

MAGNETIC PEANUT HULLS FOR METHYLENE BLUE DYE REMOVAL: ISOTHERM AND KINETIC STUDY

TAHA N.A.^{1,2} EL-MAGHRABY A.^{2,*}

¹Chemistry Department, Al-Lith University College Umm Al-Qura University, Saudi Arabia ²Department of Fabrication Technology Advanced technology and New Materials Research Institute City for Scientific Research and Technology Applications Alexandria, Egypt

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*to whom all correspondence should be addressed: e-mail: <u>maghrabyazza@yahoo.com</u>

ABSTRACT

Biosorption is an emerging technique for water treatment utilizing abundantly available biomaterials. The potential feasibility of magnetic peanut hulls particle for removal of cationic dye (methylene blue) from aqueous solution was investigated. The effects of various experimental parameters were examined and optimal experimental conditions were decided. Characterization of biosorbent was carried out by Fourier transform infrared spectroscopy (FT-IR) and thermogravimetric analyzer. FT-IR analysis showed the presence of hydroxyl, carbonyl and carboxyl groups which can involve in the biosorption process. The results in this study indicated that peanut hull was an attractive candidate for removing cationic dyes from dye wastewater. Different kinetic and equilibrium models were applied to the experimental data. Tempkin model was the most fitted isotherm as R²= 0.963. While the resulting data for the different parameters studied was suitable to be pseudo second order.

Keywords: magnetic materials, cationic dye, peanut hulls, isotherm

1. Introduction

Water quality control standards and regulations against hazardous pollutants have become stricter in many countries. Dyes are widely used in the textile, food, cosmetics, pharmaceutical, tanneries, electroplating factories and host other industries (Sayan, 2006).These colored compounds usually have a synthetic origin and complex aromatic structures. Dyes (over 7×10^5 metric tons of synthetic dyes) are produced worldwide every year for dyeing and printing purposes and about 5–10% of this quantity is discharged with wastewater (Dafale *et al.*, 2008).Most of these dyes are toxic and potentially carcinogenic in nature and their removal from the industrial effluents is a major environmental problem (Parsons, 2004).It can cause some aesthetic problems and also reflection of sunlight penetrated into the water body. Because of their complex structures and high solubility in water, the treatment of these pollutants in wastewater is troublesome (Xue, 2009).The methods of color removal from industrial effluents include biological treatment, chemical coagulation followed by sedimentation, flotation, adsorption, oxidation and photo catalytic discoloration (Ilha *et al.*, 2009; Ozkan-Yucel and Gokcay, 2010; Amin, 2008; Gong *et al.*, 2005). Among these methods, sorption processes

appear to be preferable techniques. Adsorption has been proven to be an excellent method for removing dyes from aqueous solutions because of its significant advantages. It is cheap, easily available, most profitable, easy to be used and most efficient in economic and environmental points of view compared to the conventional treatment.

Biosorption is emerging as a highly effective, economical and widely used method for the treatment of textile effluents. It is considered as a potential alternative over the traditional costly treatment technologies. The biosorption process is based on the biomaterial–contaminant interactions resulting in the sequestration of organic and/or inorganic pollutants by non-living biomaterials (Lu *et al.*, 2011). Biosorption process significantly transfers the dyes from the aqueous effluent to a solid phase, hence decrease their bioavailability to living organisms (Alencar *et al.*, 2012). This decolorized water can thus be released to the environment, or it can be reutilized in the industrial processes (Saha *et al.*, 2012). Afterwards, the biosorbent can also be regenerated and reused (Mittal *et al.*, 2009).

Peanut is a common food material in the world. Annual production of peanut in Egypt has reached 190,000 million metric tons (www.soyatech.com/peanut_facts.htm). Peanut production in the world is 34.4 billion kg/year (http://www.wikipedia.com).Peanuts are rich in mono-unsaturated fatty acids like oleic acids that prevent coronary artery disease and strokes by favoring healthy blood lipid profile; they also contain high concentrations of polyphenolic antioxidants, primarily pcoumaric acid and resveratrol, which have protective function against cancers, heart disease, and reduce stroke risk (Etherton *et al.*, 2008).In recent years, there has been an increase in the use of biological materials including agricultural and industrial solid wastes as adsorbents for the removal of heavy metals (Periasamy and Namasivayam, 1996).The advantage of using solid wastes is that it saves disposal costs while alleviating potential environmental problems. Many sorbents based on low cost agricultural by-products had been used for dye sorption from wastewater, which included banana pith (Namasivayam and Kanchana, 1992), orange peel (Namasivayam *et al.*, 2007).

Materials whose physical properties can be varied by application of external magnetic fields belong to a specific class of smart materials. In many cases magnetically responsive composite materials can be formed by modification of originally diamagnetic materials by magnetic nanoparticles, present in different types of magnetic fluids (ferrofluids). Such composite materials have already found many important applications in various areas of biosciences, biotechnology, medicine and environmental technology (Safarik and Safarikova, 2002). The application of magnetite in the field of waste water treatment is becoming an interesting area of research. Nanoparticle exhibit good adsorption efficiency especially due to higher surface area and greater active sites for interaction with metallic species and can easily be synthesized several researches have used it as an adsorbent (Hritcu *et al.*, 2009). Recently, the possible use of agricultural by-products has been widely investigated as a replacement for current costly methods of removing different types of heavy metals and dyes from wastewater. Some of the agricultural waste materials can be effectively used as low-cost adsorbents. Modification of agricultural by-product could enhance their natural adsorption capacity or add another additional value to the by-product.

The present work is concerned with the synthesis of magnetic peanut hull as a biosorbent for removal of dye. The effects of various operating parameters on biosorption such as initial pH, sorbent dosage, agitation speed and temperature were monitored and optimal experimental conditions were determined. Different adsorption isotherms (Langmuir, Freundlich and Tempkin isotherms) and kinetic models (pseudo-first-order and pseudo-second-order kinetics) were used to find out most suitable models describing our experimental findings.

2. Experimental

2.1. Materials

Peanut hulls were collected from locally available roasted peanuts. The collected biomaterial was extensively washed with tap water to remove soil and dust, sprayed with distilled water then dried in an oven at 80 °C to a constant weight. Dry peanut hull was crushed in a Willy mill and fraction smaller than 500µm was collected and used for magnetic modification. Ferrous sulfate (hyptahydrate) "Oxford", ferric chloride anhydrous "Loba Chemie" and ammonium hydroxide "Sigma–Aldrich", were used without further purification. Methylene blue (MB) supplied by Sigma–Aldrich (M) Sdn Bhd. Methylene blue in commercial purity were used without further purification (λ = 660 nm).

2.2. Preparation of magnetic adsorbent

Magnetically responsive peanut hulls were prepared by 2.1 g of ferrous sulfate and 3.1 g of ferric chloride are dissolved in 80 ml distilled water with vigorous stirring at 80 °C. Then 10 ml of ammonium hydroxide solution (25%) was added. Ten grams of powdered peanut hulls 500 μ m were suspended in the previous solution at 80 °C. The suspension was mixed on a stirrer for 30 min. The magnetically modified peanut hulls particles were then left to cool to room temperature and washed with distilled water then dried until complete dryness (Gong *et al.*, 2005).

The stock solutions of MB (1000 mg l⁻¹) were prepared in distilled water. All working solutions were prepared by diluting the stock solution with distilled water to the needed concentration.

2.3. Adsorption of dye on magnetically modified peanut hulls

The batch adsorption capability of the prepared magnetically modified peanut hulls toward methylene blue dye was investigated using their aqueous solutions. Adsorption experiments were carried out in a rotary shaker at different speed and ambient temperature, using 250 ml shaking flasks containing 100 ml different concentrations of dye solutions 5-25 mg l⁻¹. The initial pH values of the solutions were previous adjusted with 0.1 M HCl or NaOH using pH meter. Different doses of sorbent were added to each flask. After shaking the flasks for predetermined time intervals, the samples were withdrawn from the flasks and the dye solutions were separated from the sorbent by filtration then centrifugation. Dye concentrations in the supernatant solutions were estimated measuring absorbance at maximum wavelengths of dyes with a UV spectrophotometer (Shimadzu, Japan). The amount of dye per unit weight of adsorbent, $q_e (mg g^{-1})$ was calculated by the following equation:

$$q_e = (C_0 - C_e) V/W$$
 (1)

Where C_0 is the initial dye concentration (mg l⁻¹), C_e is the equilibrium dye concentration (mg l⁻¹), V is the volume of the solution (I) and W is the mass of the biosorbent (g).

The aqueous samples were taken at time intervals and the concentrations of MB were similarly measured. The amount of adsorption at time t, q_t (mg g⁻¹), was calculated by:

$$q_t = (C_0 - C_t)V/W$$
 (2)

Where C_0 and C_t (mg l⁻¹) are the liquid-phase concentrations of dye at initial and at any time t, respectively. Vis the volume of the solution (I) and W is the mass of dry adsorbent used (g).

3. Results and discussion

3.1. Characterization of prepared samples

3.1.1. FT-IR Study

Information about the physical properties of adsorbent such as pore structure is essential procedure prior to adsorption process. As a biomass, the peanut hull is a complex material consisted of polyphenol such as catechol, pyrogallic acid and m-trihydroxybenzene, mineral, lipid, and cellulose, etc (Kargi and Cikla, 2006). Chemical sorption can occur by the polar functional groups of these constitutes, which include carboxyl groups and phenolic hydroxyl as chemical bonding agents. The FT-IR spectra of native and magnetic peanut hulls biomass were studied in the range of 400–4000⁻¹ cm using (FTIR-8400 S- Shimadzu, Japan) (Fig. 1). The results of the FT-IR spectra of raw biosorbent revealed the presence of peak in the region of 2900 cm⁻¹ which is due to the C–H stretching and indicates the presence of –CH and CH groups in the structure of peanut husk biomass. The band at 1735.93 cm⁻¹ allocates the C=O stretching vibrations. Presence of broad bands in the region of 3300 cm⁻¹indicates the presence of O–H group (carboxylic acids, phenols and alcohols) on the surface of biosorbent as in cellulose (Akar *et al.*, 2009)., pectin and lignin. The presence of peaks in the region of 2370 cm⁻¹might be due to presence of C= C bonds. The peak at 1421.5 cm⁻¹ was caused by the CH₂ bending. The peak at 1259.5 cm⁻¹ is indicative of the OH in plane bending cellulose .There are three peaks in the raw peanut at 1242 cm⁻¹, 2071 cm⁻¹ and 2328 cm⁻¹ disappeared. These changes in peak intensity may be affect the adsorption of pollutants.



Figure 1. FTIR spectrum of magnetic and raw peanut hulls

3.1.2 Thermal analysis

Fig. 2 shows the pyrolysis characteristics of the samples ,the percent weight loss of raw peanut hulls are studied in N_2 atmosphere at heating rate of 10 °C/min by using thermo gravimetric analyzer (TGA – 50 Shimadzu, Japan from 25 °C to 600 °C.

As a whole, the pyrolysis process of the raw is characterized by a three-stage thermal degradation, and the decomposition of the raw mainly takes place in the second stage. The main mass loss ends at 370 °C (40%) and is followed by a slow and continuous mass changes with a long tail of devolatilization. At the first in the curve until 100 °C water release takes place. At the second stage, the mass rapidly decreases due to cellulose vitalization, and then in the third stage, the slow mass loss can be observed due to lignin decomposition (Souza *et al.*, 2009).





3.1.3 Scanning Electron Micrograph images

The surface features and morphological characteristics of the raw and magnetic peanut hulls are studied by using scanning electron microscope (SEM) (JEOL JSM 6360LA-japan)as Fig. 3 (a & b) raw peanut and(c & d) for magnetic peanut hulls.



(a)





Figure 3. SEM analysis of (a, b) unloaded peanut hulls biomass (c, d) Magnetic peanut hulls

This characteristic used to determine the particle shape and porous structure of biomass. The figures indicated the porous and fibrous texture of the raw peanut hulls with high heterogeneity that could contribute to the biosorption of the dyes. The raw hulls materials have insoluble cell walls with fibrous content and are largely made up of cellulose based structural proteins (Taha and Maghraby, 2014). A high response surface of the functional groups is also present. As shown in Fig. 3(a&b) the parent raw peanut hulls material has more fiber and more active sites, while in Fig. 3 (c&d) it showed the raw peanut hulls is completely covered with iron oxide, and all the iron oxide particles are aggregated to form a spherical and cage-like structure. The iron oxide particles exhibit magnetic behavior, creating more negative charges (Panneerselvam *et al.*, 2011).The presence of the new particles on the peanut surface not only to change material to be magnetic but also it may increase the sorption process due to increase the surface area with small particles added.

3.2. Influence of initial dye concentration

The influence of dye concentration on adsorption percentage of dye were estimated as a function of time for different initial concentrations of dye at ambient temperature, with 0.1 g of adsorbent and agitation rate of 200 r.p.m for 2 hours. It is evident from the Fig. 4, that the adsorption of dye increase in contact time increase until reaches equilibrium at 60 min. When the dye concentration was increased from 5 to 25 mg l⁻¹, the percentages of dye sorbed decreased from 71 to 30% in MB. The removal of dye was found to be dependent on the initial concentration. The amount of dye adsorbed, q_e (mg g⁻¹), increased with increase initial concentration. Further, the adsorption was rapid in the early stages and then gradually decreased and became almost constant after the equilibrium point. The dye concentration which had high adsorption of dye was 20 mg l⁻¹. At low concentrations, the ratio of available surface to initial dye concentration was larger, so the removal became independent of initial concentrations. However, in the case of higher concentrations, this ratio was low. The percentage removal then depended upon the initial concentration.



Figure 4. Influence of dye concentration on adsorption of MB by magnetic peanut hulls (sorbent dose: 0.1 g; stirring speed; 200 r.p.m; contact time: 2 h).

3.3. Effect of adsorbent dose

Biosorbent dose is an important parameter because this factor determines the capacity of biosorbent for a given initial concentration of the adsorbate (Gupta *et al.*, 2007). The effects of sorbent dose on the removal ratios of dye were checked out by varying the biosorbent dose from 0.025 to 0.3 mg for particle size 500 μ m, 20 mg l⁻¹ dye concentration with agitation rate of 200 r.p.m. at ambient temperature. The percentages of dyes

sorbed increased as the sorbent dose was increased over the range 0.025–0.3 mg (fig. 5). This can be simply attributed to the increased sorbent surface area and availability of more sorption sites (Garg et al 2004). On the other hand, the results indicated that by increasing the biosorbent dose, the biosorption capacity (mg g^{-1}) of biosorbent decreased significantly. Above 2.0 g l^{-1} of sorbent dose, the adsorption equilibrium of dye was reached and the removal ratios of dyes held almost no variety. So, the peanut hulls biomass of 2.0 mg was chosen for subsequent experiments. Maximum biosorption capacity of peanut hulls was achieved by using 0.2 g biosorbent dose.





3.4. Effect of rpm

It is normally found that with higher stroke speed, there is faster removal and higher removal at equilibrium. However, the results obtained in this work, depicted in Fig. 6, shows that the stroke speed did not affect the removal in the range of stroke speed studied, 100-500 r.p.m. This finding implies that film mass transfer is not a limiting step in the overall adsorption process. (Wang and Lin, 2008b) found that the stroke speed should be over 120 stroke/min overcome the film mass transfer resistance for the adsorption system of lead ion/rice hull ash.

3.5. Effect of temperature

Real textile effluents are mostly released at relatively higher temperatures so temperature is also an important design parameter for the real application of biosorption process. The results regarding the effect of temperature on the biosorption potential of peanut hulls biomass are shown in Fig. 7. Experiments were performed at five temperatures within the range of 20-40 °C, 0.2 mg adsorpent, 20 mg l⁻¹ methylene blue and with speed rate 200 r.p.m. The results clearly that biosorption of methylene blue dye onto peanut hulls biomass is endothermic process. The increase in temperature results increase in biosorption of dye from aqueous solution. This increase may indicates the increased tendency of MB due to an increase in its kinetic energy with increasing temperature, and this situation leads to more sorption on the surfaces of peanut hulls. Maximum dye removal was achieved at 25 °C. It can be clearly seen that the adsorption temperature did not influence the removal rate and the equilibrium removal. The report of other system, lead ion/rice hull ash, indicated that with higher adsorption temperature there was faster removal and higher equilibrium removal, though the influence was minor (Wang and Lin, 2008b).



Figure 6.Effect of agitation rate on adsorption of MB by magnetic peanut hulls (dye concentration: 20 mg l⁻¹; sorbent dose: 0.1 g; contact time: 2 h)

From this results it's clarify that the 25 °C the most suitable temperature with 200 r.p.m. speed rate for 0.2 adsorbent dose and 20 mg l^{-1} dye. Similar result has also been reported for the removal of methylene blue by adsorption on biosolid (Sarioglu and Atay, 2006).



Figure 7. Effect of temperature on adsorption of MB by magnetic peanut hulls (dye concentration: 20mg l⁻¹; stirring speed: 200 r.p.m; sorbent dose: 0.1 g; contact time: 2 h)

3.5.1. Equilibrium isotherm models

With the data in Figure (8), Langmuir, Freundlich and Tempkin equations were employed to study the sorption isotherm of MB. The Langmuir equation is based on the assumption that maximum sorption corresponds to saturated monolayer of sorbate molecule on the sorbent surface, that the energy of sorption is constant and

that there is no transmigration of sorbate in the plane of the surface. The Langmuir equation was shown as follows:

$$q_{e} = \frac{K_{L} \cdot C_{e}}{1 + .a_{L} C_{e}} \frac{C_{e}}{q_{e}} = \frac{1}{K_{L}} + \frac{a_{L}}{K_{L}} \cdot C_{e}$$
(3)

Where q_e is the amount of dye adsorbed per unit mass of sorbent particles at equilibrium; C_e is the equilibrium phase concentration of dye and a_L , K_L are Langmuir constant. Therefore, a plot of Ce/qe versus Ce, gives a straight line of slope $\frac{a_L}{K_L}$ and intercept $t\frac{1}{K_L}$.

While the empirical Freundlich equation was expressed as:

$$q_{e=} K_{F} \cdot C_{e}^{\frac{1}{n_{F}}}$$
(4)

The equation is conveniently used in the linear form by taking the logarithm of both sides as:

$$\log q_{e=} \log K_{F} + \frac{1}{n_{F}} \cdot \log C_{e}$$
(5)

Where q_e is the amount of dye adsorbed per unit mass of sorbent particles at equilibrium; C_e is the equilibrium phase concentration of dye, Freundlich constants, K_F and $1/n_F$, are related to adsorption capacity and intensity of adsorption, respectively.

And Tempkin isotherm has commonly been applied in the following form:

$$q_e = \left(\frac{RT}{b}\right) \ln \left(AC_e\right)$$
(6)

Where RT/b = B. T is the absolute temperature in Kelvin and R is the universal gas constant, 8.314 J (mol K)⁻¹. The constant b is related to the heat of adsorption. A plot of q_e versus lnC_e yielded a linear line.

Table 1 summarized the results of the isotherm constants for the different equilibrium isotherms tested. On the basis of the correlation coefficients (R^2), Langmuir model yields ($R^2 = 0.939$) while Freundlich model ($R^2 = 0.567$) and Tempkin isotherm was ($R^2 = 0.963$) which seemed to represent the equilibrium adsorption data with better fit as compared to the other isotherms.

Table 1. Isotherm parameters for removal of methylene blue by magnetic peanut hulls powder

Isotherm	Parameters						
Langmuir model	R^2 = 0.939 Q_0 = 8.9605 b = 0.7256 RL = 0.052						
				Freundlich model	$R^2 = 0.5671$		
					n _f = 0.788		
					$K_{f} = 0.277$		
Tempkin model	$R^2 = 0.9636$						
	A=0.6612						
	B = 4.7227						









(c)

Figure 8. Isotherm models of MB adsorption, a: Langmuir, b: Freundlich, c: Tempkin

3.6. Kinetic studies

The biosorption mechanism and potential rate controlling steps are important to study for design purposes during the wastewater treatment. Several kinetic models are available to describe the sorption kinetics. Mostly used models including the pseudo-first order and pseudo-second order were applied to the experimental data to evaluate the biosorption kinetic of methylene blue dye. The applicability of these kinetic models was comparing by calculating the correlation coefficients (R^2) for the different parameters studied as listed in table (2). The comparison between R^2 for pseudo first and second concludes that the reaction fitted to pseudo second order and this also can be noticed from values of q_e calculated and $q_{e, exp}$ as they are seemed to be equal.

Table 2. The pseudo-first-order, pseudo-second-order adsorption rate constants and calculated and experimental q_e values obtained at different initial MB concentrations for different parameters studied

Dye system		Pseudo first order model			Pseudo second order model		
	q _{e,exp} mg g ⁻¹	K ₁ (min⁻¹)	q e1 mg∕g	R ²	K ₂ g mg ⁻¹ min ⁻¹	q e₂ mg g⁻¹	R ²
1-Different concentrations W=0.1 g, n= 200 rpm, ambient temperature							
C ₀ = 5 mg l ⁻¹	2.18	0.045	0.5913	0.918	0.232	2.200	0.9995
C ₀ = 10 mg l ⁻¹	4.05	0.040	0.9145	0.927	0.105	4.122	0.9998
C ₀ = 15 mg l ⁻¹	6.25	0.025	1.3095	0.875	0.0694	6.2189	0.9997
C ₀ = 20 mg l ⁻¹	9	0.0492	4.4290	0.946	0.0204	9.4162	0.996
C₀ = 25 mg l ⁻¹	7.73	0.049	3.9147	0.945	0.02197	8.1566	0.999
2- Sorbent dosage							
n= 200 rpm, C ₀ =20 mg l ⁻¹ ambient temperature							
w ₀ = 0.025g	34.2	0.0454	10.527	0.958	0.0088	35.2	0.9988
w ₀ = 0.5g	17.2	0.0375	3.3159	0.942	0.03139	17.36	0.9998
w ₀ = 0.1g	9	0.0493	4.4299	0.9464	0.0204	9.416	0.996
w ₀ = 0.2g	4.589	0.0435	0.2740	0.7858	0.3757	4.6126	1
w ₀ = 0.3g	3.132	0.0286	0.1374	0.8696	0.785	3.1309	1
3 - rpm							
W=0.2 g, C₀=20 mg l⁻¹ ambient temperature							
rpm ₀ =100	4.388	0.0428	1.6626	0.8291	0.06254	4.427	0.9952
rpm ₀ = 200	4.588	0.04353	0.2740	0.7858	0.3004	4.766	0.9999
rpm ₀ = 300	4.76	0.0373	0.380	0.8948	0.3005	4.7664	0.9999
rpm ₀ = 400	4.795	0.0320	0.1569	0.9292	0.7046	4.79616	1
rpm ₀ =500	4.75	0.033	1.2387	0.9815	0.0727	4.7939	0.9988
4- Temperature							
W=0.2 g, n= 300rpm C ₀ =20mg l ⁻¹							
T ₀ = 20 °C	4.5	0.0521	1.3068	0.8742	0.1024	4.5517	0.9993
T ₀ = 25 °C	4.762	0.0373	0.380	0.8948	0.3005	4.766	0.9999
T ₀ =35 °C	4.657	0.00678	1.6315	0.7018	0.3685	4.682	0.9999
T ₀ = 40 °C	4.75	0.00392	1.527	0.9014	0.5359	4.7103	1

4. Conclusion

The efficiency of magnetic particle impregnated onto peanut hulls in removing cationic dyefrom aqueous solution has been investigated. The comparison of raw and magnetic peanut hulls indicates no vanished or disappeared in hydroxyl or carbonyl groups which always involvement in the adsorption process. Results indicate that adsorption is positively dependent on adsorbent dose and stroke speed. The percentages of dyes sorbed increased then reached maximum values as the sorbent dose was increased. The removal of dye was found to be dependent on the initial concentration of the dye. The adsorption equilibriums were reached at about 2 hr. The Adsorption data indicate the applicability of pseudo-second-order kinetics. The isothermal data were fitted the Tempkin model. The Magnetic peanut hull in present study shows good promise for practical applicability due to its easy availability of the material.

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