Diclofenac Removal with Moringa Oleífera Shell Biosorbent

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Abstract

The presence of drugs in the environment has become a global issue as, even at very low concentrations, their excessive use could lead to a resistant gene, thus rendering the use of drugs ineffective. Wastewater treatment plants still lack the necessary technology to remove low concentrations of these pollutants and therefore, a series of removal methods capable of reducing the presence of drugs in the environment have been developed. This work is aimed at studying the biosorption process. Biosorption is an adsorption process that employs biodegradable materials as adsorbents. On this occasion, Moringa oleífera shells (MO) were chosen due to their coagulant, anti-microbial and nutritional properties in order to remove residues of Diclofenac (DCF) found in the water. Studies of the pH effect of the drug solution, a determination of the adsorbent surface charge as well as a process kinetic trial have been carried out so as to determine the likely action mechanism that controls the drug and the material interactions. It has been discovered that the pH exerts a great influence on the removal percentage of DCF (88% at pH 2), the surface of the MO shells are positively charged for pH < 6 and negatively charged for the alkaline zone. In addition, the experimental results of the kinetic adsorption showed a better fit to the Pseudo-second order model ($R^2 > 0.998$), thus indicating that the mechanism of action controlling the interaction between DCF and MO could possibly be the chemisorption mechanism. The adsorption capacity obtained was 0.37 mg g^{-1} for an initial concentration of 1 mg L^{-1} at pH 2. The removal increased with contact time and equilibrium was reached after about 90 min. FTIR analysis identified several groups, including hydroxyl, carbonyl and aromatic, which are involved in the adsorption process. These results show that Moringa oleífera shells can act as a cost-effective alternative for the removal of Diclofenac from aqueous solutions.

Keywords: *Biosorption, diclofenac, kinetic, micropollutant, moringa oleífera.*

1. Introduction

The contribution of drugs in the contamination of surface water, wastewater and drinking water streams has been increasing over time [1,2]. The level of drug residues within the environment has become a global issue, because even at very low concentrations, excessive intake of this type of contaminant can cause bacterial resistance to the effects of drugs, which in turn leads to a decrease in its efficiency and therefore becomes a great threat to the area of medicine [3].

In the present work, the drug of interest is the nonsteroidal anti-inflammatory drug (NSAID) Diclofenac (DCF), also known as (2 [(2,6-dichlorophenyl) amino] phenylacetate). It is a drug prescribed to reduce pain and inflammation, that is important for treating patients with rheumatoid arthritis, or those recovering from surgery [4]. Among NSAIDs, DCF is widely used, with global comsuption estimated at approximately 940 tons annually [5]. The total consumption for Europe is estimated at 180 tons per year; meanwhile, in other countries, such as China, India and Brazil, the consumption is more than 60 tons per year [6]. Although there is a great difficulty in establishing typical values of DCF concentration. Reported studies indicate that Diclofenac concentrations in the environment vary widely, with values ranging from 0.44 to 7.1 µg L^{-1} in municipal wastewater, up to 6.88 µg L^{-1} in hospital effluents and reaching 203 µg L^{-1} in pharmaceutical industry discharges [7]. In Pakistan's rivers, a concentration of 4900 ng L^{-1} and a maximum of 1030 ng L^{-1} in water surface waters in Germany have been detected [6]. Nowadays, Diclofenac residues are widely detected in European waters, mainly due to wastewater discharges. For this reason, Diclofenac has been included in the European Union Watch List to improve monitoring and support future regulation. Recent studies have proposed environmental quality standards in the range of 0.04–0.05 µg L^{-1} for surface waters [8].

Given the widespread presence of Diclofenac in water environments, it is important to develop effective and sustainable treatment methods. Several technologies have been applied, but they are often expensive, energy intensive and can result in the generation of additional contaminants.

A series of methods has been developed to remove pollutants from water streams [9]. These technologies include biological processes such as activated sludge [10] and membrane bioreactors [11], separation techniques such as nanofiltration and reverse osmosis [12], and advanced oxidation processes such as ozonation, ultraviolet light, and UV/H_2O_2 systems [13].

One of them is the biosorption method, an adsorption process that uses biodegradable materials as adsorbents [14]. This method represents a simple, economical, and environmentally friendly solution. Besides being efficient, the addition of nutrients is not necessary, and the recovery of both the pollutant and the adsorbent is possible [15]. On this occasion, Moringa oleífera (MO) shells have been used as a biosorbent to remove low concentrations of the anti-inflammatory Diclofenac (DCF).

While a significant research on the treatment of emerging contaminants in aqueous solutions, there is still limited information on the use of Moringa oleifera shells as a biosorbent for Diclofenac removal. Most of the existing research on Moringa oleifera has focused on its seeds for the coagulation process, while the use of its shell, which is an abundant agricultural by-product, has not

been widely studied. Therefore, studying the adsorption performance of this material allows the valorisation of agricultural waste and provides a low-cost alternative biosorbent for the treatment of pharmaceutical contaminants in water.

Moringa oleífera is a tropical plant that grows in tropical and subtropical regions, and it belongs to the family Moringaceae [16]. It is recognized worldwide for its coagulant, antimicrobial, medicinal properties and its high nutritional content [16]. MO is high in protein, vitamins, carbohydrates, fiber, and fatty acids [17]. Additionally, MO is highly recognized in the wastewater treatment area. Using Moringa oleifera seeds or seed powder, it is possible to reduce water turbidity [18], remove bacteria [19], and achieve a high removal of suspended solids [18].

Various natural and low-cost materials have been used as materials for the elimination of pharmaceutical residues from water [20,21]. However, many of these materials require pre-treatment steps that may increase cost and complexity. In this context, Moringa oleifera shells represent a promising biosorbent due to their availability, biodegradability, and minimal processing requirements, making them an attractive alternative for sustainable water treatment applications.

The objective of the present paper is to understand the possible mechanism of action that controls the adsorption process of the drug. Accordingly, a study of the effect of the pH solution and an analysis of the adsorption kinetics were carried out.

2. Materials and Methods

This section describes the materials used and the methods followed in the study, including the chemicals, biosorbent preparation, surface charge determination, FTIR characterization, sample analysis, and the biosorption experiments carried out to evaluate the effects of pH and contact time.

2.1 Chemicals

The antibiotic used in this study was Diclofenac Sodium, obtained from Sigma-Aldrich Company. Also, solutions of NaOH 1M and HNO_3 1M. For all the experiences, a 1 mg L^{-1} solution of Diclofenac was diluted with distilled water.

2.2 Preparation of Biosorbent

The shells of Moringa oleífera (Fig. 1) were collected in Luanda, Angola. The shells were subjected to a drying oven process at 30 °C for one day and then pulverized through the IKA A11 grinding equipment. As it can be seen in the Fig. 2, different fractions of MO powder were separated using sieves with diameters ranging from 0.425 µm down to less than 0.075 µm). The particle size of 0.106 <µm<0.205 was selected for the experimental tests.

Fig. 1. Moringa *oleífera plant and* shells used in lab experiments.

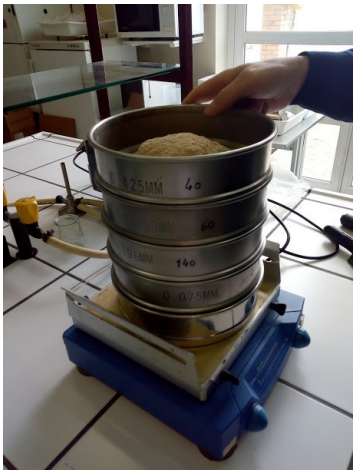

Fig. 2. Granulometric analysis (set of sieves).

2.3 Determination of Adsorbent Surface Charge

In a SHELL-LAB shaking incubator, 100 mg of MO were stirred (150 rpm) in 50 mL of distilled water at a temperature of 25 ºC. The pH was adjusted with 0.1M HCl and 0.1M NaOH using a Hanna Instruments pH meter.

2.4 Biosorbent Characterization by Fourier Transform Infrared Spectroscopy

Functional groups and biosorbent-pharmaceutical interactions were characterized via FTIR analysis. The samples were prepared as KBr pellets using a SPECAC Hydraulic Press. A Perkin Elmer Spectrum FT-IR C112095 (UATR Two) was used to obtain transmittance spectra from 4000 to 450 cm^{-1} (resolution of 4 cm^{-1}). Data processing was carried out using the Spectrum IR software, version 10.6.1.

2.5 Sample Analysis

The quantification of Diclofenac was carried out at 276 nm using an HPLC-DAD setup (VWR Hitachi ELITE LaChrom) and a Merck LiChrosorb® RP-18 column. The mobile phase, a 60:40 (v/v) acetonitrile/0.014M oxalic acid mixture, was delivered at 1.0 mL min^{-1} in isocratic mode. After injecting 20 µL samples, the target compound eluted at 4.0 min. The sensitivity of the method was confirmed by a detection limit of 0.01 mg L^{-1} and a quantification limit of 0.04 mg L^{-1}.

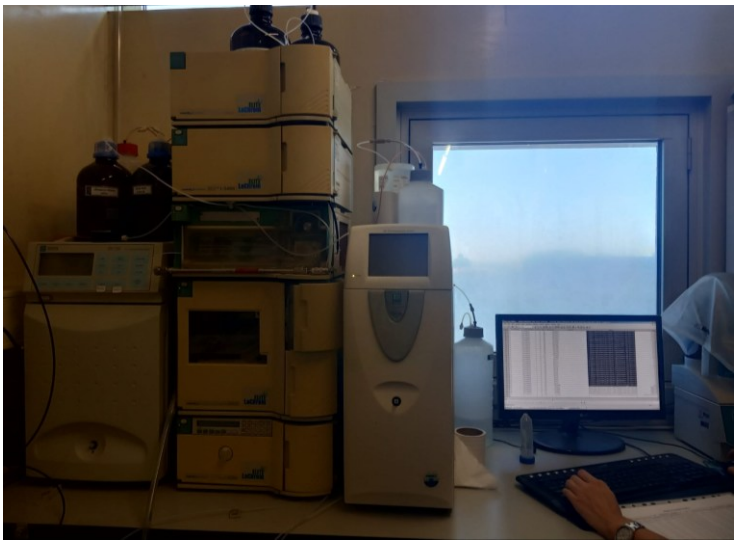

Fig. 3. HPLC equipment (LSRE-FEUP, University of Porto, Portugal).

2.6 Effect of pH

The pH level is very important in biosorption. It affects the process in two ways: it changes the chemistry of the pharmaceutical solution, and it controls the active sites on the adsorbent material, which directly impacts its ability to remove contaminants. This study tested the effect of pH between 2 and 10 (2, 5, 7, 8 and 10), maintaining all other parameters unchanged: solution volume, 50 mL; initial antibiotic concentration, 1 mg L^{-1}; adsorbent dosage, 2 g L^{-1}; stirring at 150 rpm and temperature at 25 °C.

2.7 Biosorption Kinetics

Before investigating the sorption isotherms, the equilibrium time for the batch reaction was determined through a kinetic experiment. The test was done in 50 mL Erlenmeyer flasks, each containing a solution with 1 mg L^{-1} of DCF and 100 mg of the adsorbent. The adsorption conditions were set as follows: continuous stirring at 150 rpm, a constant temperature of 25 °C, and the pH was adjusted to 2.

Samples were taken from the flaks at specific intervals: 5, 10, 20, 30, 60, 90 and 120 minutes. After collection, each sample was centrifuged (5000 rpm, 15 min) and then filtered using a syringe with a 0.45 µm filter. All essays were performed in duplicate.

Samples from the flasks were analyzed using high-performance liquid chromatography with a diode-array detector (HPLC-DAD) to measure the diclofenac remaining in the solution, and then, the biosorption removal efficiency was calculated.

The adsorption capacity of the MO, qt (mg g^{-1}) was obtained by Equation 1:

$$q_t = (C_0 - C_t) * \frac{V}{m} \tag{1}$$

where: C_0, initial DCF concentration (mg L^{-1}); C_t, DCF concentration at time t (mg L^{-1}); V, volume of aqueous solution (mL); and m, dry mass of adsorbent (mg).

In this study, three kinetic models, Pseudo-first-order, Pseudo-second-order, and Intraparticle Diffusion were applied to fit the experimental biosorption data.

Pseudo-first-order kinetic model

This model assumes that the adsorption rate is proportional to the difference between the equilibrium concentration and the amount adsorbed at any time. It is commonly used to represent processes where physical forces (e.g., diffusion) govern the adsorption mechanism, rather than chemical reactions (Equation 2).

$$q_t = q_e(1 - \exp(-k_1 t)) \tag{2}$$

where: q_t, amount adsorbed at time t (mg g^{-1}); q_e, amount adsorbed at equilibrium (mg g^{-1}); k_1, pseudo first order rate constant (min^{-1}).

Pseudo-second-order kinetic model

Commonly used to describe adsorption processes controlled by chemisorption, where chemical interactions between the adsorbate and the adsorbent play an important role. It assumes that the adsorption rate depends on the square of the number of available active sites (Equation 3).

$$q_t = (q_e^2 k_2 t)/(1 + q_e k_2 t) \tag{3}$$

where: k_2, pseudo-second-order rate constat (g mg^{-1} min^{-1}).

Intraparticle diffusion model

It is used to assess whether diffusion inside the adsorbent pores controls the adsorption rate, as described by Equation 4.

$$q_t = k_3 t^{1/2} + C \quad (4)$$

where: k_3,intraparticle diffusion rate constat (mg g^{-1} min$^{-1/2}$); C, constant related to the boundary layer thickness.

3. Results and Discussion

3.1 Surface Charge Characterization of the Adsorbent

To understand the interaction mechanism, the surface charge of Moringa oleífera was measured across the tested pH range. Results in Fig. 4 indicate a point of zero charge around pH 6, with a positive surface charge below this value and a negative one above it.

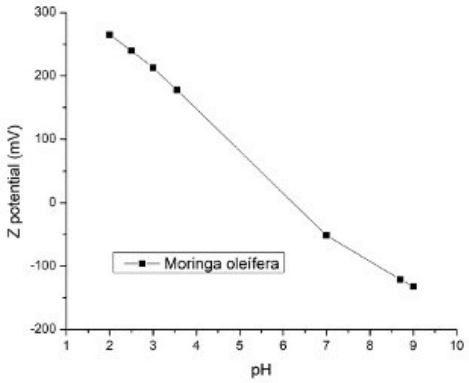

Fig. 4. Surface charge of Moringa oleifera shells at pH values from 2 to 10.

It is known that the anionic charge of the anti-inflammatory DCF [22,23]. At pH values under 6, the data indicate a powerful electrostatic attraction between the MO surface and DCF; conversely, significant repulsion is observed when the pH is greater than 6. Maximum adsorption occurred in the acidic region, with the best performance recorded at pH 2. This high efficiency reflects a great affinity between DCF and the MO particles. However, at pH 8 (the adsorbent's natural state), the removal percentage reached only about 10%. These results confirm that pH is a critical factor, driving the multiple surface interactions that control the adsorption mechanism.

3.2 Biosorbent Characterization by FTIR

The FTIR spectrum of Moringa oleífera shells showed the presence of several functional groups, indicating the complex and lignocellulosic nature of the biosorbent (Fig. 5). The main peaks identified were: a strong –OH band at 3430 cm⁻¹ (alcohols, phenols); –CH bond at 2920 cm⁻¹ (cellulose structure); carbonyl group –C=O at 1630 cm⁻¹ (amide compounds); –C=C at 1464 cm⁻¹ (aromatic compounds); carboxylic acids in the region 1384–1243 cm⁻¹; and –C–O bond at 1056 cm⁻¹ (phenols and carboxylic acids).

After DCF adsorption (Fig. 6), changes were observed at 3430 cm⁻¹ (–OH), 1637 cm⁻¹ (–C=O) and 1459 cm⁻¹ (–C=C), indicating that hydroxyl, carbonyl and aromatic groups are the main functional groups involved in DCF removal. Two new peaks appeared at 610 cm⁻¹ (–C–Cl aromatic group of DCF) and 1506 cm⁻¹ (aromatic ring of DCF), confirming the interaction between the biosorbent and the pharmaceutical.

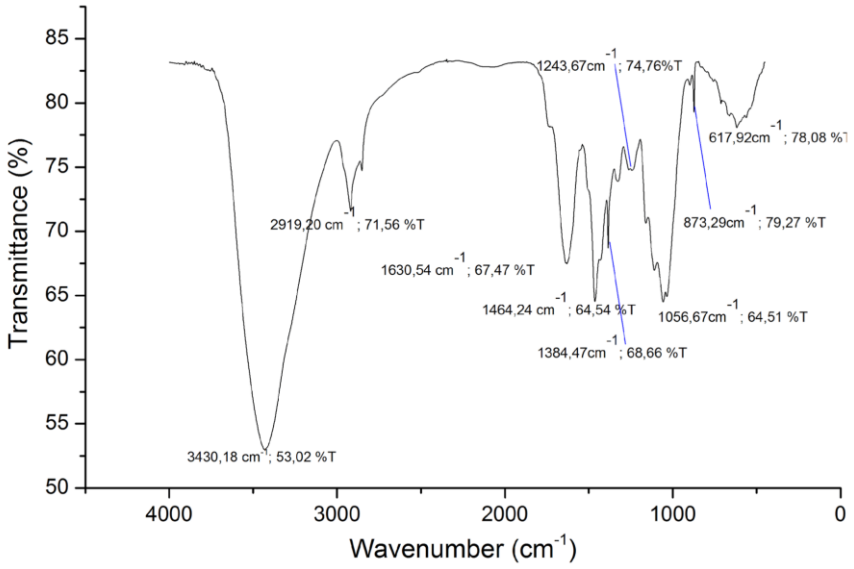

Fig. 5. Moringa oleífera FTIR spectra.

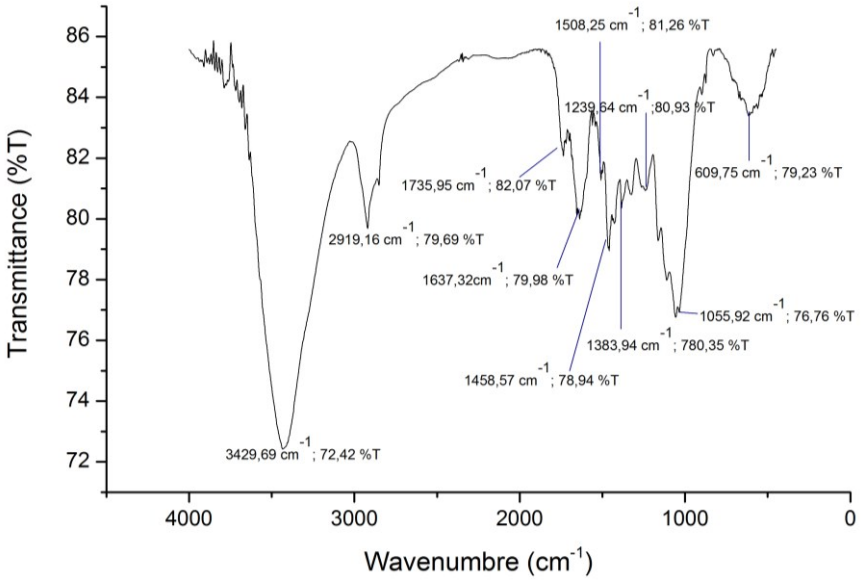

Fig. 6. Moringa oleífera FTIR spectra after the adsorption process of Diclofenac.

3.3 Effect of pH on the Biosorption Process

The solution pH is a critical factor in the adsorption of Diclofenac. The data in Fig. 7 showed that increasing the pH led to a reduction in adsorption capacity. Removal varied significantly, reaching 87.3% at pH 2 but only 4.8% at pH 8. This behaviour is associated with the surface charges of DCF and MO. At low pH values, DCF is negatively charged, while MO has a positive surface charge (Fig. 4). This creates a strong electrostatic attraction between them and favors the adsorption process. At alkaline pH, both surfaces become negatively charged, which leads to electrostatic repulsion and reduces the adsorption capacity. Similar behaviour has been reported for Moringa leaf biomass, tested for Cr(VI) removal at initial levels varying from 10 to 150 mg L^{-1}. In that study, the maximum removal was observed under strongly acidic conditions (pH 2), where electrostatic attraction favoured adsorption, while the removal efficiency decreased as the pH increased [24].

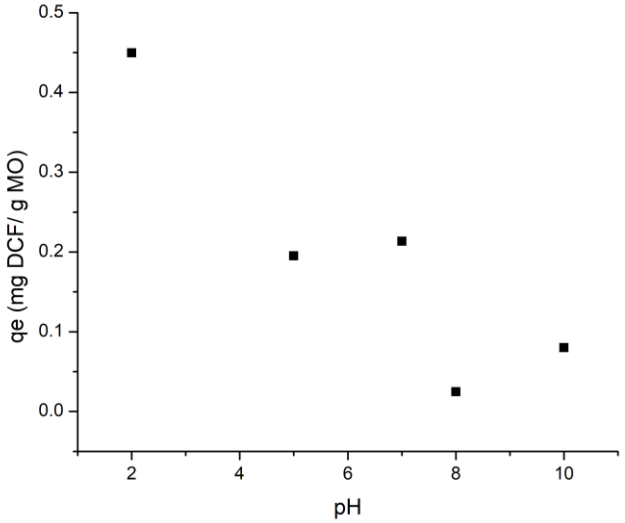

Fig. 7. Effect of pH in DCF adsorption.

The results obtained indicated that Moringa oleífera is an adequate adsorbent for Diclofenac removal from water, with a removal efficiency of 0.45 mg DCF g^{-1} MO (initial concentration of 1 mg L^{-1}).

3.4 Adsorption Kinetic Study

Fig. 8 shows the adsorption kinetics of DCF onto Moringa oleifera shells. The experiment was carried out under the conditions described in Section 2.7, using 50 mL of solution containing 1 mg L^{-1} of DCF and 100 mg of Moringa oleifera biosorbent. The removal percentage increased with contact time, reaching about 75% after 90 min. After this time, the removal increased slightly, suggesting that the adsorption process was close to equilibrium.

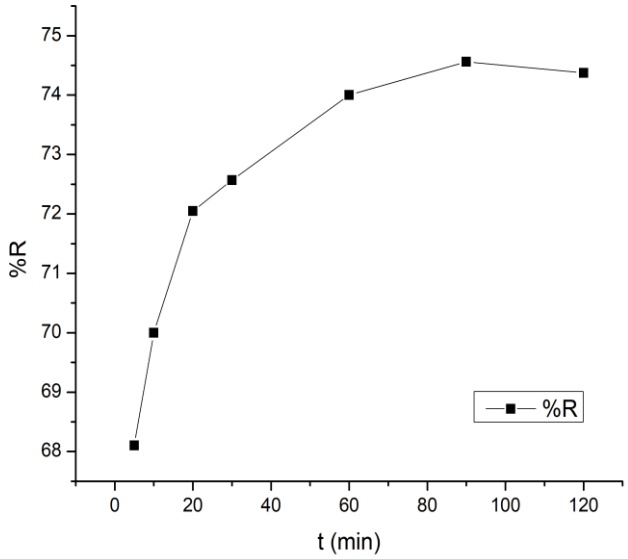

Fig. 8. Adsorption removal of Diclofenac (0.05 mg DCF, 100 mg MO).

To describe the adsorption kinetics of DCF, three models were applied: Pseudo-first-order, Pseudo-second-order, and Intraparticle Diffusion, Equations 2, 3 and 4, respectively. The kinetic parameters obtained from these models, including the rate constants, equilibrium adsorption capacities, correlation coefficients, and F-values, are presented in table 1.

Tab. 1. Kinetic parameters of Diclofenac adsorption by Moringa oleifera.

Parameter	**Diclofenac**
Pseudo-first-order	
k_1 (min^{-1})	0.4487 ± 0.0523
q_e (mg g^{-1})	0.3742 ± 0.0018
R^2	0.9623
S^2	3.12E^{-4}
Pseudo-second-order	
k_2 (g mg^{-1} min^{-1})	0.8234 ± 0.0891
q_e (mg g^{-1})	0.3731 ± 0.0009
R^2	0.9987
S^2	1.08E^{-5}
Intraparticle diffusion	
k_3 (mg g^{-1} $min^{-1/2}$)	0.0248 ± 0.0019
C (mg g^{-1})	0.2814 ± 0.0142
R^2	0.8734
S^2	1.24E^{-3}

Among the tested models (Fig. 9), the Pseudo-second-order model gave the best fit to the experimental data, with the highest R^2 value (0.9987) compared with the Pseudo-first-order model (0.9623) and the Intraparticle Diffusion model (0.8734). This result suggests that the adsorption of DCF onto Moringa oleifera shells may be mainly controlled by chemisorption, involving stronger interactions between the adsorbate and the active sites of the biosorbent surface.

The Pseudo-second-order model also provided an equilibrium adsorption capacity (q_e = 0.37 mg g^{-1}) very close to the experimental value, reinforcing the suitability of this model to describe the adsorption behavior of DCF (Table. 1). Although the Intraparticle Diffusion model also showed a relatively high fit, its lower R^2 value indicates that intraparticle diffusion was involved in the process but was not the rate-controlling step.

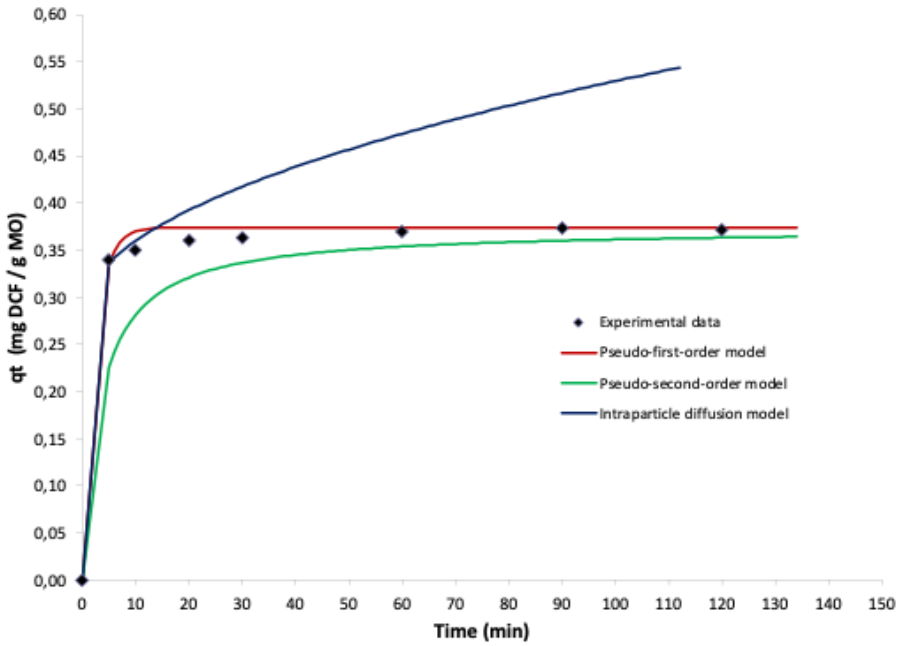

Fig. 9. Kinetic modelling of diclofenac adsorption onto Moringa oleifera shells.

The quality of each model fit was assessed using the coefficient of determination (R^2). Although R^2 gives a good indication of the fit quality, it is not enough to conclude whether the difference between models is statistically significant. For this reason, an F-test was also applied.

The test is based on the ratio of the estimated variances of the two models being compared:

$$F = \frac{S_1^2}{S_2^2} \tag{5}$$

where S_1^2 and S_2^2, variances of the two models. When the calculated F exceeds the critical value ($d_{f1} = d_{f2} = 6$; $F_c = 4.28$), the model with the lower variance fits the data significantly better. All possible pairs of models were compared at a 95% confidence level as shown in table 2.

Tab. 2. F-test results at 95% confidence level for Diclofenac adsorption (95%).

F calculated		
P1/P2	Pid/P1	Pid/P2
28.9	3.97	115

The F-test results show that the pseudo-second-order model (P2) fits the experimental data significantly better than both the pseudo-first-order model (P1/P2: F = 28.9 > F_critical = 4.28) and the intraparticle diffusion model (Pid/P2: F = 115 > F_critical = 4.28). These results confirm that the pseudo-second-order model is the most adequate to describe DCF adsorption kinetics, supporting the hypothesis that the process is governed by chemisorption.

4. Conclusions

Moringa oleífera shells show acceptable potential as a low-cost biosorbent for Diclofenac removal from water.

The FTIR analysis confirmed that MO shells have a complex lignocellulosic structure with several functional groups, including hydroxyl (–OH), carbonyl (–C=O) and aromatic compounds. After DCF adsorption, changes in these groups were observed, along with two new peaks characteristic of DCF, confirming the interaction between the biosorbent and the pharmaceutical.

The surface charge of the adsorbent is positive for pH < 6 and negative for pH > 6. DCF removal showed a strong pH dependence, with the highest removal percentage at pH 2 (87.3%) and the lowest at pH 8 (4.8%). This behaviour is explained by the electrostatic interactions between both surfaces — attraction at acidic pH and repulsion at alkaline pH.

A removal percentage of approximately 75% was reached after 90 min. Among the three kinetic models tested, the pseudo-second-order model gave the best fit (R^2 = 0.9987, $S^2 = 1.08\times10^{-5}$), with a predicted equilibrium adsorption capacity (q_e = 0.37 mg g^{-1}) very close to the experimental value.

The F-test confirmed that the pseudo-second-order model fits the data significantly better than the other two models, supporting the hypothesis that DCF adsorption is governed by chemisorption.

Acknowledgments

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