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Optimization, kinetic, equilibrium and thermodynamics parameters for Congo red adsorption from aqueous phase by untreated chicken feathers

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ABSTRACT

The study of the adsorption of Congo red dye, a diazo carcinogenic compound, onto untreated chicken feathers was conducted in batch conditions. The optimization of the adsorption was evaluated using a Box-Behnken experimental design with three independent variables. It was observed that the best adsorption conditions were at pH 5, 30°C and using a semi-ground size of chicken feathers. The Langmuir, Freundlich, Temkin and Dubinin-Radushkevich models were used to analyze the experimental data, the adsorption equilibrium was well described by the Temkin isotherm adsorption model and the maximum adsorption capacity of chicken feathers obtained by the Langmuir model was 97.8 mg g^{-1} at 30°C. The results of the thermodynamic parameters showed the spontaneous nature, the feasibility of adsorption and the exothermic process due to the negative Gibbs energy value and the enthalpy value. FT-IR analysis confirmed the adsorption of Congo red on chicken feathers, showing that carboxyl and amino protonated groups from adsorbent were involved in the adsorption process.

Keywords: Adsorption; Chicken feathers; Congo red; Optimization; Thermodynamic parameters

1. Introduction

Dyes are compounds widely used in textile, paper, plastic, food and cosmetic industries, and are common constituents of effluents discharged. The presence of a small amount of dye in water is highly visible and undesirable [1], they can significantly affect the photosynthetic activity of aquatic life due to the presence of aromatics, metals, and chlorides [2]. Physicochemical processes are generally used to treat dyes present in wastewater; these processes include flocculation, electroflotation, precipitation, electrocoagulation, ion exchange, membrane filtration, electrochemical destruction, photodegradation, and ozonation. However, sometimes these processes are expensive and cannot be used by small industries to treat a wide range of dye water [3]. Treatment through adsorption is much better than other physical techniques and has become more popular in recent years owing to their efficiency in the removal of pollutants. The treatment through adsorption technique has been found to be superior to other techniques in terms of its flexibility and simplicity of design, low initial cost, insensitivity to toxic pollutants and ease of operation [1]. Many textile industries use commercial activated carbon for the treatment of dye waste but it is very expensive [4]. On this respect, the use of biomass as adsorbent has enhanced the usefulness of the adsorption process for the removal of various organic and inorganic pollutants from wastewater and the technique has turned out to be highly efficient, versatile, and economical [5]. Recently, the use of low-cost biosorbents as agricultural wastes has been increased, because they are renewable, available in large amounts and less expensive as compared to other materials used as adsor-

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bents. Some of them have been manipulated to improve their biosorptive capacity; these manipulations may be immobilization, magnetic modification, combining with mineral sorbents and chemical or biological modifications [6–8]. Despite the apparent great potential of manipulation and surface modification techniques for biosorption, these increases the commercial cost of the biosorbents closer to the price range of man-made ion-exchange resins, which compromises feature biosorbents inexpensive as its main advantage. Additionally, most manipulations raise a number of environmental, health and safety issues such as the use of: (i) aggressive and hazardous chemicals in physical and chemical manipulations and modifications; (ii) high energy electromagnetic radiation in graft polymerization; (iii) potentially harmful microorganisms and controversial genetically-modified organisms in biological manipulations [9]. Therefore, the use of waste adsorbents without or with a minimum of processing (i.e., washing, drying, grinding) reduces the production cost by using a cheap raw material and eliminating energy costs [4]. For example, the use of bird feathers has been increased because it is an available, non-soluble and non-toxic waste material, which exhibits excellent adsorption ability to remove some toxic metal ions and some dyes [5,10,11]. Particularly, the production of chicken meat in México in 2008 was around 2500 thousands of tons, giving an annual rate of expansion in the last decade of 4.9% poultry chicken feathers, give place to environmental problems as a waste by-product of commercial poultry plants. Therefore, the use of this waste without giving any treatment in the recovery of contaminated water with azo dye is of ecological importance. Consequently, the aim of this study was to know the adsorption capacity of the Congo red dye onto untreated chicken feathers, to optimize the adsorption conditions as pH, size particle, dye concentration and contact time; and to determine the adsorption isotherm and thermodynamics parameters.

2. Materials and methods

2.1. Adsorbent material

The chicken feathers were obtained from a poultry processing plant located in the State of Mexico. Before they could be used, chicken feathers were immersed overnight in a solution of neutral detergent; afterward feathers were washed, rinsed with potable water and dried in the sun. A portion of washed and dried feathers was cut with a scissor to obtain pieces of approximately 2 cm in length (semi-ground), containing the rachis; another portion was ground in a Willey mill using a 20 mesh screen.

2.2. Optimization adsorption process

The effect of particle size (ground, semi-ground and entire); pH (5, 7 and 9) and temperature (30, 40 and 50°C) were evaluated by experimental Box-Behnken design [12]. Fifteen experiments were carried out in 15 mL Falcon tubes containing 10 mL of Congo red dye at 200 mg L^{-1} in the aqueous phase and 0.12 g of chicken feathers, under conditions of the design. After 24 h the supernatant was collected by centrifugation at 12,000 rpm for 10 min, the concentration of dye was measured by absorbance at 486 nm in a UV-Vis spectrophotometer GENESYS 105. Readings were interpolated from a Congo red calibration curve (0–10 mg L^{-1}). The Congo red dye adsorbed was calculated by Eq. (1):

$$\text{Removal percentage} = \frac{C_o - C_f}{C_o} \times 100 \quad (1)$$

where C_o is the initial concentration of Congo red in mg L^{-1}, and C_f is the final concentration in mg L^{-1} after the contact time. STATISTICA 6.0 software was used to obtain response surfaces.

2.3. Adsorption isotherms

The adsorption was performed at the optimal conditions of pH and particle size selected by design Box-Behnken, 0.1 g of the chicken feathers was added to Falcon tubes containing 10 mL of the dye (100 to 2000 mg L^{-1}), the pH was adjusted to 7, and left under stirring until the equilibrium (4 h). At the end of the adsorption period, the solutions were centrifuged at 12,000 rpm for 10 min and measured by absorbance at 486 nm. The adsorbed amount of Congo red dye per g of adsorbent (q) was calculated from the mass balance by the Eq. (2), where q is the capacity of the adsorbent in mg g^{-1}; C_o is the initial concentration of dye in mg L^{-1}; C_{eq} is the equilibrium concentration of the dye in mg L^{-1}; V is the volume in L; and W is the grams of the dry weight of chicken feathers.

$$q = \left(\left(C_o - C_{eq}\right) * V\right) / W \quad (2)$$

The Freundlich isotherm is expressed by the Eq. (3), [13], where K_F is the Freundlich constant and n (g L^{-1}).

$$q_e = K_F C_e^{1/n} \quad (3)$$

Temkin isotherm contains a factor that explicitly takes into account the interactions of adsorbent-adsorbate [14]. The model is given by the Eq. (4):

$$q_e = \frac{RT}{b}\ln\left(A_T C_e\right) \quad (4)$$

where q_e (mg g^{-1}), and C_e (mg L^{-1}) are the amount of adsorbed dye per adsorbent weight unit and unadsorbed dye concentration in solution at equilibrium, respectively; A_T is Temkin isotherm equilibrium binding constant (L g^{-1}); b, is Temkin isotherm constant; R is the universal gas constant and T is temperature (K). Meanwhile, Dubinin–Radushkevich isotherm is generally applied to express the adsorption mechanism with a Gaussian energy distribution onto a heterogeneous surface [15], Eq. (5).

$$q_e = (q_s)\exp\left(-K_{ad}\varepsilon^2\right) \quad (5)$$

where q_e is the amount of adsorbate in the adsorbent at equilibrium (mg g^{-1}); q_s the theoretical isotherm saturation capacity (mg g^{-1}); K_{ad} the Dubinin–Radushkevich isotherm constant (g^2 k J^{-2}) and ε Dubinin–Radushkevich constant. Finally, Langmuir equation is expressed as Eq. (6) [16]:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (6)$$

where q_m (mg g^{-1}) and K_L (L mg^{-1}) are the Langmuir constants related to the maximum adsorption capacity of adsorbents and the affinity between the adsorbate and the adsorbent, respectively. The constant called the separation factor (R_L) defined by Eq. (7), was also evaluated; where C_o (mg L^{-1}) is the initial concentration of dye and the K_L (L mg^{-1}) is the Langmuir constant related to the energy of adsorption.

$$R_L = \frac{1}{1 + K_L C_o} \quad (7)$$

2.4. Kinetic studies

With the aim of investigating the mechanism of adsorption, the pseudo first order equation [17] and the pseudo second-order equation of Lagergreen [18], and the intraparticle diffusion equation [19] were used at 40°C by Eqs. (8)–(10), respectively.

$$\frac{dq_t}{dt} = k_1 \left(q_e - q_t\right) \quad (8)$$

$$\frac{dq_t}{dt} = k_2 \left(q_e - q_t\right)^2 \quad (9)$$

$$q_t = k_i t^{1/2} \quad (10)$$

where q_t and q_e are the amounts of Congo red adsorbed at time t and equilibrium (mg g^{-1}), respectively, and k_1 is the pseudo first-order rate constant for the adsorption process (h^{-1}), k_2 is the equilibrium rate constant of the pseudo second order equation and k_i is the intraparticle diffusion rate constant.

2.5. Thermodynamic parameters

The thermodynamic parameters were calculated using Eqs. (11)–(13):

$$K_c = \frac{C_s}{C_e} \quad (11)$$

$$\Delta G° = -RTlnK_c \quad (12)$$

$$\Delta H° = \Delta G° + T\Delta S° \quad (13)$$

where K_c is the equilibrium constant, C_s is the concentration at equilibrium of dye in the adsorbent (mg g^{-1}), C_e is the concentration of dye in the solution (mg L^{-1}) and T is the temperature (K). ΔG°, ΔH° and ΔS° are changes in Gibbs energy, in the standard enthalpy and standard entropy, respectively.

2.6. Analysis of chicken feathers

Chicken feathers were analyzed by Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectrophotometer (Cary 630-Agilent Technologies) in a range of 650 to 4,000 cm^{-1}, with 16 scans at a resolution of 4 cm^{-1}. The feathers were also analyzed by scanning electron microscopy (JSM-7800F Field emission scanning electron microscope).

3. Results and discussion

3.1. Effect of feather size, pH and temperature

There are many factors affecting dye adsorption, the optimization of the conditions will greatly help in the development of industrial-scale dye removal treatment process [20]. The response surfaces have as an objective to track efficiently for the optimum values of the variables such that the response is maximized. In our case, the effect of feather size, pH, and temperature on the removal of Congo red dye by untreated chicken feathers is shown in Fig. 1. From Fig. 1a and 1b, it is clearly evident, as the temperature increased from 30 to 50°C, the removal of Congo red varies in the range of 74 to 96 % varying the size particle and using different pH values. Meanwhile, the pH variable has a negative effect with an increasing pH values from 5 to 9 at 30°C,

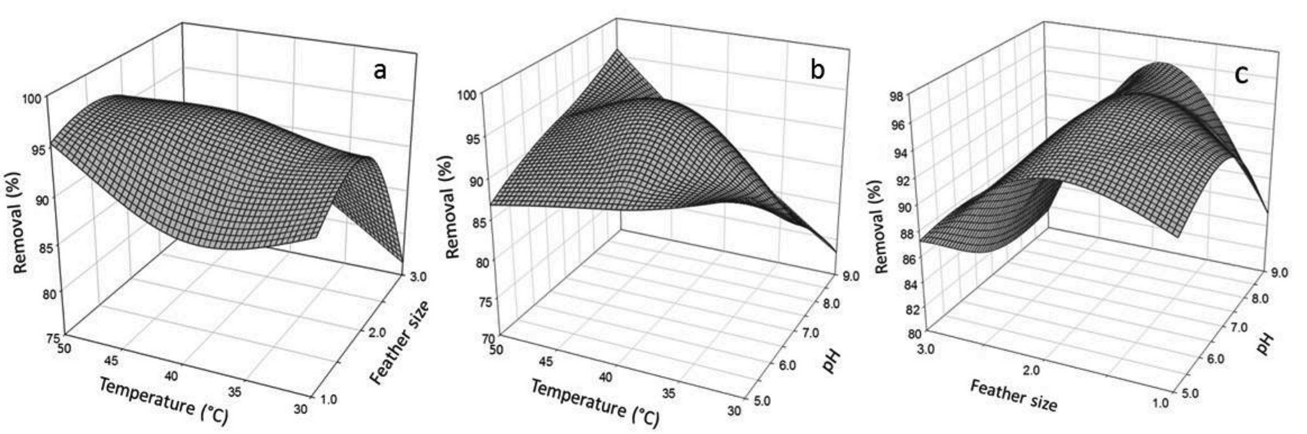

Fig. 1. Effect of temperature, pH and chicken feather size on Congo red adsorption by untreated chicken feathers. a) 3-D graphics temperature versus chicken feather size, b) temperature versus pH, c) chicken feather size versus pH.

but has a positive effect at 50°C (Fig. 1b); it can be observed that the chicken feather size affect the removal process; using the entire particle size record the lowest adsorption while the semi-ground size record high in the removal process (Fig. 1c).

Hu et al. [21], reported that the effect of pH in the removal of Congo red dye by cattail root was not affected over a pH range of 5.5–10.0, whereas Mittal et al. [11], reported that the adsorption of Congo red by oxidized hen feathers, neither the pH has an effect in the range of 3 to 11. In the present work, the particle size was the variable that caused a greater effect on dye removal; the intermediate size (number 2, semi-ground) maintained the highest dye removal (96%) compared to that obtained by the other two sizes. This last result could be due to the proportion of exposed surface of calamus, barbules, barbs and rachis in each chicken feather size. According to the response surfaces, the optimization of the removal of Congo red by untreated chicken feathers occurs at 40°C, pH 7 and using the semi-ground size.

3.2. Adsorption mechanisms

The isotherms describe the equilibrium relationships between adsorbent and adsorbate, with the remaining concentration in the solution; the correlation of equilibrium data with an equation is essential for the adsorption interpretation and prediction of the extent of adsorption. Fig. 2 shows the equilibrium adsorption of Congo red (q_e versus C_e) onto chicken feathers at 30°C (Fig. 2a) and 40°C (Fig. 2b), and Langmuir, Freundlich, Temkin and Dubinin-Radushkevich isotherm models were analyzed.

Some authors have reported that the Langmuir model is most widely used for the adsorption of pollutants from liquid solutions; however, other models have also been used to explain the process in the aqueous phase, depending on the type of adsorbent and the adsorption energy involved [22,23]. The Langmuir model showed that the maximum adsorption capacity to complete monolayer coverage on chicken feathers surface decreased as a function of temperature; that is, at 30°C was 97.85 mg g^{-1} and at 40°C was 61.55 mg g^{-1} (Table 1). This behavior was similar to the result of Mittal et al. [11], who reports the reduction of q_m from 10.6 × 10^{-5} to 3.52 × 10^{-5} mol g^{-1} (73.84 to 24.52 mg g^{-1}), due to the increase from 30 to 50°C, respectively, using oxidized hen feathers.

The removal capacity of some adsorbents previously described by other authors and the ability of the chicken feathers reported in this study is reported in Table 2, it can be seen that the obtained value in this work is comparable to others materials and in some cases was higher as banana peel, oxidized hen feathers or cattail root, it shows the potential use of chicken feathers as an adsorbent of Congo red.

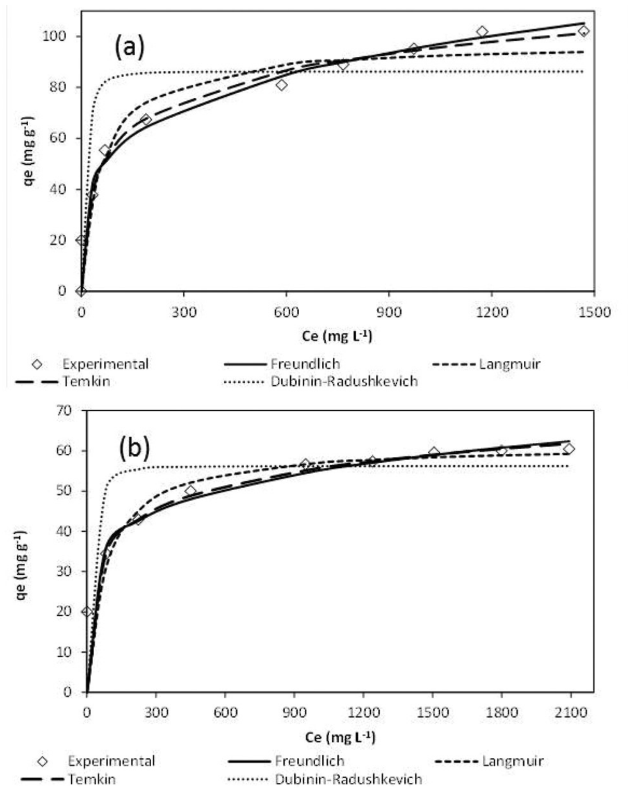

Fig. 2. Equilibrium isotherms of Congo red on chicken feathers a) 30°C and b) 40°C. Conditions: pH 5 and 10 g L^{-1} dose.

The applicability of the isotherm equation to describe the adsorption process was judged by the correlation coefficients, r^2 values. Temkin isotherm was the equation with a higher r^2 of 0.989 and 0.994 at 30 and 40°C, respectively, as compared to the other three models, which indicates that the adsorption data of the Congo red on chicken feathers were well represented by this model, which assumes that the heat of adsorption of all molecules in the layer would decrease linearly with coverage due to adsorbent–adsorbate interactions [29]. Temkin thermodynamic model is an extension of the Langmuir model; it can be interpreted in terms of two distinct physical situations: the equivalent binding sites with an adsorption enthalpy that varies with coverage due adsorbate interactions, and the second assumes a uniform distribution of heterogeneous binding sites and adsorption enthalpy that varies due to the heterogeneity of sites [30]. The Langmuir adsorption isotherm was also used to calculate the dimensionless constant separation factor. The R_L value indicates the type of isotherm; unfavorable ($R_L \geq 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). The obtained

Table 1
Langmuir, Freundlich, Temkin and Dubinin- Radushkevich isotherm constant for congo red adsorption onto chicken feathers

	Langmuir				Freundlich			Temkin			Dubinin-Radushkevich		
T (°C)	K_L ($L\ g^{-1}$)	R_L	q_m ($mg\ g^{-1}$)	r^2	K_F (mg$^{1-1/n}$ L$^{1/n}$g^{-1})	n (g L^{-1})	r^2	b	A_T (L g^{-1})	r^2	q_s (mg g^{-1})	K_{ad} (g^2 J^{-2})	r^2
30	0.016	0.020–0.17	97.85	0.9461	18.41	4.18	0.9590	154.63	0.33	0.9893	86.275	0.60	0.8749
40	0.012	0.025–0.20	61.55	0.9725	16.94	5.86	0.9870	299.01	0.73	0.9940	56.22	0.19	0.8419

Table 2
Adsorption capacities of some materials of congo red removal

Material	Maximum adsorption capacity q_m (mg g^{-1})	References
Banana peel	18.20	Annadurai et al. [24]
Bamboo dust carbon	101.90	Kannan and Meenakshisundaram [25]
Kaolin	1.98	Vimonses et al. [26]
Na-Bentonita	35.84	Vimonses et al. [26]
Coir pith carbon	6.72	Namasivayam and Kavitha. [27]
Cattail root	38.79	Hu et al. [21]
Chitin	139.00	Zuñiga-Zamora et al. [28]
Oxidized hen feathers	73.84	Mittal et al. [11]
Untreated chicken feathers	97.85	This work

values were between 0.02 and 0.2, suggesting the favorable uptake process of Congo red dye; the degree of favorability is generally related to the irreversibility of the system.

3.3. Thermodynamic parameters

Thermodynamic parameters: Gibb´s free energy change (ΔG°), entropy change (ΔS°) and enthalpy change (ΔH°) for adsorption of Congo red on chicken feathers were also determined. The negative values of ΔG° (Table 3) mean that the process is feasible and thermodynamically spontaneous; also, the higher negative values are indicative of a rapid and more spontaneous adsorption [26]. Commonly ΔG° values for physisorption are in the range of –20 to 0 kJ mol^{-1}, and those for chemisorption between –80 and –400 kJ mol^{-1}, therefore the results of the present study suggest that the adsorption is stronger than the usual physisorption. The exothermic nature process of adsorption by chicken feather is evident by the negative value of ΔH°, meanwhile the small negative value of ΔS° suggests a process slightly irreversible). Note that the adsorption process using untreated feathers proved a slightly exothermic behavior, whereas using oxidized feathers is an endothermic process [11].

3.4. Adsorption kinetics

The effect of contact time and Congo red concentration on adsorption by chicken feathers was evaluated. As shown in Fig. 3, increasing Co from 75 (Fig. 3a) to 300 mg L^{-1} (Fig. 3b), the amount of Congo red adsorbed per mass of adsorbent increased from 57.5 (Fig. 3a) to 160 mg g^{-1} (Fig. 3b), although the equilibrium time was different for different initial concentrations. In most cases, for lower concentrations of the adsorbate, the adsorption is very fast and the equilibrium time is very short. The equilibrium conditions were reached within the first 2.5 h for 75 mg L^{-1} while 4 h was needed for higher concentrations. To understand the dynamics of the adsorption process, the pseudo-first-order, pseudo-second-order rate and intra-particle diffusion models were used. The correlation coefficient (r^2) values onto chicken feathers were very similar to the four concentrations during the five hours that were studied. The q_e values calculated from the three models gave reasonable values, however, q_e values calculated by the pseudo-first and pseudo-second order were closer to $q_{e(exp)}$ values than the intra-particle diffusion model (Fig. 3). That is, the adsorption system obeys both kinetic models, maybe because both are based on the adsorption capacity [17,31]. Many biosorbents have shown that the pseudo-second-order kinetic model agreed very well with the adsorption behavior of Congo red onto cellulose waste [24], coir pith activated carbon [27] and Guava leaf powder [32] for different initial Congo red concentrations over the whole range studied; controlled by the interaction between the dye molecules and the functional groups distributed on the surface of biosorbents.

Table 3
Thermodynamic parameters for adsorption of congo red onto chicken feathers

ΔH° (kJ mol^{-1})	ΔS° (kJ mol^{-1} K^{-1})	ΔG° (kJ mol^{-1})	
		30°C	40°C
–2.28	0.147	–47.05	–48.53

3.5. Fourier-transform infrared spectroscopy analysis

The Fig. 4 displays the FT-IR spectrum of chicken feathers used to adsorb Congo red; it is very similar to the result reported by Paul et al. [33]. Some peaks are characteristics of the absorption spectra of keratin according to Sowa et al. [34] and Church et al. [35]: Amide I 1,635 cm^{-1} and Amide II 1,581 cm^{-1} which suggests the presence of a helical structure in the keratin chain, whereas the bands of Amide I (around 1,635 cm^{-1} which is merged with Amide I of a-helix) and Amide II 1,541 cm^{-1}, indicate the presence of keratin secondary structure of b-sheet type. The –C=O stretching vibration of lipids present in keratin and the C–C(=O)–O bands of lipids were at 1,165–1,090 cm^{-1}, and the envelope captured at 3,401 cm^{-1} is the overlap of –NH stretching vibration of an amide of a-helix and b-turns. This result suggests that the main functional groups involved in the adsorption process were carbonyl, carboxyl, alcoholic and amino groups, which in basic conditions become anionic in nature (carboxylate, COO–); for this reason is that under conditions of pH 9 the adsorption process was less efficient; furthermore, Congo red dye is above their isoelectric point and therefore more negatively charged. The adsorption of an anionic dye generally decreases with an increase in pH, and this phenomenon is associated not only with the negative charge

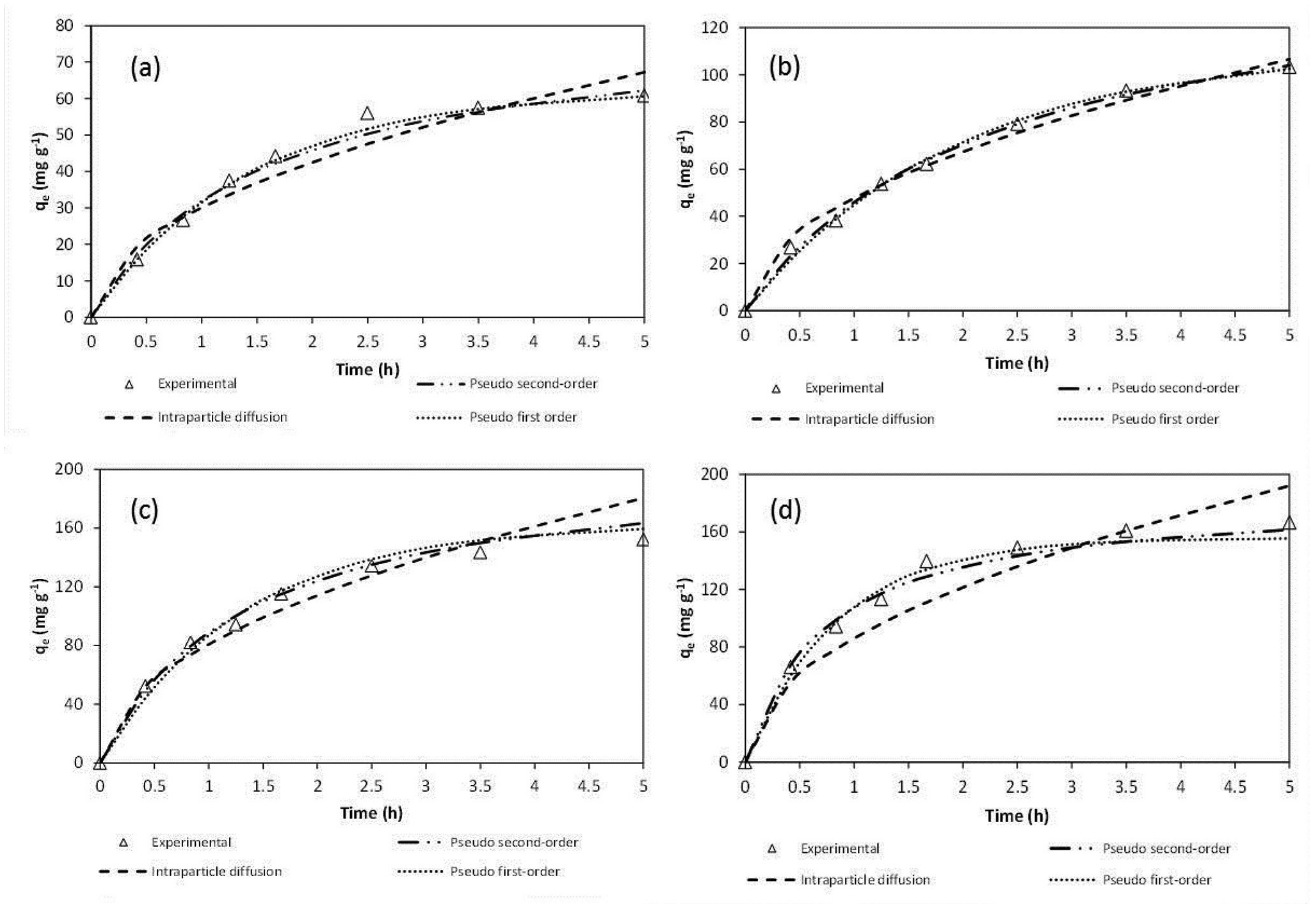

Fig. 3. Plots of q_e vs. time for Congo red adsorption on chicken feathers; initial concentration a) 75, b) 150, c) 225 and d) 300 mg L^{-1}; Conditions: pH 5 and 10 g L^{-1} dose.

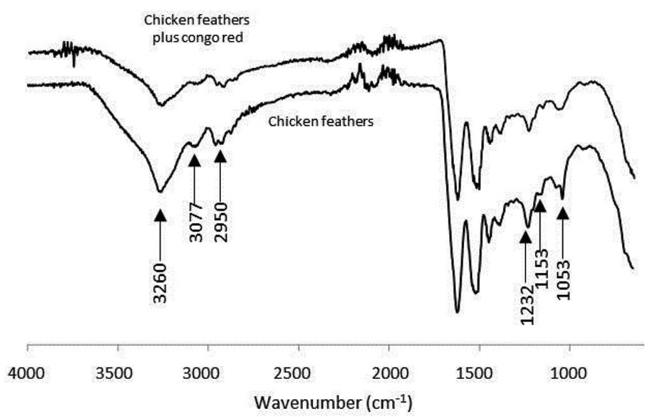

Fig. 4. FTIR Spectra of untreated chicken feathers and Congo red adsorbed on untreated chicken feathers.

on the surface of the adsorbent but also with excess OH$^-$ ions in the solution that compete for the adsorption sites [36]. Fig. 5 shows a schematic mechanism of adsorption of Congo red on chicken feathers at pH 7, where both positive and negative groups, especially groups of amino acids as arginine, alanine, threonine, glycine, praline, serine, glutamic acid, aspartic acid, tyrosine and cysteine contained in keratin could participate.

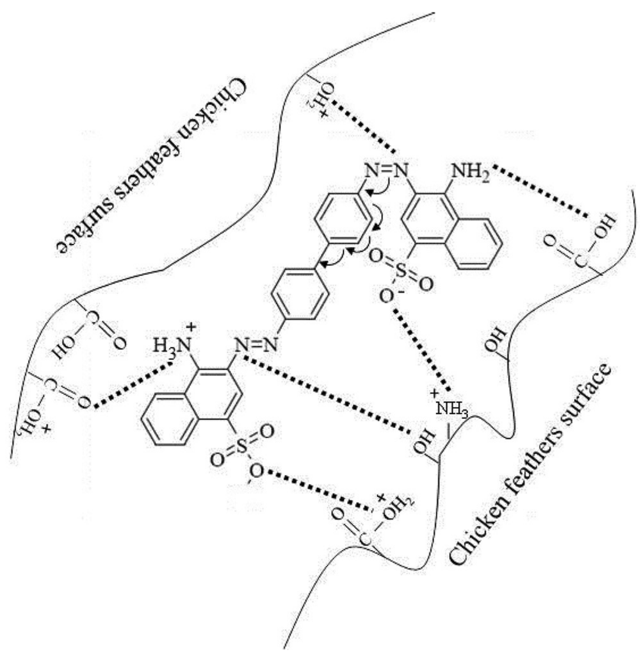

Fig. 5. Interaction proposed of the Congo red-untreated chicken feathers at pH 7, (ionic interaction and hydrogen bonding).

The analysis by FTIR shows that the main observable difference between the spectra of only chicken feather and the spectra of chicken feather containing adsorbed Congo red is a reduction or an increase in the intensities of some bands. For example, the absorption band at 3260 cm^{-1} corresponding to the stretching vibration of O–H and N–H of feathers is diminished after the Congo red dye adsorption. This could be indicative of hydrogen bond formation between the hydroxyl groups of chicken feathers and the amine group of dye molecules (Fig. 4). Furthermore, probably at pH 5 there is the protonation of carboxylic acids and hydroxyl groups of adsorbent that leads to an increase of electrostatic interactions with the dye. The increase of the peaks at 3077 and 2950 cm^{-1} could be due to–CH vibration; meanwhile the absorption band at 1232 cm^{-1} indicates that $–SO_3$ group was broadened and strengthened after adsorption. Finally, the peaks at 1153 and 1043 cm^{-1} corresponding to P=O and –C–N were also increased as a result of Congo red adsorption; although, some authors have reported also the diminished band intensities after adsorption [37,38].

Fig. 6 shows the scanning electron micrographs of chicken feathers. It can be observed that the structure of chicken feathers at different magnifications, showing the keratin fibers by which it is made, as well as porosity. It is worth mentioning that it was not possible to see through SEM any difference in structure due to the adsorption of Congo red.

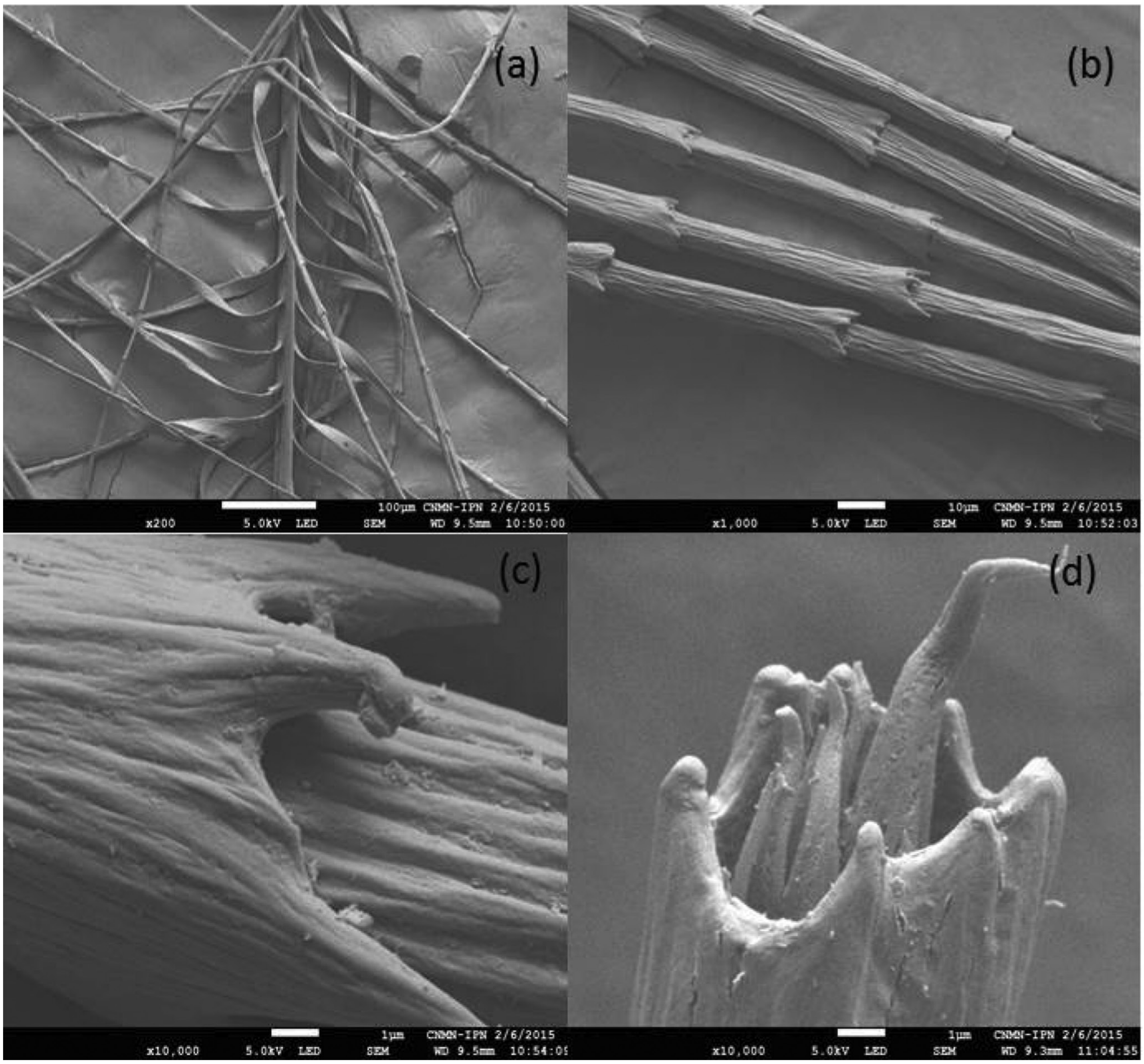

Fig. 6. Scanning electron micrographs of untreated chicken feathers at different magnifications. a) x200, b) x1000, c) x10000, d)10,000.

4. Conclusions

This research establishes that untreated chicken feather is an excellent low-cost adsorbent for the removal of Congo red. The best conditions of adsorption process were achieved using pH 5, semi-ground chicken feathers and temperature 30°C. The adsorption was found exothermic and spontaneous in nature, the maximum removal of Congo red was 97.8 mg using 1 g of chicken feathers and to establish the equilibrium was sufficient almost 4 h, the pseudo first and pseudo second order rate expression described the experimental data. The analysis by FT-IR confirmed the adsorption of Congo red onto chicken feathers and we proposed the mechanism of adsorption process through the functional groups present.

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