

Kinetics of Direct Yellow 50 Dye Adsorption onto Marble Powder Sorbents

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ABSTRACT: The aim of this present work is evaluation of the effect of marble powder, which is an industrial discharge, in the adsorption of a direct “yellow 50” dye, used in traditional industries of textile. On the other hand, various techniques for characterizing the adsorbent were used, in particular X-ray Diffraction (XRD), X-ray Fluorescence spectroscopy, Scanning Electron Microscopy (SEM), Fourier Transform Infrared spectroscopy (FTIR) and Raman spectroscopy. The effect of various experimental factors; adsorbent dose, contact time (equilibrium is established after 20 minutes), dye concentration and pH media, were studied by using the batch technique. The isotherms of adsorption data were analyzed by various adsorption isotherm models such as Langmuir and Freundlich. The dye yellow adsorption by marble powder made have excellent performance related to the adsorption potential.

KEYWORDS: Adsorption isotherm, Marble powder, Direct Yellow 50.

I. INTRODUCTION

The environmental's problem issue that arises in the textile industry is the amount of water used for wet processing of textiles and discarded rich chemical load [1], without pre-treatment. However, wastewater from printing units and textile dyeing are often colored and contain residues of dyes and chemicals products, which require appropriate treatment [2]. To meet legislative requirements, textile wastewaters are most often treated by different physicochemical [3-4] and biological [5-6] methods. Nevertheless, the elimination of textile dyeing effluents in aquatic ecosystems has a considerable interest by researchers due to reported data on the mutagenic and genotoxic potential of textile dyes [7]. Thereby, can be removed by adsorption process in which the dye binds to the adsorbent.

Color removal from effluents polluted with dyes of textile industries has been considered a challenge due to the difficulty of treating such wastewaters by conventional methods. The effluents of manufacturing and textile industries are discarded into process is one of the most effective and economically feasible methods for the removal of dyes from aqueous solution [8]. Large number of low-cost and biodegradable adsorbents can be exploited by researchers. These adsorbents can be obtained from natural resources for the elimination of various dyes at different conditions.

The adsorption by activated carbon seems to be the best prospect for the elimination of dyes. Despite its effectiveness, this adsorbent is prohibitive and difficult to regenerate after use. So it's necessary to produce relatively inexpensive adsorbents which can be used for the treatment of waste water. Recently many researches were more interested in the use of various low-cost adsorbents, which also seem to be suitable to late substitute activated carbon [9]. A wide variety of low-cost materials such as natural clay [10], oil shale ash [11], bagasse fly ash [12,13], sawdust [14], maize cob [15], peat [16,17], white rice husk ash [18], cornelian cherry, apricot kernel, almond shell [19], orange peel [20], wheat bran [21] ... has been used successfully for the removal of dyes in aqueous solution.

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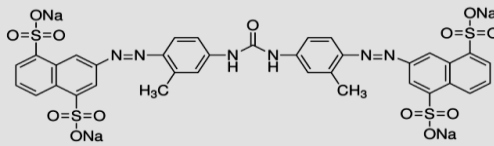
Vol. 3, Issue 10, October 2014

In this work, we were interested in the study of adsorption of the Direct Yellow 50 dye on marble powder. Various experimental parameters were analyzed: adsorbent amount, dye concentration, contact times and pH. The capacities of adsorption of marble powder were given by using the isotherms of Langmuir and Freundlich.

II. MATERIALS AND METHODS

II-1 Adsorbate

The adsorbent used is the Direct Yellow 50, provided by the company Ciba-Geigy. Table 1 represents a descriptive card of the Direct Yellow 50 dye (DY50).

Table 1	structure and characteristics of Direct Yellow 50 dye
Name	Direct Yellow 50
Chemical structure	
Molecular formula	$C_{35}H_{24}N_6Na_4O_{13}S_4$
Other name	Tetra sodium 3-[4-[[4-(4,8-disulfonatophthalen-2-yl)diazenyl-3-methylphenyl] carbamoylamino]-2-methyl phenyl naphthalene-1,5-disulfonate
Molecular weight (g/mol)	256,816
Maximum absorbance wavelength $\lambda_{max}(nm)$	294

II-2 Adsorbent

Marble powder (MP) used in this work was provided in powder form by the “Company Marble Great Northern”. This adsorbent was used without any prior treatment.

II-3 Marble powder characterization

A. Determination of pH zero charge point of marble powder

The surface chemistry characterization of the prepared MP was performed with pH drift method. This test was conducted to determine the pH of the point of zero charge, pH_{pzc} of the MP. pH_{pzc} is the pH when the charge on the MP surface is zero. For this purpose, 50 ml of 0.01N NaCl solutions were prepared and added into a series of Erlenmeyer flasks. Then, their pH values were adjusted in range between 2 and 12 using 0.01N HCl solution and 0.01N NaOH. The initial pH of the solutions were measured with pH meter and noted as pH_{initial}. After constant value of pH_{initial} had been reached, 0.15 g of the MP sample was added into each Erlenmeyer flask. The solution pH was measured after 48 h and noted as pH_{final}, then plotted against pH_{initial}. pH_{pzc} of the marble powder sample is the point when pH_{initial} = pH_{final} [22]. Measured pH was down by pH-Metre (Hach sension 2 pH ISE meter).

B. Characterization of surface chemical function of marble powder

The dosage of the acid function of surface of the MP can be carried out according to the method of BOEHM [23] wich rests on the acido-basic force of the function of surface. The samble studied is put in contact with a solution of the one of the four base of different forces: HCO₃Na, Na₂CO₃, NaOH and NaC₂H₅O. One then proposition basic excess in return by a Hydrochloric acid solution 0.1 N.

C. Experimental procedure

Adsorbent MP was characterized by various methods of analysis: XDR, of the Panalytical company, by using a Cu radiation K α ($\lambda = 1, 54060$ nm), functioning to 40 kV and 30 mA. The data were collected with $2\theta = 10^\circ-80^\circ$. XRF, uses a sequential spectrometer AXIOS PANALYTICAL with a channel of measurement bases on only one goniometry covering the complete range of measurement of B to U. FTIR of the BRUKER company (Vertex70) by using a range sweeping wavelength 400-7500 cm⁻¹. SEM (Quanta 200 FEI), and RAMAN of the BRUKER company (Sentera).

International Journal of Innovative Research in Science, Engineering and Technology

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Vol. 3, Issue 10, October 2014

The MP was tested for the adsorption of DY 50 from aqueous solutions at room temperature conducted by mixing various amount of MP for DY 50 dye removal in glass erlenmeyer flasks containing 250 ml of a dye solution at various pH using digital heating controlled magnetique stirrer (Stuart). Dye solutions were prepared using ultrapure water (MILLIPORE, direct-Q, UV3 with Pump) to prevent and minimize the possible interferences. The effect of several variable of MP dose (0,25-2 g/l), pH (5-12), contact time (0-100 min) and initial dye concentration (10-60 mg/l) were studied.

At the end of the adsorption experiment the dye concentration was determined, 4ml of suspension was taken and then centrifuged for 10 min at a speed of 4000 rpm and the supernatant was immediately dosed by UV spectrophotometer (Jasco V530).

The pH of the solution is adjusted by addition of an minor amount of HCl or NaOH (0, 1 M). At the end of the experiments of adsorption, the concentration of dye was given by measuring the absorbance of the solution with $\lambda = 394$ nm using a UV-visible spectrophotometer. All the experiments were carried out in double and the median values are presented.

The amount of equilibrium adsorption Q_e (mg/g) was calculated using the formula:

$$Q_e = \frac{C_0 - C_e}{W} V$$

Where C_0 and C_e (mg/L) are the liquid concentrations of dye initially and at equilibrium, V is the volume of the solution (L) and W is the mass of dye adsorbent (g).

The dye removal percentage can be calculated as follows:

$$\% \text{ of dye removal} = \frac{C_0 - C_e}{C_0} \times 100 \tag{2}$$

Where C_0 and C_e (mg/L) are the initial and equilibrium concentrations of dye in solution.

III.RESULTS AND DISCUSSION

III. 1 Characterization of the adsorbent

A. Determining the pH of the point of zero charge of marble powder

The Figure 1 represents the data drift pH, which pH_{PZC} of the adsorbent can be determined.

The combined influence of all the functional groups of the MP determines the pH of point of zero charge (PZC), it's the pH where the net surface charge on MP is zero. In the case where the $pH < pH_{PZC}$, the surface of the marble powder has a positive charge, whereas at $pH > pH_{PZC}$, the surface has a negative charge. [24]. The pH_{PZC} is the point where the $pH_{final} = pH_{initial}$ [22], for the marble powder used in this work, the $pH_{PZC} = 8.3$ this is in agreement with the results founded by Shaban E. Ghazy H.M. Gad and Abdullah [25].

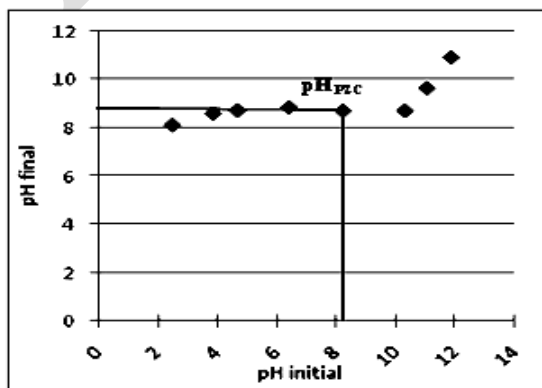


Figure 1: pH_{PZC} determination using pH drifts method

B. Characterization of surface chemical function of marble powder

Figure 2 and Table 2 summarize the results of the Boehm titration method, The surface functional groups is an important feature for any adsorbent, the data, of the Boehm titration method, indicate that in the surface of the MP, there are more acid carboxylic and lactone functions than phenol and carbonyl functions.

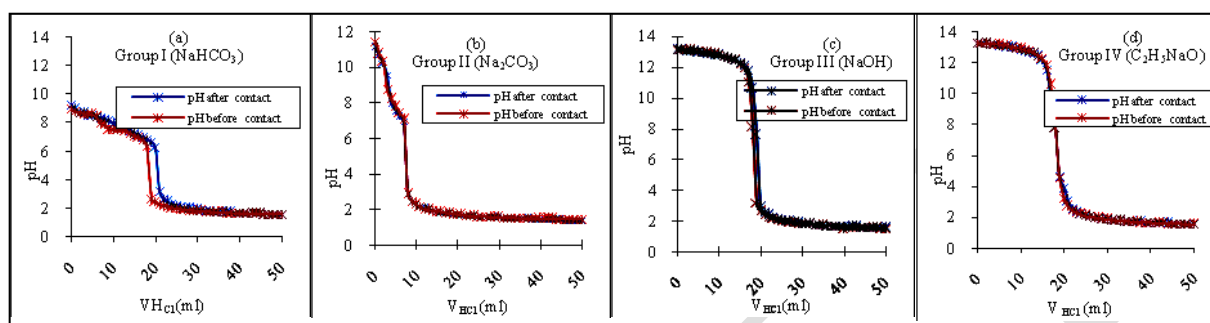


Figure 2: Evolution of neutralizing bases NaHCO₃ (a), Na₂CO₃ (b), NaOH (c) C₂H₅ONa (d) by HCl before (control) and after contact with the MP

Group	meq/g
GI : Carboxylic acid fonction	0,0105
GII : Lactone fonction	0,0105
GIII : Phénol fonction	0,0055
GIV : Carbonyl fonction	0,0055
∑ Surface fonction	0,032

Table 2: Results of the dosage of surface chemical function of the marble powder.

C- Characterization of marble powder by X-ray diffraction

Table 3 show the results of analysis by X-ray fluorescence spectrometry of the MP. The chemical composition of the MP is mainly composed of calcite and silica.

The remaining oxides are divided in two categories, those which have a little small mass percentage (MgO and SO₃) and those which are with the state of trace (Al₂O₃, Fe₂O₃, Na₂O, K₂O, Mn₂O₃ and TiO₂).

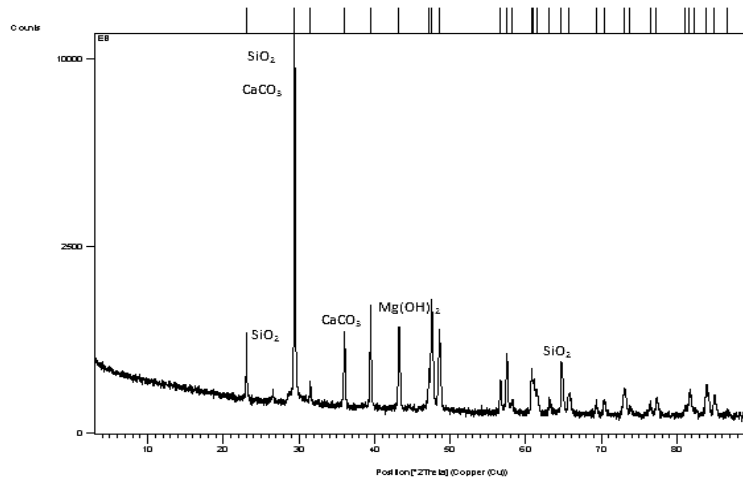
Table 3: Mineralogical composition, of Marble Powder

Chemical compound	CaO	SiO ₂	SO ₃	MgO	Al ₂ O ₃	Fe ₂ O ₃	Na ₂ O	K ₂ O	Mn ₂ O ₃	TiO ₂	Loss on ignition at 1000 °C	Total
% [OSA]	55,4	2,31	0,61	0,54	0,19	0,07	0,03	0,01	0,01	0,01	40,81	99,99

D. Characterization of marble powder by X-ray diffraction

To determine the structure of the MP, we proceeded to the recording of results X-ray Diffraction Siemens 5000 shown in Figure 3. The examination of this spectrum shows that the MP is consisting essentially by Calcium oxide (CaO) [26] and Silica (SiO₂) [26].

The analysis by di



E. Characterization of marble powder by Scanning Electron Microscope

Figure 4 show the result of analysis of MP by the SEM; the use of this technique makes it possible to visualize the morphology of the surface of marble powder. The results (Figure 4) show the presence of crystals of different sizes of calcite (than 1 to 9µm).

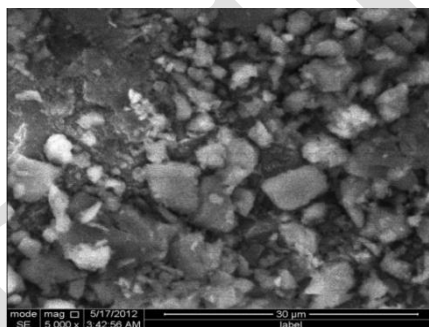


Figure 4: SEM (x 5000; 10 microns) of marble powder

F. Characterization of marble powder by Infrared spectroscopy

An examination of the MP by FTIR is illustrated in Figure 5, the analysis spectrum shows the presence of large bands located at 710 cm⁻¹, 1500 cm⁻¹, 1800 cm⁻¹ and 2500 cm⁻¹ shows to the presence of calcite [25], a band around 1600 cm⁻¹ corresponds to HOH and a band at 800 cm⁻¹ indicates the presence of Si-O [26]. The results are in perfect agreement with those of XRD.

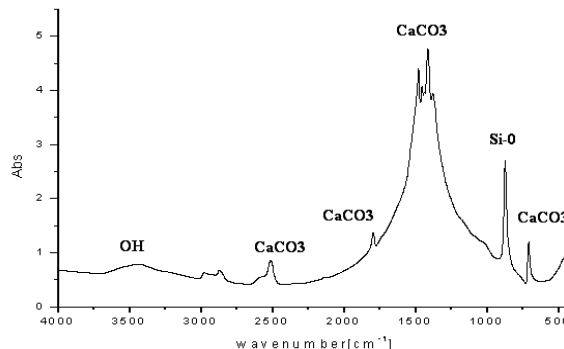


Figure 5: IR spectrum of the powder of marble.

E- Characterization of marble powder by Raman Spectroscopy

The Raman spectrum of the MP is illustrated in Figure 6. It shows the presence of a band at 1086 cm⁻¹, characteristic of calcite CaCO₃ [27], a band at 3000 cm⁻¹ characteristic of C-H, a band at 750 cm⁻¹ characteristic of C-C-C and a band characteristic of C-H and CO to 300 cm⁻¹ [28].

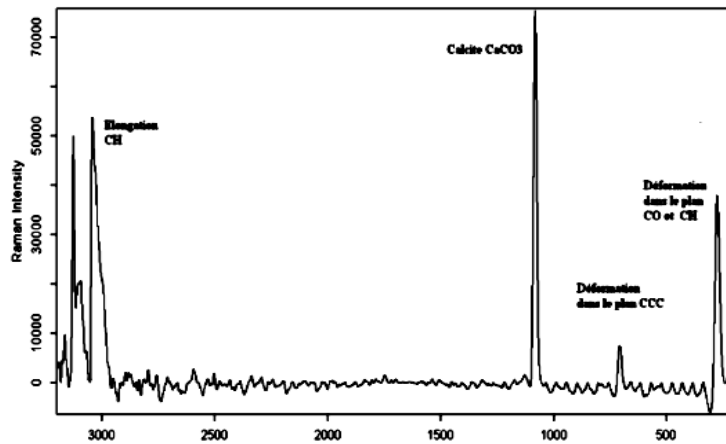


Fig.7. Raman spectrum of the marble powder

III.2. Study of effect of various parameters on the adsorption of the Direct Yellow 50

A-Influence time of contact on the adsorption of the Direct Yellow 50

The study of the kinetics of elimination of the DY 50 by Marble powder MP in aqueous solution is shown in Figure 7. The analysis of the temporal evolution of the quantity of DY50 adsorbed per gram of MP, for a given concentration (from 10 to 60 mg/l), shows that it increases rapidly during the first 10 minutes and then reached equilibrium around a stationary maximum value. The increase observed during the first ten minutes is probably due to the abundance of active sites on the surface of the adsorbent. According to the curve of Figure 7 the contact time required to reach equilibrium conditions was 20 min.

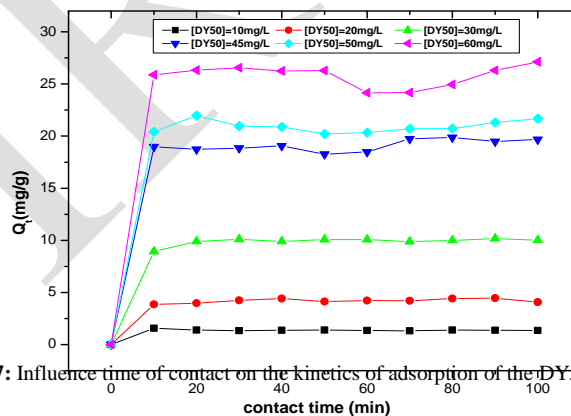


Figure 7: Influence time of contact on the kinetics of adsorption of the DY50 on MP

[Direct Yellow 50] ₀ mg.L ⁻¹	10	20	30	45	50	60
Q _{ads} (mg.g ⁻¹)	1,39	3,97	9,9	18,73	21,97	26,33

Table 4: Quantity adsorbed by MP with various initial concentrations of the DY50

B-Influence amount of adsorbent

The study's results of the mass's effect of the adsorbent on the rate of reduction are represented in Figure 8. The results show that for a dye concentration of 30mg/l and for a contact time of 20 minutes, an increase of the mass of marble from 0.25 g to 1.25g increases the percentage of removed dye (R%) in solution from 10% to 38%.

The increase in the percentage of removed dye (R%) in solution is due to the increase in the number of active sites of the MP.

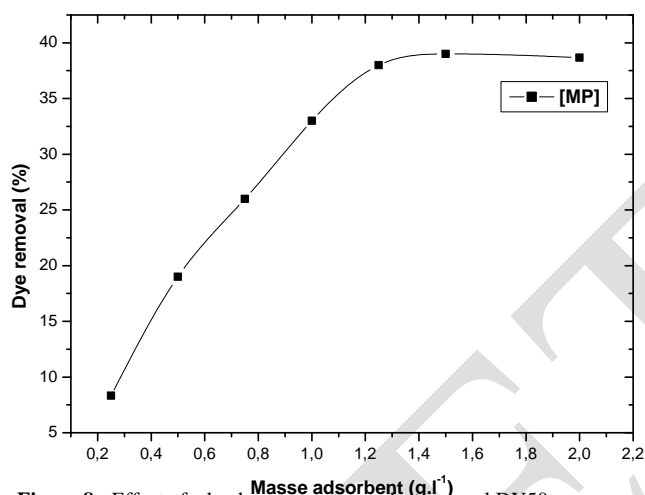


Figure 8 : Effect of adsorbent amounts on dye removal DY50

C-Effect of initial dye concentration on adsorption of the dye by marble powder

Figure 9 show the analysis of the evolution of the quantity (Q_e) of DY50 adsorbed per gram of MP. The results shows that Q_e increases from 1,39 to 26,33 mg/g when the dye concentration change from 10 mg/l to 60 mg/l. The shapes of the curves are similar and approximately independent of the initial dye concentration. A similar trend was reported for the adsorption of dyes such as reactive dye adsorption on activated sludge dried [29], metal complex dyes on pine sawdust [30] and Rhodamine-B coal active [31].

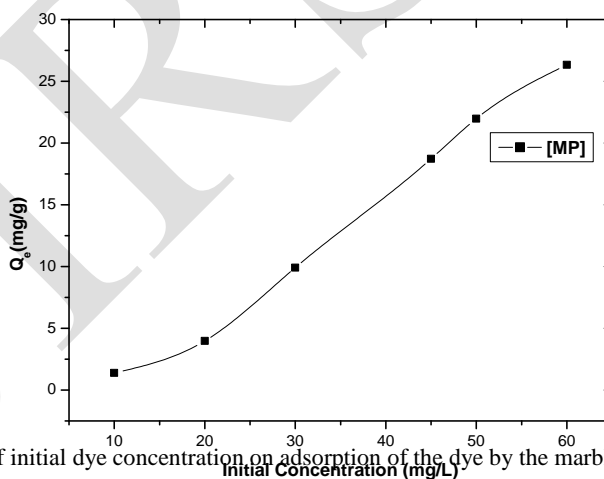


Figure9: Effect of initial dye concentration on adsorption of the dye by the marble powder

D-Effect of the pH on the holding capacity of the Direct Yellow 50 dye

The results of the study’s effect of dye pH on the quantity of DY50 adsorbed per gram of MP are shown in Figure 10. The pH value of the solution is an important controlling parameter in the adsorption process, and the initial pH of the solution has more influence than the final pH. The quantity of DY50 adsorbed per gram of MP is equal to 21,41 mg/g at pH 5 and decreases for pH basic. This trend can be explained by electrostatic interaction between the adsorbent and the dye molecule.

At pH 5 means below the pH_{PZC} (which is equal to 8.3) the H^+ ion concentration in the system increased and the surface of the marble powder acquires positive charge, by absorbing H^+ ions. As the MP surface is positively charged at low

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 3, Issue 10, October 2014

pH, a significantly high electrostatic attraction exists between the positively charged surface of the MP and an anionic dye molecule, leading to maximum dye adsorption. As the pH of the system increases ($pH > pH_{pzc}$), the number of negatively charged sites increases and the number of positively charged sites decreases. Negatively charged surface sites on the MP do not favor the adsorption of dye anions due to the electrostatic repulsion.

The result of the increase of the adsorption of the DY 50 dye by the MP in an acid solution, is the same as that found on the adsorption of the Acid Orange 10 dye on the fly ash from the bagasse [32] and the adsorption of Reactive Blue 2, Reactive Red 4 and Reactive Yellow 2 dyes on activated carbon [9].

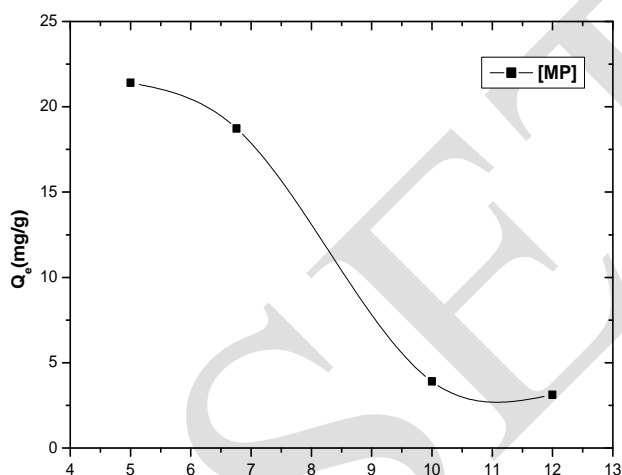


Figure 7 : Influence pH on the capacity of adsorption of the Direct Yalow 50

III.3. Adsorption kinetics

The model of the kinetics of first order is adapted the most for the weakest concentrations of aqueous solution. This model is presented by the relation of Lagergren [33], based on the adsorbed quantity, is the first equation speed established to describe the kinetics of adsorption in a liquid/solid system. This model of pseudo-first-order is represented by the following relation [34]:

$$\ln(q_e - q_t) = \ln q_e - K_1 t \tag{3}$$

Where q_t : quantity of adsorbate adsorbed at time t ($mg \cdot g^{-1}$), q_e : quantity adsorbed at equilibrium ($mg \cdot g^{-1}$);

k_1 : constant speed of adsorption of the pseudo model - first order (min^{-1}), T : time (min).

Evolution of $\ln(q_e - q_t)$ according to time, presented in our work. If the Lagergren relation is checked, while carrying $\ln(q_e - q_t)$ according to time, we must obtain a line of slope $-K_{ads}$. This indicates that the kinetics of adsorption of the DY50 on MP does not correspond to the kinetics of the pseudo-first order.

The reaction speed of pseudo-second-order depends on the quantity adsorbed on the surface on the adsorbent and the quantity adsorbed at equilibrium. The model of pseudo-second-order can, being represented in the following form [35]:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \tag{4}$$

Where k_2 : constant speed of adsorption of the model of pseudo-second-order ($g \cdot mg^{-1} \cdot min^{-1}$).

If this equation is checked, by tracing t/q_t according to t , we must obtain a line of slope $1/q_e$ and ordinate in the beginning equal to $1/(K_2 q_e^2)$.

The modeling of the kinetics of adsorption of DY50 on adsorbent MP by the model of pseudo-second order is presented on Figure 11. These results are obtained with constant operating condition: with $20^\circ C$ (agitation = 600 rpm and $[MP] = 2 g \cdot L^{-1}$).

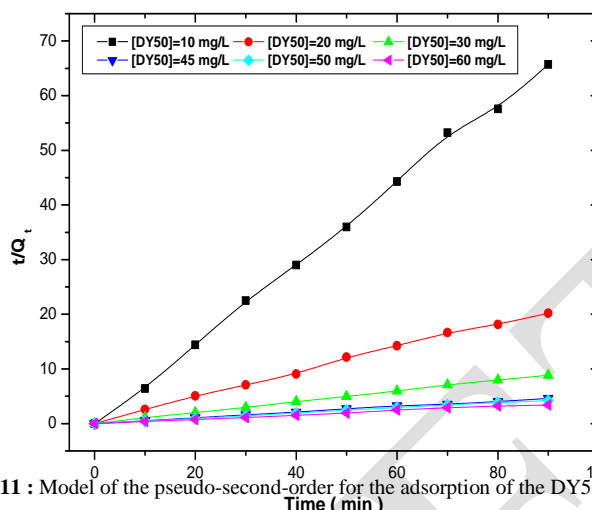


Figure 11 : Model of the pseudo-second-order for the adsorption of the DY50 on adsorbent MP

The corresponding parameters were gathered in the Table 5 which also presents the coefficients of correlation. These calculated coefficients are more about the unit for the kinetic model of pseudo second-order. That indicates that this last model described well the experimental results of the adsorption of the DY50 on the two adsorbents.

C ₀ (BM) (mg.L ⁻¹)	Q _{exp} (mg.g ⁻¹)	Kinetics of the 2 nd order		
		K ₂ (g.mg ¹ .min ⁻¹)	Q _{cal} (mg.g ⁻¹)	R ²
[DY50]=10 mg/L	1,39	25,31	1,36	0,998
[DY50]=20 mg/L	3,97	2,21	4,29	0,994
[DY50]=30 mg/L	9,9	2,10	10,10	0,999
[DY50]=45 mg/L	18,73	0,81	20,00	0,997
[DY50]=50 mg/L	21,97	1,46	21,27	0,998
[DY50]=60 mg/L	26,33	2,23	26,31	0,993

Table 5 : Parameters characterizing the kinetics of adsorption of Direct Yalow 50 on the marble powder.

III.4. Isotherms of adsorption

The last step is adsorption isotherms which study to model the curve, or more precisely to report it by a mathematical equation of the entire curve. Traditional models of Langmuir, Freundlich and Elovich will be used for their simplicity of implementation.

The Langmuir model [36] is based on the following assumptions:

- Forming a single layer of adsorbate on the surface of the adsorbent.
- The existence of the defined sites of adsorption.
- The surface is uniform without interaction between the adsorbed molecules.

The Langmuir equation can be written as follows:

$$(5) \quad \frac{1}{Q} = \frac{1}{Q_{max}} + \left(\frac{1}{KQ_{max}} \right) \frac{1}{C_r}$$

Where Q is the adsorption capacity in mmole or mg of solute per gram of adsorbent (mmol/g) or (mg/g), Q_{max} is the maximum adsorption capacity (mmol/g) or (mg/g), C_r is the concentration of residual solute (mmol/l) or (mg/l) and K is the equilibrium constant to the torque solute adsorption/adsorbent (l/mmol) or (l/mg).

The Freundlich model [37], is based on an empirical equation that reflects a variation of energy with the adsorbed amount. This distribution of interaction energies can be explained by heterogeneity of adsorption sites. Differently to the Langmuir model, the Freundlich equation does not provide an upper limit for the adsorption which restricts its

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 3, Issue 10, October 2014

$$\ln \left(\frac{C_a}{m} \right) = \ln K + \left(\frac{1}{n} \right) \ln C_r$$

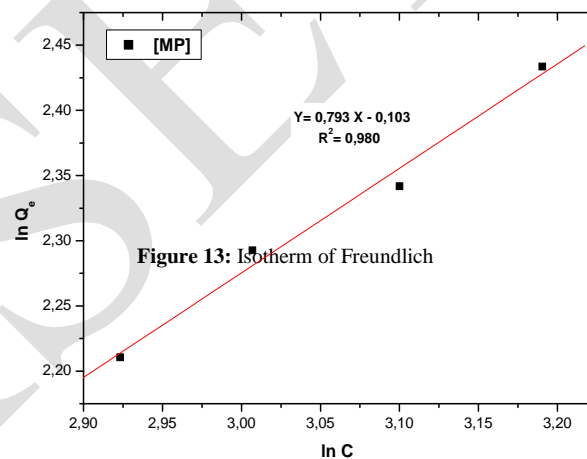
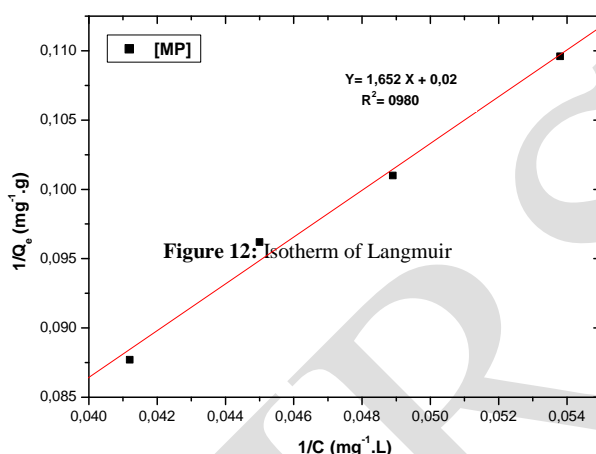
application to dilute media. However, this model admits the existence of interactions between adsorbed molecules [38]. It is of the following form:

(6)

Where m is the mass of the adsorbent (mg/l), C_a is the concentration of solute adsorbed (mmol / l) or (mg/l), K and n are the equilibrium constant to the torque solute adsorption/adsorbent and C_a/m is the adsorption capacity per unit mass of adsorbent (mmol/g) or (mg/g).

To study the adsorption isotherm of the dye at 25°C, a volume of one liter of distilled, water containing the yellow dye, at a concentration 30 g/l and the initial pH is brought into contact with different masses of MP. The stirring speed is set at 600 rpm. To determine the instantaneous concentration of the dye with time, the sample is centrifuged for 10 min at 4000 rpm, and the supernatant was immediately measured by a UV spectrophotometer.

To measure the adsorption isotherms of the yellow dye in the marble powder at 25 °C, the duration of contacting solution-adsorbent was set to 20 min, at which time it is ensured that there is no significant variation concentration of the dye in equilibrium.



The estimated parameters of the isotherms of adsorption of Langmuir and Freundlich, for the adsorption of Direct Yellow 50 dye, by using adsorbents (MP), are gathered in Table 6.

	Langmuir			Freundlich		
	Q_{max} (mg/g)	K_L (L/mg ⁻¹)	R^2	K_f (mg/g)	n_f	R^2
MP	50	0,012	0,980	0,9	1,26	0,980

Table 6 : The estimated para

IV. CONCLUSION

Marble powder has shown its effectiveness in removal of the dye in aqueous solution. The adsorption is highly dependent on various parameters such as the mass of adsorbent, contact time, pH and initial concentration of the dye. The adsorption capacity increased with increasing the concentration of the Direct Yellow dye. Or, this capacity decreased with increasing pH of the same color. The kinetics of dye adsorption on powdered marble is fast, equilibrium is reached after 20 min. The adsorption isotherms of dye powder of marble rather satisfactorily follow the Langmuir model.

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 3, Issue 10, October 2014

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International Journal of Innovative Research in Science, Engineering and Technology

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Vol. 3, Issue 8, August 2014

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