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Physicochemical characterization of Moringa *oleífera*'s shells as biosorbent for pharmaceuticals biosorption

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Abstract. The pharmaceuticals as emerging contaminants have become one of the most controversial environmental issues at global scale. Over the years, the presence of antibiotics and anti-inflammatory drugs inside rivers, lakes, and even inside drinking water streams has increased. The wastewater treatment plants (WWTPs) lack the necessary technology to remove concentrations between the range ng/l-mg/l and therefore, the need to develop new methods able to remove contaminants in an effective, low cost and environmental friendly way arises. The present work is focused on studying the potential adsorption capacity of Moringa *oleífera* (MO) to remove Diclofenac (DCF) and Oxytetracycline (OTC) from wastewater. Through different experiences, it was possible to characterizes the main functional groups of MO and determine the principal responsible of the biosorption process.

Keywords. *Pharmaceuticals, Diclofenac, Oxytetracycline, biosorption, Moringa oleífera.*

Introduction. Biosorption is an adsorption process that uses biological material as "biosorbent" (1). Moringa *oleífera* (MO), the best-known variety of the genus Moringacea, is a tree native to the southern foothills of the Himalayas, the north of India, Bangladesh, Afghanistan and Pakistan (2). It is considered one the most famous plants worldwide. So far, it is known that it has application in the areas of human and animal nutrition (3), medicinal use (4) and wastewater treatment (5). The objective of this work is to determine the main functional groups of the MO shells before and after the biosorption process of DCF and OTC.

Methods and materials

The Moringa *oleífera* (MO) shells were taken from Luanda, Angola, Africa. The pharmaceuticals were Oxytetracycline hydrochloride (>95% crystalline) and Diclofenac Sodium, both obtained by Sigma-Aldrich Company. In all the experiences, it was prepared 1 mg/l solution, diluted with distilled water.

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Biosorbent preparation

MO's shells were dried in oven at 30ºC for one day and pulverized into powder through IKA A11 basic analytical mill. Moringa powder was separated according to the size of the grain through a series of sieves with different diameters (0,425 µm, 0,250 µm, 0,106 µm, 0,075 µm and lower than 0,075 µm) ordered in column. The experiences were done with the granulometry 0,106 < µm < 0,205.

Characterization of MO by FTIR

Fourier transform infrared spectroscopy (FTIR) was performed to determine the functional groups present in the MO and the effect of the interaction between the pharmaceuticals and the biosorbent. For this analysis, it was prepared KBr pellets, through the use of the SPECAC Hydraulic Press and it was used the UATR Two Perkin Elmer Spectrum FT-IR C112095. The transmittance spectra were obtained in a wavelength range between 4000-450 cm^{-1} and with a resolution of 4 cm^{-1}. The data were processed using Perkin Elmer Spectrum IR Software Version 10.6.1.

Results and discussion

The **Erro! Fonte de referência não encontrada.Figure 1**shows the presence of many functional groups, indicating the complex nature of Moringa *oleífera* shells.

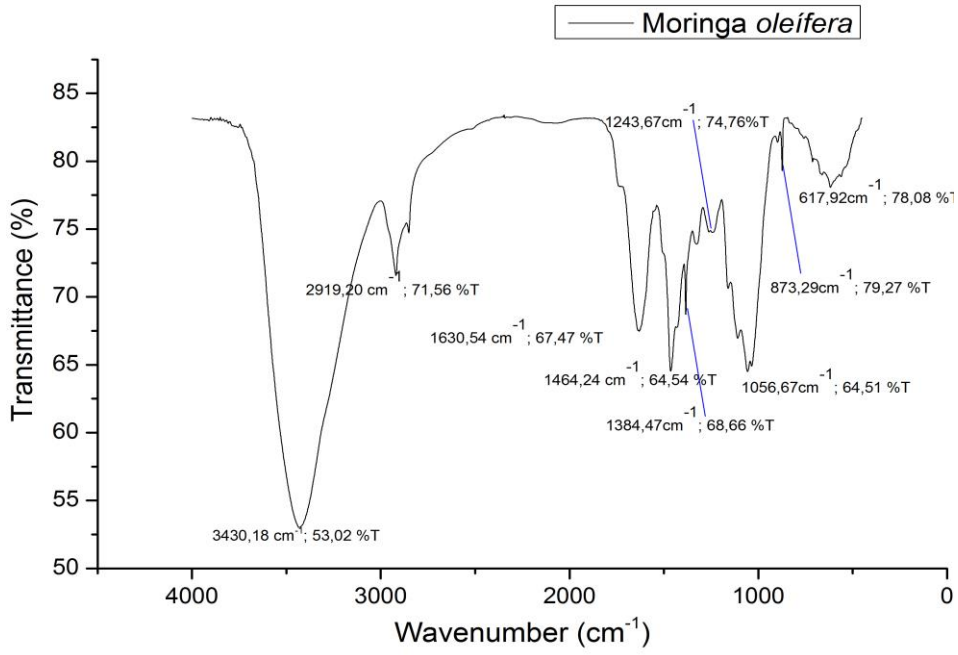

Figure 1. Principal functional groups of Moringa *oleífera*

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A strong peak at 3430 cm^{-1} indicates the presence of the hydroxyl group (-OH) which could belong to the proteins, fatty acids, carbohydrates and phenolic compounds (6,7, 8). The peak at 2920 cm^{-1} indicates the presence of -C-H bond of the CH_2 group, which could be related to the cellulose structure. The peak at 1630 cm^{-1} is due to the carbonyl group (-C=O) that could belong to the primary or secondary Amide compounds (NH_2CO). The band at 1464 cm^{-1} corresponds to the -C=C of Aromatics compounds. In the region of 1384-1243 cm^{-1}, it is found a series of weak peaks that could correspond to the presence of carboxylic acids. The strongest band is near to the wavelength of 1056 cm^{-1} and is attributed to the -C-O bond, as a prove of the presence of phenols compounds, carboxylic acids and also showing the lignocellulosic structure of the biosorbent (9).At least, the weak bands between 873-618 cm^{-1} could correspond to the -C-H bond of aromatics compounds.

From

Figure 2 can be seen that after the adsorption process, the peak at 3430 cm^{-1}, which represents the -OH- group, the band at 1637 cm^{-1} (-C=O) and the peak at 1459 cm^{-1} (-C=C) have been changed. These changes indicate that the hydroxyl, carbonyl and aromatic groups are the responsible for the DCF removal. Also that could generate a strong interaction between the negatives charges of the groups of the adsorbent with the positives charges, as for example of the amine group, of the anti-inflammatory.

It can be observed two new peaks, the first one at 610 cm^{-1}, which belongs to the aromatic group (-C-Cl) of DCF and another one at 1506 cm^{-1} which confirms the presence of its aromatic ring (10).

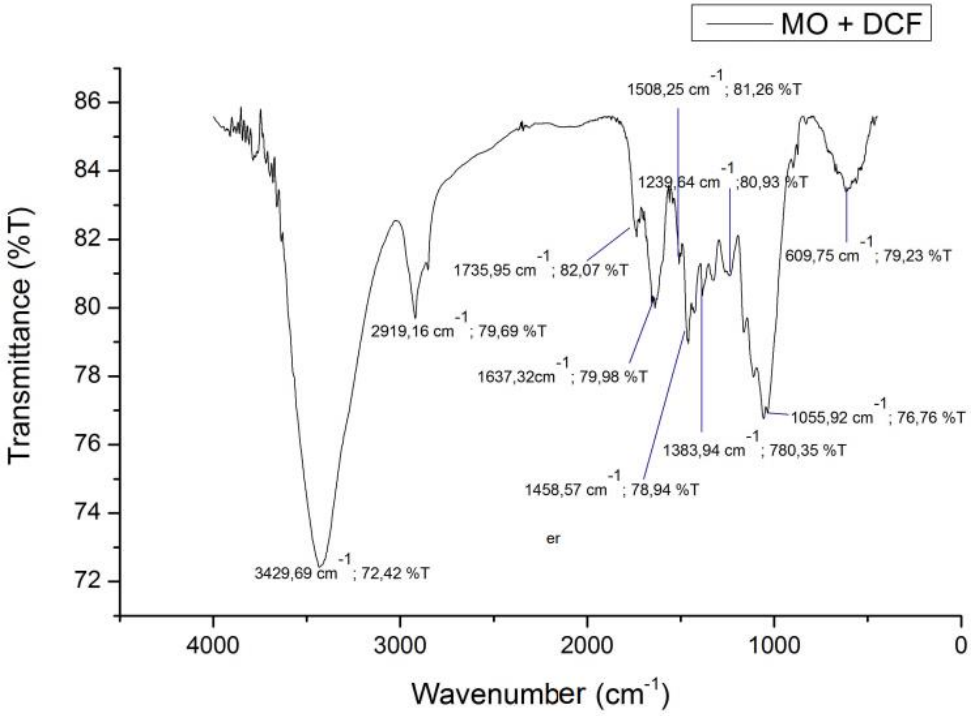

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Figure 2. Moringa *oleífera* shells composition after adsorption process of DCF from water.

The **Figure 3** illustrates the results of the adsorption process of Oxytetracycline solution. Knowing the complex composition of the antibiotic (amides, carbonyl, amines, hydroxyl and aromatic groups), the main differences can be seen at the following peaks: 3435 cm^{-1} (-OH), 1633 cm^{-1} (-C=O), 1465 cm^{-1} (-C=C), 1260 cm^{-1} and 1055 cm^{-1} of carboxyl acids, indicating that the responsible of the adsorption, also in this case, are the hydroxyl, carbonyl and aromatic groups.

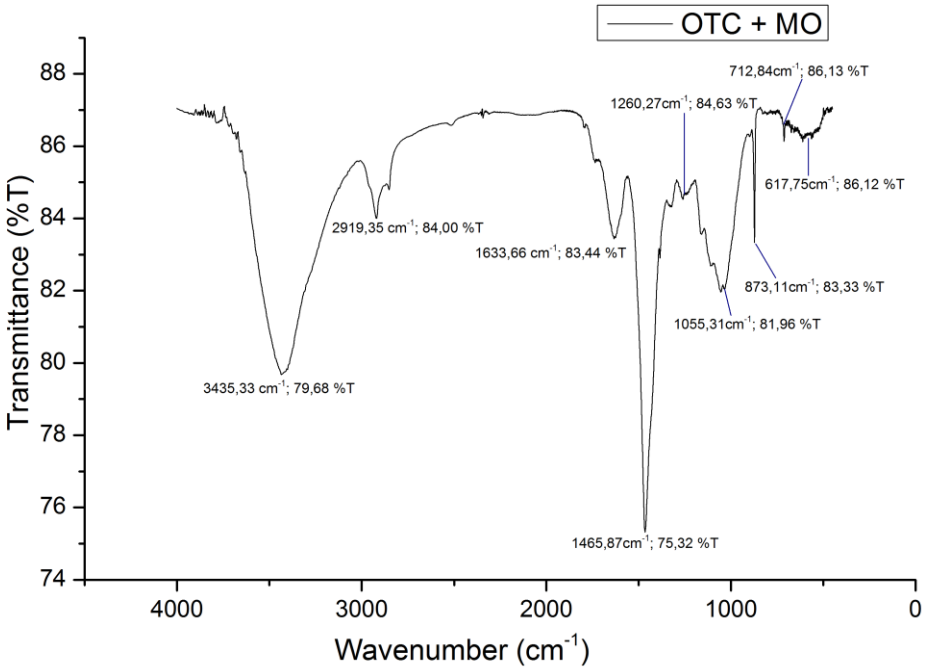

Figure 3. Moringa *oleífera* shells composition after adsorption process of OTC from water.

The lower absorption at 3435 cm^{-1} could be related to hydrogen bonding between the functional groups of the adsorbent and the antibiotic. The peak at 712 cm^{-1}could indicate the complex formation between the OTC and the adsorbent or also the presence of aromatic group of the antibiotic (11).

Conclusion. By FTIR analysis, it is known that Moringa *oleífera* shells present a complex nature and a huge variety of functional groups, such as hydroxyl (-OH), -CH bonds, carbonyl group (-

C=O), -C=C of aromatic compounds, carboxylic acids, amines, amides and phenol compounds. After DCF and OTC adsorption processes. It was possible to determine that, in both cases, the possible responsible are the hydroxyl (-OH), carbonyl (-C=O) and aromatic groups (-C=C).

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Disclosure. The authors report no conflicts of interest in this work.

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