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Removal of Astrazon yellow dye from aqueous solutions by sorption onto Stipa tenacissima L Alfa fibers as a natural adsorbent

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ABSTRACT/RESUME

Abstract*: This work aims to explore the use of alfa fibers as a natural and low-cost adsorbent for the removal of astrazon yellow dye (AY) from aqueous solution on industrial textile effluent. The alkaline procedure was used to remove none cellulosic substances such as pectin, lignin and hemicellulose. The influence of alkaline treatment on fiber morphology is analyzed by FT-IR and SEM, showing physico-chemical changes in the treated material. The observations show that an interesting mesoporous structure was developed. The specific surface was notably increased to 62.48 m2/g. Batch adsorption experiments, at 25 C° and agitation speed of 150 rpm, were carried out and the effects of operational parameters including contact time, initial dye concentration, adsorbent dose, and pH solution on adsorbed amount were investigated and discussed.*
Batch adsorption shows that equilibrium was obtained after 40 min, and the adsorbed dye amount increases with pH. The highest removal rate was about 83 %. The experimental data of modelization showed that data were best fitted with the Freundlich model. The correlation coefficient values (R2) showed that the adsorptions Kinetic were well described by the pseudo-second-order model. The adsorption thermodynamics and isotherms are consistent with spontaneous endothermic physicosorption. The results indicated that alfa fibers could be used effectively and advantageously to adsorb astrazon yellow dye from aqueous solutions.

I. Introduction

Textile industry wastes represent enormous harm for human health, especially dyes that are used in excess, as a result wastewater is highly concentrated in dye molecules which make biodegradability hard [1]. Thus, the removal of dyes from colored effluents, particularly those from textile industries, is one of the major environmental concerns nowadays. The treat dyeing wastewater methods can be classified into two types: physical and chemical processes; adsorption by activated carbon is the most common one. Although the process is highly effective, the running costs are high with the need for regeneration after each sorption cycle [2]. Recently, the usage of agricultural wastes or industrial by-products as low-cost alternative adsorbent is receiving a considerable attention.
A number of investigations have shown that agricultural by-products such as clay [3], wheat shells [4], orange peel [5], peanut husk [6], pistachio hull [7], wool and cotton fiber [8] have the potentialities to be used as low-cost adsorbent for the removal of dyes in wastewater. Also, some researchers have proved the success of several cheap materials such as apricot stone [9], wheat bran [10] and sepiolite [11] for the removal of astrazon yellow from aqueous solutions, but no studies were conducted about the alfa capacity to

capture it. Knowing that alfa or esparto (*Stipa tenacissima* L.*)* is a perennial tussock grass widely distributed in semi-arid ecosystems of the southern and western Mediterranean basin and mainly in Maghreb zone [12].

The present research aims to develop inexpensive and effective adsorbent from *Stipa tenacissima* L. fibers for the removal of astrazon yellow dye from aqueous mediums to replace existing marketing materials.

In this study, alfa cellulosic fibers were extracted from *S. tenacissima* L. grass using alkaline procedure. They were then studied as an adsorbent for astrazon dye. The influence of experimental parameters such as pH, initial concentration and temperature of the adsorption efficiency has been investigated. Adsorption isotherm and corresponding kinetic curves were obtained, several adsorption models were used to verify experimental data and to determine the adsorption mechanism. Thermodynamics studies were also carried out to estimate free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) changes.

II. Materials and Methods

II.1. Materials

The *S. tenacissima* L. plants used in this study were collected from Boussaada (Algeria). Astrazon Yellow 7GLL, was provided from Dystar. Its general chemical formula is $C_{20}H_{25}N_2$ and color Index: basic yellow 21 with a molecular weight of *293 g/mol*. It absorbs light at *416 nm*.

II.2. Extraction of cellulose fibers from *S. tenacissima* L.

Alfa stems of *50-80 cm* length were manually cut into approximately *2-3 cm* pieces. Cellulosic fibers were extracted from it using a chemical process following El Ghali A. and *al.* (2010) method [13]. First peroxide bleaching was conducted in an around-bottomed flask, where was placed *5 g* of fresh Alfa, *200 ml* of distilled water, and *10 ml* of H_2O_2 (*30 % (w/w)*). The mixture is stirred for more than *6 h* at *90 C*°. Then the bleached alfa was washed with hot then cold water and dried at room temperature. Cellulose was extracted from Alfa plant with *400 ml* toluene/ethanol mixture (*2/1, V/V*) for *6 h* using Soxhlet apparatus [14]. The second step involved the extraction of cellulosic alfa fibers using alkaline procedure. For the alkaline boiling process, the temperature of extraction and the concentration of NaOH solution were fixed, respectively, at *50 C*° and *0.25 M* for *4 h*. The fibers were extracted using a liquid to plant ratio of *10:1*. At each step of the different treatments, the insoluble residue was extensively washed with distilled water and fibers were dried at room temperature until constant weight.

The extraction efficiency (*R %*) represents the variation of sample weight before and after the extraction; it was calculated as Yield of extraction as shown by equation (1) :

$$R(\%) = 100 * \frac{mf}{ma} \quad (1)$$

Where: m_a and m_f are the weight of dried alfa before and after extraction, respectively. The value of *R* was *58 %*, which can be considered acceptable compared with other studied extraction methods [15].

II.3. Characterization of alfa fibers

II.3.1. Determination of pH_{PZC}

The zero point of charge (pH_{PZC}) of alfa fibers was determined according to Belaid et *al.* [16] method. *50 ml* of *0.01 M* NaCl solution was prepared in distilled water. Solutions with different *pH* ranging from *2-12* were prepared using HCl (*0.1 M)* or NaOH (*0.1 M*). Then, *0.15 g* of fibers was added to each medium solution, and the final *pH* was measured after *48 h* under agitation at room temperature. The pH_{PZC} is the point where the curve of final *pH* vs initial *pH* crosses the line fitted by final *pH* equal initial *pH*.

II.3.2. Plant analysis

Alfa fibers were characterized by elemental analyses, FT-IR and SEM. FT-IR spectra were recorded on 4100 JASCO FT-IR spectrophotometer in the range of *4000-400 cm^{-1}* for qualitative identification of grass plant and extracted fibers constituents. The SEM micrographs of extracted alfa fibers were taken using ESEM Philips XL30.

The adsorption-desorption isotherms of the treated alfa samples were carried out in a BET apparatus on Nova Station A – QuantaChrome instrument. The adsorption gas is nitrogen, and measurements are made at *77.3 K*, the sample weight is equal to *88.7 mg* and the sample volume equal to *0.12677 cc*. The degassing time was of

14 h at *200 C*°, and the analysis time was about *144.8 min*.

II.3.3. Batch adsorption experiments

Adsorption measurements on alfa fibers were carried out in batch process to evaluate the effects of various parameters, such as initial dye concentration, contact time, pH and temperature. In each adsorption experiment, *0.5 g* of alfa fibers is added to *100 ml* of astrazon yellow dye solution. The desired pH was achieved by adjustment with HCl (*0.1 M*) or NaOH (*0.1 M*). Experiments were conducted using jar test (P SELECTA), where the mixture is stirred for *2 h* at *150 rpm*, the aliquots

are collected and filtered at pre-determined time intervals, the DO of the limpid solutions is measured by a UV- Vis spectrometer (PG instruments CT60) at *416 nm*. The adsorbed dye quantity at equilibriums (q_e) and the removal efficiency of alfa fibers was determined by equations (2) and (3) respectively:

$$qe = \frac{(C_o - Ce)V}{m} \quad (2)$$

$$R\% = \frac{(C_o - Ct)V}{C_o} \quad (3)$$

Where; q_e (*mg/g*) and *R %* are the adsorption capacity and removal efficiency, respectively. C_o (*mg/L*) is initial dye concentration, C_e (*mg/L*) is dye concentration at equilibrium, *m* (*g*) is the adsorbent mass, and *V* (*L*) is dye solution volume.
The temperature influence was conducted by immersing solution in a thermostatic bath, regulated at work temperatures (*20*, *30*, *40* and *50 C°*) and stirred at *150 rpm*.

II.4. Kinetic and isotherm studies

The adsorption kinetics is determined by analyzing the quantity of adsorbed dye from aqueous solution at different time intervals. To investigate the effect of the initial dye concentration on the adsorption kinetic different test solutions were prepared with dye concentration between *20* and *50 mg/L*. Adsorption Isotherms at various adsorbent dosages (*2-20 g/L*) are analyzed to determine the equilibrium adsorption capacity. Thermodynamics parameters were calculated from equilibrium adsorption data at different temperatures.

III. Results and discussion

III.1. Characterization of extracted fibers

III.1.1. Infrared spectroscopy (FT-IR)

FT-IR was carried out to observe the chemical changes occurring on the alfa plant induced by chemical modification (figure 1). A broad absorption band at *3274-3500 cm^{-1}* is mainly due to OH groups, which is lower in intensity after treatment. The band at *2916 cm^{-1}* corresponds to CH band present in all-natural fibers; the intensity of this peak has decreased during the elimination of hemicelluloses. It is also noted that the *1731.9 cm^{-1}* band which corresponds to the non-cellulosic constituents (pectin, lignin, and hemicelluloses) has disappeared as a result of the treatment like found by Benyahia, A. *et al.* [17], also a band at *1630 cm^{-1}* was found indicating the existence of a C=O band of lignin acetyl groups which is partially reduced on fibers surface. Finally, C-O band is observed at *1050 cm^{-1}*, it decreases after treatment [14].

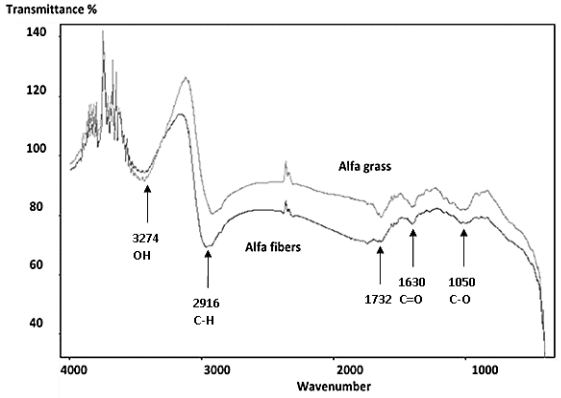

Figure 1. *The infrared spectrum of alfa grass and fibers.*

III.1.2. Scanning Electron Micrographs (SEM)

Alfa fibers are composed of *48 %* of cellulosic chains, which are the primary reinforcement material, leading to microfibrils, which are held together by amorphous hemicelluloses giving fibrils. The fibrils are assembled like numerous layers to build up the structure of the fiber. Fibers or cells are cemented together by lignin, which can be dissolved by alkalinity [18]. The morphology of alfa fibers was investigated by SEM (figure 2); figure 2.a show that fibers were totally separated by chemical treatment and the adsorbent surface (figure 2.b), present a heterogeneous aspect with some apparent macro-pores of less than *10 µm*. Those pores were probably naturally present in raw material but were partially masked by non-cellulosic coating like cited by many authors [17-19], this fact does not reflect the real adsorption potentialities of our material, that is why we needed to explore adsorption-desorption isotherms (BET).
It can be seen above BET surface area results that after the alkaline treatment, the mesoporous structure was developed (*20.88 A°*), increasing the specific surface (*62.48 m^2/g*) compared to native alfa plant estimated by Boumehdi Toumi, L. et *al.* [20] about (*3.29 m^2/g*).

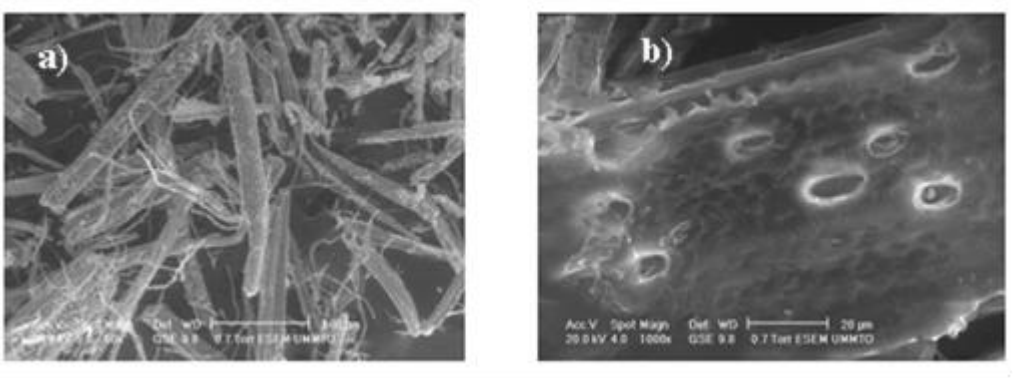

***Figure 2**. Scanning electron micrographs of alfa fibers.*

III.2. Adsorption studies

III.2.1. Effect of initial dye concentration and contact time

The effect of initial dye concentration on the adsorption is shown in figure 3; the amount of (AY) adsorption was quickly increased over the first *20 min* then it slows down till *40 min*. The equilibrium was acquired within
40 min. In this second step, the concentration gradient is reduced due to the saturation of the adsorption sites by the dye particles (observed at the SEM) [20]. The increase in loading capacity of the adsorbent with a relation with dye ions is probably due to the high driving force to mass transfer. An increase in the initial dye concentration enhances the interaction between AY and adsorbent, resulting in greater adsorption capacity [21, 22].

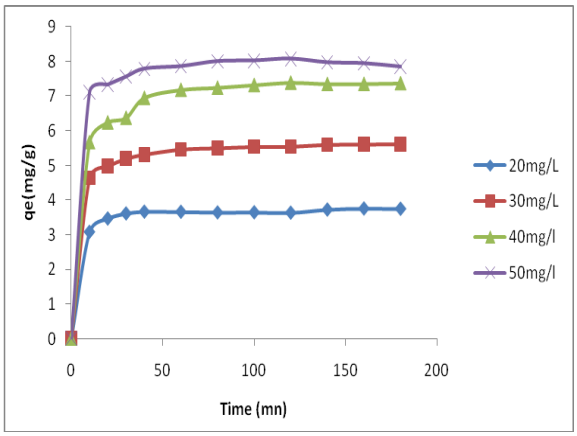

***Figure 3.** Effect of contact time on the adsorption of AY dye on alfa fibers for various initial dye solution concentrations (pH=7. alfa fibers mass 0.5 g)*

III.2.2. Effect of adsorbent dosage

In adsorbent dosage experiments, the chosen weights of adsorbent were treated with *100 ml* AY dye solution with a constant concentration of *30 mg/L*. Figure 4 shows the effect of alfa fibers dose on AY dye removal. As a result, indicates the removal efficiency increased from *66.99* to *98.34 %* with increasing dosage from *2* to *3 g/L* and then remained almost constant. As a result, indicates (figure 4) dye removal percentage increases gradually. Such a trend is mainly due to the increase of adsorptive surface area and the availability of more adsorption sites. Therefore, *5 g/L* of alfa fibers was chosen as optimum dosage for AY dye removal; This suggests that after a specific dose of adsorbent, maximum adsorption is reached and therefore the amount of pollutant remains constant even after further addition of the adsorbent dose [23].

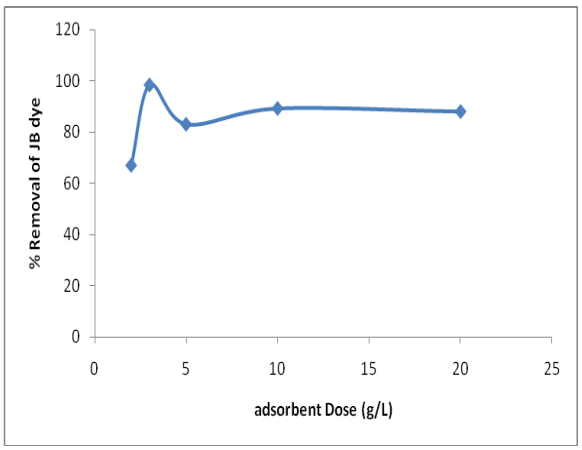

***Figure 4.** Effect of alfa fibers dose on AY dye removal for the initial dye concentration (C0= 30 mg/L. pH=7 and contact time 120 min)*

III.2.3. Effect of temperature

Temperature controls two significant adsorption aspects; the swelling behavior of adsorbent and solubility of the adsorbate molecules, which affects the rate of diffusion through the solution to the

adsorbent [13]. Figure 5 shows that the adsorption capacity decreases with increasing temperature, above *40 C°*; this is due to the ionic force established, between alfa fibers and AY dye, which is destabilized with the increase in temperature. This phenomenon may be due to the exothermic effect of the surrounding during the adsorption process [24, 25].

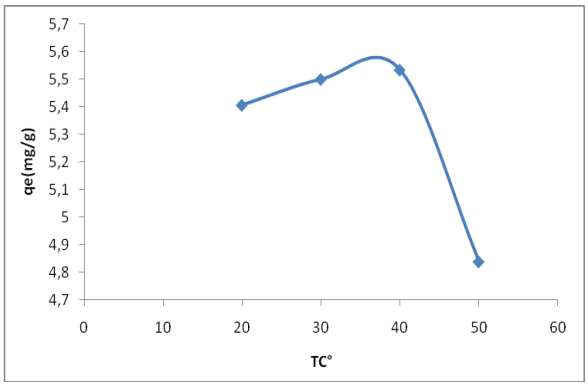

Figure 5. *Effect of temperature on AY dye removal by alfa fibers (C0= 30 mg/L. alfa fibers mass 0.5 g and contact time 120 min).*

III.2.4. Effect of pH

One of the most critical factors controlling the adsorption capacity in aqueous solutions is *pH*. Figure 6 (b) shows the influence of the *pH* solution on alfa fibers adsorption of dye; the amount of adsorbed AY dye was found to increase with an increase in *pH*; this can be explained by the value of the pH_{PZC} (pH_{PZC}*=7.7)* of alfa fibers as shown in figure 6 (a). As the pH of the system increased beyond the pH_{PZC} of the adsorbent, the number of negatively charged adsorbent sites increased too, which enhances the adsorption of positively charged AY dye through electrostatic attraction forces [26]. Similar pH_{ZPC} values for cotton cellulose was reported by Baghdadi, M. *et al.* [26].

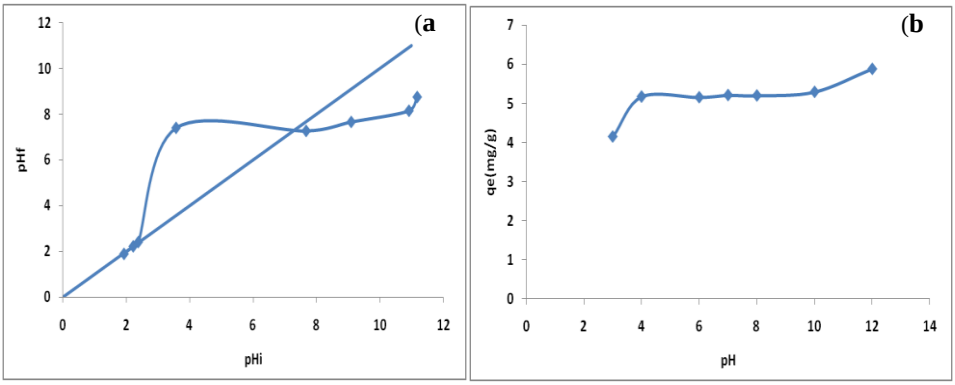

Figure 6*. a) Zero point of charge (pHZPC) of the used adsorbent for the adsorption experiment. b) Effect of pH on the adsorption of AY dye on alfa fibers (C0= 30 mg/L. alfa fibers mass 0.5 g and contact time 120 min).*

III.3. Adsorption isotherms

The adsorption isotherm indicates how the adsorbed molecules are distributed between the liquid and the solid phases at equilibrium. Adsorption isotherms parameters obtained from different models provide important information on the adsorbent surface properties and its affinity with the adsorbate. In this study, Langmuir and Freundlich's isotherms were used for the treatments of the equilibrium adsorption data. The Langmuir adsorption isotherm is the best-known linear model for monolayer adsorption and most frequently used to determine the adsorption parameters. Langmuir model is represented by the following equations (4)-(5):

$$q_e = \frac{K_L \times q_m \times C_e}{1 + K_L \times C_e} \quad (4)$$

$$\frac{1}{q_e} = \left[\frac{1}{K_L \times q_m}\right] \times \frac{1}{C_e} + \frac{1}{q_m} \quad (5)$$

Where q_e is the amount of adsorbed dye (*mg/g*), it represents the adsorptive capacity of the adsorbent for the equilibrium effluent concentration, q_m is the maximum amount adsorbed (*mg/g*), k_L is a Langmuir's constant signifying energy of adsorption (*L/mg*) and C_e is the equilibrium concentration of AY dye in the aqueous phase (*mg/L*). The Langmuir equations make several assumptions such monolayer adsorption on a surface containing a limited number of sites, predicting a homogenous distribution of adsorption energies. The plots of $1/q_e$ vs $1/C_e$ are linear. The values of q_e and K_L have been evaluated from the intercept and slope of these plots (table 1). The maximum adsorption capacity of the monolayer was found to be *19.60 mg/g* of alfa fibers.

Table 1. *Characteristic parameters obtained by the Freundlich and Langmuir equations*

Langmuir constant			Freundlich constant		
q_m (mg.g^{-1})	K_L (L.mg^{-1})	R^2	K_F	1/n	R^2
19.6	0.065	0.931	0.900	1.03	0.983

The essential characteristics of Langmuir isotherm parameters can be used to predict the affinity between the adsorbate and adsorbent using separation factor or dimensionless equilibrium parameter R_L [27]. Ho and McKay [28] established that the value of R_L indicated that adsorption Langmuir type could be irreversible when ($R=0$), favorable when ($0<R_L<1$), linear when ($R_L=1$) or unfavorable when ($R_L>1$). The R_L was found to be *0.338* for the concentration of *30 mg/L* of AY dye. It means that the adsorption is favorable and the process reversible.

Adsorption capacity based on the Langmuir isotherm, q_m , is usually used as an essential parameter to evaluate adsorbent performances. The q_m values of some adsorbents used for basic dyes removal, listed in table 2 Are compared to obtained results.

Table 2*. A comparative evaluation of the adsorbent capacities of by some adsorbents*

Adsorbent	Name of the dye	q_{max} (mg/g)	References
Sepiolite	Astrazon Yellow	62.5-82.5	Tekbaş et al., 2009 [11]
Wheat bran	Astrazon Yellow	69.09	Sulak et al., 2007 [10]
Modified clay	Astrazon golden Yellow	523.6	Çakmak et al., 2017 [29]
Clinoptilolite	Basic Yellow	59.6	Yener et al., 2006 [30]
Amberlite	Basic Yellow	8.7	Yener et al., 2006 [30]
modified zeolites	Basic Yellow	21.2–17.8	Çalıskan Y. et al., 2019 [31]
Alfa fibers	Astrazon Yellow	19.6	This study

Moreover, the Freundlich model is an empirical equation that assumes heterogeneous adsorption sites due to the diversity sites. The Freundlich equation (6):

$$q_e = K_F C e^{1/n} \quad (6)$$

Equation (6) can be linearized as equation (7):

$$\ln q_e = \ln k_F + \frac{1}{n} \ln C_e \quad (7)$$

Where q_e is equilibrium adsorption capacity (*mg/g*), C_e is the equilibrium concentration (*mg/L*) of AY dye; K_F and *1/n* are empirical Freundlich constants which indicate adsorption capacity of adsorbent and intensity of adsorption (*mg/g*), respectively. They are determined from the *ln* q_e versus *ln* C_e plot. The value of K_F and *1/n* are presented in table 1. The *1/n* value in the Freundlich equation was found to be *1.03* for alfa fibers.

III.4. Adsorption kinetics

The study of adsorption kinetics describes the solute uptake rate, and this rate controls the residence time of adsorbate uptake including the diffusion process at the solid-solution interface; the mechanism of adsorption depends on the physical and chemical characteristics of the adsorbent as well as on the mass transfer process [27]. In order to understand the controlling mechanism of the adsorption process, the pseudo-first-order, the pseudo-second-order and intraparticle diffusion models were applied to examine the experimental data. The pseudo-first-order rate expression of Lagergren is given as equation (8):

$$log(q_e - q_t) = log q_e - \frac{k_1}{2.303} t \quad (8)$$

Where K_1 (min^{-1}) is the rate constant of the pseudo first order adsorption process which is obtained from the slop of the plot of $\log(q_e - q_t)$ vs time and the intercept gives q_e (equilibrium or maximum adsorption capacity) .

The pseudo second order kinetics given by Ho's equation as equation (9):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (9)$$

Where k_2 ($g.mg^{-1}.min^{-1}$) is the pseudo-second-order rate constant; the slopes and intercept of plots of t/q_t versus time were used to calculate the second-order rate constant k_2 and q_e.

The possibility of intra-particle diffusion was explored by using an intra-particle model. The corresponding relation is given as equation (10):

$$q_t = K_{id} t^{1/2} + C \quad (10)$$

Where: C is the intercept constant, and K_{id} is the intraparticle diffusion rate constant ($mg/g\ min^{1/2}$).

Kinetic parameters of the applied models and correlation coefficients are summarized in table 3.

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The correlation coefficients (R^2) for the pseudo-first-order kinetic models are low. Moreover, a significant difference in equilibrium adsorption capacity (q_e) obtained from the experimental and calculated values were observed, indicating a poor pseudo-first-order fit to experimental data. R^2 of the second-order kinetics model was more sweetable; besides, calculated q_e values were entirely in agreement with experimental data in this case; this suggests that the adsorption data are well represented by pseudo-second-order kinetics [28]. The analysis of kinetic data by other researchers has also shown that the pseudo-second-order equation can be used to simulate with a good agreement the adsorption of AY dye [32-34]. The values of K_2 constants decrease with the increase of initial AY concentration; this behavior can be attributed to the decreasing of competition phenomenon on the surface adsorption sites at a lower concentration

***Table 3**. Kinetic parameters of AY adsorption onto alfa fibers*

	pseudo-first order				pseudo-second order			Intraparticle Diffusion		
C_0 ($mg.L^{-1}$)	q_{exp} ($mg.g^{-1}$)	k_1 (min^{-1})	q_e ($mg.g^{-1}$)	R^2	k_2 ($L.mg^{-1}.min^{-1}$)	q_e ($mg.g^{-1}$)	R^2	K_{id} ($mg.g^{-1}.min^{-0,5}$)	C	R^2
15	2.875	0.055	0.401	0.689	-0.724	2.857	0.999	0.016	2.815	0.613
25	4.544	0.192	1.728	0.98	0.197	4.716	0.999	0.12	3.777	0.614
30	5.213	0.142	3.016	0.9	0.089	5.494	0.999	0.147	4.293	0.915

III.5. Thermodynamic parameters

To study the effect of temperature on thermodynamic parameters such as the changes in Gibb's free energy ($\Delta G°$), entropy ($\Delta S°$) and enthalpy ($\Delta H°$) were also investigated and calculated for adsorbent using the following equations (11)-(12)-(13):

$$\Delta G = - RT\ln K_d \quad (11)$$

$$K_d = \frac{C_{Ad}}{C_e} \quad (12)$$

$$\ln k_d = \left(\frac{\Delta S°}{R}\right) - \left(\frac{\Delta H°}{R}\right)\frac{1}{T} \quad (13)$$

Where k_d is the equilibrium constant, C_{Ad} is the amount of dye adsorbed on solid at equilibrium (*mg/L*), C_e is the equilibrium concentration of dye in the solution (*mg/L*), R is the gas constant (*8.314 J/mol.K*), and T is the temperature in kelvin. The values of $\Delta H°$ and $\Delta S°$ can be obtained from the slope and intercept of Vant'Hoff plot of $ln\ k_d$ versus $1/T$.

AY dye adsorption on alfa fibers increased when the temperature was increased from *293* to *313 K*. The process is thus endothermic. The plots were used to calculate the values of thermodynamic parameters (table 4). The positive $\Delta H°$ value (table 4) suggests the endothermic nature of adsorption and the negative $\Delta G°$ values indicates the feasibility and spontaneity of the adsorption process; the low value of this heat *(<20 K.Joule/mole*) confirms that it is physical adsorption [35]. The positive value of $\Delta S°$ shows the increase in randomness at the solid-solution-interface during adsorption [10].

***Table 4.** Thermodynamic parameters for AY dye adsorption on alfa fibers.*

T(K)	$\Delta G°$ ($kJ.mol^{-1}$)	$\Delta S°$ ($J.mol^{-1}.K^{-1}$)	$\Delta H°$ ($kJ.mol^{-1}$)
293	-5.383	41.682	6.757
303	-6.038		
313	-6.44		

IV. Conclusion

The experimental study was conducted to investigate the potential of alfa fibers as an adsorbent for the removal of astrazon yellow dye from aqueous solutions. The alkali treatment improved the adsorption features of alfa by increasing its specific surface and forming mesoporous structure.

The operating parameters such as *pH* solutions, adsorbent dose, contact time, initial AY dye concentration and temperature were optimized to achieve high adsorption efficiency that was very interesting; with neutral pH conditions and short equilibrium time (*40 min*) and an adsorbent dose of *0.5 g/L*. Freundlich isotherm model gives the best fit to the experimental data indicating diversity and heterogeneity in the adsorption sites, which well describe natural fibers structure. The kinetic data showed that AY adsorption on alfa fibers follows a pseudo-second-order behavior, and the phenomenon has an endothermic and spontaneous character. For all those reasons, alfa fibers were found to be an economical and environmental friendly substrate, as they are effective adsorbents to removal astrazon yellow dye from aqueous solutions under soft conditions.

V. Acknowledgements

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