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Pretreated Fishbone as Low Cost-Adsorbent for Cationic Dye Adsorption from Aqueous Solutions: Equilibrium, **Optimization, Kinetic and Thermodynamic Study**

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Abstract. The present study investigated the use of pretreated fish bone (PTFB) as a new surface, natural waste and low-cost adsorbent for the adsorption of Methyl green (MG, as model toxic basic dye) from aqueous solutions. The functional groups and surface morphology of the untreated fish bone (FB) and pretreated fish bone were characterized using Fourier transform infrared (FTIR), scanning electron microscopy (SEM) and Energy dispersive X-ray spectroscopy (EDS), respectively. The effect of operating parameters including contact time, pH, adsorbent dose, temperature, and inorganic salt was evaluated. Langmuir, Freundlich and Temkin adsorption isotherm models were studied and the results showed that the adsorption of basic dye followed Freundlich isotherm. Kinetic modeling of the data at different temperatures confirmed pseudo-second-order(P-2-O) model, along with calculated thermodynamic parameters depicted that the adsorption process is spontaneous and endothermic in nature. Diffusion studies suggested that intra-particle diffusion is not the singular rate-controlling factor. The results indicated that 92% of MG capable of being sequestered under optimum adsorption conditions: pH 10.64, adsorbent dose 0.1 g/L, and 60 min contact time. Finally, the results showed that the pretreated fish bone can be effectively used as a proper adsorbent for the adsorption of cationic dye from aqueous solutions.

Keywords: Fishbone, Methyl green, Temkin isotherm, Thermodynamic parameters, Intra-particle diffusion

1. Introduction

The pollution of water stands for a serious environmental problem for human and aquatic systems. Many applications such as printing, tanning, food, textile, and cosmetics industries typically employ biological molecules as dyes to color their products. It has been supposed that cationic dyes have been in general more toxicity levels as compared with anionic dyes [1]. Cationic (basic) dyes have typically employed in silk, nylon, acrylic and wool dyeing in textile production owing to noble solubility of synthetic dyes, and may be existing in trace quantities in built-up wastewaters. The existence of cationic dyes in aquatic streams averts light from water penetrating and has a passive influence on photosynthesis. Features like poisonousness and non-biodegradability stand for the consequence of complex dyes structures. Consequently, they represent against risk to environmental health and human. Therefore, the removal of cationic dye from aquatic system before discharging into water bodies is needed [2]. Methyl green (MC), a cationic dye, is one of the most frequently used dye stuff and it causes hazardous problems both water environments and human life.

Many treatment systems have been used for the removal of different kinds of dyes from aqueous solutions such as, flocculation [3], photocatalytic degradation [4], microbiological decomposition [5], fungus biosorbent [6], and adsorption [7].

Recently, adsorption method has been initiated as being economical and more effectual as compared with physical and chemical techniques. Its process has succeeded in eliminating organic and inorganic pollutants [8], as a result of being cost-effective, astraight forward process and simple design.

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Recently, diverse adsorbent materials were employed for removing organic material and color. Naturally, cheap and in effect materials like agronomic materials, food manufacturing by-products and animal bones like fish bones were employed as adsorbents for the removal of organic and inorganic contaminates in recent times. Several economical absorbents in the literature were used for dyes removal including lignocellulosic material [9], sunflower seed hull [10], coconut coir [11], bauxite clay [12], and fish bone [13]. Bones have 70 % inorganic and 30 % organic phases by weight [14]. The hydroxyl apatite Ca10(PO4)6(OH)2 creates the foremost portion of inorganic phase and it is an effectual adsorption material as a result of its capability or removing completely charged contaminates based on ion exchange reacting with calcium ions on a bone surface [2].

The main objective of the present research is to use fish bone (FB) as new adsorbent and to study its capability for the adsorption of cationic dye from aqueous solutions. As a model dye, methyl green was selected for the adsorption analyses. In our current study, the surface categorization of PTFB will be examined using Fourier transform infrared (FTIR), scanning electron microscopy (SEM) and Energy dispersive X-ray spectroscopy(EDS). Additionally, contact time of adsorption, adsorbent dose, temperature, pH, and salt effect parameters were investigated. The adsorption kinetic, isotherm behaviors were compared and the thermodynamic parameters were determined as well.

2. Experimental

2.1 Pretreatment of fish bones sorbent

Fish bones was collected from a local fish shops in Al-Kadhumyia city, Iraq. Firstly, the fish bones were washed for a number of times with hot purified water for removing the soluble and residue impurities from the bones. Then, the washed fish bones was dried up in the oven at 90 °C and ground into fine powder by a mortar.

2.2. Pretreatment of fish bones sorbent

Fifteen gram of ground fish bones was treated with100 mL of 0.1 M aqueous solution of NaOH and stirred at room temperature for 4 hours. Then, 3g ofAl₂O₃ gradually added and stirred at room temperature for 5 hours. The treated fish bones were filtered, washed several time with distilled water, and then dried in an oven at 80°C for 6 hours; milled and sieved in the size \leq 75µm. Lastly, the treated fish bones powder was kept in the air tight container to be ready for a use.

2.3. Adsorption experimental method

Basic dye, Methyl green (MG), was employed without additional purification. The dye features and chemical structure are listed in Table 1. The stock solution was prepared by dissolving 1 g of dye in 1000 mL distilled water. The pH solution was adjusted to 7.42 for MG as optimum value obtained from the optimization study conducted in our previous study[15].NaOH (Merck) and HCl (Merck) have employed for adjusting the pH of the solutions. NaCl that have employed for studying the influence of mineral salt on dye removal has as well gotten from Merck. To examine the effect of temperature, 0.1 g of fish bones with 10mL MG dye solutions of known concentration (60-140mg.L⁻¹) in 100 mL Erlenmeyer flasks, shaken on a temperature-controlled shaker at 25, 35 and 45 °C. After the equilibrium time elapsed, the suspension was centrifuged at 3000 rpm for 5 min., then filtered to be analyzed for MG concentration using UV–Vis spectrophotometer (double beam, T-80, England) at a maximum wavelength of 618 nm. The MG uptake loading capacity (mg/g) of fish bones for each concentration at equilibrium was determined as follows:

$$q_e = (C_o - C_e) \times V/W_t$$

(1)

Here, C_o and C_e (mg/L) are for the initial and equilibrium solution concentrations of MG, respectively, V (L) is for the volume of the dye solution, and W_t (g/L) represents the dose of used adsorbent. The MG removal percent was calculated for each run by the following equation:

$$R\% = (C_o - C_e)/C_o \times 100$$

IUPAC Name	Molecular Formula	Molecular Weight	Synonyms	color
[4-[[4-(dimethylamino)phenyl]-(4- dimethylazaniumylidenecyclohexa-2,5-dien-1- ylidene)methyl]phenyl]- trimethylazanium;dichloride	C ₂₆ H ₃₃ N ₃ Cl ₂	458.471 g/mol	Basic Blue 20, Methyl green chloride	Blue- green
~ N. 	×− ×	CI-	Cl	

Table 1. Dye structure and characteristics.

(2)

2.4. fish bondes Characterization

Characterization of the fish bones has investigated using scanning electron microscopy (T-SCAN Mira3 France) and Energy dispersive X-ray spectroscopy (EDS, Hitachi, Ltd., Tokyo, Japan)to investigate the surface morphology of fish bones. Fourier transform infrared (FTIR) analysis (Shimadzu 8400, Japan) in the scanning range of(4000–500 cm⁻¹)was used to identify the functional groups of fish bones surface.

3. Results and Discussion

3.1. FTIR spectroscopy analysis

The FTIR analysis of untreated Fish bones and PTFB is given in (Figure 1.a,b). The band in 'Figure1a' observed at 3344.68 cm⁻¹ can be assigned to the O-H stretching bond. The two bands at 2922.55 and 2852.81 cm⁻¹ were attributed to the C-H stretching bond. The stretching band of C=O groups is placed at 1741.78 cm⁻¹ [2]. The bands at 1020.38 and 960.58 cm⁻¹ are related to vibrations of $-PO4^{-3}$ groups. The stretching mode of N=O is attributed to band at 1456.30 cm⁻¹. The band at 871.85 cm⁻¹ is assigned to C-C stretching [16]. After treatment with NaOH and Al₂O₃ 'Figure 1b', the band at 3344.68 cm⁻¹ shifted and changed the intensity of this band at around 3473.91 cm⁻¹. The band at 981.80 cm⁻¹ is assigned to Al–O bond vibrations. The bands at 848.71 and 642.32cm⁻¹ are characteristics of vibrational modes of alumina O-Al-O bond [17].







Figure 1b. FTIR spectra of pretreated fish bones surface (PTFB).

3.2. SEM analysis

The SEM images for characterizing fish bones surface before and after treatment are depicted in 'Figure2 a,b'. Before treatment 'Figure2a', the fish bone shows a clean surface with several numbers heterogeneous pores and holes, which create a huge free surface area [2]. After treatment 'Figure2b', the morphology of fish bones has roughness surfaces and the distinct openings of pores as well enable the dye ions accessibility into the internal bones part [16,18].



Figure 2.SEM images of fish bones surface(a) untreated FB (b) PTFB.

3.3. Elemental analysis

Elemental analysis of untreated FB and PTFB surfaces was examined by energy dispersive X-ray spectroscopy (EDS) analysis. Detailed composition of untreated FB and PTFB samples with EDS diagram is given in 'Figure3a, b'. In the EDS spectrum 'Figure 3 a' of untreated FB (C, O, Ca and P) were detected. The EDS spectrum after treatment with NaOH and Al_2O_3 revealed the existence of Al element (17.58 W_t%) of FB surface 'Figure 3 b'. The content of O, P and Ca increased from 34.29 W_t%, 8.59 W_t% and 10.31 W_t% to 37.08 W_t%, 12.62 W_t% and 15.62 W_t%, respectively, while the content of C declined from 46.45 W_t% to 17.10 W_t%. It was an evident that most of Al is deposited on the fish bones surface.



Figure 3.Energy dispersive X-ray analysis images and composition of fish bones surface (a) untreated FB (b) PTFB.

3.4. Effect of sorbent dose

Regulating the influence of adsorbent dose in experiments, one of the useful significant parameters influencing adsorption, have accomplished with a primary dye concentration of 80 mg/L and adsorbent dose in the interval 0.05-0.8g/L at 25° C. The experiment consequences have illustrated in 'Figure4'. As the adsorbent dose has increased, the MG dye removal efficiencies have raised from 79.3% to 98.6%. Actually, the amount of existing adsorption sites rises by raising the adsorbent dose and volume of pores wherever adsorption takes a place. Accordingly, the highly applicable adsorbent dose for MG dye removal has adjusted to 0.8 g/L[19]. Similar observations have been made earlier [20, 21].



Figure 4.Effect of adsorbent dose on adsorption of MG onto PTFB surface(initial MG conc. 80 mg/L, pH 7.42 and 25 °C).

3.5. Salt effect

The effect of inorganic salt (i.e. NaCl) on adsorption capacity of MG on PTFB surface is shown in Figure 5.The dye adsorption increased from 7.11 to 7.94 mg/g with the increasing NaCl concentration ranging from 0.1 to 0.3 M.This MG attachment to the solid can be regulated through electrostatic interactions increasing along with its adsorption capacity. The increased MG adsorption capacity under salt adding is based on the dye molecules aggregation generated by the salt ion actions, and this raises the adsorption capacity of dye onto PTFB surface [22].



Figure 5. Effect of ionic strength on MG adsorption (initial MG conc. 80 mg/L, pH 7.42 and 25 °C).

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3.6. Effect of pH

The pH effect on MG adsorption by PTFB surface was studied at 25°C, initial concentration of 80 mg/L and adsorbent dose of 0.1 g. To obtain the desired pH a few drops of 0.1 M NaOH or HCl was added. Figure6 explains the influence of pH on the adsorption of MG at pH range of 1.88–10.64. The dye removal was increased with increasing pH. The low value of dye removal at pH 1.88 is may be as a result of the protonation of functional groups that creates the adsorbent surface in more positive and investigate the repulsion among dye cations and adsorbent [2]. When the pH is increased, the amount of negatively charged sites on the surface of adsorbent surface increased and the removal percentage of MG as well rises. This might be due to the increasing in electrostatic attractions among the MG cation and the adsorbent's functional groups [23]. Related performance has stated by other scholars for the adsorbing basic dye in [24, 25].



Figure 6.Effect of pH on MG adsorption(initial MG conc. 80 mg/L and 25°C).

3.7. Kinetic modeling

A kinetics study of adsorption was necessary to investigate the effect of contact time. The resulting parameters of adsorption kinetic, as shown in 'Figure 7', after a contact time of 30min, there was no change in cationic dye (MG) adsorption capacity on PTFB surface. Thus, we selected 60 min as the best contact time for all the MG adsorption experiments on the adsorbent surface [26]. The rapid uptake of MG dye molecules from aqueous solution refers to the adsorption process efficiency. Moreover, it is evident that the adsorption capacity of PTFB surface increased rapidly with increasing of contact time due to the availability of active sites on the surface [27].



Figure 7.The effect of contact time of MG adsorption onto PTFB surface (initial MG conc. 80 mg/L, pH 7.42 and 25 °C).

The adsorption process kinetics were studied using the pseudo-first-order (P -1-O)equation 3 [28], and the pseudo-second-order(P-2-O) equation 4 [29] models

$$\ln(q_{e}-q_{t}) = \ln q_{e}-k_{1}t$$
(3)
$$\frac{t}{qt} = \frac{1}{k_{2}q_{t}^{2}} + \frac{1}{q_{e}}t$$
(4)

Here, $k_l(\min^{-1})$ and $k_2(g \cdot mg^{-1} \cdot min^{-1})$ are the kinetic rate constants, respectively. q_e and q_t (mg/g) are the MG adsorbed concentrations at equilibrium and at time of adsorption t (min), respectively. The P -1-O constants (k_1 and q_e) were calculated from slopes and intercepts as given in Figure 8a. P -2-O constants k_2 and q_e were determined from the slopes and intercepts from 'Figure 8 b', respectively. The results of experimental data with the (P -1-O) and (P -2-O) for adsorption of MG onto PTFB have shown in Table2.Consequences indicated great correlation coefficient magnitudes by ranging (0.9999-0.9997) for system and a good agreement between (q_e cal.) and (q_e exp.) magnitudes specifying the applicability of the P-2-O kinetic model for PTFB system.[30].Related consequences have found in [31,32].



Figure 8. Pseudo-first order (a) and Pseudo-second order(b) kinetics for MG dye adsorption onto PTFB surface at different temperatures.

Table2. Kinetics parameters for the adsorption of MG onto PTFB surface ($C_0 = 80 \text{ mg/L}$).

			Pseudo-first order			Pseudo-second order		
Adsorbent	T(K)	$K_1(min^{-1})$	q _e , _{exp} mg/g	q _{e,calc} mg∕g	R^2	$K_2(g \cdot mg - 1 \cdot min^{-1})$	q _{e,calc} mg/g	R^2
	298	0.0747	6.7381	0.5387	0.9578	0.1112	6.8965	0.9997
PTFB	308	0.0579	7.0686	2.6093	0.9970	0.1361	7.1428	0.9999
	318	0.0448	7.9889	2.4783	0.9266	0.1547	8.0000	0.9998

3.8. Kinetic model validity

The fitting and applicability of the isotherm model to the kinetic parameters have compared through judging from the R^2 magnitudes and the normalized standard deviation Δq_t (%) determined from equation 5. The normalized standard deviation, Δq_t (%) has employed for confirming the used kinetic model to refer to the adsorption process. It is given as:

$$\Delta \mathbf{q}_{t} = 100 \frac{\sqrt{\Sigma(\frac{q_{exp} - q_{cal}}{q_{exp}})^{2}}}{n-1}$$
(5)

Where n is the data points number, q_{exp} and q_{cal} (mg/g) stand for the experimental and calculated adsorption capacity magnitudes. Lower magnitude of Δq_t specifies very well agreement between experimental and calculated data. The Δq_t magnitudes for p-2-O are 4.2666%, 3.3900% and 2.7166% at 25°C, 35°C and 45°C, respectively. Thus, the pseudo-second-order kinetic model appears to be the best fitting model for adsorption of MG dye onto PTFB surface[33].

3.9. Adsorption mechanism

Adsorption kinetics can be typically controlled by various mechanisms. The highly general ones are based on the diffusion mechanisms that can be clarified by intraparticle diffusion model suggested by Weber and Morris [34]. Intraparticle diffusion model is given as

$$q_t = k_{id} t^{0.5} + I (6)$$

 k_{id} , the intra-particle diffusion rate constant (mg/g min^{0.5}) can be obtained from the slope of the linear plot of q_t versus $t^{1/2}$ and I (mg/g) is a constant 'Figure9'. The interceptions of plot reveals the boundary layer influence. Greater magnitudes of intercept means bigger contribution of surface sorption in the rate-controlling step. If the regression of q_t against $t^{0.5}$ is linear and passes through the origin, then intraparticle diffusion is the solitary rate-limiting step. In the case of linear plots at every concentration didn't pass through the origin, it shows that the intraparticle diffusion wasn't only rate controlling step [35]. The magnitudes of k_{id} and I and the resultant regression coefficients have recorded in Table 3. The magnitudes of K_{id} and I listed in Table3 explain that the constants I values are not zero. This specified that the intraparticle diffusion is not the only rate-limiting step but other process is feasibly be involved in the adsorbing process [8].



Figure 9. The intra-particle diffusion for MG dye adsorption onto PTFB surface at different temperatures.

Table 3. intraparticle diffusion rate constants for the adsorption of MG onto PTFB surface at different temperatures.

Adsorbent	T(K)	k_{id} (mg/g min ^{0.5})	I (mg/g)	\mathbb{R}^2
PTFB	298	0.1548	5.7654	0.9747
	308	0.1347	6.1739	0.9969
	318	0.1187	7.1374	0.9435

3.10. Equilibrium study

For optimizing the design of adsorption system for the adsorbates adsorption, it is essential to create the most suitable correlation for the equilibrium curves. Diverse isotherm models have been reported to refer to the equilibrium features of adsorption. Several of these models are Langmuir, Freundlich, and Temkin models.

Langmuir isotherm is effective for monolayer adsorption. The linearized equation can be represented as follow [36]:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_e} \tag{7}$$

Where, $C_e(mg/L)$ is equilibrium concentration of MG in solution, $q_m (mg/g)$ is the Langmuir monolayer adsorption capacity and $K_L(L/mg)$ is Langmuir constant. q_m and K_L values can be determined from the slope and intercept of the linear plots of C_e/q_e against C_e .

The Freundlich isotherm model, which is frequently employed for describing non-specific adsorption, was employed to clarify the adsorption data. The linear form of the Freundlich equation as given as below [37]:

$$logq_e = logK_f + \frac{1}{n}loqC_e \tag{8}$$

Where, K_F is Freundlich coefficient related to adsorption capacity(mg/g) and n(related to adsorption intensity) were obtained from the values of slope and intercept of the plots of log q_e against log C_e

Regarding Temkin adsorption isotherm, the energy of adsorption is in a linear function of the surface coverage because of adsorbent-adsorbate interactions. Temkin isotherm equation has in general been adopted as [38]:

$$q_e = B \ln \ln A_T + B \ln \ln C_e \tag{9}$$

Where B = RT/b, b stands for Temkin constant and associated with heat of adsorption (J/mol), A represents Temkin isotherm constant (L/g), R for the gas constant (8.314 J/mol K), and T is the absolute temperature (K). The magnitudes of (B) and (A_T) can be determined from the slope and interception by the graph plotted between q_e and lnC_e.

The isotherm parameters for the three models are introduced in Table 4. In terms of R^2 values, Freundlich isotherm skwed the highest values for dye within the temperature range used.

Isotherm	298 K	308 K	318 K
Langmuir			
$K_L(L/mg)$	-0.0362	-0.0321	12.3456
$q_m(mg/g)$	-8.1967	-17.2413	13.5135
R^2	0.9751	0.9043	0.9930
Freundlich			
$K_{\rm F}$	0.0492	0.2513	10.6340
1/n	1.9555	1.5170	1.0267
\mathbf{R}^2	0.9919	0.9925	0.9952
Temkin			
B _T (J/mol)	15.6479	12.7073	1.3572
$A_T(L/g)$	0.1262	0.1969	2921.9310
R^2	0.9597	0.9683	0.9820

Fable 4. Isotherm	parameters for	MG adsorption	onto PTFB surface.
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3.11. Thermodynamic analyses

The uptake of MG dye by the PTFB rises with raising the temperature ratifying the endothermic nature of the adsorption step. The variation in standard free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) of adsorption have computed by [39]

$$\Delta G^{\circ} = -RT \ln K^{\circ} \tag{10}$$

Where R is the gas constant $(8.314 \text{J} \cdot \text{mol}^{-1} \text{K}^{-1})$ and T is the absolute temperature. The distribution adsorption coefficient (K_d) [40] is determined by the following equation:

$$K_d = \frac{c_o - c_e}{c_e} \times \frac{v}{w} \tag{11}$$

Where C_o is the initial MG concentration and C_e is the equilibrium MG concentration in (mg/L). V is the sample volume (mL) and W_t is the weight of the adsorbent (g/L).

The standard enthalpy change (ΔH°) and the standard entropy change (ΔS°) were calculating using equation 12:

$$\ln K^{\circ} = (\Delta S^{\circ}/R) - (\Delta H^{\circ}/RT)$$

(12)

The equilibrium constant (K[°]) was determined by plotting lnK_d against C_e and extrapolating C_e to. The value of the intercept is that of lnK° [12].

Thermodynamic parameters obtained are summarized in Table5. The negative values of ΔG° obtained reveal that the adsorption process is favorable and that it is spontaneous and is observed to decrease with increase in the temperature, signifying that the adsorption process of MG on PTFB surface is more advantageous under higher temperatures [41]. Positive magnitude of ΔH° indicates endothermic features of the adsorption process. The positive ΔS° magnitude for MG adsorption on PTFB indicates the randomness and reveals the affinity of adsorbent surface concerning MG[42].

Adsorbent	T(K)	$\Delta G^{\circ} (kJ.mol^{-1})$	ΔH°	ΔS^{o}
		. ,	(KJ. mol)	(J. K mol.)
	298	-13.3819	214 0082	757 5009
PTFB	308	-15.8513	214.0082	/3/.3908
	318	-28 7596		

Table5. Thermodynamic parameters for the adsorption of MG.

4. Conclusions

Fish bones, one of the most numerous fish-processing industry waste and natural products, were used for MG adsorption. With many specific advantages such as low- cost, availability, natural origin, and high adsorption capacity. In this study, the adsorption of basic dye (methyl green) from aqueous solutions was based on pretreated fish bone as a new adsorbent surface. Kinetic, isotherm, and thermodynamic parameters of the adsorption process were investigated. The resultant data of adsorption process are fitted in good manner to Freundlich isotherm. The second-order-model has been in highly agreed to the acquired results. The adsorption process has been spontaneous and endothermic in nature in relation to the negative and positive magnitudes of ΔG° and ΔH° , respectively. The influence of different parameters including, pH, contact time, temperature, inorganic salt and adsorbent dose was studied. The resultant has depicted that pretreated fish bones can be efficiently employed as a low-cost adsorbent for the adsorption of basic dyes from aqueous solutions and could contribute to the protection of the environment.

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