



Adsorption study for removal of sunset yellow by ethylenediamine-modified peanut husk

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ABSTRACT

Peanut husk crosslinked with epichlorohydrin, followed by modification with ethylenediamine, was used as a new adsorbent for removal of sunset yellow from aqueous solution. Factors affecting the adsorption characteristics of the said adsorbent, namely, pH value, adsorption time, temperature, and concentration of initial sunset yellow were dealt with in the whole experiments in a batch system. It was concluded that the maximum adsorption capacity of this adsorbent for sunset yellow was 117.7 mg/g at 313 K, which was improved 4.3 times as much as that of the unmodified peanut husk. The Langmuir isotherm model could provide a better description for the adsorption equilibrium when compared with the Freundlich model under the conditions of the present study. The calculated thermodynamic parameters showed that the adsorption of sunset yellow onto peanut husk biomass was feasible, spontaneous, and endothermic under examined conditions. Due to the examination of controlling mechanisms of the process, the Lagergren-first-order model, the pseudo-second-order model, and the intra-particle diffusion model were used to correlate the kinetic data. It was found that the intra-particle diffusion is the significant controlling step under the experimental conditions. It showed that the peanut husk modified with ethylenediamine had good performance for removal of sunset yellow and could be used as a highly efficient biomass adsorbent to treat dyes-containing wastewater.

Keywords: Peanut husk; Ethylenediamine; Modification; Adsorption; Sunset yellow

1. Introduction

Synthetic dyes and pigments are widely used in various industries, such as food, paper, printing, leather, textiles, plastics, and cosmetics. As dyes are generally designed to resist bio-degradation, they may cause severe ecological and environmental problems. In addition, some of the dyes or their metabolites are either toxic, mutagenic, or even carcinogenic. Such

effluent must be treated before it is discharged into waterbody. Among the most useful dyes, sunset yellow is widely used as an additive in pharmaceutical tablets and capsules, in confectionery items, etc. It has been shown that sunset yellow may cause increasing risk of cancer and many other diseases due to its carcinogenic and mutagenic characteristics.

There are many processes available to remove dyes, such as advanced oxidation, combined chemical and biochemical process, adsorption membrane

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filtration, aerobic, and anaerobic digestion. Nowadays, activated carbon works as adsorbent to remove dyes in most commercial systems because of its high adsorption ability. But the processing costs are still expensive. So people try to develop cheaper and effective adsorbents to remove dyes and find an alternative method from different starting materials such as bagasse pith [1,2], rice husk [3,4], water hyacinth [5], hen feather [6], sawdust [7], polar leaf [8], papaya seed [9], etc.

Peanut husk, an agricultural by-product available in large quantity in China, is often burned and discarded directly, which produced waste gas and dust. Fortunately, a possible solution to utilize peanut husk as an adsorbent has been found to remove contaminants in wastewater. Peanut husk was introduced directly as a low-cost adsorbent for the efficient removal of sunset yellow dye in aqueous solution [10]. However, raw peanut husk cannot be used as a good natural adsorbent due to two major limitations. Firstly, on its contact with water, there is a leaching of yellowish color into solutions; secondly, on its prolonged contact with water, peanut husk tends to disperse in the whole solution. Researchers have studied the removal of heavy metals [11], phenol [12], and dyes [13–18] by adsorbent based on peanut husk. The purpose of this work was to produce a new adsorbent from peanut husk chemically modified with ethylenediamine and the adsorption of sunset yellow on it was investigated on account of the initial concentration of sunset yellow, pH, adsorption time, and temperature. The adsorption equilibrium was modeled with the Langmuir and the Freundlich isotherms. The kinetic parameters and intraparticle diffusion were also determined for this process.

2. Materials and methods

2.1. Preparation of ethylenediamine-modified peanut husk (EMPH)

The peanut husk used in this study was obtained from a farmers' market in Chongqing, Rep. of China. The peanut husk was first washed copiously with distilled water to remove dust and impurities and dried at 60°C in an air circulating oven. In order to receive uniform modification and reproducible results, the peanut husk was sieved into 40–60 meshes before modification. 2.0 g (dry weight) of the raw peanut husk was mixed with aqueous NaOH (45 mL, 1 mol L⁻¹) and epichlorohydrin (25 mL), then the mixture reacted for 4 h at (40 ± 1)°C. The biomass residue was rinsed with distilled water and oven-dried.

Then 2.0 g (dry weight) of the epichlorohydrin crosslinked peanut husk was heated at 65°C with 100 mL of sodium carbonate solution (0.1 mol L⁻¹) and 2.5 mL of ethylenediamine for 2 h. The peanut husk modified with ethylenediamine (EMPH) was washed thoroughly with distilled water, filtered, and then oven-dried and stored in a desiccator.

The general reaction scheme can be expressed as Fig. 1 [19].

2.2. Chemicals

Stock solution was prepared by dissolving 1.0 g of sunset yellow dye in 1,000 mL of twice-distilled water. The test solutions were prepared by diluting the stock solution to the desired concentration. All reagents used in this study were of analytical reagent grade and used without further purification. The initial pH was adjusted to the pre-determined value (2.00–12.00) ± 0.10 with NaOH or HCl solutions (1.0 mol L⁻¹) prior to the addition of adsorbent.

2.3. Batch adsorption studies

Batch adsorption experiments were carried out in 100 mL conical flasks containing 0.1 g of EMPH and sunset yellow solutions (30 mL) of the desired concentration at constant temperature. The flasks were oscillated on a shaker (SHA-C Digital Display Thermostatic Oscillator, Jiangsu Dazhong Instrument Co. Ltd, China) at constant 150 rpm shaking rate for given time. Samples were taken out from mixture and centrifuged and the supernatant liquid was analyzed spectrophotometrically at 482 nm to determine the residual dye concentration in the system (UV1102 Spectrophotometer, TECHCOMP, China). All the experiments were done twice in parallel and average values were calculated further. For kinetic studies, a series of solution with different initial concentration were prepared, each data point at pre-determined time interval was measured from an individual flask and therefore, no correction was necessary due to withdrawal of sampling volume. For isotherms studies, a series of solution containing 30 mL of sunset yellow in the range of 250–1,000 mg L⁻¹ were prepared. EMPH (0.1 g) was added to each solution and then the mixtures were agitated at temperature of 293, 303, and 313 K, respectively, for 24 h.

The adsorption capacity q (mg g⁻¹) was calculated as follow:

$$q = \frac{v(c_0 - c_t)}{m} \quad (1)$$

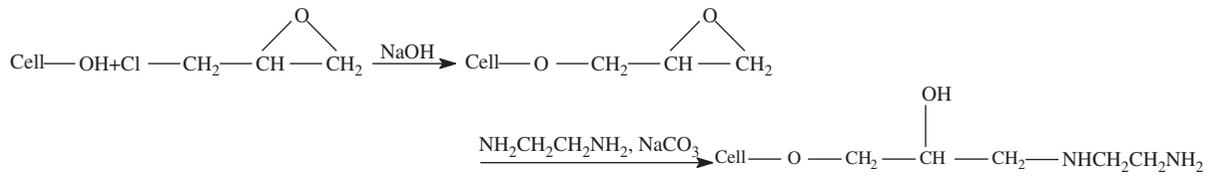


Fig. 1. The general reaction scheme.

where c_0 and c_t are the initial concentration and the concentration at t moment (mg L^{-1}); v is the volume of sunset yellow solution used (L); and m is the mass of the dry EMPH used (g).

2.4. Adsorption isotherms

The Langmuir and the Freundlich isotherms were used to analyze the equilibrium phenomenon of the present system. The linear form of the Langmuir equation is generally accepted in the following variation:

$$\frac{c_e}{q_e} = \frac{1}{K_L q_{\max}} + \frac{c_e}{q_{\max}} \quad (2)$$

where c_e is the equilibrium concentration of dye solution (mg L^{-1}), q_e is the equilibrium capacity of dye on the adsorbent (mg g^{-1}), q_{\max} is the maximum monolayer adsorption capacity of the adsorbent (mg g^{-1}), and K_L is the Langmuir adsorption constant (L mg^{-1}).

The Freundlich isotherm equation is a semi-empirical one employed to describe heterogeneous system:

$$\ln q_e = \ln k_F + \frac{1}{n} \ln c_e \quad (3)$$

where k_F (L mg^{-1}) and n (dimensionless) are the Freundlich adsorption isotherm constants, indicating the capacity and intensity of the adsorption, respectively.

2.5. Determination of thermodynamic parameters

Thermodynamic parameters such as free energy change (ΔG), enthalpy (ΔH), and entropy change (ΔS) can be estimated by using equilibrium constants changing with temperature. The isosteric enthalpies ΔH at different adsorption levels can be easily calculated from the Clasius–Clapeyron equation [20]:

$$\ln c_e = \frac{\Delta H}{RT} + \text{constant} \quad (4)$$

where c_e is the equilibrium concentration (mg L^{-1}), T is the absolute temperature (K), R is the universal constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$).

For the systems where the Freundlich isotherm could be fitted to the experimental data, the Gibbs free energy values ΔG (kJ mol^{-1}) of the adsorption process can be calculated using the following equation [21]:

$$\Delta G = -nRT \quad (5)$$

where T is the absolute temperature (K), R is the universal constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$), n is the Freundlich constant.

The entropy change ΔS ($\text{J mol}^{-1} \text{ K}^{-1}$) associated with the adsorption system can be calculated from the Gibbs–Helmholtz equation [21].

$$\Delta S = (\Delta H - \Delta G)/T \quad (6)$$

2.6. Adsorption kinetics

Because of examination of the controlling mechanism of this process, pseudo-first-order, pseudo-second-order and intraparticle diffusion kinetic equations were used to correlate the experimental data. The pseudo-first-order kinetic model was suggested by Lagergren [22] for the adsorption of solid/liquid systems and its integrated form is given below:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (7)$$

where q_t (mg g^{-1}) is the adsorption capacity at time t (min^{-1}) and k_1 (min^{-1}) is the rate constant of the pseudo-first-order adsorption.

The kinetic data were further analyzed with Ho's pseudo-second-order kinetics model [23]. expressed as:

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \quad (8)$$

The adsorption process follows generally three consecutive steps of external diffusion, intraparticle diffusion, and actual adsorption. One or more of these steps control the adsorption kinetics altogether or individually. The kinetic data were also analyzed by intraparticle diffusion kinetics model [24] to determine the rate-controlling step:

$$q_t = k_p t^{1/2} + C \quad (9)$$

where k_p ($\text{mg min}^{-1/2} \text{g}^{-1}$) is the intraparticle diffusion rate constant and C (mg g^{-1}) is a constant, k_2 ($\text{g mg}^{-1} \text{min}^{-1}$) is the rate constant of the pseudo-second-order adsorption.

For elucidation of the adsorption rate-controlling mechanism, the Wünlwald-Wagner intraparticle diffusion model was proposed to give the intraparticle diffusion coefficient, D ($\text{cm}^2 \text{s}^{-1}$) [25]:

$$\log\left(1 - \frac{q_t}{q_e}\right) = \log\left(\frac{6}{\pi^2}\right) - \frac{4\pi^2 D}{2.303d^2} t \quad (10)$$

where d (cm) is the mean particle diameter.

3. Results and discussion

3.1. Effect of Initial pH of solution

The pH value of the solution was an important factor in the adsorption process. The initial pH of the working solutions was adjusted to between pH 2.0–12.0 by addition of dilute HCl (1.0 mol L^{-1}) or NaOH solution (1.0 mol L^{-1}). The effect of pH on the adsorption of sunset yellow was shown in Fig. 2.

As shown in Fig. 2, a sharp decrease in the adsorption capacity occurs as the pH of the initial solution was increased from 2.0 up to 4.0, beyond which the adsorption capacity remains almost zero. The higher adsorption capacity of sunset yellow at low pH may result from enhanced protonation of $-\text{NH}_2$ at the surface of the EMPH. It is contributed to the preferential adsorption of the negative sunset yellow dye ions to positive active sites and facilitates the diffusion process. With increasing pH, protonation reduces and electrostatic repulsive force becomes dominant, which inhibits the diffusion and adsorption of sunset yellow onto EMPH [6,9].

3.2. Effect of contact time

Removal of sunset yellow by EMPH was performed with three different initial concentrations of

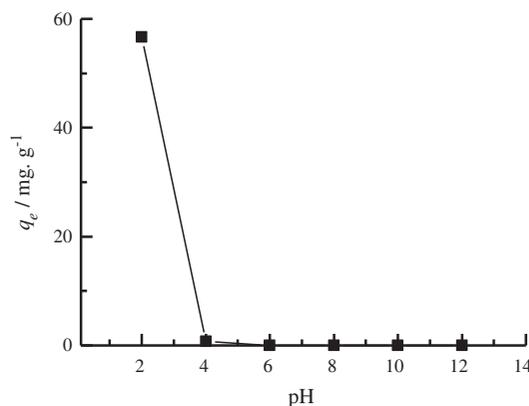


Fig. 2. Effect of pH on the adsorption of sunset yellow ($T = 303 \text{ K}$, $c_0 = 199.1 \text{ mg L}^{-1}$, contact time = 24 h, rpm = 150).

sunset yellow at temperature 303 K. According to the results (Fig. 3), the removal of sunset yellow was faster in initial 300 min and then it became slower and finally reached equilibrium at approximate 1,200 min. During initial stages, a large number of vacant sites were available on the surface of EMPH and the adsorption of sunset yellow was very quick. Then, the adsorption process became slower due to gradual occupancy of vacant sites. With increasing time, the occupation of the remaining vacant sites became more difficult due to increased repulsive forces between dye molecules and bulk solution [7,9].

Fig. 3 also shows that the equilibrium capacity of EMPH increased from 76.02 to 93.01 mg g^{-1} as increasing initial concentration of sunset yellow because the initial dye concentration provides an important driving force to overcome all mass transfer resistance [6–8]. The increase of loading capacity of EMPH with increasing concentration of sunset yellow may also be derived from the higher interaction between sunset yellow and EMPH.

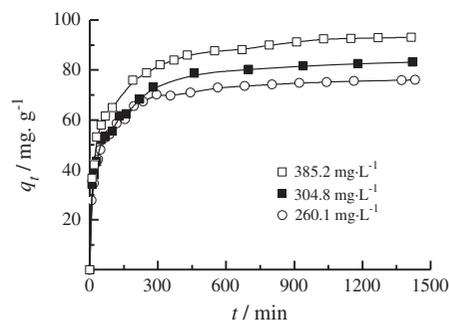


Fig. 3. Effect of contact time on the adsorption of sunset yellow ($T = 303 \text{ K}$, pH 2.0, rpm = 150).

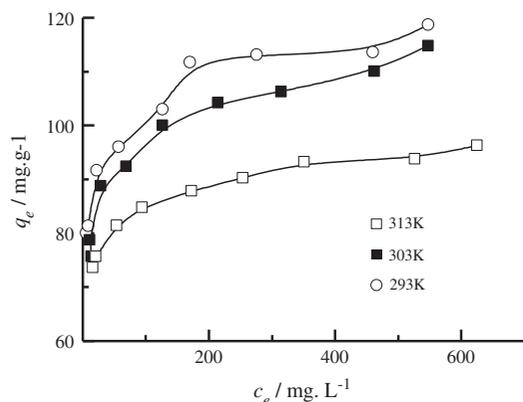


Fig. 4. Adsorption isotherms of sunset yellow on EMPH (pH 2.0, contact time = 24 h, rpm = 150).

3.3. Adsorption isotherms parameters

The adsorption isotherms of EMPH at different temperatures are depicted in Fig. 4. According to Fig. 4, the adsorption of sunset yellow onto EMPH was enhanced by increasing the temperature from 293 to 313 K at different initial dye concentration. The Langmuir and the Freundlich adsorption isotherms were used to evaluate the adsorption equilibrium data. The Langmuir plot of c_e/q_e against c_e yields straight lines. The isotherm constants and correlative coefficients were tabulated in Table 1. The correlative coefficient R^2 values in Table 1 confirm that the adsorption equilibrium data are fitted very well with the Langmuir model under the studied conditions (all above 0.99). This indicates uniform adsorption and strong dye-adsorbent interactions over the surface of the adsorbent. The maximum monolayer capacity of EMPH is determined as 96.2, 114.9, and 117.6 mg g^{-1} for 293, 303, and 313 K, respectively, which suggests an endothermic process. The values of Freundlich

Table 1
Langmuir and Freundlich isotherm constants of sunset yellow on EMPH

T (K)	Langmuir constants			Freundlich constants		
	q_{\max} (mg g^{-1})	K_L (L mg^{-1})	R^2	n	k_F	R^2
293	96.2	0.092	0.9993	14.3	61.2	0.9941
303	114.9	0.078	0.9982	10.3	61.5	0.9723
313	117.6	0.11	0.9987	11.5	68.7	0.9779
313 ^a	27.2	0.15	0.9892	11.9	5.6	0.9209

^aIsotherms regression data of the unmodified peanut husk at 313 K.

constant, k_F , increased with increasing temperature and showed easy uptake of sunset yellow by EMPH. All n values were found to be greater than 1, which indicated a favorable adsorption condition in this process [7].

Table 1 also showed that the maximum adsorption capacity of EMPH for sunset yellow was 117.6 mg g^{-1} at 313 K, which was improved 4.3 times as much as that of the unmodified peanut husk (27.2 mg g^{-1}).

3.4. Thermodynamic parameters

The isosteric enthalpies ΔH at different adsorption levels can be easily calculated from Eq. (4). The plotting of $\ln c_e$ vs. $1/T$ yields a straight line with slope equal to $\Delta H/R$ (Fig. 5). The calculated values of ΔH are listed in Table 2.

Based on the analysis of Table 2, an endothermic physical adsorption process is confirmed by the positive values of all enthalpies. The numerical value of ΔH ($<40 \text{ kJ mol}^{-1}$) also predicts the physisorption behavior which is in agreement with the adsorption theory of Langmuir mode. The decline in enthalpies with loading suggests a heterogeneous surface of the adsorbent and indicates that the higher energy sites are favored and dominate at lower loading [21].

For the systems where the Freundlich isotherm could be fitted to the experimental data (Table 1), the Gibbs free energy values ΔG (kJ mol^{-1}) were calculated using Eq. (5). The negative values of ΔG indicate the spontaneous nature of sunset yellow adsorption onto EMPH.

The entropy change ΔS ($\text{J mol}^{-1} \text{K}^{-1}$) was calculated from Eq. (6). The positive value of ΔS reflects the affinity of EMPH to sunset yellow and also indicates the increased randomness at the solid/solution

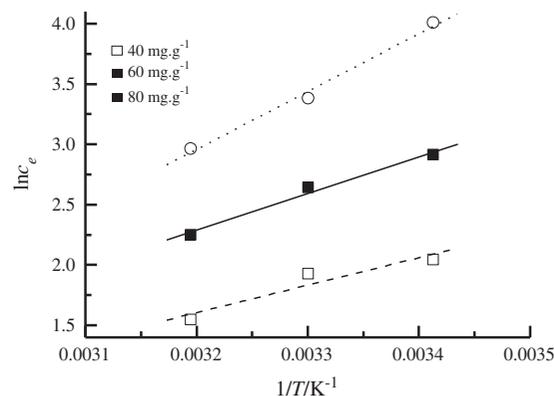


Fig. 5. Determination of isosteric enthalpies of adsorption of sunset yellow on EMPH.

Table 2
Thermodynamic properties of the systems tested

q_e (mg g ⁻¹)	ΔH (kJ mol ⁻¹)	ΔG (kJ mol ⁻¹)			ΔS (J mol ⁻¹ K ⁻¹)		
		293 K	303 K	313 K	293 K	303 K	313 K
40	39.88	-34.8	-25.84	-30.05	254.89	216.90	223.42
60	25.28				205.06	168.71	176.78
80	18.87				183.18	147.56	156.30

interface during the adsorption process. Similar results were reported in literature [10,17].

3.5. Kinetic parameters of adsorption

For investigation of the adsorption process of sunset yellow onto EMPH, the pseudo-first-order, the pseudo-second-order and the intraparticle diffusion models were used to correlate the experimental data of Fig. 3.

The values of k_1 , q_e and correlative coefficients of the pseudo-first-order model were listed in Table 3. Low correlative coefficients and a large difference of equilibrium adsorption capacity (q_e) between the

experiment and calculation reveal a poor fit, which indicate that the film mass transfer can be ignored under the present experiment conditions.

The rate constant k_2 , the q_e value and correlative coefficient R^2 of the pseudo-second-order kinetics were calculated from the linear plots of t/q_t against t and the results were given in Table 3. The correlative coefficients are all greater than 0.99 and the calculated q_e values are also very close to the experimental data, which indicated that the pseudo-second-order model can provide a perfect fit to the experimental data. The adsorption of sunset yellow onto EMPH can be considered as a chemical reaction between sunset yellow and EMPH based on electron exchange or charge sharing [26–28].

Table 3
Statistical results of the application of the kinetic models

Model			Initial sunset yellow concentration (mg L ⁻¹)		
			260.1	304.8	385.2
First-order kinetic	k_1	Rate constant (min ⁻¹)	3.2	2.5	2.8
	$q_{e,cal}$	Equilibrium capacity (mg g ⁻¹)	27.2	35.1	37.4
	R^2	Correlation coefficient	0.9232	0.8968	0.9291
Second-order kinetic	k_2	Rate constant (g mg ⁻¹ min ⁻¹)	4.5	3.3	3.1
	$q_{e,cal}$	Equilibrium capacity (mg g ⁻¹)	76.9	84.7	94.3
	R^2	Correlation coefficient	0.9996	0.9989	0.9992
Intraparticle diffusion	k_p	Rate constant (mg min ^{-1/2} g ⁻¹)	2.1	2.8	4.5
	R^2	Correlation coefficient	0.9764	0.9946	0.9598
Wünwald-Wagner intraparticle diffusion	D	Effective diffusion coefficient (cm ² s ⁻¹)	4.7	5.1	6.7
	R^2	Intercept	-0.9369	-0.8585	-0.6975
	$q_{e,exp}$	Correlation coefficient	0.9937	0.9988	0.9634
		Experimental data of the Equilibrium capacity (mg g ⁻¹)	77.5	84.9	94.9

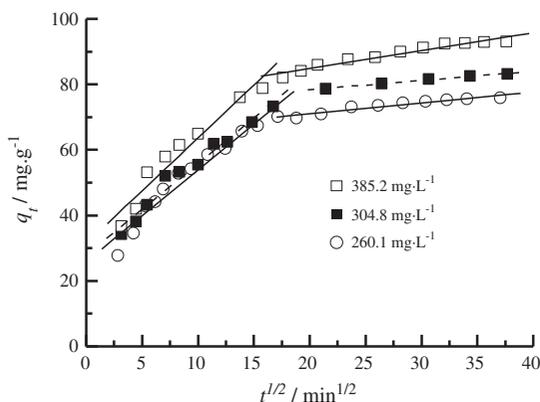


Fig. 6. Intra-particle diffusion model plots at different initial concentrations.

The possibility of intraparticle diffusion cannot be overlooked because of the long adsorption equilibrium time in the present study. For obvious illustration of this point, intraparticle diffusion model was proposed to describe the adsorption process (Fig. 6). The time dependence of q_t in Fig. 6 could present two straight lines which could be well fitted linearly. The multilinearity suggested that the intraparticle diffusion was dominant in sunset yellow adsorption. The q_t in the first portion showed a rapid increase with time, which was attributed to the rapid external diffusion of dyes to the surface of EMPH. The second portion corresponded to the intraparticle diffusion effect. The values of k_p and correlation coefficients were also shown in Table 3. It is found that correlation coefficients for the intraparticle diffusion model were all greater than 0.95. However, the linear plots did not pass through the origin of coordinates, which indicated that the intraparticle diffusion was not the sole rate-controlling step. A similar discovery was observed for the adsorption of methylene blue by polar leaf and the adsorption of sunset yellow by peanut husk [8,10].

Table 3 also listed the calculated results of Wünlwald-Wagner intraparticle diffusion model. The values of the internal diffusion coefficient fall within the magnitudes reported in literature [25], which suggested that the adsorption of sunset yellow should be governed by intraparticle diffusion mechanism.

3.6. Comparison of the q_{max} of various adsorbents

A list showing the maximum adsorption capacity of different materials for the adsorption of sunset yellow from its aqueous solution is given in Table 4. From Table 4, it was observed that the adsorption capacity of EMPH for sunset yellow was comparable

Table 4

Comparison of adsorption capacities of various adsorbents for sunset yellow

Absorbent	Langmuir q_{max} ($mg\ g^{-1}$)	T ($^{\circ}C$)	Refs.
Ethylenediamine-modified sawdust	117.6	40	Present work
Unmodified peanut husk	27.2	40	Present work
Unmodified peanut husk	28.9	20	[10]
Chitosan-coated illite	135.6	25	[29]
3-Chloro-2-hydroxypropyltrimethylammonium chloride modified <i>Ramulus mori</i>	18.2	25	[30]
Macroporous resin D301T	31.5	55	[31]
Glutaraldehyde crosslinked arginine	151.5	25	[32]

to other low-cost adsorbents. This provided strong evidence for the potential of EMPH in technical applications of toxic dye removal from aqueous solutions.

4. Conclusions

The adsorption of sunset yellow dye onto ethylenediamine modified peanut husk (EMPH) was carried out in batch mode. The results indicated that the adsorption capacity of the EMPH was considerably affected by pH value, initial dye concentration, and temperature. Based on the correlative coefficients, the Langmuir isotherm model can provide a better description when compared with the Freundlich equation. The intraparticle diffusion kinetics gave two linear regions, which indicated that the intraparticle diffusion was not the sole rate-controlling step. The results of the study indicated EMPH can be used as an effective adsorbent for dye removal.

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