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Chicken feather fibres waste as a low-cost biosorbent of acid Blue 80 dye

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ABSTRACT

Chicken feathers (CFs), waste from the poultry industry, were tested as biosorbent for colour removal of synthetic coloured water. Biosorption kinetics and isotherms were determined to provide an explanation to the physicochemical behaviour of the biosorption process of C.I. Acid Blue 80 dye on CFs. Up to 80% of the dye was biosorbed at 50˚C demonstrating the biosorbent potential of the waste for the removal of organic dyes. Moreover, two mathematical approaches have been used to estimate the thermodynamic parameters such as the enthalpy, the entropy and the affinity of sorption. The first approach uses the Langmuir equation (affinity of sorption of 27,514 J/mol at 50˚C) whereas the second approach calculates the affinity directly using the chemical potentials. In both cases, the affinity of the system is positive indicating that the biosorption process occurs spontaneously. In addition, the infrared spectroscopy and scanning electron microscopy results shown that the chemical structure and the morphology of the fibres were not significantly affected by the biosorption step.

Keywords: Chicken feathers; Waste; Acid dyes; Equilibrium; Isotherms; Thermodynamics; Affinity

1. Introduction

Synthetic dyes are worldwide used in textile, printing, cosmetic and food industries generating effluents of particular environmental concern since these compounds provide an undesirable colour to the waters. Moreover, some of them are harmful compounds which may originate dangerous by-products after oxidation, hydrolysis or other chemical reactions that can take place in the water phase, risking the ecosystem they are contacting and becoming a priority to eliminate before discharging to the environment. But the complex aromatic molecular structures of the dyes make them more stable and more difficult to biodegrade [1–3]. For the treatment of these wastewaters, different techniques as sorption, biological processes, coagulation, ultrafiltration, chemical and electrochemical oxidation and photocatalytic oxidation have been used [4–7]. Among them, sorption is gaining attention with sorbents that can be obtained from industrial or agricultural wastes [8–10]. The search for biosorbents has increased due to their low cost and commercial availability

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[11–13]. Biosorption can be defined as the ability of biological materials to accumulate compounds at their surface from aqueous or gaseous phases through metabolically mediated or physico-chemical pathways of uptake.

Each European eats every year an average of 17 kg of chicken while the Americans 44 kg [14]. Taking into account that approximately a 10% of the weight of each chicken corresponds to the feathers, the chicken food industry generates thousands of tonnes of chicken feathers (CFs) which become a low-cost residue that are usually incinerated or composted [15]. Poultry feathers are a biodegradable and renewable source that during the last few years has found several applications such as composite materials [16] or also as biosorbents due to their interesting sorption properties [17]. Among the poultry feathers, CFs have already been proposed as a low-cost biosorbent to eliminate metals and some organic pollutants from wastewaters showing promising results [18, 19]. CFs are a keratin-based material, constituted by a 91% of keratin, 1% of lipids and 8% of water [16], that due to the amino acid composition show amphoteric properties according with the degree of dissociation of mainly the carboxyl and the amino side-chain groups. In addition, CFs contain two differentiable parts, quill or rachis and fibre or barb. Taking on board the above-mentioned properties, CFs can be a potential biosorbent for sorption of organic dyes such as the C.I. Acid Blue 80. Concurrently, the reuse of CFs would benefit both the processing industry (revalorization of a waste) and the environment (removal of hazardous compounds). After the biosorption step, the resulting waste of CFs loaded with dye would follow the established regulations of each country for their disposal and treatment, for example incineration.

In the present work, the dye Acid Blue 80 has been chosen as model pollutant to study the sorption onto CF fibres. This dye is a diprotic acid with sulphonic groups which suffers dissociation in aqueous solution. Among the different families of dyes (reactive, disperse, acid, basic, sulphur…), acid dyes are extensively used in the textile industry for the dyeing of wool, silk and polyamide and also, to some extent, for paper, leather, ink-jet printing, food and cosmetics [11]. The principal chemical structures of these dyes are azo, anthraquinone, triphenylmethane, azine, xanthene, nitro and nitroso being most of them soluble in acidic water generating so polluted, acidic and coloured effluents which need to be further treated to reduce their environmental impact. Hence, the interest of the application of biomass such as CFs for removing acid dyes from wastewaters.

To sum up, the purpose of this study was to study the feasibility of valorization of CFs waste as a biosorbent for removing organic dye molecules from wastewaters.

2. Materials and methods

2.1. Biosorbent preparation

CFs were kindly supplied by a waste management company located in Catalonia (Spain). The CFs were stored at −80°C to avoid degradation by microorganism until they were used. CFs were washed and sanitized by immersion into a bath of 3,300 ppm of hydrogen peroxide (Panreac, Spain) during 3 min, to sterilize them, at a liquor ratio of 40/1 and at 35°C. After that, CFs were filtered, washed with deionized water under vacuum and subsequently dried in an oven at 60°C for 48 h. Finally, CFs were dried during 30 min under vacuum. For this study, the fibre fraction of the CFs was separated from the quill for homogenization of the biosorbent samples.

2.2. Water uptake of the fibres

Water absorption experiments were carried out to know the water uptake (WU) capacity of the CFs fibres. To do so, the material was immersed into water at the three selected working temperatures (30, 50 and 70°C). Then, the capillary water was removed by centrifugation for 5 min. The WU was calculated by Eq. (1).

$$\mathrm{WU} = \frac{W_s - W_i}{W_i} \times 100 \quad (1)$$

where W_i and W_s are the weight of dry and hydrated sample, respectively.

2.3. Titration curve

CF fibres were characterized in terms of proton absorption capacity of the functional groups responsible of the amphoteric properties, mainly the carboxyl and the amino groups. To do so, fibres were immersed in HCl solutions of different concentration and with a pH of 0.75, 1.53, 2.23, 2.73, 3.16, 3.59, 4.35, 4.69, 5.32, 5.85 and 6.52. The experiments were carried out at 25°C for 48 h which was confirmed to be an adequate period of time to reach the absorption equilibrium without significant chemical hydrolysis of the fibres. Proton absorption capacity of the CFs fibres was

expressed as mol of protons absorbed in equilibrium by weight of fibre (mol H^+/kg CF).

2.4. Kinetic and isotherm studies

A preliminary kinetic study was carried out at 30 and 70°C, the extreme temperatures later used at the biosorption study, to select the appropriate equilibrium time for the subsequent biosorption isotherms study at equilibrium. Kinetic experiments were performed in batch condition using a thermostatic bath. The bath was prepared using a liquor bath ratio (mg of fibre/ml of bath) of 1/400, a dye concentration of 0.0675 g/l (which represent a 3% on weight fibre) and without any addition of electrolyte. The dye C.I. Acid Blue 80 (C.I. 61585, Fig. 1), from Ciba-Geigy (Spain), was used in the commercial form without further purification (51% w/w purity) as it would be used in industry. The CFs fibres and the dye solution were mixed and allowed to interact for 72 h. The pH of the solution was always kept at the natural pH of the acidic dye (otherwise specified), that is pH 6, and measured with a CRISON GLP 21 pHmeter (Spain). Then, the absorbance of the solution was measured using a Shimadzu UV–vis 2410 PC spectrophotometer (Spain) at the wavelength of the maximum absorbance of the dye, 626 nm.

The biosorption studies were carried out in batch conditions using a shaking thermostatic bath (Memmert, GmbH) at 30, 50 and 70°C and for the different initial dye bath concentrations of 1, 2, 3, 5 and 9% on weight of fibre. The liquor bath ratio was kept at 1/400, and the bath was continuously shaken to ensure good contact between the fibres. Once the equilibrium was reached, the absorbance of the solution was measured to calculate the dye uptake in the CFs fibres.

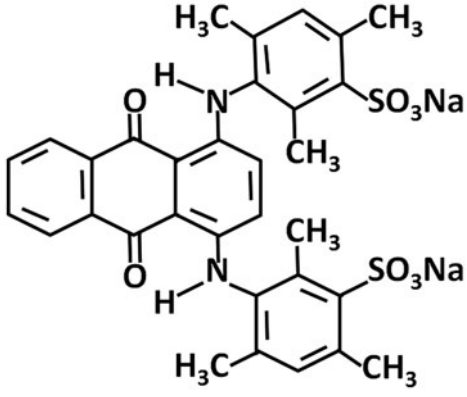

Fig. 1. Chemical structure of C.I. acid blue 80 dye (Colour index C.I. 61585).

All experiments were performed in duplicate, and the average value was plotted.

2.5. Adsorption isotherm modelling

Langmuir isotherm equation has usually been applied to model the sorption at equilibrium of acid dyes onto solid sorbents [1] and to estimate the affinity of the adsorbate by the sorbent. In particular, Langmuir model offers an explanation to the mechanism of interaction between acid dyes and keratin-based fibres, which is based on the phenomena of sorption of the dye on specific sites of the fibre [20]. The Langmuir equilibrium constant is given by Eq. (2):

$$K = \frac{[D]_f}{\left([D]_{SAT} - [D]_f\right)[D]_s} \tag{2}$$

where K represents the equilibrium constant (l/mol), $[D]_f$ is the dye concentration in the fibre (mol/kg), $[D]_s$ is the dye concentration in the solution (mol/l) and $[D]_{SAT}$ is the dye concentration at the saturation limit (mol/kg). In order to obtain the equilibrium constant (K), the linear representation of the Langmuir equation is commonly used (Eq. 3):

$$\frac{1}{[D]_f} = \frac{1}{[D]_{SAT}} + \frac{1}{K[D]_{SAT}[D]_s} \tag{3}$$

Once obtained the value of the equilibrium constant (K), the affinity or standard chemical potential ($-\Delta\mu^\circ$) of the biosorption process is calculated using Eq. (4) [20].

$$-\Delta\mu^\circ = RT \ln K \tag{4}$$

where R is the gas constant and T is the temperature.

On the other hand, a second approach based on chemical potentials considerations can be used to estimate the affinity of the biosorption process. In this model, which takes into account the pH changes of the medium and the polyacidic nature of the dye molecules, the affinity is calculated using Eq. (5) [20].

$$-\Delta\mu^\circ_{H_zD} = RT \ln\left(\frac{\theta_H}{1-\theta_H}\right)^z \left(\frac{\theta_D}{1-\theta_D}\right) - RT \ln([H_s]^z[D_s]) \tag{5}$$

where $-\Delta\mu^\circ_{H_zD}$ is the standard affinity of the sorption process (J/mol), $[H_s]$ is the protons concentration in

the solution, z represents the negative charges in the dye (in this case $z = 2$ from the two sulphonic groups), and θ_H and θ_D are the occupied sites fraction for protons and dye, respectively, or the rate of occupied sites by the total quantity of available sites (θ_T) (i.e. $\theta_H = [H]_f/\theta_T$ and $\theta_D = [D]_f/\theta_T$).

For polyacid dyes and following the assumption of Gilbert–Rideal, the occupied sites fraction can be assumed to be $\theta_{H=}z\theta_D$, that is the polyacid dye molecule is to be regarded as occupying only one available site [20]. With these assumptions, Eq. (5) is rewritten as Eq. (6):

$$-\Delta\mu^{\circ}_{H_zD} = RT\ln\left(\frac{Z\theta_H}{1-Z\theta_H}\right)^z\left(\frac{\theta_D}{1-\theta_D}\right) - RT\ln([H_s]^z[D_s]) \quad (6)$$

The standard enthalpy and entropy were calculated by plotting the known van't-Hoff equation described by Eq. (7):

$$\frac{\Delta\mu^{\circ}}{T} = \frac{\Delta H^{\circ}}{T} - \Delta S^{\circ} \quad (7)$$

The above-mentioned equations proposed to model the biosorption step (Langmuir- and potential-based equation) has been selected taking into account that the biosorption is based on an ionic interchange mechanism between the ionizable groups of CFs (–COOH and $–NH_2$) and the sulfonic groups ($–SO_3H$). It is well known that processes based on that mechanism follow the two equations proposed in the manuscript. Hence, others sorption isotherm, such as Freundlich or Nerst, were not considered for the analysis.

2.6. Fourier transform infrared microscopy

Chemical structure of CFs both before and after biosorption steps was analysed by Fourier transform infrared spectroscopy (FTIR) using a Nicolet Avatar 320 spectrophotometer (Nicolet Instrument Corporation, United States). Samples were prepared by mixing 1 mg of previously cut fibres in a matrix of 300 mg of KBr followed by pressing at 167 MPa. The FTIR spectrum was recorded in the range of 400–4,000 cm^{-1} with 40 scans and with a resolution of 4 cm^{-1}.

2.7. Scanning electron microscopy

The morphology of chicken fibres both before and after biosorption steps was inspected by scanning electron microscopy (SEM) using a Phenom Standard SEM (Phenom-World BV, Eindhoven, The Netherlands). Previous to the analysis, samples were covered with a metallic coating.

3. Results and discussion

3.1. Kinetic studies

Kinetic of biosorption of acid dye 80 in CFs is shown in Fig. 2. As it can be seen, the rate of bisorption is high during the first 250 min and after that time the rate of biosorption decreases, levelling off for time higher than 3,000 min. When the temperature increases, the percentage of uptaken dye increases from 53 to 76%, confirming the endothermic nature of the biosorption process. To ensure that the sorption equilibrium is reached, a time of 72 h (4,320 min) was chosen for all the further isotherms studies.

Besides kinetic data, equilibrium data for the bisorption of the dye on CFs are another factor to consider when establishing both the maximum sorption capacity of the CFs and the involved biosorption mechanism of interaction between dye and fibre. Hence, the equilibrium isotherms of biosorption were studied following the experimental plan previously stated in Section 2.4. The biosorption isotherm results are shown in Fig. 3.

From the results, it can be observed that at 50˚C, the biosorption was always higher than at 70˚C as it was expected, taking into account the endothermic nature of the biosorption process. On the contrary, dye sorption at 30˚C lied always below the sorption obtained at 50˚C. This latter behaviour could be explained by the combination of the following two factors: firstly, the major or minor contribution of the physi- or chemi-sorption phenomena on the dye biosorption or secondly, the degree of swelling of the fibre at a particular temperature. To confirm or reject the influence of this latter factor, the effect of the

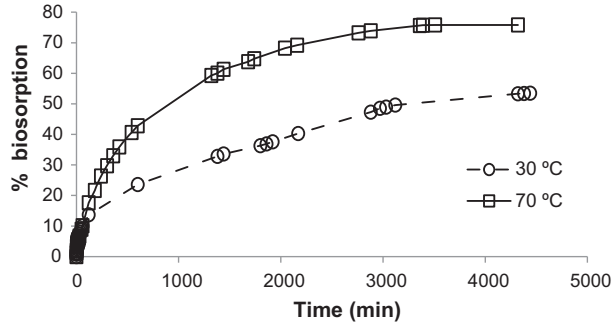

Fig. 2. Kinetic of biosorption of Acid Blue 80 in CFs at 30–70˚C (3% o.w.f.).

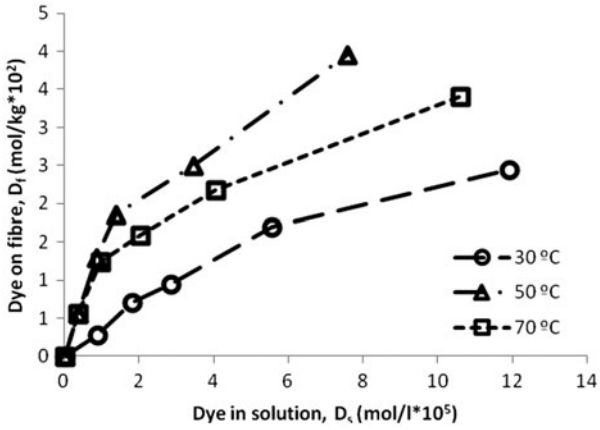

Fig. 3. Equilibrium isotherm of the biosorption of C.I. Acid Blue 80 in CF fibres at 30, 50 and 70˚C (D_f: Concentration of dye sorbed into the fibre at equilibrium; D_s: Concentration of dye in solution at equilibrium).

temperature on the WU of the CF fibre was analysed at 30, 50 and 70˚C. The results shown in Table 1 pointed up that the water sorption capacity of the fibre decreases when increasing the temperature selected for the WU test. It is important to note that the observed trend for the WU is in accordance with the thermodynamic nature of the process but it differs from the previously observed trend for the adsorption of dye at 30–50˚C (Fig. 3). In this latter case, the increase of temperature from 30 to 50˚C increases the biosorption of dye in contrast with the decrease of the sorption of water that has been observed in the former case. Consequently, it can be concluded that the above-mentioned sorption behaviour of dye induced by temperature (Fig. 3) is not caused by the insufficient fibre swelling and therefore can be justified by the mechanism of biosorption itself.

So, discarded the influence of insufficient swelling at low temperatures on the biosorption trend, the effect of the temperature can be explained by means of the combination of the following two different mechanisms: Chemisorption and physisorption [21]. The first one is predominant in the range between 30 and 50˚C and involves the chemical union between the dye molecule and the fibre producing an increase of the sorption when the temperature rises within the range (Fig. 3). The second one, physisorption, consists on the absorption of the dye molecules on the fibre without chemical union which is predominant in the range 50–70˚C, because the increase of the molecules mobility produces a decrease on the sorption rate when increasing temperature (Fig. 3). In brief, it can be said that at high temperatures, the physisorption would be the predominant mechanism while at low

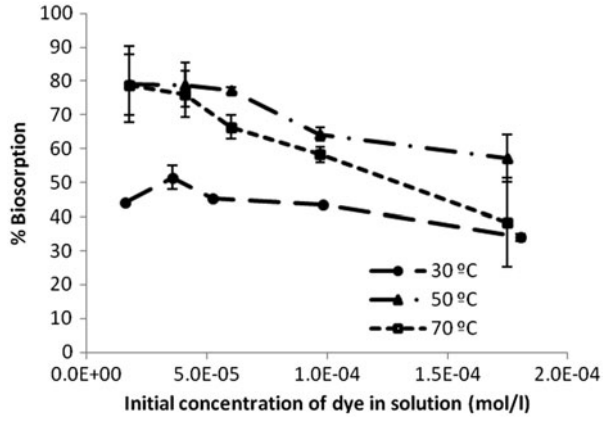

Fig. 4. Percentage of biosorption at equilibrium of C.I. Acid Blue 80 in CF fibres at 30, 50 and 70˚C.

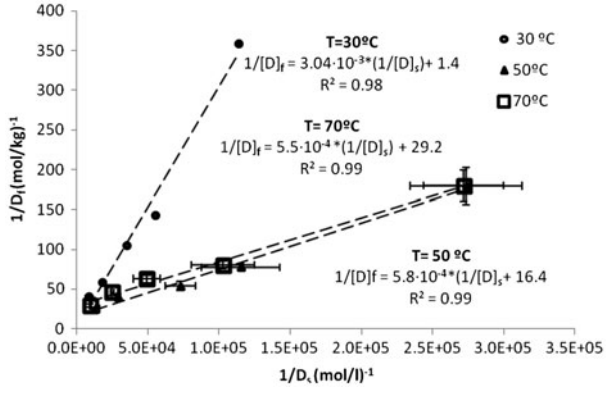

Fig. 5. Simple linear regression of the experimental results based on Langmuir isotherm (Eq. (3)).

Table 1
WU of CF fibres determined at 30, 50 and 70˚C

Temperature (˚C)	Water uptake (%)
30	61 ± 6
50	44 ± 3
70	40 ± 3

Table 2
Equilibrium constants estimated from linear regression of the data according to Langmuir model

Temperature (˚C)	$[D]_{SAT}$ (mol/kg)	K (l/mol)
30	0.71	466
50	0.06	28,044
70	0.03	52,927

Table 3
Standards affinity, enthalpy and entropy of the biosorption process

	Langmuir approach (Eqs. (1) and (3))			Potentials-based approach (Eq. (5))		
	30℃	50℃	70℃	30℃	50℃	70℃
$-\Delta\mu^{\circ}$ (J/mol)	15,484	27,514	31,029	64,952	78,040	83,242
ΔH° (J/mol)	103,732			74,963		
ΔS° (J/(mol *K*))	397.4			465.4		

temperatures, the chemisorption would be the controlling step what is in accordance with other published works [21].

In addition to that, the percentages of biosorption were calculated from the isotherms of biosorption data and the results are plotted in Fig. 4. The data show that the higher percentage of biosorption of CFs fibres was reached at 50℃ with a value of 80% at 50℃. On top of that, the percentage of biosorption was found maximum at low initial dye bath concentration ($<5\times10^{-5}$ mol/l). Also, it is worth to note that increasing the concentration of dye a decrease of the biosorption was produced, especially when the temperature was above 50℃, due to the saturation of the CFs biosorbent and to the major effect of the physisorption mechanism which induces molecules mobility.

Once the isotherm results have been discussed, their modelization has been considered in order to investigate the mechanism of interaction between dye and CF fibre pair. Modelization of the biosorption results was performed fitting the results to two approaches: (i) the Langmuir model given by Eq. (3) and (ii) the chemical potential-based model given by Eq. (6).

Linear regression of the data for each temperature was conducted based on Langmuir isotherm model (Eq. (3)), and the results are plotted in Fig. 5.

The fitted straight lines in Fig. 5 confirm the applicability of this approach with a coefficient of determination (R^2) higher than 0.98. Additionally, from the fitting, the equilibrium constant, *K* (l/mol), and the dye concentration at the saturation limit, $[D]_{SAT}$ (mol/kg), were estimated for each temperature and the values were shown in Table 2, which were directly affected by the temperature. It can be seen that $[D]_{SAT}$ decreases with the temperature whereas the *K* increases. Consequently, an increase of the calculated sorption affinity of the dye–CFs system was also observed with temperature (Table 3).

Concurrently, the biosorption affinity was calculated using the second thermodynamic approach given by Eq. (6) and based on the chemical potentials. In this case, the required total quantity of available sites parameter (θ_T) was estimated from the titration curve of the CF fibres with hydrochloric acid which was experimentally carried out. The titration results are plotted in Fig. 6, and a value of θ_T 0.9 mol/kg (pH 2) was selected which is in close agreement with the previously reported value for wool fibres [20], demonstrating certain similarity between the CFs and wool fibres, mainly because both have a comparable chemical nature and composition [22].

Finally, standard enthalpy and entropy were estimated by linear regression of the standard affinity

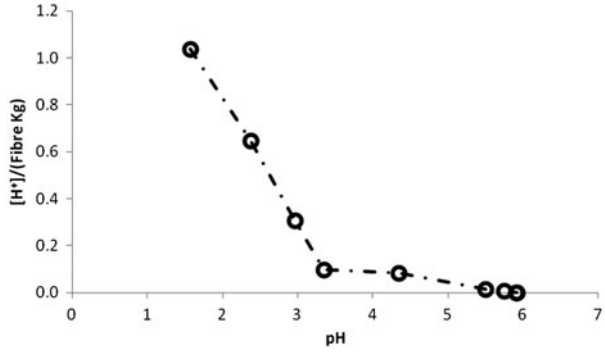

Fig. 6. Titration curve of CF fibre carried out with hydrochloric acid at room temperature.

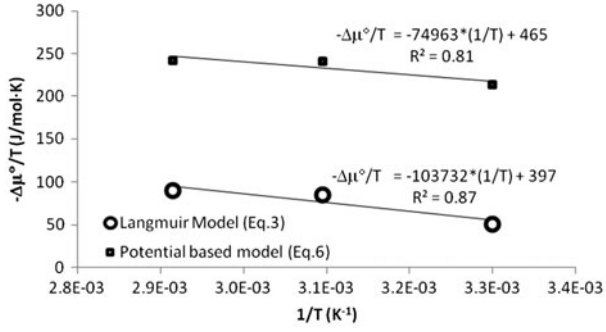

Fig. 7. Graphical representation of $-\Delta\mu^{\circ}/T$ (J/mol *K*) vs $1/T$ ($1/K$) to determine standard enthalpy and entropy of biosorption using Eq. (7).

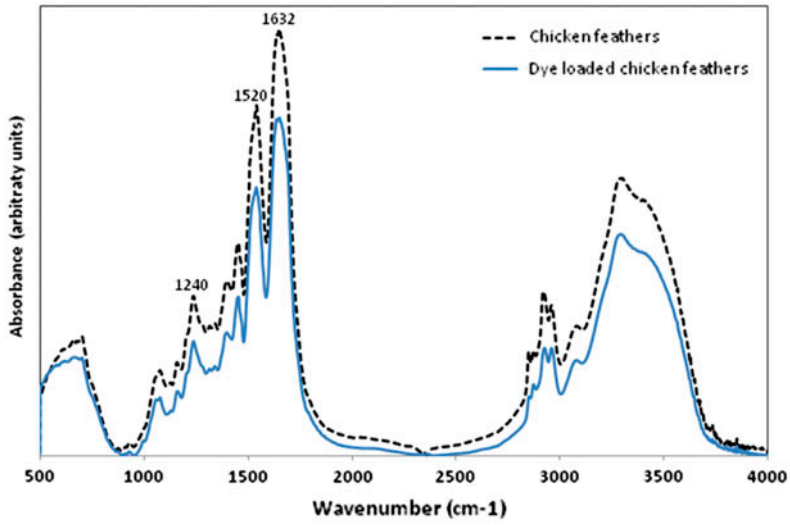

Fig. 8. Fourier transform infrared spectra of CF fibres (dotted) and dye-loaded CF fibre (line). The dye-loaded CF fibre has been treated in a bath with 5% o.w.f. of dye at 70˚C during 72 h.

results using Eq. (7). The simple linear regression was plotted in Fig. 7.

It can be seen in Fig. 7 as the two approaches studied converge approximately in the estimation of the enthalpy and entropy, but a significant different value was estimated for standard affinity. Thus, we can conclude that the process is endothermic ($\Delta H^\circ > 0$) and entropic ($\Delta S^\circ > 0$), but to determine accurately a sole value of affinity of sorption was not possible due to the significant differences between the values estimated for each model. Comparing these results with those of similar studies which used wool as sorbent [23], it was found that calculated enthalpy and entropy values for CFs fibres and the C.I. Acid Blue 80 dye were up to 50% higher. Consequently, in the case of CFs fibres—Acid dye Blue 80 system—it was necessary to provide more heat to produce the biosorption which will occur spontaneously owing to the fact that in both approaches CFs fibres shown affinity for the C.I. Acid Blue 80 dye.

As regards to the effect of the biosorption process on the chemical structure of the CFs fibres, FTIR spectra before and after the sorption of dye were collected and analysed (Fig. 8). As it is shown in Fig. 8, the broad and medium intensity bands ranging from 3,000 to 3,600 cm^{-1} are indicative of the stretches of the bonds belonging to the carboxylic acid (–COOH), the alcohol (–OH) and the amino acid ($–NH_2$) groups. The IR peaks located at 1,632 cm^{-1} (Amide I), 1,520 cm^{-1} (Amide II) and 1,240 cm^{-1} (Amide III) are related to typical amino

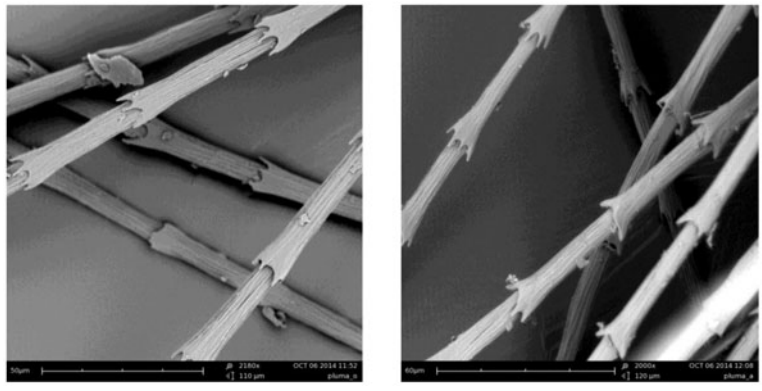

Fig. 9. Scanning electron microscopy of the barbules of CF fibres (left, magnification ×2,180) and dye-loaded barbules CF fibre (right, magnification ×2,000). The dye-loaded CF fibre has been treated in a bath with 5% o.w.f. of dye at 70˚C during 72 h.

acids of keratin-based fibres such as wool. The FTIR spectrum of the CFs was compared to the spectrum obtained for the dye-loaded CFs, and no differences were noticed for the main absorption bands. As a result, it can be concluded that there was not significant effect of the biosorption step on the chemical structure of the CFs fibres. In addition, the FTIR technique was not sensitive enough to corroborate the presence of specific interactions between the chemical groups of the CFs and the dye molecules.

In addition to the FTIR analysis, SEM was carried out for samples loaded and non-loaded with dye molecules. The SEM micrographs are shown in Fig. 9 revealing the morphology of both fibres. The microphotographs do not reveal significant differences in their morphology. Both images show the typical pattern of the barbules of CF fibres that exhibit their characteristic growth knots and the very fine striations on the surface.

4. Conclusions

CF fibres have proven to be an effective biosorbent for the removal of C.I. Acid Blue 80 dye from water and thus placing the biosorption process as a potential application for the revalorization of CF wastes.

Up to 80% of dye molecules could be biosorbed using CFs waste, and the degree of biosorption was significantly influenced by the temperature and the initial concentration of dye in solution.

Equilibrium data corroborated that chemisorption and physisorption were competitive mechanisms influenced by the temperature that control the dye degree of biosorption. The amount of sorbed dye into the CF fibres at equilibrium is higher at 50˚C than at 30 or 70˚C because between 30 and 50˚C, the chemisorption controls the sorption process while between 50 and 70˚C, the physisorption is the controlling step.

The enthalpy and the entropy of the biosorption process have converged using the two thermodynamic models. However, the affinity value estimated using Langmuir-based model was lower compared with the calculated by the model based on potential assumptions. Nevertheless, it is noteworthy that both models show the same trend with temperature.

Overall, the results confirm that there were chemical interaction between CFs and organic dyes, such as C.I. Acid Blue 80, since the biosorption took place spontaneously with a high efficacy degree. Consequently, sorption system based on the use of CFs biomass could be developed for the treatment of effluents loaded with organic dyes and reusing a part of the huge amount of CFs waste that is daily generated worldwide and what would be beneficial from the environmental point of view. Moreover, taking into account that the generation of CFs is ubiquitous, the technological development of CFs-based biosorbent system could be feasible and simply implemented where necessary, promoting its applicability on the field of the wastewater treatment.

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