills during the milling process for the separation of rice from the grain .In the present study, rice hulls with fresh biomass was collected from nearby rice mill. This obtained rice hulls was grounded into fine powder and washed four times with tap water. The rice hulls were dried at 105^{o}C for 2hr in the oven (EL-Maghraby and EL Deeb, 2011). The rice hulls was sieved to (< 1.18mm) and used for Batch experiment.

3.2. Adsorbate (Methyl red):

Methyl red (MR) dye was chosen as a model system because of its intense colour in aqueous system and low biodegradability due to the presence of benzene rings. Methyl red is an azo dye (also known as (2-{(E)-[4-(dimethylamino) phenyl]diazenyl}benzoic acid) is a pH indicator, it is red in pH under 4.4 yellow in pH over 6.2, and orange in between. The molecular structure of Methyl red is depicted in Figure (1), (Adawei *et al*, 2012). The chemical formula of methyle red is $C_{15}H_{15}N_3O_2$ with molecular weight of 269.31g/mole and melting Point between 179-182 °C (Azhar *et al.*, 2005).

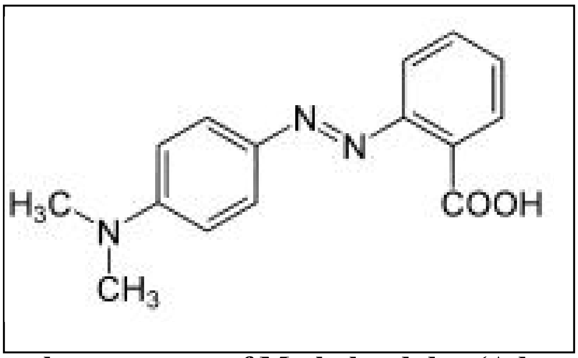

Fig. (1): Molecular structure of Methyl red dye (Adawei *et al.*, 2012)

3.3. Preparation of dye solution :

The stock solution of the dye was prepared by dissolving 0.5g of dye in 1000ml distilled water to make a stock solution of 500 mg/L.The experimental solution was prepared by diluting definite volumes of the stock solution to get the desired concentration. For absorbance measurements a spectrometer UV.VIS double beam (JENWAY) was used.

3.3. Adsorption parameters studied:

- Adsorbent dosages are (5, 7, 10, 12, 15) gm.
- contact times are (20, 40, 60, 80, 100, 120, 140, 160, 180) min.
- Solution pH (3, 4, 6, 8, 9).
- Initial dye concentrations are (100, 200, 300, 400 and 500) mg/l.
- Adsorbents particle size (0.4, 0.6, 1.18 and 2.38) mm.

4. Experimental procedure

All batch experiments were performed in a set of conical flasks of (250 ml) in volume , containing different amounts are (5, 7, 10, 12 and 15) gm. of rice hulls with the particle size of (0.5-2.38) mm mixed with 100ml of methyl red solution of 500 mg/l initial concentration.The whole set was then placed on a wrist shaker for 100 min at 26 ^{o}C for reaching equilibrium.The pH of the solution was equal to 3.Methyl red dye analyzed using a UV/Visible spectrophotometer at 450 nm wavelength, then the dye concentrations were calculated from calibration curve.

The adsorption capacity of the methyl red dye was calculated using the following general formula: (Azhar *et al.*, 2005; Gulipalli *et al.*, 2011and Morcali *et al.*, 2013).

$$q_e = \frac{V}{m} * (C_o - Ct) \qquad \ldots\ldots\ldots 1$$

in which q_e is the amount of methyl red adsorbed at equilibrium per unit weight of sorbent (mg/g), C_o and C_t (mg/L) are the dye concentrations in the solution before and after adsorption, V is the solution volume (in liter (L)), and m is the amount of sorbent (in gram (g)) used in the adsorption experiment.

The dye percent removal (%) was calculated using the following equation:

$$E = \frac{C_o - Ct}{C_o} * 100 \qquad \ldots\ldots\ldots\ldots 2$$

5. Results and Discussion

5.1 Effect of contact time :

To investigate the effect of contact time, an experiment was carried out by mixing (100)ml of methyl red solution of (500)mg/L concentration with (10)gm. of rice hulls adsorbent for (20, 40, 60, 80, 100, 120, 140, 160 and 180)min with agitation speed of 150 (rpm) at pH 3.

The effect of contact time on the adsorption of methyl red onto rice hulls is represented in a plot of percentage adsorption versus adsorption time as shown Fig.(2). It is evident from the figure that the removal efficiency is dependant on the equilibrium time. It is clear that the extent of adsorption is rapid in the initial stages and becomes slow in later stages till saturation of the adsorbent is allowed. The final dye concentrating did not vary significantly after (100 min) from the start of adsorption process. This shows that equilibrium can be assumed to be achieved after (100 min).

This is obvious from the fact that a large number of vacant surface sites are available for the adsorption during the initial stage and with the passage of time, the remaining vacant surface sites are difficult to be occupied due to repulsive forces between the solute molecules on the solid phase and in the bulk liquid phase (Gulipalli *et al*., 2011).

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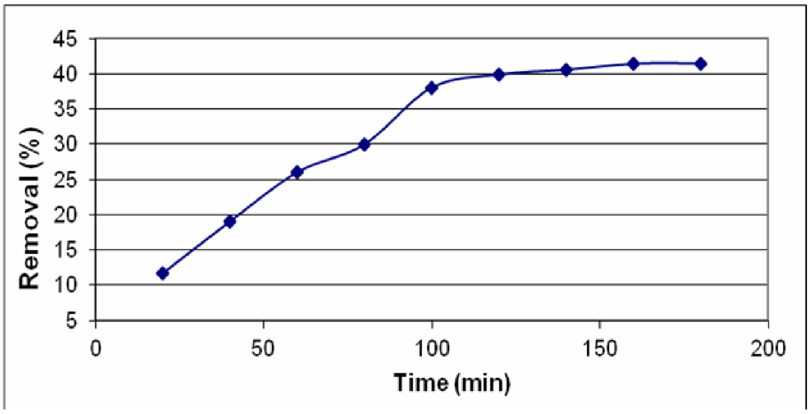


Fig.(2): Effect of contact time on the adsorption of Methyl red dye (C_o=500mg/L)

5.2. Effect of sorbent dosage:

The effect of rice hulls dosage on the uptake of methyl red was studied with methyl red concentration of (500 mg/L) at different adsorbent dosages (5-15) gm., and at a fixed agitation speed (150 rpm) for an equilibrium time of (100 min). The results obtained from this study are presented in fig.(4). The amounts of dye sorbed per gram of the rice hulls q_e (mg/g) (5.89, 4.6, 3.57, 3.08, 2.54), decreased with the increase in the dosage of the adsorbent.

The increase in dye removal percentage with adsorbent dose can be attributed to increased adsorbent surface area and availability of more adsorption sites. At the beginning of the process the rate of dye removal by the rice hulls was fast and then decreased gradually.(EL-Maghraby and . EL Deeb, 2011).

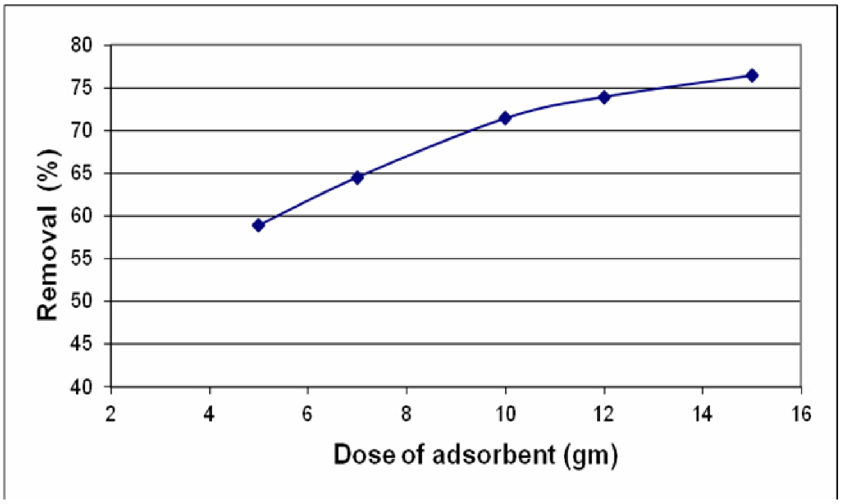


Fig. (3): Effect of sorbent dosage on the equilibrium uptake of the dye Methyl red

5.3. Effect of Initial concentration of MR:

A dosage of 10 grams from the adsorbent material were mixed with 100ml of methel red solutions of different concentrations of (100, 200, 300, 400 and 500)mg/L and agitated for 100 min. in time and pH equal to 3.

The amounts of dye adsorbed versus varying initial dye concentrations were plotted in figure.(4). It was found that the up take capacity increased with increase of initial concentration of the adsorbate (0.36, 1.08, 1.88, 2.68, 3.68, and 3.67) mg/g. Also, it was found that an increasing in the dye concentration had caused the decreasing in the percentage of dye removal.

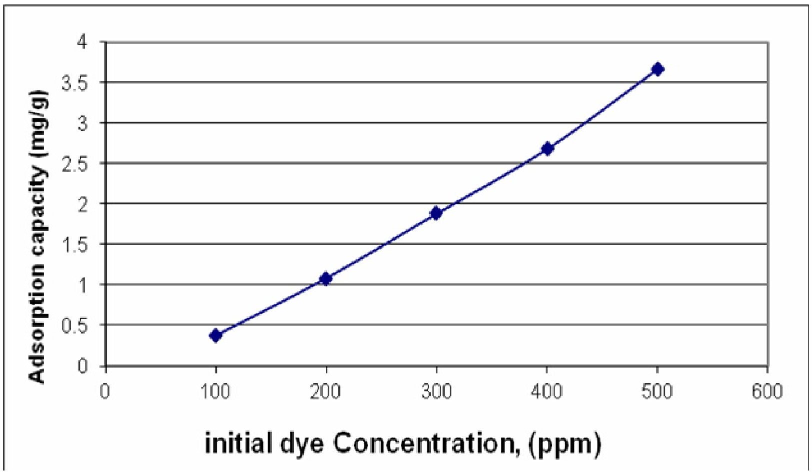


Fig. (4): Effect of Initial methyl red concentration on the adsorption capacity of rice hulls

5.4. Effect of pH :

To study the effect of pH on the adsorption of methyl red dye, a solutions of (500)mg/L concentration and different pH values (3-9)was prepared and mixed for (100) min. with (10)gm. from rice hulls adsorbent of particle size (1.18)mm.

Initial pH value may enhance or depress the uptake. This is attributed to the charge of the adsorbent surface with the change in pH value. The effect of solution pH on methyl red adsorption was investigated and the results are illustrated in Figure (5).

It is evident from this figure that the percentage adsorption decreased with increase in PH, reaching minimum value at pH equals 6. And farther that the percentage adsorption increased with increase in pH, reaching maximum value at pH equals to 9. The pH of the system exerts profound influence on the adsorptive uptake of adsorbate molecules presumably due to its influence on surface properties of the adsorbent and ionization/dissociation of the adsorbate molecule. The increasing in the adsorption of the dye with decreasing of pH values is due to the attraction between the azo dye and excess H^+ ions in the solution (Santhi *et al.*, 2010).

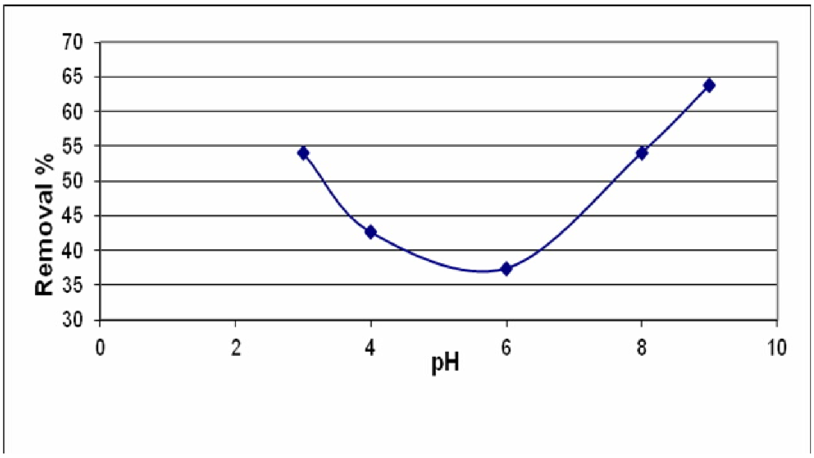


Fig. (5): Effect of pH on the adsorption of Methyl red dye

5.5. Effect of particle size:

The influence of the adsorbent particle sizes of (2.38, 1.18, 0.6 and 0.4)mm with 100 ml of methyl red dye solution of pH 3 and dye solution concentration of 500 mg/L for 100 min mixing time on the adsorption rate was studied .

Figure (6)shows the effect of adsorbent particle size on the adsorption capacity of rice hulls. The higher uptake of dye (3.33 mg/g) obtained by using (0.4 mm) particle size diameter comparing with the other particle sizes (0.6, 1.18, 2.38) mm which gave (2.99, 2.95, 2.63) mg/g respectively.

The lower particle size presents a larger surface for adsorption, which tends to increase the dye sorption rate at the initial stages (Ofomaja, 2008), therefore, the increase in the uptake by smaller particles was due to the greater accessibility to pores and to the greater surface area for bulk adsorption per unit mass of the adsorbent as obtained by (Krishna and Swamy, 2012).

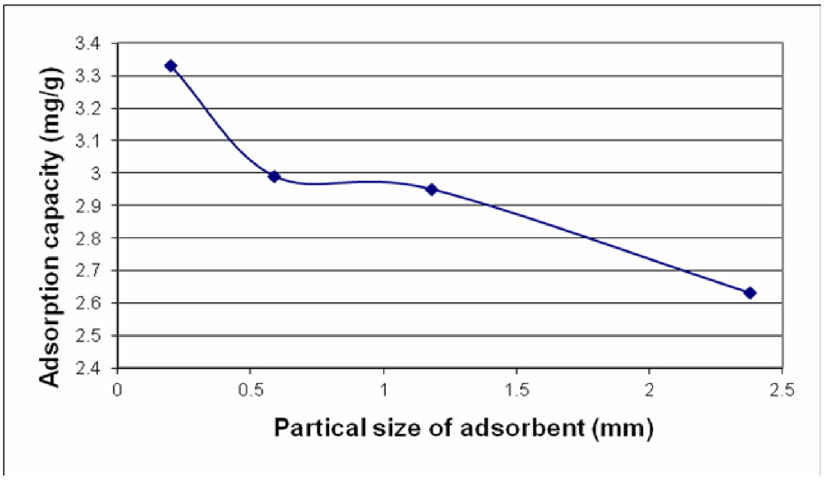


Fig. (6): Effect of rice hulls Particle size on the adsorption rate of Methl red

6.Adsorption Isotherms:

The relationship between the amount of substance adsorbed at constant temperature and its concentration in the equilibrium solution is called the adsorption isotherm. The adsorption isotherm is important from both a theoretical and a practical point of view. In order to optimize the design of an adsorption system to remove the dye, it is important to establish the most appropriate correlations of the equilibrium data of each system. Equilibrium isotherm equations are used to describe the experimental adsorption data. The parameters obtained from the different models provide important information on the adsorption mechanisms and the surface properties and affinities of the adsorbent.The most widely accepted surface adsorption models for single-solute systems are the Lingmuir and Freundlich models (Santhi *et al*., 2010), figure (7) represents the equilibrium adsorption isotherms of methyl red on rice hulls adsornent.

adsorption isotherms are important for the description of how molecules or ions of adsorbate interact with adsorbent surface sites and also, are critical in optimizing the use of adsorbent. Hence, the correlation of equilibrium data using either a theoretical or empirical equation is essential for the adsorption interpretation and prediction of the extant of adsorption (Ozacar *et al*., 2008).

Langmuir and Freundlich isotherms (Langmuir, 1916; Freundlich,1906) as cited in(Nasuha *et al.,*2011) are commonly used to describe the equilibrium characteristic of adsorption.

Langmuir isotherm refers to homogeneous adsorption, which adsorption can only occur at a fixed number of definite localized sites, with no transmigration of the adsorbate in the plane of the surface. The Langmuir model can be given as

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{1}{q_{\max}} C_e \quad \text{...........3}$$

where q_e is the amount of adsorbate in the adsorbent at equilibrium (mg/g), C_e is the equilibrium concentration (mg/L), and q_{max} (mg/g) and K_L are the Langmuir isotherm constants related to free energy. The above equation can be linearized to get the maximum capacity, q_{max} by plotting a graph of C_e/q_e vs. C_e.

R_L, equilibrium parameter, a dimensionless constant, is used to determine whether an adsorption is favourable or not (Reddy *et al*., 2013, Noroozi *et al*., 2004) and is calculated by

$$R_L = \frac{1}{1 + K_L C_o} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots 4$$

Values of RL	Type of isotherm
RL >1	Unfavorable
RL =1	Linear
0< RL <1	Favorable
RL =0	Irreversible

The linear form of the Freundlich isotherm model is derived assuming a heterogeneous surface of adsorption capacity and adsorption intensity with a non-uniform distribution of heat of adsorption. The Freundlich model can be given as:

$$q_e = K_F C_e^{\frac{1}{2}} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 5$$

Equation 4 can be Linearized in logarithmic form of Freundlich isotherm model equation and is represented by

$$\log q_e = log K_F + \frac{1}{n} log C_e \qquad \ldots\ldots\ldots 6$$

where K_F and 1/n are Freundlich isotherm constant (mg/g) (dm3/g)n related to adsorption capacity. A plot of log q_e vs log C_e yields a straight line, with a slope of 1/n and intercept of K_F, fig.(8) represents the adsorption equilibrium isotherm of Methel red dye on rice hulls.

the suitable R_L values were found to be between 0 and 1. In the present study, the calculated value of R_L is in the range of 0-1, indicating that the adsorption process is favorable for the adsorbent.

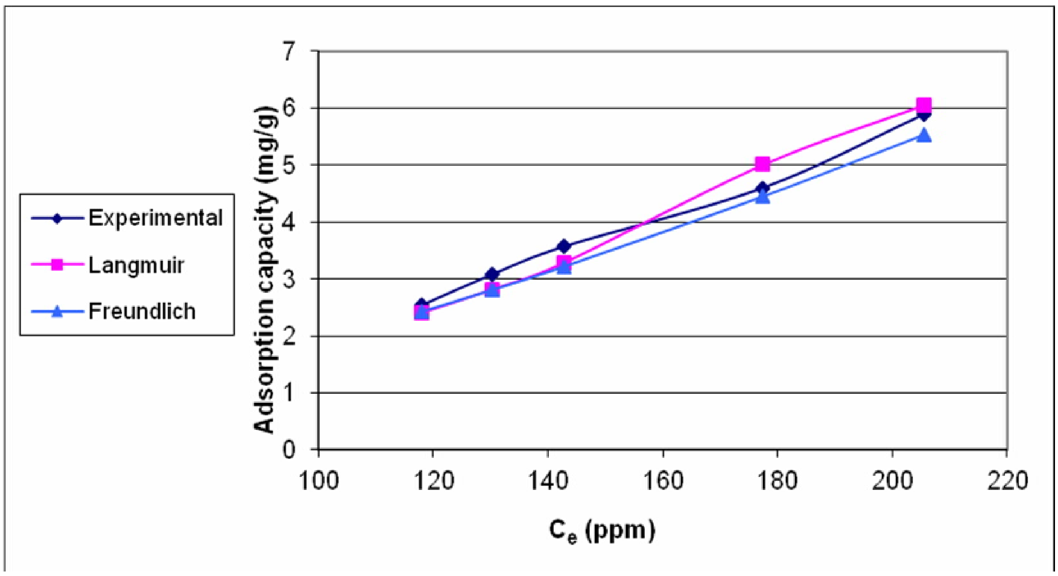
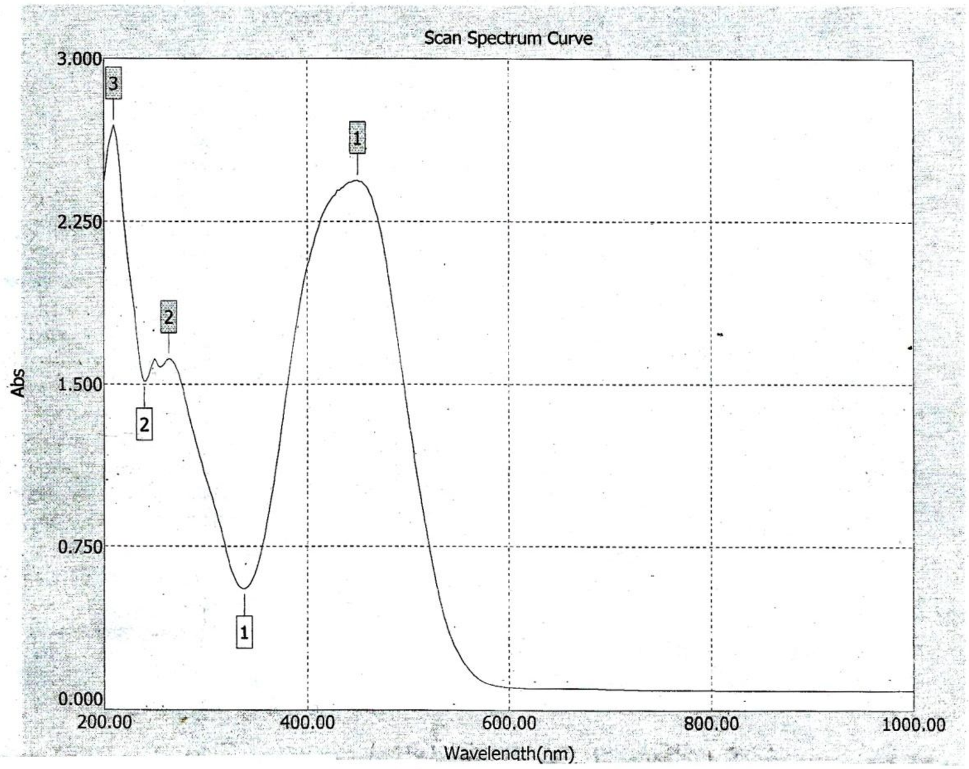


Fig. (7): equilibrium adsorption isotherms of methyl red on Rice hulls

7.Conclusions

The present investigation showed that rice hulls can effectively be used as a cheap abundant for the removal of dyes from aqueous solutions under different operating parameters. The adsorption isotherm was fitted well with both Langmuir and Freundlich isotherm models, and the type of adsorption of methyl red dye on rice hull was of favorable type. The removal of methyl red increases with increase of adsorbent dosage used and initial dye concentrations.



- **Instrument Performance**
  Model : UV-VIS Spectrophotometer
  Number : 18-1884-01-0103
  Spectral Bandwidth : 2.00 nm
- **Scan Spectrum Performance**
  Scan Range : 200.00 to 1000.00 nm
  Measure Mode : Abs
  Interval : 2.00 nm
  Speed : Fast
  Data File : dye.spd
  Create Date/Time : 25 كانون الأول، ٢٠١٢ ٠٩:٥٩:٠٨ ص
  Data Type : Original
  Method File:
- **Analyse Note**
  Analyser : Administrator
  Sample Name :
  Comment :

No.	P/V	Wavelength(nm)	Abs	Comment
1	Peak	450.00	2.439	
2	Peak	264.00	1.616	
3	Peak	210.00	2.691	
1	Valley	338.00	0.555	
2	Valley	240.00	1.513	

Fig.(8): Scan Spectrum Curve of methyl red dye

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