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Biochar from orange waste as a filter medium for domestic effluent treatment aimed at agricultural reuse

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ABSTRACT

Brazil is the world's leading orange grower, and this agro-industrial sector produces a sizable amount of by-products. Because biochar has the potential to be used as a filtering medium in wastewater treatment systems, it can be advantageous to produce biochar from orange trash as an efficient way to use these resources. The purpose of this study was to assess how temperature affects the synthesis of biochar and to describe the adsorptive qualities of the material for use in filtration systems for agricultural reuse. The produced material was examined for its chemical composition, crystalline structure, morphological changes brought about by the conversion of biomass into biochar, surface area, and average pore size. The pyrolysis temperatures used to produce biochar ranged from 350 to 650°C for 60 minutes. Based on the advantageous physicochemical characteristics for usage as an adsorbent, 550°C was determined to be the ideal temperature. The orange biochar system outperformed the gravel system in terms of macronutrient removal, according to the filtration results using septic tank effluent. The elimination of magnesium (62.09%) and total phosphorus (31.58%) was noteworthy. These findings indicate a promising and long-term wastewater treatment option by indicating that the treated effluent may be suitable for use in some crops.

Keywords: biochar, horizontal flow, sewage treatment, wastewater.

Biocarvão de resíduos de laranja como meio filtrante para tratamento de efluente doméstico visando reúso agrícola

RESUMO

O Brasil é o maior produtor mundial de laranjas, gerando um volume significativo de subprodutos desse setor agroindustrial. Para utilizar esses recursos de forma eficaz, a produção de biocarvão a partir de resíduos de laranja pode ser valiosa devido ao seu potencial uso como meio filtrante em sistemas de tratamento de águas residuais. Este estudo teve como objetivo avaliar a influência da temperatura na produção de biocarvão e caracterizar suas propriedades adsorventes para sistemas de filtração no reúso agrícola. As temperaturas de pirólise para produzir o biocarvão variaram de 350 a 650°C durante 60 minutos, e o material obtido foi analisado quanto à sua composição química, estrutura cristalina, mudanças morfológicas decorrentes da transformação da biomassa em biocarvão, além da determinação da área superficial e do tamanho médio dos poros. A temperatura ótima foi estabelecida em 550ºC de acordo com as propriedades físico-químicas favoráveis para uso como adsorvente. Os resultados da filtração com efluente de fossa séptica mostram que o sistema de biochar de laranja obteve melhor remoção de macronutrientes do que o sistema de brita. Destacaram-se a remoção de fósforo total (31,58%) e magnésio (62,09%). Esses resultados sugerem que o efluente tratado pode ser viável para uso em determinadas culturas, revelando uma alternativa promissora e sustentável para o tratamento de águas residuais.

Palavras-chave: água residuária, biochar, fluxo horizontal, tratamento de esgoto.

1. INTRODUCTION

Brazilian citrus cultivation plays a vital role in the national economy, with the country being the world’s largest producer of oranges. For the 2023/24 harvest, production is projected at 408 million boxes of 408 kg each (USDA, 2024). The orange juice production process generates waste, which accounts for approximately 50% of the total mass of the fruit, consisting mainly of peels and pulp. When this waste is not repurposed, it is often discarded without treatment, leading to economic losses and posing environmental risks (Sugimoto, 2018).

An alternative to recycling the waste from orange processing is the production of biochar, which can be used to treat household wastewater. Biochar is a solid material obtained through the pyrolysis of plant biomass in an enclosed space (muffle furnace) at temperatures between 300 and 800ºC (IBI, 2018). During pyrolysis, aromatic structures gradually form as the temperature rises, which increases the biochar's surface area. The physical properties of this material depend not only on the raw material and the pyrolysis conditions, but also on the handling before and after pyrolysis (Maia, 2011). This material is characterized by its high carbon content, stability, alkalinity, and porous structure consisting of meso and macropores.

According to the National Sanitation Information System (Brasil, 2021), only 56% of the Brazilian population has access to a sewage network, and only 52.2% of the wastewater produced in the country is adequately treated. Thus, the reality remains that half of the country's wastewater is disposed of irregularly and without proper treatment. Therefore, investing in studies and developing effective and economically viable techniques to treat this wastewater is essential. One promising option for wastewater treatment is using a filtration system that uses biochar from orange waste as a filter medium. Filters are tanks filled with sand and other filter media through which wastewater flows in a downward stream, promoting the removal of pollutants through biological and physical processes (ABNT, 1997).

Filtration works by separating substances based on their distinct physical and chemical properties. In this process, solid particles are removed from a mixture of solids and liquids, and the filter medium is responsible for retaining these particles (Geankoplis, 1993). Porous materials must be used in filtration to achieve satisfactory results, retaining solid particles without complex operation and maintenance (ABNT, 1997). This approach is characterized by its feasibility due to its low implementation costs, ease of operation, and maintenance (Junior *et al.*, 2016). Additionally, the application of filtration systems for wastewater treatment should also be highlighted in the context of agricultural irrigation.

Reusing wastewater for irrigation presents numerous benefits, including the conservation of water resources, prevention of untreated wastewater pollution in water bodies, direct recycling of nutrients and organic matter, and economic advantages through reduced fertilizer use (Libutti *et al.*, 2018). In this context, this study aimed to extract and analyze biochar from orange waste to evaluate its adsorption capacity and feasibility in agricultural reuse processes.

2. MATERIAL AND METHODS

2.1. Biochar Production and Characterization

In the biochar production phase, the orange waste was dried in an oven at 105ºC for 48 h, ground in a macromill, and sieved through a 10-mesh sieve. Four tests were conducted with dried orange waste at temperatures ranging from 350 to 650ºC to identify the optimal temperature for biochar production and optimize the carbonization phase. The material was weighed before and after carbonization for each temperature to determine the process yield. It was stored in the muffle furnace until the set temperature was reached, and then it was held for 60 min.

The material was filtered to determine the carbonization temperature conditions that would result in biochar with the best properties. The color and pH of the filtrate were observed at the four temperatures tested. Once the ideal carbonization conditions were determined, the biochar production process was repeated until the required amount for the study was reached.

After biochar production, the material was characterized by ash content in the muffle furnace and moisture content indirectly via the solids by drying in an oven at 105ºC to a constant mass. The volatile matter content was determined in a muffle furnace at 980ºC for 3 min, and the difference between the sum of the volatiles and the ash content calculated the fixed carbon. The composition of the material was analyzed by X-ray diffraction (XRD). The morphology of the transformed biochar was observed by surface analysis using scanning electron microscopy (SEM), and the pore sizes and surface area were analyzed using the Brunauer-Emmett-Teller (BET) isotherm (Brunauer *et al.*, 1938).

2.2. Construction of the Filtration System

The experiment was conducted using a completely randomized design (CRD) with 5 test units for each treatment, totaling 15 units. The treatments were as follows:

- Treatment 1 (F): filter with gravel 0 and gravel 1;
- Treatment 2 (BF): filter with gravel 0, gravel 1, and a layer of biochar;
- Treatment 3 (BGF): filter with gravel 0, gravel 1, and a layer of biochar interspersed with gravel 0.

PVC pipes measuring 40 cm in height and 15 cm in diameter were used for all treatments, as shown in Figure 1. The filter medium consisted of biochar and gravel. In the system without biochar (F), 20 cm of gravel 1 (with a diameter between 12.5 and 22 mm) was placed on top, and 15 cm of gravel 0 (with a diameter between 5 and 12 mm) on the bottom. In the BF system, the configuration included a 15 cm layer of gravel 1 at the bottom, 8 cm of gravel 0, 6 cm of biochar, and 5 cm of gravel 0. The BGF system was similar to the BF system, but the biochar layer also contained gravel 0.

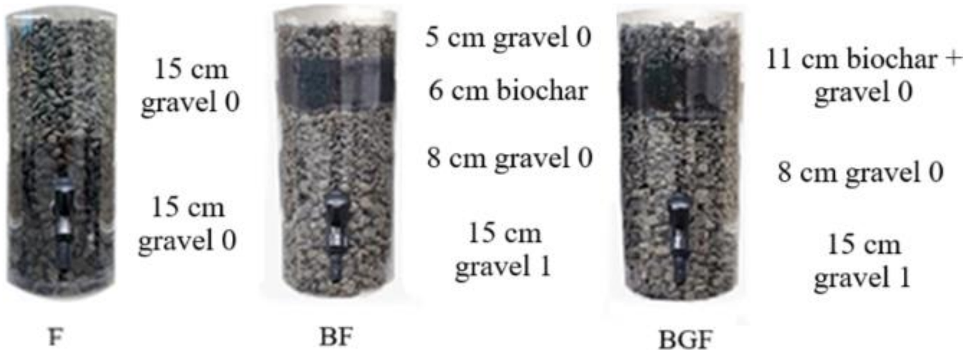

Figure 1. Assembly scheme of the filters in the acrylic tubes used in the development of experiments.

Wastewater was introduced at the top via a drip system, and a tap was installed at the outlet to facilitate sampling.

2.3. Physical, Chemical, and Biological Analyses of the Influent and Effluent

Routine monitoring involved taking samples of raw and treated wastewater after it passed through the filter bed. These samples were subjected to physical, chemical, and biological analyses based on the methodology described in the Standard Methods for the Examination of Water and Wastewater (APHA *et al.*, 2012). Routine sampling was performed on average weekly for one month, including analysis of parameters such as pH, electrical conductivity (EC), turbidity, total organic carbon (TOC), total nitrogen (TN), total phosphorus (TP), potassium (K), calcium (Ca), magnesium (Mg), sodium (Na), and sodium adsorption ratio (SAR).

3. RESULTS AND DISCUSSION

3.1. Characterization of Orange Biochar

To determine the most suitable temperature, the color and yield of the filtrate were observed. It was found that a clear filtrate was obtained at 550 and 650ºC, indicating that the biochar did not release combustion by-products into the filtrate. With a yield of 26.03% and a pH of 8.98, 550ºC was considered the most suitable for producing biochar, as it is more economical in terms of energy consumption than the higher temperature of 650ºC, making the process more sustainable. Carvalho *et al.* (2018) recommend a pH close to neutrality as it provides a balanced environment. The alkaline pH observed in biochar production can be attributed to carbonate formation and the release of alkali salts during pyrolysis (Gonzaga *et al.,* 2018).

The results demonstrated that the material has a low water content (1.53%), which means that the material is less hygroscopic, contributing to increased resistance (Guilhen *et al.*, 2020), Table 1. The ash content indicates the amount of inorganic substances present in the material and varies depending on the composition of the raw material. The presence of minerals affects the properties and can reduce biochar yield (Enders *et al.*, 2012).

Table 1. Average carbon, volatile matter, ash, and moisture in orange biochar samples.

Components	Contents (%)	Standard deviation (%)
Moisture	1.53	± 0.005
Ash	16.75	± 0.060
Volatile material	29.56	± 0.072
Fixed carbon	52.16	± 0.061

With an ash level of 16.75%, the biochar outperformed the orange biomass reported by Devens *et al.* (2018), which had an ash content of 9.26%. This outcome varies depending on the source material's composition; the more nutrients there are, the more ash is present. Certain elements volatilize about 500ºC or so, which could lead to a decrease in the amount of ash.

The pyrolysis process is inextricably tied to the volatile matter and fixed carbon concentrations. Because the concentrations of volatile matter and fixed carbon are inversely correlated, a slower pace of this process leads to a lower concentration of volatile matter and a larger concentration of fixed carbon.

A result of the tests conducted was a volatile matter content value of 29.56%. Since the pyrolysis process releases volatile chemicals with temperature increases, this result can be regarded as predicted for the material. Accordingly, the release decreases with decreasing temperature (Pinheiro *et al.*, 2005).

The charring process contributes to the release of volatile compounds. At lower temperatures, the concentration of volatiles is lower, and the concentration of fixed carbon is higher, which is the remaining mass after the release of volatile compounds, water content, and ash. These contents are related to the calorific value of the material, and the higher the fixed carbon, the longer the material can withstand combustion (Aich *et al.*, 2020).

Figure 2 shows the diffraction patterns of materials. The broad peaks are characteristic of amorphous regions (Stachurski, 2011) that remained in the biochar as Mafra *et al.* (2013), who mention the predominance of amorphous areas over crystalline ones in biochar due to the presence of organic matter. Both analyses show peaks at 2θ = 21° and 45°, which refer to the planes 002 and 100, these peaks indicate that the material has a partially crystalline structure but may still contain a significant amount of amorphous carbon (Gonzalez-Canche, 2021). The crystalline peaks are less intense in biochar, indicating a disordered or random structure in which the atoms do not repeat regularly (Stachurski, 2011).

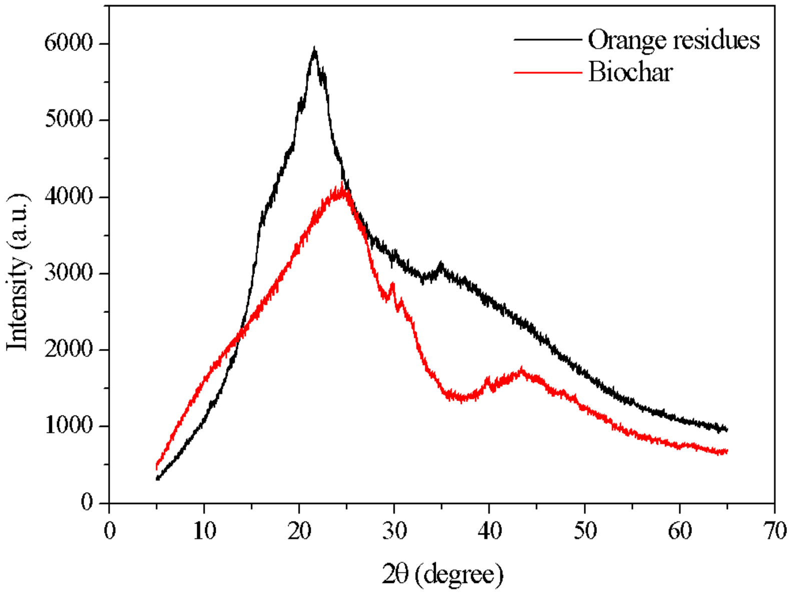

Figure 2. X-ray diffraction patterns of orange waste and biochar materials.

The change in intensity between the peak in orange residues and in biochar occurs due to the increase in temperature during the pyrolysis process. As the temperature increases, the cellulose peaks lose intensity and become broader (Keiluweit *et al.*, 2010). Subjecting the biomass to a high-temperature carbonization process, the resulting biochar shows low crystallinity, indicating the occurrence of graphitization in the samples (Zhang *et al.*, 2022). This graphitization is evidenced by the broad peak observed in the samples at 45°, attributed to the graphitic carbon planes C(100). Thus, the XRD results confirm that the biochar has a highly graphitic structure (Yan *et al.*, 2021).

Figure 3 shows the surface morphology of materials. It was possible to observe the change in morphology of the orange waste and the biochar. The orange residues have a homogeneous and flat surface, while pore development can be observed due to carbonization in the biochar. The greater porosity of the biochar is an essential property of the material, as it supports the adsorption process (Wang *et al.*, 2020).

The pore size and surface area of materials were determined by the isothermal Brunauer-Emmett-Teller (BET) technique, Figure 4. The biochar exhibited a Type III isotherm, typical of non-porous solids, macroporous solids, or materials with mesopores. Besides, the volume adsorbed by the biochar was higher compared to the orange residues. The surface area of

0.251 m² g^{-1} and pores with a diameter of 15 nm classified the biochar as mesopores, higher than orange waste, which has a surface area of 0.109 m² g^{-1} and pores with a diameter of 3.2 nm, Table 2. According to IBI (2018), biochar must have a large surface area and high porosity to be considered a suitable material for use as an adsorbent in wastewater treatment. Accordingly, orange biochar is an ideal material for use as an adsorbent and agrees with the values reported in the literature (Maia *et al.*, 2017; Carvalho *et al.*, 2018).

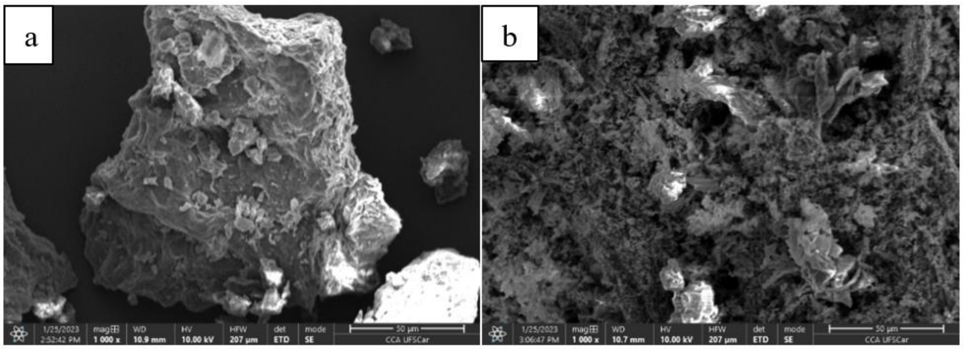

Figure 3. SEM images at 1000X magnification: (a) orange waste; (b) biochar.

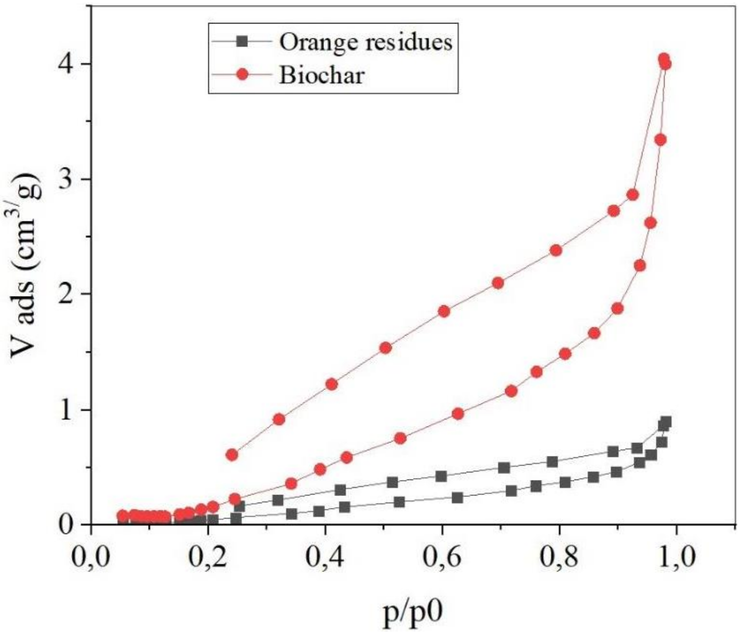

Figure 4. Adsorption-desorption isotherms graph for orange waste and orange biochar at 550°C.

Table 2. Determination of surface area, volume and pore size for orange residues and biochar.

Sample	Surface area (m² g^{-1})	Pore volume (cm^3 g^{-1})	Average Diameter/Average Porosity (nm)
Orange residues	0.109	0.002	3.2
Biochar 550ºC	0.251	0.005	15

3.2. Characterization of the Effluent

Table 3 presents the average characterization of the effluent after the filtration process, comparing the raw effluent (RE) and the effluent treated with a gravel filter (F), a biochar filter (BF), and a combined biochar and gravel filter (BGF). An analysis of this data shows that most parameters have superior system efficiency, indicating a significant reduction of these macronutrients in the three systems evaluated. In particular, the macronutrients P and Mg in the biochar showed a higher reduction. In contrast, the EC and turbidity parameters after treatment showed no significant changes in the results compared to raw sewage, indicating that equilibrium was reached (Souza *et al.*, 2023). However, for certain crops, supplementing nutrients and controlling some parameters may be necessary to ensure adequate irrigation results.

Table 3. Average characterization of the effluent after the filtration process.

Parameters	Treatment			
	RE	**F**	**BF**	**BGF**
pH	7.62	8.92	9.28	9.16
EC (dS m^{-1})	0.910	0.920	0.906	0.846
Turbidity (NTU)	3.40	1.84	1.78	1.85
TOC (mg L^{-1})	12.09	21.01	35.56	22.69
TN (mg L^{-1})	75.32	79.92	69.25	79.84
TP (mg L^{-1})	7.30	2.49	1.67	2.23
K (mg L^{-1})	79.0	72.1	54.05	55.4
Ca (mg L^{-1})	44.0	62	35.45	53.75
Mg (mg L^{-1})	37.0	19.81	10.3	16.25
Na (mg L^{-1})	65.50	52.4	54.16	56.28
SAR	1.75	1.48	2.06	2.07

RE – Raw effluent ; F – Filter with gravel; BF – Biochar filter; BGF – Filter with biochar and gravel.

The pH values showed no significant differences between the treatments. They remained elevated, possibly due to the ability of the gravel to make the water alkaline, which may decrease over time. Another factor that may contribute to the increase in pH in the BF and BGF filters is the inherent alkalinity of the biochar (Silva *et al.*, 2018).

According to Almeida *et al.* (2010), a normal pH range for irrigation in agriculture is between 6.5 and 8.4. Restrictions are required at pH values above 8, as this can lead to clogging of the emitters during local irrigation. Therefore, to use this wastewater for irrigation, the source of the pH increase would need to be investigated, or the pH would need to be adjusted after treatment.

As for the EC analysis, the values for the three filters reached the value of the influent and fluctuated close to 0.91 dS m^{-1}, indicating no gain or loss, which means that equilibrium was reached in terms of the concentration of dissolved ions in the water (Souza *et al.*, 2023). According to Technical Instruction No. 32/2006 of the Companhia de Tecnologia de Saneamento Ambiental do Estado de São Paulo (CETESB), treated wastewater with EC from 0.75 to 2.90 dS m^{-1} can only be used in soils with good drainage capacity (CETESB, 2006).

Regarding the turbidity values, it should be noted that the value remained below 2.0 NTU for all three filters, indicating that the filtration efficiently reduced the turbidity. The ideal values for human consumption recommended by the CETESB (2019) are below 5.0 NTU, with values below 1 NTU indicating a higher efficiency of water treatment. High turbidity is caused by the presence of suspended particles, which serve as a protective shield for pathogenic microorganisms (Brasil, 2006).

The optimal turbidity value for reusing wastewater in agricultural irrigation, according to Braga *et al.* (2014), should be less than 2 NTU. A value over this cutoff point indicates the existence of suspended particles, which can impede the irrigation system and harm plants.

A significant increase in TOC is observed due to the characteristics of amorphous and rich carbon material (Mafra *et al.*, 2013). Although biochar is effective in water purification, it is important to emphasize that it can release small amounts of carbon into the water. To minimize this release, it is necessary to pre-treat the biochar. While the increased TOC value was observed in the filter analysis containing only biochar (BF), the other two filters showed values close to 20 mg L^{-1}. This indicates a greater release of carbon when only orange biochar is used as the filter material. A high total organic carbon (TOC) concentration in irrigation water can serve as a source of plant nutrients and help improve soil stability and structure (Pluske *et al.*, 2024)

There was an initial increase in nitrogen values, but from 21 days onward, these values decreased and approached raw wastewater levels when the gravel system was used and dropped below this level when biochar was used. When treated with biochar, it can adsorb nitrogen due to its high C/N ratio. Under certain conditions, however, this adsorbed nitrogen can be desorbed back into the water, leading to a temporary increase in nitrogen concentration during the analysis (Petter *et al.*, 2012).

Regarding phosphorus, the raw wastewater originally contained 7.3 mg L^{-1}, and after analyzing the treated wastewater, values between 1.78 and 1.85 mg L^{-1} were found, corresponding to a removal efficiency of 77.16% by biochar. This demonstrates that biochar exhibits a good capacity for phosphorus adsorption. Phosphorus is essential for plant energy transfer and storage and also plays a crucial role in root development. Phosphorus deficiency can lead to stunted growth, abnormal coloration, and reduced flower and fruit production (Ferro *et al.*, 2015).

Potassium (K) is an important macronutrient for the growth and development of plants. After filtering the wastewater, it can be observed that the values do not differ between BF and BGF. Compared to F, a lower reduction of potassium can be observed if no biochar was used. The adsorption of potassium by biochar from oranges can occur due to the physical and chemical properties of the biochar as well as the presence of compounds in the orange peels that can interact with potassium. When the K concentration in the wastewater is high, adsorption by the biochar may be more evident due to the surface area of the biochar, which ensures greater adhesion of the nitrifying microorganisms (Luna *et al.*, 2013). Another factor that can influence the adsorption of potassium is the increased pH of the biochar. In alkaline solutions, the acidic functional groups may be less protonated, resulting in a negative surface charge that favors the adsorption of positive ions such as potassium.

Between the analyses at 14 and 21 days, the filtering media showed a calcium removal efficiency of 9.84% for filter F, 25.9% for filter FB, and 43.93% for filter FBB. According to Almeida (2010), the appropriate calcium levels in irrigation water for many plants are between 50 and 200 mg L^{-1}, so the values obtained after filtration are within the range necessary for plant growth, development, and resistance. Calcium deficiency can lead to problems such as reduced resistance to diseases and pests and growth disorders.

The magnesium (Mg) removal data showed that the biochar filter (BF) was efficient and contained about 10 mg L^{-1} during the days of analysis. In contrast, the values for the gravel filter (F) and the combined filter (BGF) were above 10 mg L^{-1}. Despite the differences between the results, a reduction was observed in both cases compared to the raw wastewater. The magnesium limit in irrigation water for sensitive plants should be around 50 mg/L (Mkilima, 2023). However, it is noted that the raw effluent presented a low concentration of Mg; thus, the filters showed a removal efficiency ranging from 46.45% to 72.16%.

High concentrations of sodium (Na) can be harmful to plant development as they cause ion toxicity and impair the uptake of essential nutrients such as potassium and calcium. The filtration of the three treatments showed a decreased Na content with a variation between 14% and 20%. From the EC data, an increase in the EC values of the filters was observed, close to that of the influent, supporting the growth in Na. Consequently, the EC value increases with increasing sodium concentration. (Santana *et al.*, 2007). The increase in Na concentration occurred after the filter became saturated, leading to the accumulation of Na following the filtration process.

From the Na, Mg, and Ca values, the sodium adsorption ratio (SAR) can be derived, an essential parameter for assessing water quality for irrigation (Ayers and Westcot, 1999). If the Na to Ca and Mg concentration is high, soil salinization can occur, significantly impairing plant development and productivity. Although the SAR values were higher than the raw wastewater baseline, all values obtained did not exceed 2.6. Therefore, according to Braga *et al.* (2014), water quality should be safe when using this water for irrigation.

4. CONCLUSIONS

Based on the production and characterization results of orange biochar after the pyrolysis process at different temperatures, the material treated at 550ºC was found to have promising properties for use as an adsorbent in wastewater treatment. The statistical analysis of the results revealed that the biochar filter (FB) is particularly effective in removing macronutrients compared to the results obtained with Only gravel. The high removal efficiency of total phosphorus (31.58%) and magnesium (62.09%), combined with its ability to maintain parameters such as electrical conductivity (EC) and turbidity within acceptable limits, highlights the potential of this material as a sustainable and efficient solution for effluent treatment. This indicates that biochar is an effective alternative for wastewater treatment, especially for agricultural reuse, aiming at the recovery of nutrients present in the effluent.

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