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Equilibrium and Thermodynamic Studies for the Removal of Anionic Congo Red Dye by *Carpobrotus Edulis* Plant as Low-Cost Biomaterial

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ABSTRACT

Organic synthetic dyes are widely used in textile industry. The presence of dyes in the effluents is a major concern due to their effect on health and environment. Thus, these pollutants must be removed from wastewaters by appropriate treatments before their evacuation. In this regard, much attention has recently been paid to dried plants as low cost biomaterials. In the present study, we aimed to investigate the removal of Congo red dye (CR) from aqueous solutions by *Carpobrotus edulis* plant as natural adsorbent using batch adsorption technique at room temperature. The effect of contact time, pH, dye concentration, and adsorbent dose on the removal of dye was also studied. The experimental data were analyzed by using mathematical models to determine the thermodynamic parameters. The negative values of free energy change indicated the spontaneous nature of the adsorption and negative value of enthalpy change suggested the exothermic nature of the adsorption process. The characterization of the dried plant was studied by using different methods such as; BET method, SEM-EDX and Elemental analysis techniques. Overall the results of the present study show that C. edulis plant can be successfully used for removal of anionic textile dyes from industry effluents.

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Keywords: Adsorption, Congo Red Dye, *Carpobrotus Edulis* Plant, Natural Adsorbent, Wastewater Treatment.

Introduction

Organic synthetic dyes are widely used in textile industry. The dye affinity for textiles depends on their chemical structures and the nature of fibers employed. Industrial wastewater containing organic pollutants have been widely studied in order to treat before discharge into the environment. These colorful discharges pose an aesthetic and health problem because many of dyes are toxic [1, 2]. Several techniques have been employed for the removal of dyes from industrial effluent including precipitation, coagulation-flocculation, reverse osmosis, filtration, ozonation, and adsorption process [3-5]. The adsorption is one of the most used for this disposal techniques to treat textile waste effluents because it is easy availability, most profitable, essay of operation and most efficient [6, 7]. A wide variety of materials have been tested for the removal of dyes from aqueous solutions [8, 9]. However, most of these adsorbents do not require relatively long adsorption contact times. Several low cost adsorbents namely, agricultural waste peels, biomass-based activated carbon and industrial by-products have been discussed in a number of research papers for the removal of water pollutants and their mechanisms [10-14]. In our laboratory, work is underway to evaluate the possibility of using natural materials for the management of industrial wastewater pollution [15-16, 4]. Many studies on the use of the *Carpobrotus edulis* plant as an adsorbent have been tested by our laboratory [16-19]. The objective of this study was to determine the optimal conditions for the removal of Congo red dye by *C. edulis* plant. The effect of contact time, temperature, pH, dye concentration, and adsorbent dose on the removal of dye was studied. The characterization of the dried plant was also presented.

Material and Methods

Preparation of the Natural Adsorbent

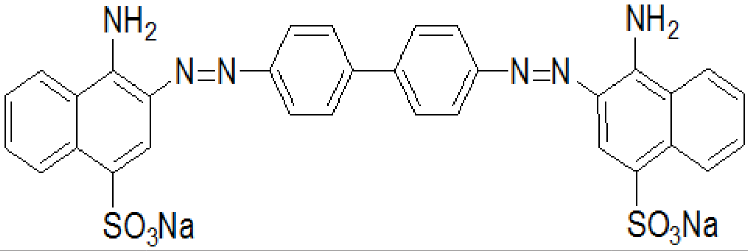
C. edulis plant was collected from the region of Agadir (Mediterranean zone of Morocco) in June 2018. The dried stems and leaves of *C. edulis* plant were converted into micro-particles by grinding them in a Mechanical grinder. These micro-particles of this plant were used as adsorbent materials. They are known to have a rich polypeptides content, the importance of which has been demonstrated in previous work, regarding the ion binding [17, 20].

Preparation of Dye Solutions

Congo red (CR) is a benzidine-based, direct, anionic diazo dye prepared by coupling tetrazotised benzidine with two molecules of napthionic acid. Its molecular structure and physicochemical characteristics are presented in Table 1. Congo red is the first synthetic azo dye produced that is capable of dying cotton directly. Congo red containing effluents are generated from a number of industrial activities: textiles, printing and dyeing, paper, rubber, plastics industries [4, 21, 22]. Besides, CR has been widely used for staining (bacteria, carious tissue, fungi, polyglutamine protein, skin, etc.) and biological applications (detecting bacteria; Alzheimer's desease, etc.) [23].

The anionic Congo red dye was purchased from Loba chimie. A stock solution of 1000 mg/L in Congo red dye was prepared with double distilled water .The working solutions were obtained by diluting the stock solution.

Table 1: Properties of Congo Red Dye

Molecular structure					
					
Molecular formula	Molar mass (g/mol)	C.I. number	CAS number	Type	λ_{max} (nm)
$C_{32}H_{22}N_6Na_2O_6S_2$	696.66	22120	573-58-0	Anionic	497

Characterization of Natural Adsorbent

In order to know the structure sight of the dried plant, scanning electron microscopy (SEM) was generally employed to observe samples morphology. The micro-particles structure and morphology of dried C. edulis plant was obtained by using Scanning Electron Microscopy (SEM, Tescan, Vega3, CAC-Cadi Ayyad University), with an accelerating voltage of 20 kV. The elemental analysis of dried plant was analyzed by SEM coupled with Energy dispersive X-ray (EDX) analyzer employed to divulgate the different elements presenting in the micro-particles of the dried plants.

Specific surface area of the dried plant was evaluated through N_2 adsorption at 77 K, using a Micromeritics ASAP 2010 instrument (IEMM, Montpellier, France). The Brunauer–Emmet and Teller (BET) model was applied to fit nitrogen adsorption isotherm and evaluate the surface area (S_{BET}) of the adsorbent.

Experimental Procedure

The removal of Congo red dye was performed by batch adsorption experiments at room temperature. Well established amounts of *C.edulis* plant were placed in Erlenmeyer glass flasks of 100 ml containing 40 ml of dye solution of known concentration and pH. The solutions were vigorously stirred for a given time period to reach equilibrium. The agitation speed was kept constant for each run to ensure equal mixing. After different contact times (t_c), the resulting solutions were centrifuged at 5000 rpm for 10 min and the supernatant was filtered through a 0.45µm membrane filter and the filtrate was analyzed. The residual concentration of dye solution was determined by using UV-Visible (Tech Comp UV 2300 Spectrophotometer) at the respective λ_{max} value, which is 497 nm for CR.

The Congo red dye uptake (mg.g^{-1}) and the removal dye percentage (%) were determined as follows:

$$q_t = \frac{V(C_i - C_t)}{m} \qquad (1)$$

$$\%Removal = \frac{100(C_i - C_t)}{C_i} \qquad (2)$$

Where C_i (mg/L) and C_t (mg/L) are the initial and final dye concentrations respectively. q_t(mg/g) is the amount removed per mass unit of the bio-adsorbent at time (t); m (g) is the amount of the bio-adsorbent and V(l) is the working solution volume.

Results and Discussion

Characterization of Natural Adsorbent Material

A detailed characterization of *C. edulis* plant adsorbent has been done by different techniques as it was described in our previous report [17, 19, 24]. The scanning electron microscope (SEM) has been a primary tool for characterizing the surface morphology and fundamental physical properties.

Generally, the adsorbent with porous morphology has hight adsorption capacity [17, 25]. The SEM of *C. edulis* plant (Figure 1) shows a homogeneous surface and porous structure.

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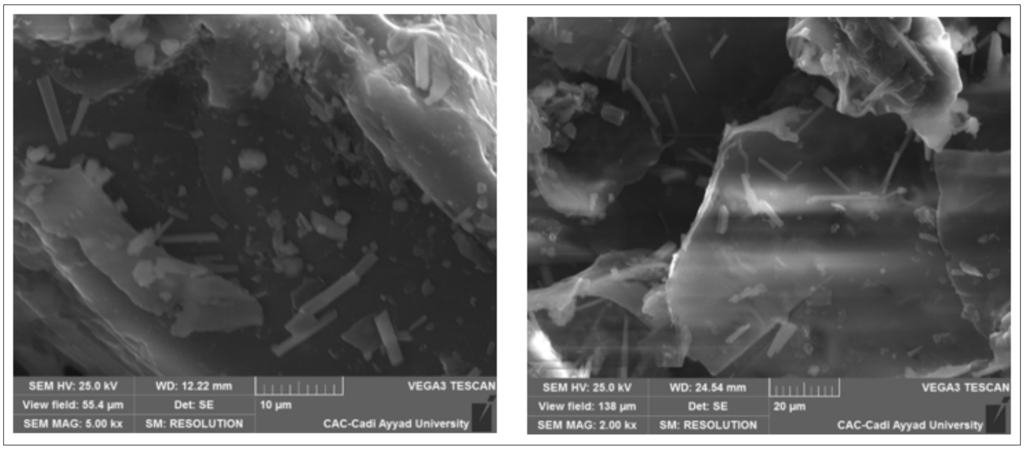

Figure 1: SEM of *C. edulis* plant, particle size < 500 µm

The results of EDX analyses showed that the contents of Carbon and Oxygen in dried *C. edulis* plant were higher suggesting that the former carbon possessed more oxygen-containing surface functional groups. However, the specific surface area of the *C. edulis* plant is in order of 2.6 $m^2.g^{-1}$ [19].

Effect of Contact Time on Dye Removal

The effect of contact time on the adsorption of Congo red dye was performed for initial concentration, C_i=20 mg/l. The impact of contact time on the adsorption of CR dye solution at room temperature (23 ±2 °C) and initial pH value 6.4 was shown in Figure 2.

It is noted that the uptake adsorbed of Congo red dye retaining by *C. edulis* surface increases with contact time. This adsorption is very fast in the first 90 min. Then, the adsorption equilibrium is reached after a contact time of about 120 min. The percentage removed of Congo red dye at equilibrium is about 70 %. This result could be due to the high availability of free active sites at the beginning of adsorption process [19, 26, 27]. This study revealed that the contact time is an important parameter for the adsorption of Congo red dye on *C. edulis* plant. So, taking into account these results, the contact time was fixed to 120 min for the rest of the batch experiments. The Figure 2 also shows that the pH at equilibrium does not change with respect to time.

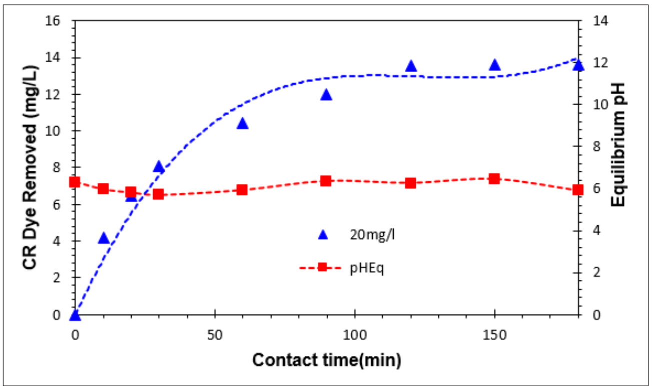

Figure 2: Effect of contact time on the removal of CR onto *C. edulis* plant: m/V = 20g/l, Ci =20 mg/L pH i=6.4 and T = 23±2°C.

Effect of Initial Dye Concentration

The initial concentration of Congo red dye varied from 20 mg/l to 300 mg/l at two temperatures and a fixed initial pH. The results are illustrated in Figure 3. The increase in the adsorption capacity is probably due to greater interaction between the adsorbate and adsorbent. At equilibrium (after 120 min of contact time the adsorption capacity of *C. edulis* plant increased with the initial Congo red concentration in the solution which may be due to more availability of acid dyes molecules and the active sites of the adsorbent have not reached saturation even at initial dye concentrations more than 200mg/l. The similar results was reported by other researches [28].

The higher amount of dye adsorption at higher concentrations is probably due to increased diffusion and decreased resistance to dye uptake [29, 30]. These results indicate a great potential application of *C. edulis* plant particles, as a bio-adsorbent to the treatment of industrial wastewaters.

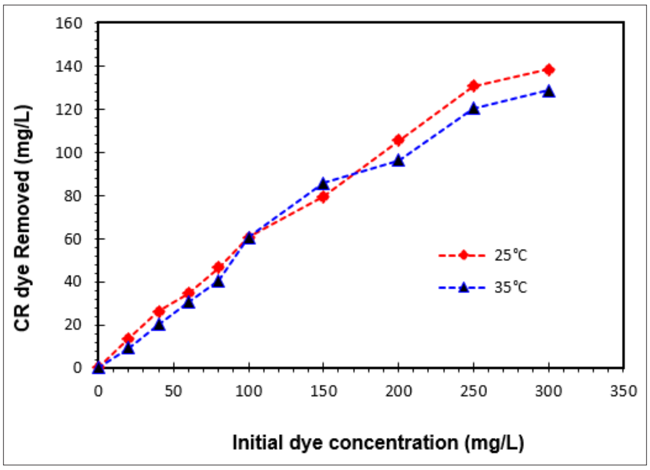

Figure 3: Effect of initial dye concentration on the removal of Congo red: Ratio m/V=20 g/L, pH=6.4, t_c =120 min

Effect of Initial pH Solution

The effect of solution pH was investigated according to the following procedure. A mass of 0.8 g of dried *C. edulis* was added a number of glass bottles containing 40 ml of dye solution. The pH of the dye solutions was adjusted over the range pH2 to pH12 using 0.1 M HCl or 0.1 M NaOH solutions.

The results presented in Figure 4 indicate that Congo red removal is considerably affected by the initial pH of solution. From these results, the Congo red uptake, first increased slightly from pH 2 to 8 and then after decreased with further increase in the pH up to 12. The point of zero charge of dried *C. edulis* plant was found to be 7.6 [19, 24].

So at pH less than pH_{pzc} the surface of the dried plant is predominated by positive charges, the surface has a high positive charge density, so the uptake of negatively charged Congo red dye would be high. while at pH greater than pH_{pzc} the surface is predominated by negative charges, the surface has a high negative charge density, so the uptake of negatively charged Congo red dye would be low. The maximum dye removal for adsorbent was observed at a pH value of 4. The pH effect on the dye removal may be due to the chemical and electrostatic interactions between the adsorbent surface and the dye molecules [31] (Figure 4).

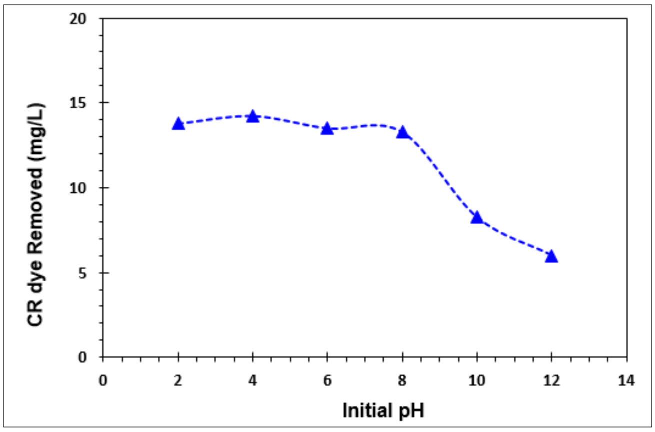

Figure 4: Effect of initial pH on adsorption of Congo red dye: Ci = 20 mg/l, m/V=20 g/L, T=23±2°C, and, t_c = 120 min

Effect of Ionic Strength

Ionic strength is an important factor that can enhance or adsorb anionic dyes such as CR dye. Therefore, a study was applied to measure the adsorption of dyes in the presence of NaCl salt at concentrations in the range of 0.01-0.50 mol/L. The results show that the adsorption of the anionic CR dye in the presence of salt remains approximately constant with increasing NaCl concentration (Figure 5). This shows that there is no effect of ionic strength on the adsorption capacity of Congo red dye. A similar study by Al-Degs et al.,2008 on the removal of some anionic dyes (Reactive Yellow 2, Reactive Red 4 and Reactive Yellow 2) on activated carbon showed that the percentage removal of reactive dyes increases with the addition of small amounts of salt, and then it remains constant [32].

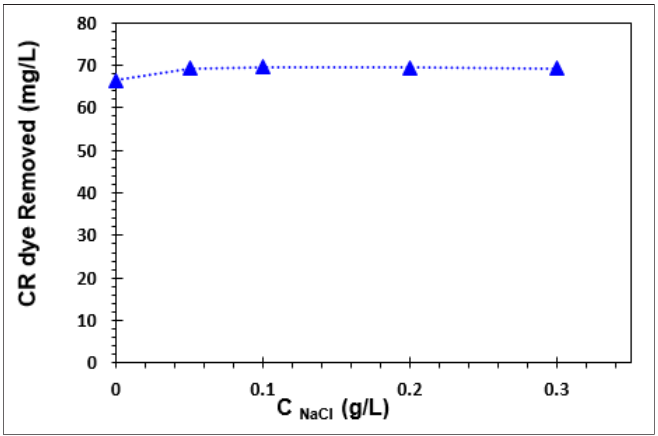

Figure 5: Effect of the ionic strength on the adsorption capacity of CR dye onto *C. edulis* plant: Ci = 20 mg/l, m/V=20 g/L, pH = 6.4, and t_c= 120 min

Effect of Temperature

The temperature of the dye solution played an important role in the whole adsorption process and particularly on the adsorption capacity. The effect of temperature on the adsorption of Congo red dye by *C. edulis* plant was studied in the temperature range of 25°C to 40°C. The results are illustrated in Figure 6. It was observed that the adsorption capacity is slower at higher temperatures and the decrease of adsorption capacity of Congo red with increasing temperature, this result indicates that the adsorption process nature is an exothermic.

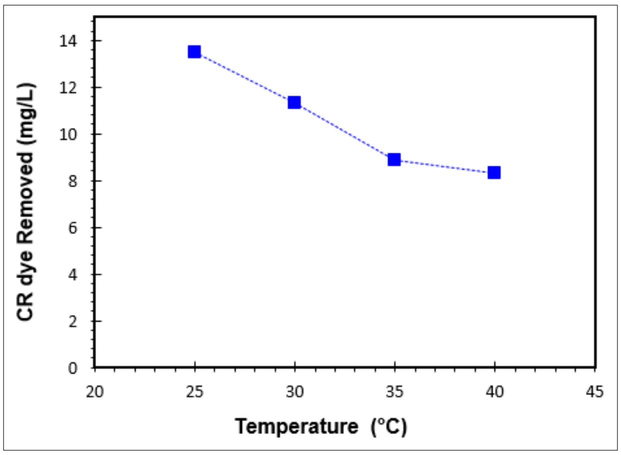

Figure 6: Effect of temperature on Congo red adsorption by C. edulis plant: Ci = 20 mg/l, m/V=20 g/L, pH = 6.4, and t_c= 120 min

Adsorption Thermodynamic

The thermodynamic study was carried out to have an idea about the nature of adsorption CR dyes on *C. edulis* plant as natural adsorbent. The thermodynamic parameters such as Gibbs energy change (ΔG °) (J mol^{-1}), enthalpy change (ΔH °) (J mol^{-1}) and entropy change (ΔS °) ($J.K^{-1}\ mol^{-1}$) were determined by these equations [33]:

$$\Delta G^{\circ} = -RTLn(K_0) \text{ Eq. (3)}$$

$$Ln\ K_0 = \frac{\Delta S^{\circ}}{R} - \frac{\Delta H^{\circ}}{R}\left(\frac{1}{T}\right) \text{ Eq. (4)}$$

$$\Delta G^{\circ} = \Delta H^{\circ} - T\ \Delta S^{\circ} \text{ Eq. (5)}$$

Where K_0, T (K) and R ($J.mol^{-1}\ K^{-1}$) are the thermodynamic equilibrium constants, the absolute temperature and the universal gas constant (8.314 $J.mol^{-1}\ K^{-1}$) respectively.

The enthalpy and entropy values were obtained from the linear plot of the variation of Ln (K_0) vs 1/T (Figure 7); ΔH°/R and ΔS°/R are the slope and intercept, respectively.

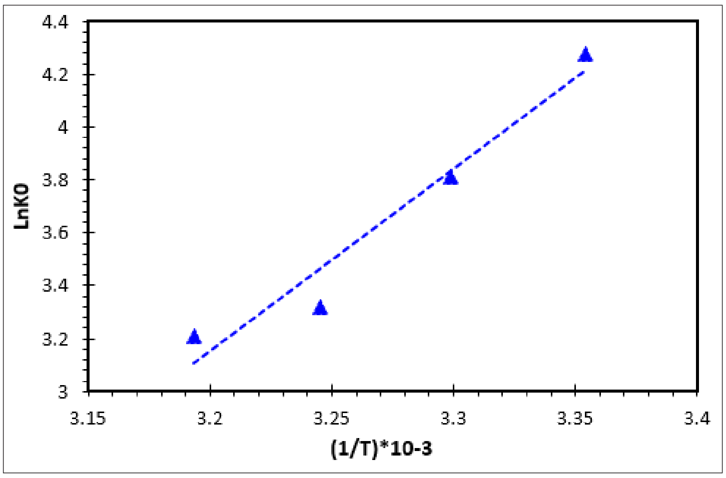

Figure 7: Variation of LnK_0 as a function of 1/T for the adsorption of CR dye on *C. edulis* plant (adsorbent dose = 20 gL^{-1}, C_i = 20 mg L^{-1}, t_c = 120 min, T=25 °C, pH CR=6,4)

The values of thermodynamic parameters (ΔH°, ΔS° and ΔG°) for the adsorption of Congo red dye are presented in Table 2.

Table 2: Thermodynamic Parameters of Adsorption

System	ΔG° (Kj.mol^{-1})				ΔH° (KJ.mol^{-1})	ΔS° (J.K^{-1}.mol^{-1})
	25 °C	30 °C	35 °C	40 °C	-57,52	-157,89
C. edulis plant /CR dye	-10,59	-9,60	-8,50	-8,35		

According to these results, we found that the value of ΔG° at different temperatures is negative, which indicates that the adsorption of CR on the C. edulis plant as natural adsorbent is spontaneous [34, 35]. In this study, all ΔG° values are between 0 and -20 kJ/mol, which clearly shows that the adsorption to be physical process in nature (physisorption). The ΔH ° values are found to be negative confirming that the adsorption is exothermic. The negative values of ΔS° show that adsorption occurs with increasing order at the solid-solution interface [36, 37].

Comparison of Adsorption Capacity with Different Adsorbents Reported in Literature

In order to justify the validity of *C. edulis* plant as natural bio-adsorbent materials for adsorption processes, its adsorption potential must be compared with other various adsorbents used for this purpose. The values of maximum adsorption capacity of the adsorbents for the removal of Congo red dye reported in the literature are given in Table 3. The direct comparison of the adsorbent capacity of *C. edulis* plant with other adsorbents capacity reported in the literature is difficult due to the varying experimental conditions employed in those studies (Initial concentration, Ratio m/V, pH of solution, contact time…). However, *C. edulis* plant in this study possess reasonable adsorption capacity in comparison with other adsorbents.

Table 3. Maximum C. edulis adsorption capacities (q_m, mg/g) of Congo red dye compared with other natural adsorbents (in mg per g of bio-adsorbent).

Adsorbent	q_m(mg/g)	Reference
Orange peel	14	[38]
Red mud	4.05	[39]
Biogas waste slurry	9.50	[40]
Bagasse fly ash	11,88	[41]
Acid activated red mud	7.08	[42]
Activated carbon (Laboratory grade)	1.88	[41]
Coir pith	6,72	[43]
Industrial waste egg shells	19.50	[44]
C. edulis plant	7.86	This work

Conclusion

This study indicates that micro-particles obtained from dried *C. edulis* plant can be used as adsorbent for the treatment of wastewater contaminated with dyes. The adsorption process has been shown to be affected from experimental conditions such as initial pH, initial dye concentration and contact time. Batch adsorption studies under varying experimental conditions showed that the adsorption of Congo red dye is fairly rapid in the first 90 min and thereafter increases slowly to reach equilibrium in about 120 min. The percentage removal of Congo red dye from aqueous solutions by dried C. edulis plant was 70%. The thermodynamic study showed that the adsorption of Congo red dye using the batch equilibration technique is spontaneous, physical and exothermic. The results of present investigation show that the dried *C. edulis* plant could be used as low cost and eco-friendly material for the removal of industrial dyes such as Congo red.

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