

EXPERIMENTAL ACID-BASE PURIFICATION OF CARBONIZED RICE HUSKS: EXPLORING THE ADSORPTION EFFECTIVENESS OF UNVALUED RICE INDUSTRY RESIDUE

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ABSTRACT

The burning of rice husks produces natural silica and generates charcoal with excellent properties for the adsorption of different pollutants. The objective of this study was to enhance the adsorption properties of this industrial residue from burning rice husk through acid and base purifications. The raw coal (RC) was initially modified with HCl(aq.) and then with NaOH(aq.), and subsequently renamed MC. Efficiency was verified by the adsorption of methylene blue (MB) dye, considering a concentration of 10 mg L⁻¹, dosage of 0.05 g L⁻¹, pH 6.7

and 120 min. MB removal was 78% for RC and 97% for MC, with experimental capacities of 144.78 mg g⁻¹ and 180.78 mg g⁻¹, respectively. The kinetics were better adjusted by Avrami (R² 0.99, error <2.8%). The properties of charcoal improved with the modification, presenting a more porous structure, greater surface area and functional groups that contributed to adsorption, proving that simple purification methods, such as acid-base purification, can be crucial for waste reuse.

KEYWORDS: activated carbon, modification, removal, dye, methylene blue.

PURIFICAÇÃO ÁCIDO-BASE EXPERIMENTAL DE CASCAS DE ARROZ CARBONIZADAS: EXPLORANDO SUA EFICÁCIA NA ADSORÇÃO DE RESÍDUOS NÃO VALORIZADOS DA INDÚSTRIA DE ARROZ

RESUMO

A queima da casca de arroz produz sílica natural e gera um carvão com excelentes propriedades para adsorção de diferentes poluentes. O objetivo deste trabalho foi aprimorar as propriedades de adsorção desse resíduo carbonizado através de purificação ácida e básica. O carvão bruto (RC) foi modificado, primeiramente com HCl(aq.) e em seguida com NaOH(aq.), sendo renomeado MC. A eficiência foi investigada pela adsorção do corante azul de metileno (MB), considerando concentração 10 mg L⁻¹, dosagem 0,05 g L⁻¹, pH 6,7, e 120

min. A remoção de MB foi de 78% para RC e 97% para MC, com capacidades experimentais de 144,78 mg g⁻¹ e 180,78 mg g⁻¹, respectivamente. As cinéticas foram melhor ajustadas por Avrami (R² 0,99, erro <2,8%). As propriedades do carvão melhoraram com a modificação, apresentando estrutura mais porosa, maior área superficial e grupos funcionais que contribuíram para a adsorção, provando que métodos simples de purificação podem ser cruciais para a reutilização de resíduos.

Palavras chave: carvão ativado, modificação, remoção, corante, azul de metileno.

1. INTRODUCTION

Rice husk charcoal is a porous and amorphous material obtained by the pyrolysis of rice husks, exhibiting an extremely wide surface area, which makes it a highly effective adsorbent. Its properties include a high surface area, allowing it to accommodate a large amount of contaminants, and good adsorption capacity, which enables the removal of impurities from water, air, and combustion gases. Furthermore, its multiple applications in areas such as water treatment, air purification, and contaminant removal highlight its versatility (Goodman, 2020; Karam *et al.*, 2022; Pode, 2016). The use of rice husk charcoal is also a sustainable and renewable option to improve environmental quality. Thus, studies have demonstrated the potential of rice husks as a raw material in the production of activated charcoal with various applications (Ahmad, Khan, Giri, Chowdhary, and Chaturvedi, 2020; Bushra and Remya, 2020; Goodman, 2020; Karam *et al.*, 2022; Tabassam *et al.*, 2022).

In this context, rice husks emerge as a promising alternative, as they are an abundant agricultural byproduct in Brazil, often underutilized and frequently discarded. The burning of rice husks is widely used for various purposes, such as a source of renewable energy, production of natural silica, agricultural uses, construction materials, among others (Asadi *et al.*, 2021; Braga *et al.*, 2013; Campos *et al.*, 2017; Goodman, 2020; Hossain, Mathur and Roy, 2018). This burning generates a residue that consists of a mixture of ashes (mainly composed of silicon oxide) and high-carbon content charcoal (with excellent properties) (Asadi *et al.*, 2021; Pode, 2016). This residue has the potential to be reused in other processes, such as adsorption, due to the properties of charcoal that make it efficient in removing unwanted substances in various industrial processes and water treatment (Asadi *et al.*, 2021; Goodman, 2020; Karam *et al.*, 2022). This alternative makes the process even more sustainable, offering the possibility of a circular economy.

The treatment of rice husk charcoal is essential to produce activated charcoal with improved properties. This process can involve various treatments (pre-treatment, activation, drying) with conditions ranging from mild to intense, including chemical, physical, or a combination of both (Dada, Inyinbor, Tokula, Bello and Pal, 2022; Grefa *et al.*, 2023; Samsalee, Meerasri and Sothornvit, 2023; Wazir, Ullah and Yaqoob, 2023). Gentler modifications that can be done on-site or at the industrial set, favor the valorization of an industrial residue, making it possible to reduce landfill disposal costs and generate profits with a value-added product for practical applications. The modification creates pores that increase the adsorption capacity of the charcoal. As a result, treated rice husk charcoal becomes efficient in removing contaminants (Tabassam *et al.*, 2022; Wang *et al.*, 2023). Motlagh, Sharifian and Asasian-Kolur (2021) studied the utilization of rice husk residues as a raw material for producing of high-quality activated carbon through chemical activation. The activation process was performed using K_2CO_3 as the activating agent under controlled conditions of impregnation and activation temperature (1000 °C). The results revealed a highly porous activated carbon with a specific surface area exceeding $2000 \text{ m}^2 \text{ g}^{-1}$ and significant microporosity. Evaluation of the adsorption properties demonstrated an efficient removal capacity for a wide range of contaminants, highlighting the potential of this material for water purification and environmental remediation applications. Homagai, Poudel, Poudel and Bhattarai (2022) used rice husks to produce charcoal and investigated by introducing a xanthate group by chemical treatment. The researchers evaluated the capacity of the adsorbents to remove crystal violet dye from aqueous solutions. The xanthated rice husk exhibited superior dye adsorption capacity than the charred rice husk. This suggests a promising adsorbent to treat water contaminated with dyes. In summary, the study demonstrated that modifying rice husks resulted in effective adsorbents,

highlighting the feasibility of economically and ecologically sustainable solutions for water pollution.

The adsorption technique using activated charcoal has proven highly effective in removing contaminants from water, thanks to its vast surface area and porous structure, which enable it to adsorb various types of contaminants, such as volatile organic compounds, dyes, heavy metals, and pesticides (Ahmad *et al.*, 2020; Diehl *et al.*, 2023; Grabi *et al.*, 2022; Sah *et al.*, 2022; Streit *et al.*, 2023). Dyes are one of the main sources of water pollution, and their presence is due to their extensive use in industrial processes such as paper, textiles, electroplating, pulp and paper, food, cosmetics, rocks, and minerals. An example of this is the dyeing and valorization of rocks and minerals in Rio Grande do Sul, which requires large volumes of water and uses different dyes, generating high concentrations of dyes in effluents that, if not treated properly, can cause harm to the ecosystem. One example is methylene blue, a cationic dye that is highly resistant to degradation due to its complex aromatic structure and xenobiotic properties, it can be harmful to aquatic life and humans when there is significant exposure to it (Benjelloun, Miyah, Evrendilek, Zerrouq, and Lairini, 2021; Sah *et al.*, 2022).

The growing concern for sustainability and the conservation of natural resources has driven research toward more environmentally friendly solutions. Therefore, studies on the development of adsorbent materials using alternative raw materials have been conducted (Ahmad *et al.*, 2020; Diehl *et al.*, 2023; Grabi *et al.*, 2022; Sah *et al.*, 2022; Streit *et al.*, 2023). These findings underscore the importance of ongoing research in the development of more accessible, sustainable, and efficient water treatment technologies to combat pollution caused by dyes and other industrial contaminants. The use of rice husks as a precursor for the production of activated charcoal is one of the promising solutions, not only from an economic perspective but also in the context of sustainability and environmental preservation. Thus, the objective of this study was to evaluate the valorization of a carbonaceous industrial residue derived from rice husk burning for the production of natural silica through a simple modification with acid and alkaline washing to enhance the material's adsorption properties and the removal of methylene blue dye.

2. METHODOLOGY

2.1. Material treatment/preparation

Raw coal (RC) naturally impregnated with sodium silicate, a residue from the burning of rice husks to produce natural silica, was supplied by the company Orysazil (Itaqui/RS). This coal was modified, named MC, through an acid and basic washing to remove organic and inorganic compounds present in the coal. First, the RC (density 0.315 g mL^{-1}) was mixed with a 1.0 mol L^{-1} hydrochloric acid (HCl) solution at a ratio 1:1.5 (v/v), with magnetic stirring for 1.0 h. Subsequently, successive washes with distilled water were performed until neutralization. Then, the material was dried in an oven at 100°C for 24 h. This dried material was mixed with a 1.0 mol L^{-1} sodium hydroxide (NaOH) solution, and the same process described previously for acid washing was carried out.

2.2. Characterizations

The surface area, pore volume and average pore size were determined by nitrogen adsorption/desorption isotherms (MICROMERITCS, ASAP 2020) using the Brunauer Emmett Teller (BET) and Barret Joyner Halenda (BJH) methods. The functional groups of the materials were

determined by Fourier Transform Infrared Spectroscopy (FTIR) on a spectrophotometer (SHIMADZU, IRPrestige-21), using the KBr pellet transmittance method and reading range of 4000 to 400 cm^{-1} .

2.2. Adsorption assays

The MB dye (CAS N° 61-73-4, $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$, 319.85 g mol^{-1}) was purchased from Sigma-Aldrich. The concentrations of MB dye were determined by spectrophotometry (SHIMADZU, UVmini-1240), according to the standard curve determined at 664 nm ($[\text{MB}] = 6.2513 \times \text{absorbance}$; R^2 0.9979). The kinetic adsorption test of the MB dye was conducted at 25°C and pH 6.7, with 100 mL of dye solution at a concentration of 10 mg L^{-1} and adsorbent dosage of 0.05 g L^{-1} . Aliquots were withdrawn at interval from 0 to 120 min at determined times. Dye removal and adsorption capacity were determined according to Equations (1) and (2), respectively.

$$R (\%) = \frac{C_i - C_t}{C_i} \times 100 \quad (1)$$

$$q_t = \frac{C_i - C_t}{m} \times V \quad (2)$$

Where: R is the removal percentage (%), C_i is the initial concentration of the contaminant (mg L^{-1}), C_t is the concentration of the contaminant at time t (mg L^{-1}), q_t is the adsorption capacity at time t (mg g^{-1}), m is the mass of adsorbent (g), V is the volume of the liquid phase (L).

The kinetic behavior was correlated to first-order pseudo-reaction models, Equation (3) (Lagergren, 1898) and second-order, Equation (4) (Ho & McKay, 2000; Ho & McKay, 1999), Avrami, Equation (5) (Avrami, 1939) and Elovich, Equation (6) (Elovich & Larionov, 1962). The adjustments of the kinetic models parameters to the experimental data were performed using the OriginPro 7 Software (OriginLab Corporation, USA).

$$q_t = q_1 \times (1 - \exp(-k_1 \times t)) \quad (3)$$

$$q_t = \frac{t}{\left(\frac{1}{k_2 \times q_2^2}\right) + \left(\frac{t}{q_2}\right)} \quad (4)$$

$$q_t = q_{AM} \times (1 - \exp(-k_{AM} \times t)^n) \quad (5)$$

$$q_t = \frac{1}{\alpha} \times l(1 + \alpha\beta t) \quad (6)$$

Where: q_t is the adsorption capacity in time (mg g^{-1}), q_1 e q_2 and q_{AM} are the theoretical adsorption capacity (mg g^{-1}), k_1 is the pseudo-first order rate constant (min^{-1}), k_2 is the pseudo-second order rate constant ($\text{g mg}^{-1} \text{min}$), k_{AM} is the avrami rate constant (min^{-1}), n is the avrami exponent, t is the time (min), α is the initial adsorption rate ($\text{g mg}^{-1} \text{min}$), β is the desorption constant (mg g^{-1}).

3. RESULTS AND DISCUSSIONS

3.1. Material characterizations

The surface properties of RC and MC are shown in Table 1.0, an increase in the properties of RC is observed after acid and basic modification, presenting a more porous structure with greater surface area. The surface area increased approximately 1.83 times (486.29 to 894.18 $\text{cm}^2 \text{g}^{-1}$), the total pore volume by 1.98 times (0.284 to 0.584 $\text{cm}^3 \text{g}^{-1}$), and the average pore diameter 1.21 times (3.7 to 4.5 nm). The pore size values indicate that RC and MC are mesoporous materials (2 to 50 nm). In this way, it shows that the modification was efficient in removing impurities (organic and inorganic), mainly ash, which contributed to improving the adsorption properties of the material. A similar result was obtained by Wazir *et al.* (2023) who obtained a surface area of 729.4 $\text{m}^2 \text{g}^{-1}$ for rice husk charcoal impregnated with sodium hydroxide, after activating it for 1 h in a nitrogen environment at 900 °C.

Table 1: Surface analysis of materials.

Sample	Surface área ($\text{m}^2 \text{g}^{-1}$)	Pore volume ($\text{cm}^3 \text{g}^{-1}$)	Average pore diameter (nm)
RC	486.29	0.284	3.7
MC	894.18	0.565	4.5

The visual aspect of the materials is shown in Figure 1, as can be seen, there were no visual changes with acid and basic washing. In Figure 2 shows the FTIR spectra of RC and MC used to identify the functional groups. The bands in common in RC and MC were at 3430, 1630 to 1650, 1040, 782 and 465 cm^{-1} , but with lower intensity in MC compared to RC, showing a successful modification, as observed in the surface analysis. The bands 3430 cm^{-1} and 1630 to 1650 cm^{-1} correspond to stretching and bending of the OH hydroxyl group (Ahmad *et al.*, 2020; Sah *et al.*, 2022). Furthermore, the bands at 1630 to 1650 cm^{-1} are also associated with C=O/C=C, aromatic ring bonds or carboxylic acid (Sah *et al.*, 2022; Tabassam *et al.*, 2022). The bands at 1040, 782 and 465 cm^{-1} correspond to Si-O bonds (Ahmad *et al.*, 2020; Sah *et al.*, 2022; Tabassam *et al.*, 2022). New bands emerged after the modification, such as the bands 2920 cm^{-1} (CH asymmetric stretching), 2860 cm^{-1} (CH symmetric aliphatic stretching), 2360 to 2330 cm^{-1} attributed to CH and C≡C stretching, 1107 cm^{-1} (CH /CO) and 655 cm^{-1} related to the phenyl group (Sah *et al.*, 2022; Tabassam *et al.*, 2022). These new bands demonstrate that the modification was not only efficient in removing groups related to impurities in the coal but also contributed to the emergence of new functional groups. These new groups enhance the interaction with contaminants, favoring the adsorption process.



Figure 1: Photography of materials - a) RC; b) MC.

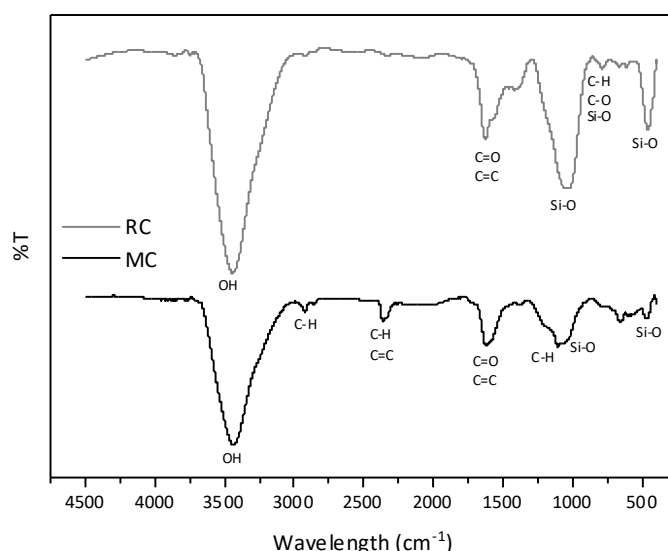


Figure 2: FTIR spectra of the materials.

The results confirm that the residue properties improved with acid and basic modification. It features a more porous structure, with greater surface area and functional groups that contribute to an efficient material for adsorption. The same was verified by Bhardwaj *et al.* (2022) in modifying a rice straw charcoal leached with acetic acid, Tabassam *et al.* (2022) who synthesized a rice husk charcoal sensitized by cinnamic acid, and Wang *et al.* (2023) who modified a rice straw charcoal with $\text{Ca}(\text{OH})_2$.

3.2. Methylene blue adsorption

The removal of MB dye by RC and MC is shown in Figure 3, and Figure 4 a and b shows the dye solutions before and after removal at 30 and 120 minutes. As can be seen for RC, the removal gradually increased, from the beginning, reaching 78% in 120 min. As for MC, it is noted that removal of more than 50% in the first few minutes, after which it gradually increased. In 120 minutes the removal was 97%, higher than the removal for RC, demonstrating that the modification led to an increase in the removal of the MB dye due to the greater surface area. Consequently, more active adsorption sites were available for interaction. Similar results were obtained by Wazir *et al.* (2023), who removed 80% of MB in 120 min, under pH 8 and initial dye concentration of 50 mg L^{-1} , using a rice husk-based charcoal impregnated with NaOH. Tabassam *et al.* (2022) modified a rice husk-based charcoal with cinnamic acid and removed 99.9% of the AM dye in 100 min, under conditions of pH 7, 25°C , 120 rpm, concentration of 200 mg L^{-1} and mass of 0.2 g.

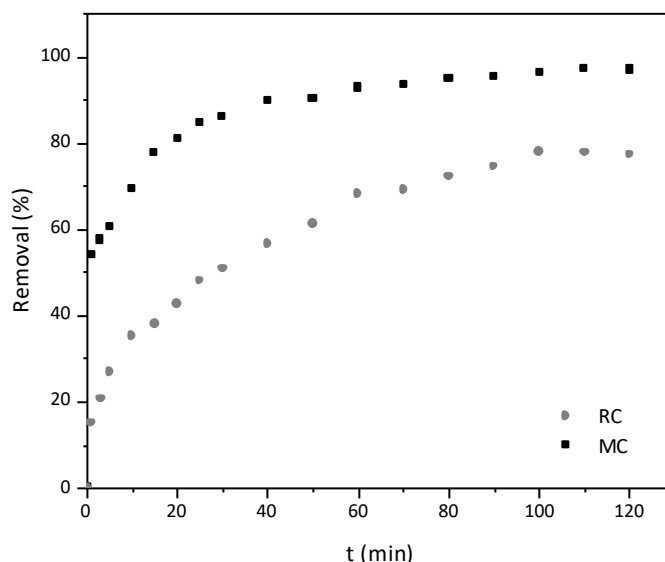


Figure 3: Removal of MB dye by RC and MC ($C_0 = 10 \text{ mg L}^{-1}$; pH 6.70, 25°C).

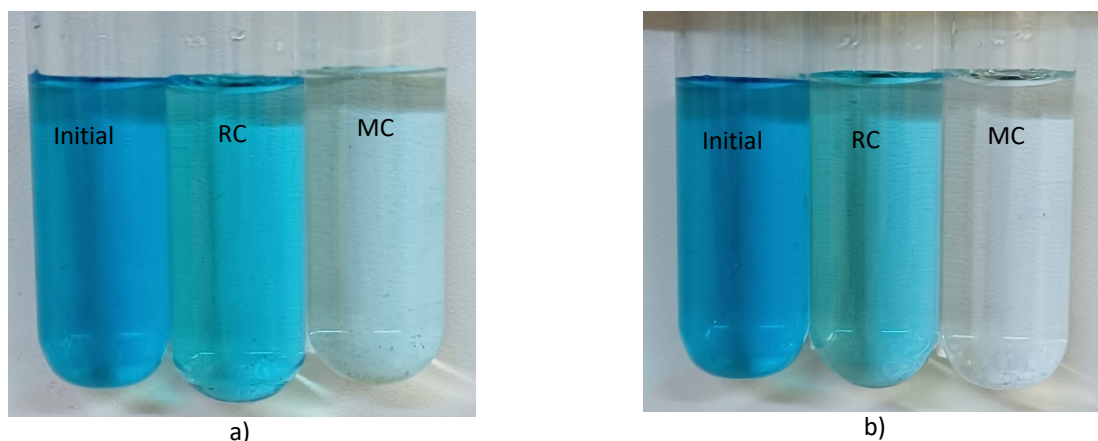


Figure 4: Removal of MB dye by RC and MC during – a) 30 min; b) 120 min.

Figure 5 a and b show the adsorption kinetics of MB dye in terms of adsorption capacity are presented. In the first few minutes, a rapid adsorption rate is observed for both materials, but with a difference for MC, which presented a high adsorption capacity compared to RC. This fact can be justified by the higher surface area of MC, which facilitates the transfer of MB molecules into the material, contributing to a greater and faster interaction between the dye and the active sites of the MC. Over time, both adsorbents showed a gradual increase in adsorption capacity. However, for MC, after 60 min a slower adsorption was noted, where the adsorption capacities varied little, indicating the occupation of available sites (saturation) of the material by the adsorbed MB, visibly presenting a plateau. While for RC it was observed that adsorption was slower after 100 min. The highest experimental adsorption capacities of the MB dye were 144.78 mg g^{-1} for RC and 180.78 mg g^{-1} for MC, showing that MC was more efficient than RC and that the modification process was effective. These adsorption capacities were higher than those reported in the literature for the removal of MB dye and similar carbons. For example, Lesbani, Siregar, Palapa, Taher and Rivanti (2021) obtained an adsorption capacity of 1.937 mg g^{-1} ($C_0 15 \text{ mg L}^{-1}$, pH 3-4, 200 min) using rice husk charcoal and a capacity of 15.58 mg g^{-1} ($C_0 75 \text{ mg L}^{-1}$, pH 3-4, 200 min) for double hydroxide modified coal in Zn/Al layer. Bhardwaj *et al.* (2022) studied a rice straw charcoal leached with acetic

acid to remove MB, the adsorption capacity found was 51.34 mg g^{-1} in conditions of C_0 dye of 135 mg L^{-1} , pH 9, dosage of 2 g L^{-1} and 25°C . Wang *et al.* (2023) obtained adsorption capacity of 242.4 mg g^{-1} in 1440 min by a carbon modified with $\text{Ca}(\text{OH})_2$ derived from rice straw, using a dosage of 0.5 g L^{-1} and initial concentration of 150 mg L^{-1} .

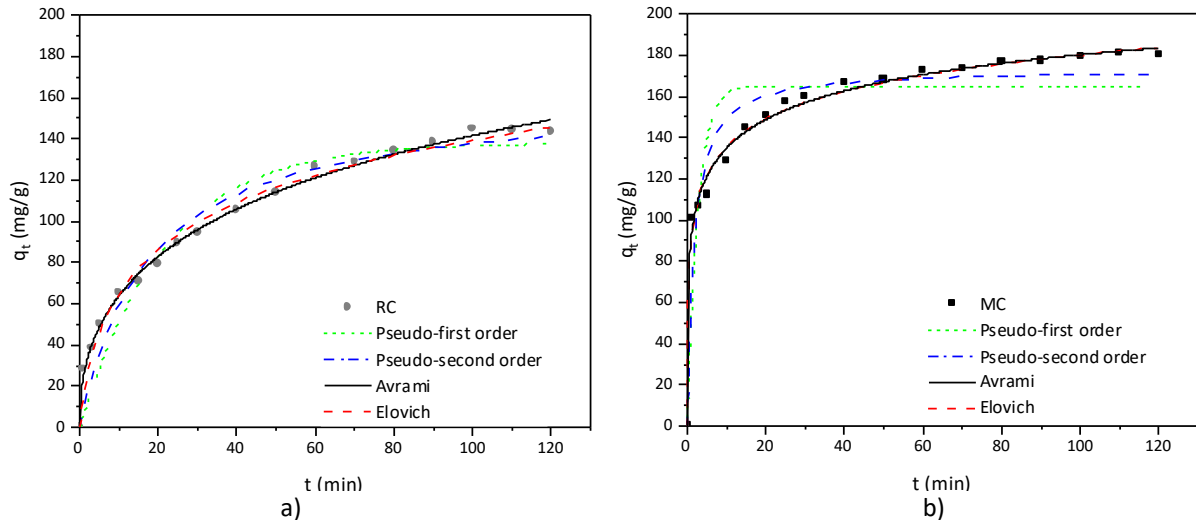


Figure 5: MB dye adsorption kinetic data and adjusted kinetic models ($C_0 = 10 \text{ mg/L}$; pH 6.70, 25°C) - a) by RC; b) by MC.

The Kinetic data were modeled by fitting pseudo-first order, pseudo-second order, Avrami, and Elovich kinetic models. The parameters values of the kinetic models are presented in Table 2. According to the coefficients of determination R^2 and adjusted R^2 , both greater than 0.99, it can be observed that the Avrami model provided the best fit for both, RC and MC. Furthermore, the average relative error (ARE) values were less than 2.8%, demonstrating that the estimated adsorption capacities are close to the experimental capacities. Regarding the rate constants (k_{AM}) and adsorption capacities (q_{AM}), it is possible to infer the same trend observed in the kinetic curves. The kinetics of MC were faster than RC, given the k_{AM} was 5.0 times greater, as well as the higher adsorption capacity for the AM dye, which was 256.54 mg g^{-1} for MC and 230.18 mg g^{-1} for RC. The parameter n demonstrates that the adsorption follows a fractional order reaction. Avrami's model considers that the process can tend to multiple orders, where the adsorption rates can change during adsorption (Benjelloun *et al.*, 2021).

Table 2: Parameters of the kinetic models for MB dye adsorption by RC and MC.

Parameters	RC	MC
Pseudo-first order		
$k_1 (\text{min}^{-1})$	0.0463	0.3704
$q_1 (\text{mg g}^{-1})$	137.96	165.15
R^2	0.9372	0.8251
R^2 adjusted	0.9333	0.8141
ARE (%)	15.350	10.783
Pseudo-second order		
$k_2 (\text{mg mg}^{-1} \text{ min})$	0.00036	0.0035
$q_2 (\text{mg g}^{-1})$	161.40	173.39
R^2	0.9652	0.9228

R ² adjusted	0.9631	0.9179
ARE (%)	11.829	7.0446
Avrami		
k _{AM} (min ⁻¹)	0.1020	0.5094
q _{AM} (mg g ⁻¹)	230.18	256.54
n	0.4481	0.2385
R ²	0.9963	0.9918
R ² adjusted	0.9958	0.9907
ARE (%)	2.7261	2.3725
Elovich		
α (g mg ⁻¹)	0.02895	0.0517
β (mg g ⁻¹ min)	19.243	2142.9
R ²	0.9877	0.9902
R ² adjusted	0.9869	0.9896
ARE (%)	6.6406	2.5885

Due to the data obtained in the MB dye adsorption kinetics, it was possible to verify that the adsorption capacity increased with the washing carried out on the residue. Therefore, CM emerged as the better material for MB adsorption. This highlights that the removal of impurities, such as Si-O groups, contributed to improving the properties of the coal, as well as increasing the surface area, favoring greater and faster adsorption of the dye. Thus, acid and basic washing was a simple and efficient alternative for valorizing industrial residue from rice husks burning.

4. CONCLUSION

The characterization results demonstrated that the surface area and pore volume of raw coal (486.29 m² g⁻¹ and 0.284 m³ g⁻¹) increased after modification with acid and base to 894.18 m² g⁻¹ and 0.565 m³ g⁻¹, respectively. The analysis of functional groups demonstrated that specific bands decreased, indicating that the modification was efficient in removing impurities (organic and inorganic groups), mainly Si-O groups. The removal of methylene blue, up to 120 min, was 78% for raw charcoal and 97% for modified charcoal, with the modified charcoal achieving 50% removal rate the first few minutes (less than 5 min). The adsorption kinetics showed that the adsorption by the modified coal was faster than raw coal, with higher adsorption capacity. In the time studied, the experimental adsorption capacities were 144.78 mg g⁻¹ for raw coal and 180.78 mg g⁻¹ for modified coal. The kinetic parameters for both materials were best fitted by the Avrami model (R² and adjusted R² greater than 0.99). The capacities estimated by the model were 230.18 and 256.54 mg g⁻¹ for raw coal and modified coal, respectively. The rate constants showed that the adsorption of the modified carbon was 5.0 times faster. These facts highlight the efficiency of simple acid and, basic washing of the residue, which contributed to improving its adsorption properties and consequently, the valorization of an industrial residue derived from the rice husks burning. Therefore, modified coal is a sustainable and promising option as a more accessible and sustainable adsorbent material for the industry, with efficient application for combating pollution caused by dyes and other contaminants.

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