

Analysis of the use of coffee husk biochar columns in the removal of sulfamethoxazole from wastewater in urban areas

Marco Aurelio Calixto Ribeiro de Holanda

Universidade Federal de Pernambuco

Manuella Virgínia Salgueiro Gondim

Universidade Federal de Pernambuco

Antonio Celso Dantas Antonino

Universidade Federal de Pernambuco

Willames de Albuquerque Soares

Universidade de Pernambuco

José Romualdo de Souza Lima

Universidade Federal de Pernambuco

Maurício Costa Goldfarb

mauricio.goldfarb@upe.br

Universidade de Pernambuco Escola Politecnica de Pernambuco <https://orcid.org/0000-0002-0909-1514>

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ABSTRACT

Due to the widespread human and veterinary use of sulfonamides in the treatment of diseases related to the gastrointestinal, urinary, and respiratory tracts, the presence of sulfamethoxazole (SMX) has been frequently detected in soil and water samples. Aiming to reduce the load of this pollutant in the surface waters of the Recife Metropolitan Area (RMR), this study aimed to analyze the performance of biochar derived from coffee husk pyrolysis as a mitigation measure. For this purpose, kinetic, sorption isotherm, and column assays were performed, and the results were compared with values simulated using Hydrus-1D. The results of the batch assays showed that equilibrium was reached in just over 36 hours, and adsorption was 59.7%, occurring in monolayers, as the Langmuir model provided the best fit for the isotherms. However, the experiments performed in columns showed a lower average adsorption value (26.3%) compared to the batch tests. This may be associated with the shorter contact time between the adsorbent and the adsorbate, as well as the arrangement of biochar particles within the column, which prevents contact between all active sites of the biochar and the SMX molecules present in the solution. Thus, due to the high toxicity and high bioavailability of SMX, any measure aiming to reduce the concentration of this contaminant in nature becomes important.

Keywords: Environmental pollution; Sulfamethoxazole; Biochar filter; Coffee husk.

1 INTRODUCTION

For some time, there has been discussion regarding the arrival of a global water crisis, as the supply capacity, which is governed by the hydrological cycle, will not be able to compensate for the mismanagement of available water resources and the large influx of pollutants into these waters (Ray and Shaw, 2016). Furthermore, the migratory increase of the population from rural to urban areas is also a worrying factor, as it is expected that by 2050, 70% of the world's population will be residing in urban centers, demanding even more water (OECD, 2013).

Moreover, water quality has been directly affected by the input of emerging contaminants, such as antibiotics, which, due to their vast applicability, have been widely administered to humans and animals, both pets and livestock (Chu; Hashim, 2023). The alteration of water quality has generated concerns, especially regarding the capacity of contaminants to cause various environmental disorders (Chu; Hashim, 2023). In this

context, sulfonamides, particularly Sulfamethoxazole (SMX), have been widely found in water and soil (Lyu et al., 2020; Rahim et al., 2024).

The entry of antibiotics into the environment occurs in several ways, such as through the mixing that occurs between conduits carrying untreated sewage and rainwater. Additionally, the low availability of basic sanitation in urban centers has facilitated the open runoff of wastewater, graywater, and blackwater and their consequent infiltration into the soil, reaching groundwater (SNIS, 2019; Oliveira; Soares; Holanda, 2021; Bouchaala et al., 2025). According to SNIS (2019), more than half of the population of the Recife Metropolitan Area (RMR) lacks access to basic sanitation, favoring these situations of rainwater contamination. Besides these, another entry point for antibiotics is hospitals, with Nazari et al. (2016) classifying them as one of the main sources of contamination.

Another access route is inefficient sewage treatment plants (WWTPs), which fail to remove all antibiotics, especially SMX (Duan et al., 2022), and discharge these treated waters into water bodies (Juella et al., 2021). These treatment plants remove only part of the SMX through biological degradation, allowing SMX to be found in the environment in concentrations ranging from micro to nanograms per liter and above 1.8 mg kg^{-1} in soil (Wang; Wang, 2018; Rodrigues et al., 2020; Li et al., 2023, Bouchaala et al., 2025).

Water contamination by SMX is linked to a series of environmental and health risks, such as the fact that it alters gene expression, changing RNA metabolic processes (Xu; Xie; Li, 2022), and increases the resistance of cyanobacteria and green algae (Zhang et al., 2023). Zhou et al. (2024) also showed that SMX can cause damage to the growth of fish (*Danio rerio*), as the animals presented liver problems upon contact with water contaminated by this antibiotic. In humans, it has been found that SMX acts by causing severe skin reactions (Wu et al., 2024) and, in some cases, when combined with Trimethoprim, can cause patient death due to respiratory system failure (Miller et al., 2023).

To minimize the risks associated with the introduction of SMX into the environment, several studies have been conducted proposing mitigation measures, such as the use of filter layers (Reguynal; Sarmah; Gao, 2017; Shen et al., 2018; Juella et al., 2022). Among the most common materials used as filter layers are biochar, manure, cellulose, microplastics, and others, which typically present high availability and, for some, low cost (Lun et al., 2023; Jin et al., 2023; Yu et al., 2023; Yuan et al., 2024).

Among these materials, biochar has been the most utilized due to its low cost. Authors such as Zamiri and Niu (2024), when studying the sorption of SMX by a biochar produced from reed canary grass (*Phalaris arundinacea*), showed that the adsorption capacity was high, reaching 147.9 mg g⁻¹. Ma et al. (2024) also showed that biochar was capable of adsorbing up to 89.2% of the SMX present in waters originating from surface runoff. Thus, the objective of this study was to analyze the performance of biochar produced from coffee husks in the adsorption of sulfamethoxazole present in the surface waters of the Recife Metropolitan Area.

2 MATERIALS AND METHODS

2.1 Study area

The capital of the state of Pernambuco, Recife, has nearly 2 million inhabitants and a climate similar to that of all the cities comprising its metropolitan region. Located in northeastern Brazil, it has a hot and humid tropical climate, with rainfall predominating between April and July (Wanderley et al., 2018). Annual precipitation exceeds 2000 mm, with the highest inputs occurring in June (Holanda; Soares, 2019).

The Recife Metropolitan Area (RMR) is composed of 15 cities (Abreu e Lima, Araçoiaba, Cabo de Santo Agostinho, Camaragibe, Goiana, Igarassu, Ilha de Itamaracá, Ipojuca, Itapissuma, Jaboatão dos Guararapes, Moreno, Olinda, Paulista, Recife, and São Lourenço da Mata), which are home to approximately 4 million inhabitants. A large part of this population still lacks access to basic sanitation (PDUI, 2018; SNIS, 2019). Therefore, the mixing of surface runoff with contaminated wastewater occurs frequently (Oliveira; Soares; Holanda, 2021).

2.2 Chemicals

The chosen contaminant is an antibiotic used for humans and animals, Sulfamethoxazole (SMX), which has been frequently found in surface runoff, groundwater, and wastewater in urban centers and aquatic environments around the world (Guwy, 2008; Huang et al., 2022). SMX is used to treat infections caused by microorganisms, such as: bronchitis, sinusitis, otitis, cystitis, urethritis, chancroid, prostatitis, genital infections, typhoid fever, cholera, diarrhea caused by enterotoxigenic *Escherichia coli*, boils, abscesses, brucellosis, among others. Its use must be under medical prescription, as it is contraindicated for patients with renal insufficiency, people

with sensitivity to sulfonamides, and for premature infants and newborns up to one and half months old (Sulfametoxazol, 2019). Furthermore, it can also cause skin irritation, serious eye irritation, and respiratory difficulties, and it is very toxic to aquatic organisms, potentially causing reproductive disorders and even cancer (Bacanli; Basaran, 2019).

The SMX molecule has a weight of $253.28 \text{ g mol}^{-1}$, and its molecular formula is $\text{C}_{10}\text{H}_{11}\text{N}_3\text{O}_3\text{S}$. Like SDZ, SMX has the appearance of a white powder but with a melting point between 166 and 169°C or between 439 and 442 K (Kolpin et al., 2002), and has a pKa_1 of 1.6 and a pKa_2 of 5.7 (Santos et al., 2011). The SMX used was acquired from Sigma-Aldrich® (São Paulo, Brazil) (CAS no.: 723-46-6, EC no.: 211-963-3).

To study water movement in the porous medium, a tracer was used, bromide (Br^-), which was obtained in salt form (KBr) and acquired from Vetec Química Fina Ltda. (Rio de Janeiro, Brazil). The SMX stock solution was prepared from 2.532 g of the solid compound, with 99.80% purity, which was diluted in 100 mL of methanol. This resulted in a final solution with a concentration of 25.32 g L^{-1} . From this solution, an aliquot was taken and diluted in distilled water to obtain the 10^{-4} M solution. For Br^- , the solution was made by diluting KBr in distilled water at a concentration of 3.47 g L^{-1} .

2.3 Biochar

The biochar used was made from coffee husks originating from an agro-industry in the coffee-growing hub of the Pernambuco Agreste region. Being a residue of coffee processing, the feedstock gives the biochar a low production cost, in addition to providing a sustainable destination for this residue. For this purpose, the coffee husks were pyrolyzed at 530°C for 12 h in the presence of low amounts of oxygen. The pyrolytic material produced had its physicochemical characteristics analyzed according to Lima et al. (2018).

To prevent intermittent problems from occurring in the columns containing quartz sand and biochar during saturation (Topic 2.6 - Solute transport)—such as the inability of the support layer contained in these columns to completely maintain the physical structure of the filter layer in some repetitions—it was necessary to perform a procedure to standardize the assembly and saturation of the columns.

Thus, the biochar underwent washing using the same solution used in column saturation (CaCl_2 and distilled water at a concentration of 1 g L^{-1}). For this, columns containing only biochar were set up (Figure 1) and connected to peristaltic pumps, where the solution was injected at the lowest flow rate allowed by the pump (0.1 mL min^{-1}). The

flow was maintained for approximately 3 days. After this period, the washed biochar was dried in an oven at 100 ± 5 °C for 24 h.

Figure 1. Column containing only biochar for the passage of the saline solution for washing.



2.4 Biochar characterization

Before and after washing, the biochar was also characterized regarding its surface functional groups using Fourier Transform Infrared Spectroscopy (FTIR), model VERTEX 70 (Bruker), performing 16 scans with a resolution of 4 cm^{-1} in the range of $4000\text{--}400\text{ cm}^{-1}$ and atmospheric compensation.

2.5 Batch assays

2.5.1 Sorption kinetics

Sorption kinetics were performed following the methodology described by Martins and Mermoud (1998), using a 1:10 ratio (washed biochar:contaminant solution) in darkened flasks. These small reactors were agitated on an orbital shaker at 200 rpm for the following times: 0, 30 min, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 8 h, 10 h, 12 h, 24 h, 48 h, 60 h, and 72 h. At each of these time points, a 1.5 mL aliquot was withdrawn, filtered (0.22

µm PVDF filter), and analyzed using High-Performance Liquid Chromatography (HPLC).

2.5.2 Sorption Isotherm

For the sorption isotherm, the same ratio of washed biochar and SMX used in the kinetics (1:10) was employed. However, the SMX concentration varied from one reactor to another to encompass concentrations higher and lower than 10^{-4} M (2.5×10^{-4} , 10^{-4} , 7.5×10^{-5} , 2.5×10^{-5} , 10^{-5} , 5×10^{-6} , 10^{-6} M). These samples were agitated on an orbital shaker for 48 h at 200 rpm. The preparation and analysis of the samples followed exactly the same process used in the kinetics. The assays were performed in triplicate, and the means of the obtained results were used.

2.5.3 Sorption Data Analysis

To analyze the results observed from the sorption kinetics, two models were used: Pseudo-First Order and Pseudo-Second Order (PFOM and PSOM, respectively), according to Equations 1 and 2:

$$Q_t = Q_e(1 - e^{-k_1 t}) \quad (1)$$

$$Q_t = \frac{k_2^2 Q_e^2 t}{1 + k_2 Q_e^2 t} \quad (2)$$

where k_1 and k_2 represent the sorption rate constants of the PFOM and PSOM [h^{-1}], respectively; Q_e is the sorption capacity at equilibrium [mg kg^{-1}]; Q_t is the sorption capacity at any time t [mg kg^{-1}]; and t is the time [h] (Ho, 2006).

The modeling of the sorption isotherm was performed using three distinct models: Linear, Freundlich, and Langmuir, which are represented by Equations 3–5, respectively:

$$Q_e = K_d C_e \quad (3)$$

$$Q_e = K_f C_e^{1/n} \quad (4)$$

$$Q_e = \frac{q_m K_l C_e}{1 + K_l C_e} \quad (5)$$

where q_m represents the maximum sorption capacity [mg kg^{-1}]; n is an empirical value; and K_d , K_f , and K_l are the constants or coefficients of the Linear, Freundlich, and Langmuir models, respectively.

For the determination of SMX concentrations, Equation 6 was used:

$$S = (C_0 - C_e) F d \quad (6)$$

where C_o and C_e represent the initial and equilibrium concentrations of SMX, respectively; S represents the concentration of contaminant that was sorbed; and Fd is the dilution factor, which is obtained through the expression: $Fd = 10$ (representing the solution/soil ratio).

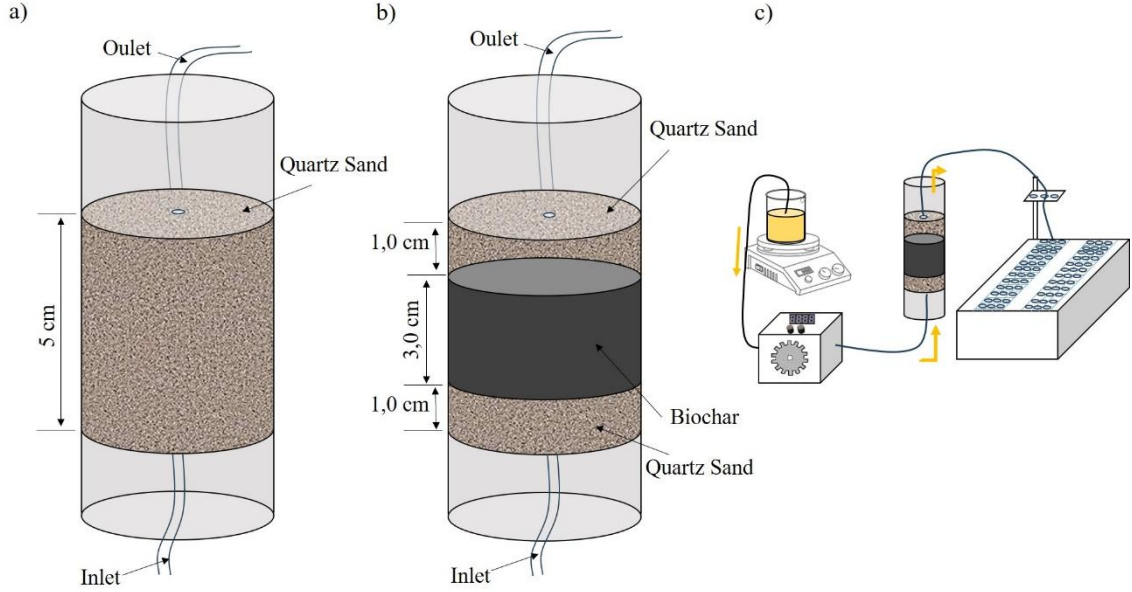
2.6 Solute transport

The experimental setup used for SMX transport consisted of glass chromatographic columns 50 mm in length and 26 mm in internal diameter (Cytiva® – XK 26/20), a peristaltic pump (Ismatec® – 78001-22), a fraction collector (Spectra/Chrom® – CF-2), and digital balances (Shimadzu® – BL3200H) to determine the mass of displaced solution applied to the soil columns.

For column assembly, small amounts of biochar and quartz sand were added and compacted until cohesive layers of five centimeters were obtained, containing either only sand (Figure 2A) or biochar and quartz sand (Figure 2B), divided into three portions: two of sand and one of biochar. The sand layers were used as an envelope for the biochar.

The quartz sand used was sieved to separate the fraction containing particles of 0.5 mm in diameter. This fraction was chosen to provide a more homogeneous flow of the solution injected into the columns, whether containing the contaminant (SMX) or the tracer (Br^-), and to prevent the biochar from being transported out of the column in the presence of a solution pulse, which would lead to a change in the structural composition of the column.

Figure 2. Column assembly scheme: a) quartz sand and b) quartz sand and ground and washed biochar; and c) scheme of the experimental apparatus.



The columns were saturated with a very low flow (0.1 mL min^{-1}), in upflow mode, with a solution of water and CaCl_2 at a concentration of 1 g L^{-1} (Saturation solution). The pH of the produced saturation solution was 7.3, which is similar to the average pH of rainwater in the city center of Recife and of the water arriving at sewage treatment plants (Oliveira et al., 2021). For the KBr and SMX injections, a flow rate of 0.3 mL min^{-1} was used, which represents a rainfall intensity of 36 mm h^{-1} . Rains of this intensity are considered very strong for the RMR and usually cause various problems (Wanderley et al., 2018). For the amount of injected KBr and SMX solution, 1 pore volume was used.

The column transport assays were performed in triplicate, and the means of the obtained results were used. Samples from the column effluents were collected at regular time intervals and then analyzed by HPLC.

The modeling of the tracer and contaminant elution curves was performed using CXTFIT within the STANMOD software (Toride et al., 1999; van Genuchten). In this case, three distinct models were used: the Convection-Dispersion Equation (CDE), the Two-Region Non-Equilibrium CDE (Mobile-Immobile Water Model - CDE-MIM), and the Two-Site Sorption CDE, which were created to fit the elution curves, Equations 7–9:

$$R \frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial z^2} - \bar{v} \frac{\partial c}{\partial z}, \text{ onde } R = 1 + \frac{\rho_d K_d}{\theta} \quad (7)$$

$$\beta R \frac{\partial c_1}{\partial t} + (1 - \beta) R \frac{\partial c_2}{\partial t} + \mu_1 c_1 = \frac{1}{P} \frac{\partial^2 c}{\partial z^2} - \frac{\partial c_1}{\partial z} \quad (8)$$

$$(1 - \beta) R \frac{\partial c_2}{\partial t} = \omega (c_1 - c_2), \quad \text{were} \quad \omega = \frac{\alpha(1-\beta)RL}{v} \quad (9)$$

where R represents the retardation factor; $\partial c/\partial z$ represents the solute concentration gradient; D is the dispersion coefficient [$\text{cm}^2 \text{h}^{-1}$]; v is the average apparent velocity [cm h^{-1}]; ρ is the bulk density [kg cm^{-3}]; Kd represents the soil-solution partition coefficient [$\text{kg}^{-1} \text{cm}^3$]; c is the solute concentration; c_1 and c_2 are the concentrations at equilibrium and non-equilibrium sites; β represents the fraction of sorption sites that have instantaneous equilibrium with the soil solution, and $(1-\beta)$ represents the fractions where equilibrium occurs only over time (kinetic); α is the first-order kinetic rate for non-equilibrium sites [h^{-1}]; L represents the column length [cm]; P is the Peclet number; T is the dimensionless time ($T = vt L^{-1}$); and μ is the degradation constant [h^{-1}].

To analyze the correlation between the values calculated by the models and those observed experimentally, the Root Mean Square Error (RMSE) was used, Equation 10.

$$EQM = \left(\frac{\sum_{i=1}^N (C_i - E_i)^2}{N} \right) * \frac{100}{\bar{E}} \quad (10)$$

where C_i represents the values calculated using the model; E_i represents the values obtained experimentally; $\bar{E}$ is the mean of the experimental values; and N is the number of results.

2.7 Quantification of SMX and KBr

To determine the quantity of SMX in the aliquots collected from the column effluents, High-Performance Liquid Chromatography (HPLC) was used. In this case, the chromatograph used was the Waters ArchHPLC, coupled to a Photodiode Array Detector (PDA), model 2998, and a Waters C18-XSelect chromatographic column, 150 mm in length by 4.6 mm in diameter with a stationary phase film thickness of 5 μm . The mobile phase used was composed of 20% methanol, 20% acetonitrile, and 60% water, with the pH adjusted to 3 using formic acid. This was injected at a flow rate of 1 mL min^{-1} , and the PDA detector was programmed with a wavelength of 268 nm. In the case of Br^- , a Thermo Scientific Orion Star A214 ion meter was used with the 9635BNWP electrode.

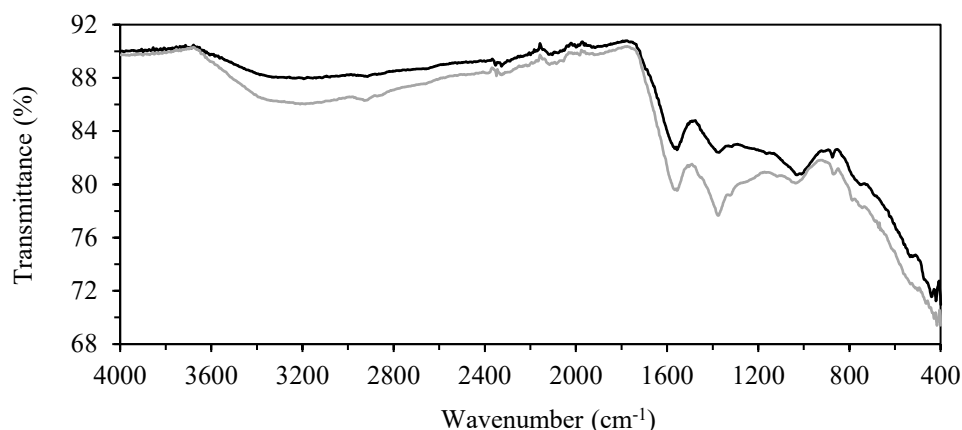
3 RESULTS AND DISCUSSION

3.1 Biochar characteristics

Physicochemical analyses showed that the biochar had an alkaline pH (10.31), the specific surface area was 244 $\text{m}^2 \text{g}^{-1}$, and the percentages of C and N were 67.11 and 2.05%, respectively, while the cation exchange capacity was 22.54.

In the case of FTIR, two samples of coffee husk biochar were analyzed by Fourier Transform Infrared Spectroscopy (FTIR): the ground biochar and the same sample after washing with the saturation solution. Figure 3 presents the FTIR spectrum of the ground biochar and the washed biochar sample.

Figure 3. FTIR spectra of the ground coffee husk biochar samples (gray) and the washed one (black).



Analyzing Figure 3, some relevant peaks are observed in the spectrum of the ground biochar (gray line), which are attributed to the presence of residual structures of caffeine, hemicellulose, cellulose, and lignin after husk pyrolysis (Cruz et al., 2013; Veiga et al., 2017). The 3600–3200 cm^{-1} range is mainly associated with O-H and N-H bonds, suggesting the presence of remaining moisture with hydrogen bonds or hydroxyl groups (Cruz et al., 2013; Veiga et al., 2017).

The variation in the 2400–1900 cm^{-1} range is generally attributed to the presence of triple bonds, suggesting the presence of $\text{C}\equiv\text{N}$ and $\text{C}\equiv\text{C}$. The absence of peaks in the 1750–1650 cm^{-1} range suggests that the ground biochar has a small amount of carbonyl and -CN groups (Giorcelli; Bartoli, 2019; Luperón et al., 2020). The peak observed at 1370 cm^{-1} probably comes from the aliphatic stretching of C-O in compounds derived from hemicellulose, and the small band centered around 1030 cm^{-1} is likely associated with C-OH bending.

The comparison of the two spectra, gray and black, suggests that the washing of the ground coffee husk biochar samples has only few effects on the characteristics of the FTIR spectrum, with the most important change being the general increase in transmittance, which oscillated between 2 and 4%. Furthermore, the broad bands become

weaker with washing, which suggests a reduction in the quantity of functional groups after the washing process.

It is also possible to observe that the peak at 2926 cm^{-1} in the ground biochar sample (referring to C-H type bonds) almost disappeared from the spectrum of the washed biochar sample, as well as the peaks at 787 , 688 , and 671 cm^{-1} , which are generally associated with aromatic arrangements (Giorcelli; Bartoli, 2019; Luperón et al., 2020). These findings allow the authors to assume that the washing and drying process of the coffee husk biochar promotes a thermal cracking and hydrogenation process, resulting in a less aromatic and more carbonaceous biochar.

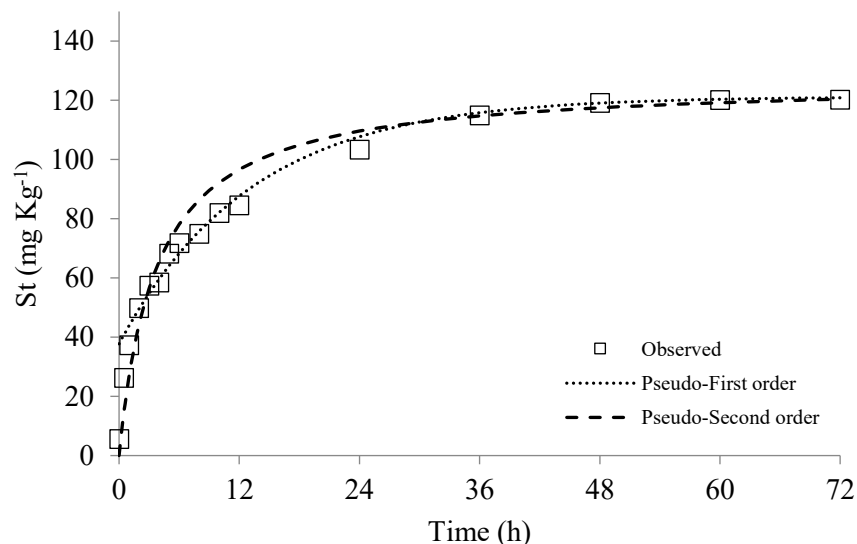
3.2 Batch assays

The sorption kinetics assays showed that in the first hours of contact between the biochar and the solution containing SMX (10^{-4} M), sorption increased rapidly. From 0 to 30 min, sorption went from 5.66 to 26.24 mg kg^{-1} , and by 4 h of the assay, this value reached 58.52 mg kg^{-1} , as observed by Huang et al. (2022) when analyzing SMX sorption kinetics in reactors containing biochar. However, sorption slowed down, so that equilibrium was only reached after just over 36 h of the assay had elapsed.

This rapid increase in sorption occurring in the first hours of the assay is a reflection of the large amount of active sites available on the biochar for π - π and hydrophobic bonds (Ren et al., 2018). After reaching equilibrium, SMX sorption reached a maximum value of 120.2 mg kg^{-1} , representing a retention of 59.7%. This corroborates the results obtained by Cheng et al. (2023), who, studying SMX sorption by phosphoric acid-activated biochar in different proportions, showed that depending on the amount of acid used and the biochar production temperature, about 42 to 96% of the SMX was adsorbed.

Huang et al. (2020), studying SMX sorption by different types of biochar, showed that depending on the type of feedstock and pyrolysis temperature, SMX sorption efficiency can be lower than that obtained in this work (59.7%), but it can also be higher, reaching 83.3% for ground biochar produced from hickory chips at $450\text{ }^{\circ}\text{C}$.

Figure 4. Observed and modeled values with PFOM and PSOM for SMX sorption kinetics (10^{-4} M).



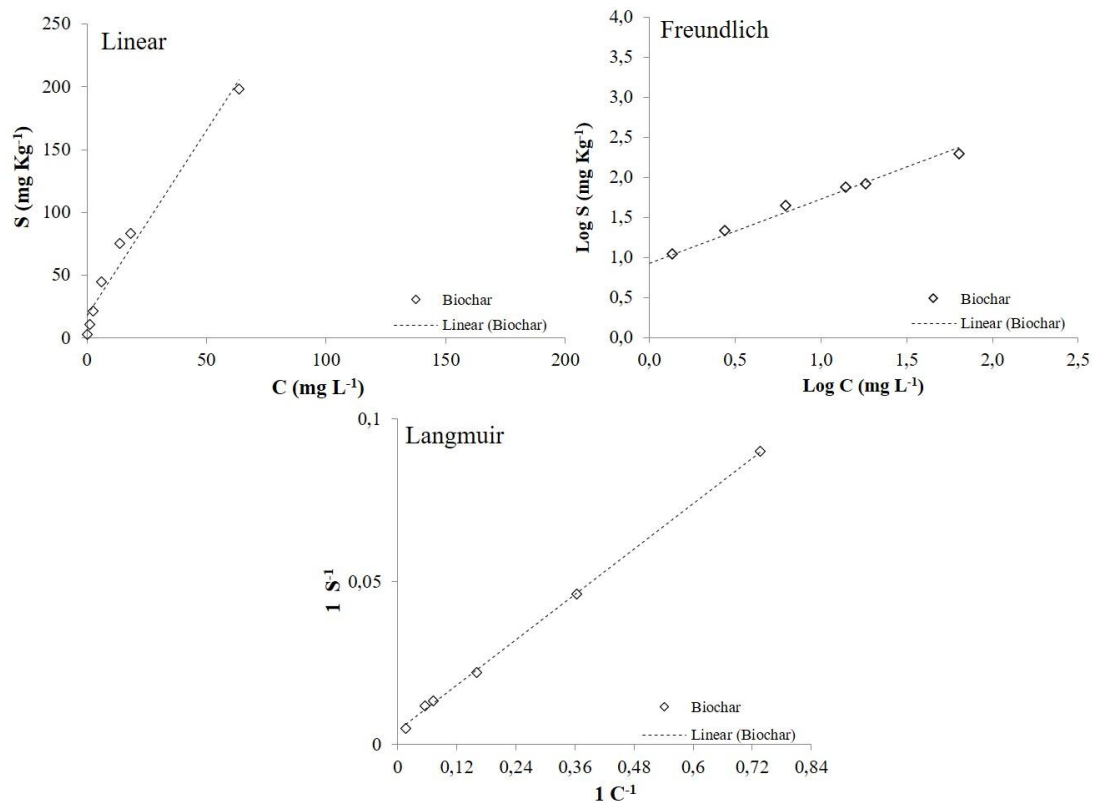
The modeling of the observed data showed that the PSOM presented a better fit to the experimentally observed data compared to the PFOM, since the coefficient of determination (R^2) of the PSOM was 0.996, while that of the PFOM was 0.982. Analyzing R^2 values alone, one could say that both models would be capable of providing good fits; however, observing the curves in Figure 4, it is noted that for the beginning of sorption kinetics, the Pseudo-First Order model does not fit as well, as it estimates sorption for $t = 0$ h with a value of $37.758 \text{ mg kg}^{-1}$. This value ends up being very different from that observed during the experimentation stage, where $S_0 = 5.656 \text{ mg kg}^{-1}$, meaning the PFOM overestimates sorption values for the first two hours of the experiment. The PSOM, on the other hand, presented an S_0 of 3.312 mg kg^{-1} .

As in this study, where SMX concentrations are low (10^{-4} M), Reguyal, Sarmah, and Gao (2017) showed that the PSOM presented better fits than the PFOM when studying SMX sorption by a FeCl_2 -impregnated biochar. Furthermore, the low values of the rate constants ($k_1 = 0.0760$ and $k_2 = 0.0021 \text{ h}^{-1}$) point to rapid adsorption of SMX on active sites, hindering adsorption as the experiment proceeds (Reguyal; Sarmah; Gao, 2017). It is worth noting that adsorption percentages were calculated using the mass balance.

Huang et al. (2020) showed that for a ground biochar, SMX sorption occurred in just 12 h, but with only 8 h of assay, 75% of the contaminant was adsorbed. However, in contrast to what was observed in this work, these authors showed that neither the PFOM nor the PSOM were capable of providing good fits, whereas the model proposed by Elovich simulated the observed data well, with an R^2 greater than 0.96.

For the sorption isotherm (Figure 5), experiments were conducted for 48 h, since the kinetics showed that equilibrium was reached at 36 h. In this case, comparing the results of the three adjustment models used (Linear, Freundlich, and Langmuir), it was observed that the Langmuir model was better, with an R^2 of 0.999. However, the Freundlich model also presented good fits, with an R^2 of 0.988. These results are very similar to those obtained by Huang et al. (2020), as the Langmuir model also showed a slight superiority compared to Freundlich ($R^2_L = 0.981$ and $R^2_F = 0.977$) for SMX.

Figure 5. SMX sorption isotherms on ground and washed biochar.



However, the results obtained with the three adjustment models in this work contrast with those obtained by Ngigi, Ok, and Thiele-Bruhn (2019), who showed that for SMX isotherms, the best fit was obtained with the Freundlich model. Studying the sorption of seven antibiotics, including SMX, by three biochars produced with different feedstocks (biosolids, cattle manure, and spent coffee grounds), Stylianou et al. (2021) showed that for the biochar produced from biosolids, the Langmuir model presented the best fits, whereas for the other two, the Freundlich model provided the best fits for SMX sorption isotherms.

The values of K_d , K_f , and K_l (2.951, 8.45, and 0.037, respectively) are also in agreement with those obtained by Huang et al. (2020) and Tang et al. (2021), where the Freundlich and Langmuir models presented very similar fits. However, in both cases, these authors obtained values one order of magnitude higher than those obtained in this study.

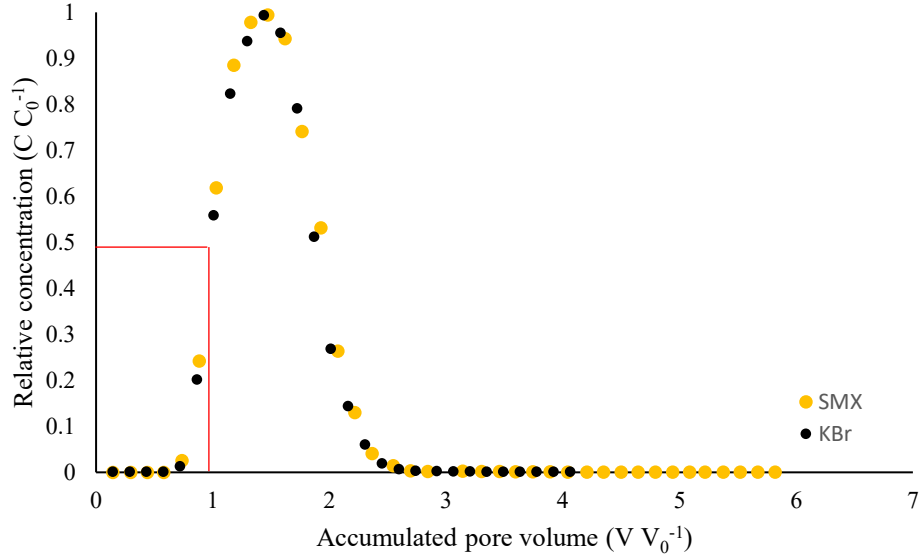
Thus, due to the fact that the Langmuir model proved to be the best among the three, it can be said that monolayer chemisorption of SMX occurred, as observed by Alsaedi et al. (2020). However, since the Freundlich model also presented a good result, it is suggested that this adsorption may also have occurred in multilayers.

3.3 Column assays

The results obtained from the batch assays are not sufficient to describe contaminant transport in as much detail as column assays do (Shi et al., 2019). This is due to the fact that column assays better represent the conditions found in the field, since the contact time between the adsorbent and the adsorbate and the structure of the layer's void network (whether soil or another filter material) are more similar. Besides these, another important factor differentiating the two assays is that, in columns, it is possible to control the flow rate at which the solution containing the contaminant is injected.

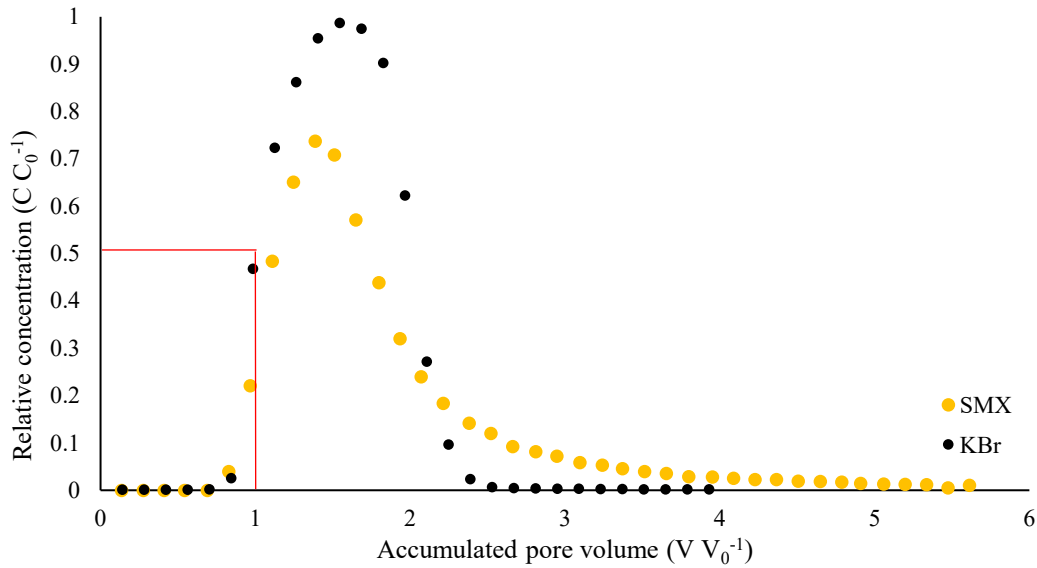
Thus, KBr and SMX injections were performed in saturated columns containing only quartz sand to analyze this material as a possible envelope for the filter layer (biochar). With this, it was found that the KBr elution curve (Figure 6) passes through the point $V/V_o = 1$ and $C/C_o = 0.5$, its relative concentration peak approaches 1, and the retardation factor is also approximately 1. Therefore, this implies that it is an ideal tracer, as it did not undergo interactions with the quartz sand (Biggar; Nielsen, 1967). Regarding the analysis of the contaminant (SMX) elution curve, it is noted that it also passes through the point $V/V_o = 1$ and $C/C_o = 0.5$ and also reaches a relative concentration close to one, proving that the quartz sand does not interfere with sorption or with the SMX transport mechanisms. Therefore, quartz sand can be used as an envelope for the biochar layer, preventing it from being washed out of the column (Figure 2b), unlike what occurs with a column containing natural soil (Shi et al., 2019).

Figure 6. Elution curves of KBr and SMX in columns containing only quartz sand.



For solute transport in columns containing quartz sand and ground washed biochar (Figure 2b), the elution curves shown in Figure 7 were obtained. In this case, it is observed that, just as in the column containing only quartz sand, the KBr elution curve passes through the point $V/V_0 = 1$ and $C/C_0 = 0.5$; thus, it also did not undergo any interaction with the ground washed biochar. The SMX elution curve, however, presented asymmetry with a long tail, indicating that there was no chemical equilibrium in its transport within the column (Alves et al., 2022). It is also noted that for KBr, slightly more than 2.5 pore volumes were sufficient to leach the entire injected concentration, while for SMX, slightly more than 5.5 pore volumes were necessary.

Figure 7. Elution curve of observed values of KBr and SMX for columns containing quartz sand and ground washed biochar.



Furthermore, it is also evident that the efficiency of the ground washed biochar within the column (26.3%) in adsorbing SMX was much lower than that obtained in the batch assays (59.7%). This difference occurs mainly due to the differences in conditions under which batch and column transport assays are performed (Zheng et al., 2019). That is, in addition to a longer contact time between the adsorbent and the adsorbate, most of the biochar pores are in contact with the SMX in batch assays, favoring sorption. Sarker et al. (2023), studying Sulfamethoxazole adsorption mechanisms, showed that contact time is a preponderant factor. Huang et al. (2019) also showed that biochar efficiency in removing SMX was higher in batch assays than in column assays due to these aforementioned differences.

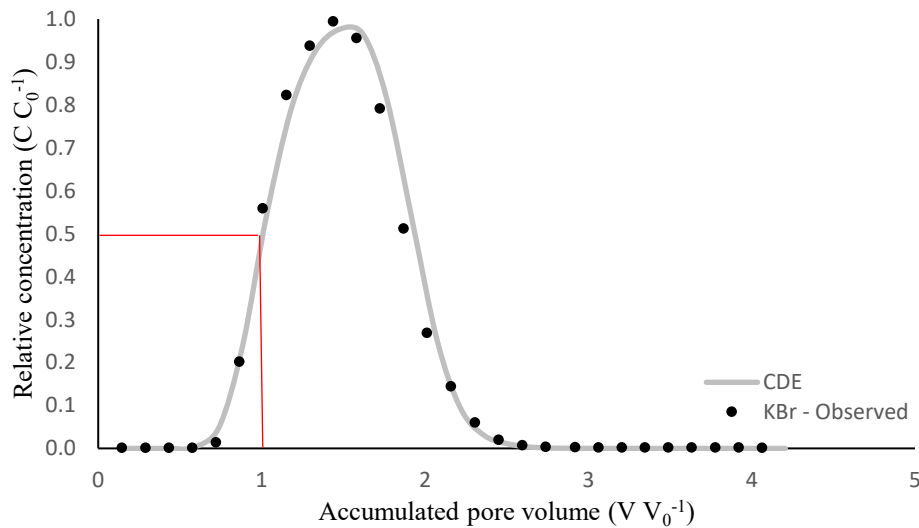
After the tracer passage, it was observed that the elution curve passes through the point $V/V_0 = 1$ and $C/C_0 = 0.5$, the retardation factor is practically unitary, and the relative concentration at the peak is practically 1, thus indicating that sorption was practically nonexistent, thereby demonstrating that there was no interaction between the biochar and the KBr, as observed in the batch assay. This behavior differs from what was observed by Barros et al. (2023) when studying KBr transport in columns containing an alluvial soil. Dousset et al. (2007) also noted a different behavior, since the KBr elution curve obtained by them for columns of more clayey soil also presented a shift to the right of the point $V/V_0 = 1$ and $C/C_0 = 0.5$. The results of Carmo et al. (2013), on the other hand, corroborate the findings of this work. Furthermore, Srinivasan and Sarmah (2014) stated that the elution curve of an ideal tracer, like KBr, should start between 0.85 and 1.05 pore

volumes, as happened in this study during the passage of this solute in columns containing only quartz sand and sand plus ground washed biochar.

Simulations of the observed KBr values for saturated columns containing quartz sand and biochar were performed using the CDE model, utilizing CXTFIT (van Genuchten et al., 2012), and the mean elution curve is shown in Figure 8. This model presented a good fit for the tracer elution curve, representing the entire curve well, from the beginning to the end of the long tail, such that the coefficient of determination was greater than 0.997 and the retardation factor (R) was practically equal to one.

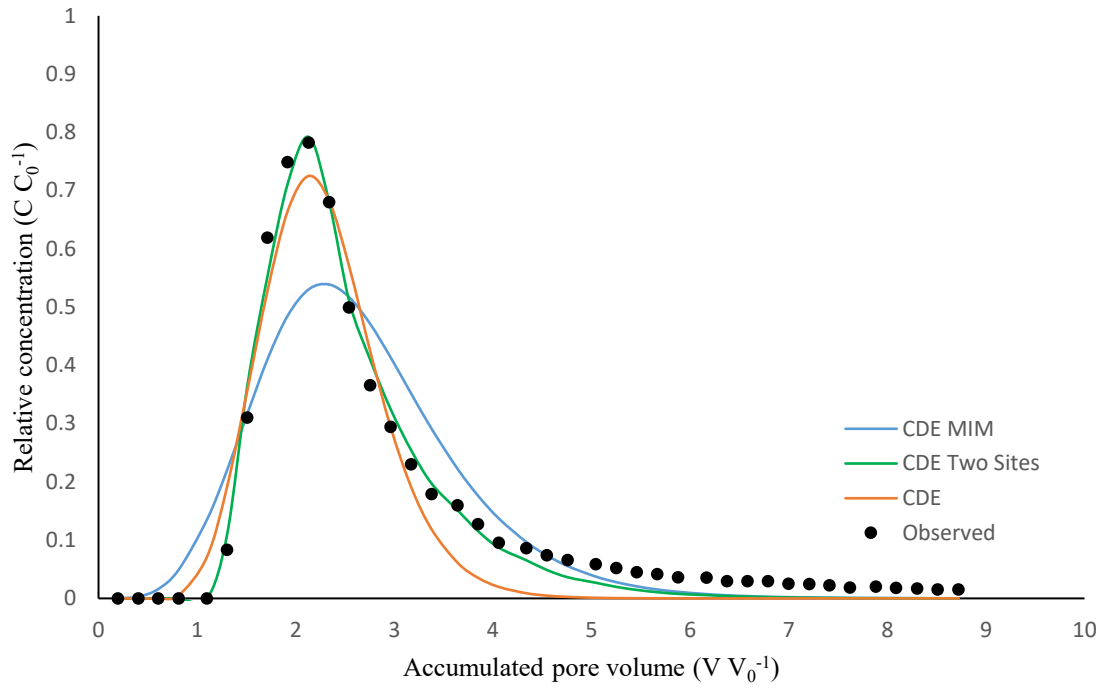
These results corroborate what was observed by Alves et al. (2022), who, studying KBr transport in saturated columns containing alluvial soil from the Capibaribe River, which runs through Recife-PE, noted that KBr transport in columns can be well simulated using the CDE model. Barros et al. (2023) also showed that the CDE model provided good fits to the KBr elution curve when studying the transport of this tracer in columns containing soil.

Figure 8. Elution curve of observed and fitted values of KBr for columns of quartz sand.



When evaluating the simulations of the SMX elution curve using the three models (CDE, CDE-MIM, and CDE Two Sites), presented in Figure 9, it is evident that the chemical non-equilibrium model (CDE - Two Sites) presented the best fits. The mobile-immobile water model (CDE - MIM) presented the worst results, as the model failed to fit the simulated values well for either the beginning or the end of the assay.

Figure 9. Elution curves of SMX injection in columns containing quartz sand and ground washed biochar (observed and simulated).



Despite presenting similar R^2 values (0.9423 and 0.9375 for CDE and CDE-MIM, respectively), Table 1, the CDE and CDE-MIM models present completely distinct results. The curves generated from these models show very distinct behaviors from each other, with the CDE model presenting simulated values more coherent with what was observed experimentally; that is, it presented a slightly better behavior. However, it is still noted that the CDE model fails to describe the final portion of the elution curve well. Unlike these two, the CDE-Two Sites model presented a higher coefficient of determination ($R^2=0.9806$), consistent with the way the simulation replicates the observed data. Such results corroborate what was observed by Zhao, Wu, and Yu (2022) when they showed that the CDE-Two Sites model fit the observed values best, with an R^2 of 0.986.

Table 1. Parameters fitted by the CDE, CDE - Two Sites, and CDE - MIM models for the fitting models of the elution curves of SMX injection in columns containing quartz sand and ground washed biochar.

Adjustement Model	R	D [cm ² h ⁻¹]	β	ω	R ²	P _e	MSE
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CDE	1,83	0,513	-	-	0,9423	6,029	0,002932
CDE – Two Sites	4,83	0,100	0,674	0,294	0,9806	30,927	0,001070
CDE - MIM	1,53	0,089	0,889	0,5479	0,9375	34,832	0,008740

Values of the parameter $\beta = 0.674$ for the CDE-Two Sites model indicate that 67.4% of the site fractions participate instantly in SMX adsorption, while 11.1% of these fractions remain immobile within the column (CDE-MIM). Such low values of immobile water fractions indicate why this model does not present a good fit, and why the CDE model fits better than it (Zhao; Wu; Yu, 2022).

According to Afshari et al. (2018), Pe values between 10 and 100 indicate that advection contributes directly to dispersion, along with molecular diffusion. Thus, since the Peclet number values for the CDE-Two Sites and CDE-MIM models were 30.927 and 34.832, respectively, it can be said that these advective and dispersive processes predominate. Values of ω lower than one also indicate a condition of chemical non-equilibrium (Braskaran et al., 1996), reaffirming the fact that the CDE-Two Sites model presented the best results. The Mean Squared Error (MSE) values also show that the CDE-Two Sites was the best among the three models used, since the closer the MSE is to zero, the better.

The values of the retardation factor, R , vary greatly with the nature of the filter layer. Analyzing the same contaminant as in this study, SMX, Srinivasan and Sarmah (2014) obtained, with the CDE model, R values ranging from 1.06 to 7.53 for columns containing two different soils as filter layers. However, unlike what was obtained here in this study, these authors stated that the CDE-Two Sites model did not present good results in fitting the elution curves, for despite fitting the peak values of the elution curves well, the rest was not well modeled. In any case, the R values obtained in this work are not far from those obtained by Srinivasan and Sarmah (2014).

Qin et al. (2016) also obtained good results with the CDE-Two Sites model for fitting levofloxacin elution curves in columns, such that many of the parameters obtained by them are in agreement with what was observed here in this study. Thus, these results show that the chemical non-equilibrium model (CDE-Two Sites) has presented the best fits for elution curves from column assays.

4 CONCLUSIONS

The batch experiments showed that the adsorption of this solute by low-cost filter layers, such as biochar, can be a solution for reducing water contamination in urban centers. The adjustment performed with the pseudo-second order model presented the best results, describing well both the beginning and the end of the sorption kinetics, indicating that chemisorption is a dominant process and that it occurs in a monolayer, since the Langmuir model was the one that best fitted the observed values ($R^2 = 0.999$). However, the Freundlich model also presented satisfactory results ($R^2 = 0.988$).

Furthermore, the sorption kinetics also showed that although equilibrium only occurred after 36 h, at the beginning of the assay, SMX adsorption by the biochar occurs faster, indicating that a large part of the sorption sites are filled within the first few hours, thus demonstrating the importance of this work in understanding the mobility and bioavailability of SMX in the environment.

It also became evident that the column assays presented lower adsorption values than the batch assays (26.3% and 59.7%, respectively), proving that the characteristic conditions under which each assay is performed directly influence SMX adsorption. Thus, it can be said that the absence of physical structure of the filter layer used in batch assays and the longer contact times between the adsorbent and the adsorbate make the batch assay an optimized test when compared to column tests. However, column assays more closely resemble the conditions found in the field, since a hydraulically connected pore network is maintained, with a well-defined bulk density and a flow similar to that of a rainfall event.

The FTIR analyses also showed that sorption values, both in batch and column assays, could be better if it were not necessary to wash the biochar. However, for the standardization of the experiments, this was a necessary step that culminated in the reduction of the aromaticity of the coffee husk biochar, making it more carbonaceous.

Declarations

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All authors contributed to the conception and planning of the research that resulted in this article.

Finally, all authors also declare that they have no relevant financial or non-financial interests to disclose.

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