Circular Economy and Sustainability (2026) 6:15
https://doi.org/10.1007/s43615-026-00786-6

ORIGINAL PAPER

Synthesis and Application of Activated Carbon from Pecan Nutshells (*Carya illinoinensis*) for the Removal of Excess Copper in Distilled Beverage

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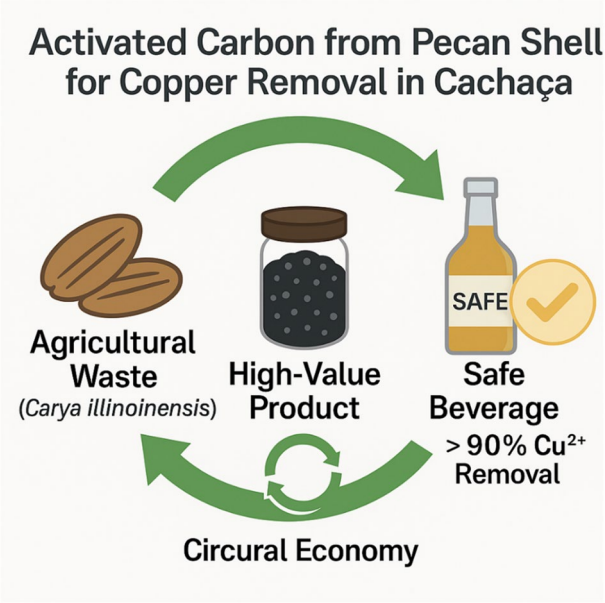
Received: 19 May 2025 / Accepted: 3 November 2025 / Published online: 11 February 2026
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Abstract

This study aimed to develop activated carbon from pecan nutshells for use as an adsorbent to remove excess copper from artisanal cachaça produced in small-scale distilleries. The process included grinding the shells, classifying particle sizes, chemically activating the material with $ZnCl_2$ (1:0.5 w/w), drying at 105 ± 1 °C, carbonizing in a muffle furnace, and washing with 6 M HCl followed by vacuum filtration. The resulting activated carbon was characterized in terms of moisture content, degree of carbonization, and iodine number. Adsorption tests were performed using two particle size fractions (10 g/L), agitating 0.1 L of cachaça with the adsorbent at room temperature (25 ± 1 °C) for up to 60 min. The initial copper concentration in the beverage was 11.30 mg/L, which exceeds regulatory limits. The activated carbon showed significant adsorption capacity, particularly the sample with the highest iodine number, achieving over 90% copper removal and reducing the copper concentration to 0.97 mg/L. These findings demonstrate the potential of pecan shell-derived activated carbon as an effective, sustainable solution for treating copper contamination in distilled beverages. A preliminary cost analysis indicated an estimated production cost of US$ 3–5 per kg, considerably lower than that of commercial activated carbons, further supporting the practical applicability of the proposed material.

Extended author information available on the last page of the article

Graphical Abstract



Keywords Pecan nut · Activated carbon · Cachaça · Copper · Adsorption · Iodine index

Introduction

The pecan tree (*Carya illinoinensis*) belongs to the Juglandaceae family and thrives under better cultivation conditions in temperate regions. It is native to the United States and Mexico and is cultivated in certain regions across different continents such as Asia, South Africa, and South America. Uruguay, Argentina, Chile, Peru, and Brazil are the countries with the highest production [1]. In Brazil, Rio Grande do Sul is the leading state in pecan production, accounting for approximately 70% of the total cultivated area nationwide, totaling approximately 7 thousand hectares. The remaining cultivated areas in the country are located in the states of Santa Catarina and Paraná, with 22% and 8%, respectively [2].

In recent years, this activity has been gaining traction thanks to growing government support and the assistance offered by the Brazilian Pecaniculture Institute (IBPE), based in the state of Rio Grande do Sul, which offers courses and guidance for better understanding by producers. Data presented by the Foreign Agricultural Service of the United States Department of Agriculture (FAS USDA) in February 2024 demonstrates an annual growth of approximately 26.44% in production between the 2019/2020 harvest (8700 MT – inshell basis) and the 2023/2024 harvest (11000 MT – inshell basis) [3].

Being a large tree, the pecan tree has a very high productive capacity, so that a small area of it brings a significant yield of fruit that can be marketed both with the shell and shelled. A negative aspect regarding the commercial market is the undervaluation of the shell, making it a waste product, often used as a substrate of little value. In this sense, the search for new

alternatives that can provide a more profitable destination for pecan shells becomes highly valuable, and for this purpose, the possibility of using them as a raw material for the production of activated carbon stands out as a promising option.

The utilization of waste, such as pecan nutshells, as adsorption carbon, is closely related to several United Nations Sustainable Development Goals (SDGs). This practice demonstrates an innovative and sustainable approach to dealing with agricultural waste while offering environmental, economic, and social benefits. Particularly, the use of waste as an energy source contributes to SDG 7 (Clean and Affordable Energy) by providing a renewable alternative to fossil fuels. Additionally, this approach aligns with SDG 9 (Industry, Innovation, and Infrastructure) by encouraging innovation in the use of available resources and promoting the development of sustainable technologies. By adopting responsible consumption and production practices, as advocated by SDG 12, the use of waste such as pecan nutshells helps reduce waste and minimize pressure on natural resources. Furthermore, by mitigating greenhouse gas emissions and promoting action against climate change (SDG 13), this practice contributes to protecting marine and terrestrial ecosystems (SDGs 14 and 15), thus preserving biodiversity. Finally, the utilization of waste as a valuable resource not only fosters job creation but also stimulates economic development in interconnected fields such as recycling and waste management, thereby contributing to the advancement of Sustainable Development Goal 8, which focuses on promoting decent work and fostering economic growth. This integrated approach highlights how waste utilization can be an effective strategy for promoting sustainable development across various areas [4].

As a porous material, activated carbon has gained significant prestige in adsorption methodology and has spread across various fields, being present in pharmaceuticals, hygiene products, food, and laboratory analyses, for example [5]. Its porosity is considered its main characteristic, responsible for its utilization direction and adsorption capacity. Its classification is controlled by the IUPAC (International Union of Pure and Applied Chemistry), described in two methodologies: based on the diameter of the constituent pores, namely micropores (0 to 2 nm), mesopores (2 to 50 nm), and macropores (diameter > 50 nm), and based on the shape of the pores, open pores (holes that communicate with the external surface of the carbon) and closed pores (isolated holes). These characteristics depend on the raw material used, production mechanisms, and the type of activation [6].

The production of activated carbon involves the dehydration of its precursor, along with carbonization/pyrolysis and activation, which can be either physical (to remove impurities) or chemical. In the physical method, the sample is gasified after prior carbonization at temperatures between 800 °C and 1100 °C. On the other hand, the chemical activation involves the impregnation of a dehydrating chemical agent, such as H_3PO_4, $ZnCl_2$, KOH, NaOH, HCl, H_2SO_4, among others, onto the precursor before carbonization. Regarding these methods, chemical activation is more suitable as it does not require high temperatures during processing and yields higher production, potentially exceeding physical activation by up to 30% in mass [7].

Furthermore, the excess of copper in artisanal cachaça becomes a relevant subject of study because, at elevated concentrations, it can pose a threat to human health, potentially increasing the risk of diseases such as Wilson's disease (accumulation of copper in the liver and brain), Menkes disease, Alzheimer's disease, as well as sclerosis and cancer (due to the formation of ethyl carbamate, a substance formed by the cyanide ion in distilled beverages where copper facilitates its formation) [8].

According to data, the production of cachaça in the country revolves around 1.3 billion liters per year, with 25% coming from artisanal production and 75% from industrial methods. The majority of this amount is consumed domestically, with only 1% to 2% being exported, with main buyers including Germany, Italy, Portugal, Paraguay, and Uruguay [9]. However, like any other product intended for human consumption, this beverage must adhere to parameters established by Decree No. 2314, dated September 4, 1997, which determines and regulates its standardization and classification. This is because, to be designated as cachaça (cana, caninha, or aguardente), the distilled spirit must be made from sugarcane as its raw material and have an alcohol content ranging between 38% and 58% by volume [10].

Despite representing a smaller percentage of production, artisanal cachaça produced on a small scale, usually made in copper stills through batch distillation, which provides greater sensory and chemical quality for the beverage, is the one that faces the most difficulty in complying with the parameters required regarding the maximum allowed copper content in cachaça (2 mg/L for export and 5 mg/L for domestic commercialization) [10], due to the lack of quantitative laboratory control. Therefore, for the produced cachaça to be commercialized without any issues, it is necessary to direct it to a mechanism that can align it with the parameters provided by current legislation.

Thus, contributing to both topics discussed, the use of activated carbon produced from pecan nutshells as an adsorbent medium for removing excess copper present in artisanal cachaça is highlighted [11]. The adsorption method, being a surface event of molecular adherence, from the adsorbent (activated carbon), can remove certain components existing in a fluid, whether liquid or gaseous, called adsorbate (copper ions). This adherence occurs due to the formation of bonds between the molecules being adsorbed and the adsorbent, which can happen in two different ways: physically caused by Van der Waals forces and chemically induced by molecular interaction that provides irreversible electron transfer [12]. Through this method, copper molecules are adsorbed by the carbon, allowing the metal concentration in cachaça to meet the limits specified in legislation. Given these challenges, the present study investigates the use of pecan shell-derived activated carbon as an effective, sustainable, and low-cost adsorbent to reduce copper concentrations in artisanal cachaça from small rural distilleries.

Materials and Methods

Raw Material and Sample Preparation

Pecan nutshells were sourced in August 2023 from a family farm located in Carlos Gomes, Rio Grande do Sul, Brazil. The shells were initially ground using a knife mill (MA 340, Marconi) with a sieve size of 1.70 mm (88 × 50 mm). Particle size classification was performed using a series of Tyler sieves. Moisture content was determined by heating the samples at 105 ± 1 °C until constant mass, using an infrared balance (ID 200, Marconi, Brazil). Figure 1 depicts the stages of the activated carbon production process.

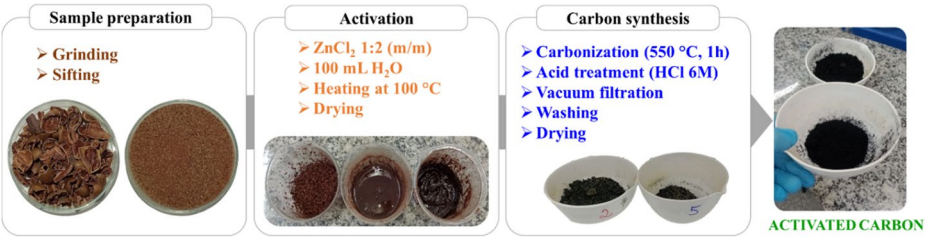


Fig. 1 Schematic representation of the production of activated carbon from pecan nutshells

Activation

Three particle size ranges (925, 390, and ≤53 µm) were selected for carbon activation. Chemical activation was performed using $ZnCl_2$ at a 2:1 ratio (sample: $ZnCl_2$, w/w) in 100 mL of deionized water. The mixture was heated on a magnetic stirrer at 100 °C (1800 rpm) until water evaporation. The pH was measured with a pH meter (DIGIMED DM-20), and samples were dried in an oven at 105 ± 1 °C for 90 min.

Synthesis of Activated Carbon

Following the methodology of Lopes et al. [13], carbonization, acid washing, filtration, and drying were performed (Fig. 1). Samples were placed in porcelain crucibles and pyrolyzed in a muffle furnace (Fornos Lavoisier 400 C) at 550 °C for 1 h, with a heating rate of 5 °C/min. After carbonization, samples were washed with 200 mL of 6 M HCl via vacuum filtration, followed by rinsing with 200 mL of boiling distilled water (98.5 ± 0.5 °C). Residual inorganic and organic impurities were thereby removed. Finally, samples were oven-dried at 105 ± 1 °C to constant mass.

Characterization of Activated Carbon

The following parameters were determined: moisture content, carbonization degree (Eq. 1), yield (Eq. 2), and iodine number (Eq. 3). Moisture was measured as in Sect. 2.1.

Degree of Carbonization

$$\mathrm{GC}\,(\%) = \frac{\mathrm{m}_0 - \mathrm{m}}{\mathrm{m}_0} * 100 \tag{1}$$

where m_0 is the initial mass of the raw material, and m is the mass after carbonization.

Yield

$$\eta\,(\%) = \frac{\mathrm{Xi}}{\mathrm{Xo}} * 100 \tag{2}$$

where X_0 is the initial mass and X_i the final mass of the product.

Iodine Number

For the iodine number, the Iodine Number Determination method [14] was employed. The tests were performed with 1 g of each activated carbon, which was placed in Erlenmeyer flasks along with 20 mL of a standard iodine solution 0.1 N. Then, the mixtures were stirred and left for solid material sedimentation. Afterward, 10 mL of the supernatant from each sample was collected for titration with a standard solution of thiosulfate 0.1 N using a 1.0% starch solution as an indicator. Titration was controlled by changing the color from blue to colorless. The amount of thiosulfate used in titration was used to calculate the iodine number present in the sample using Eq. 3.

$$\mathrm{X/M} = \frac{(\mathrm{A} * \ 12693 - (2.2 * \ (\mathrm{B} * \ 126.93)) * \mathrm{V})}{\mathrm{P}} \tag{3}$$

where *X/M* is the iodine number without correction factor (mg/g), *A* is the normality of the iodine solution (0.1 N) multiplied by 12,693, *B* is the normality of the sodium thiosulfate solution (0.1 N) multiplied by 126.93, *V* is the volume of sodium thiosulfate solution used in titration (mL), and; *P* is the mass of the sample (g).

Application of Activated Carbon

Collection and Characterization of Cachaça

The cachaça used in the study was obtained from a family-owned farm located in the municipality of Carlos Gomes, in the state of Rio Grande do Sul, Brazil, in September 2023. This cachaça was produced during the 2021/2022 harvest from sugarcane cultivated on the same farm, using natural fermentation. The process began with the use of sugarcane juice to initiate fermentation. Subsequently, the fermented must was distilled in a copper still to produce the cachaça.

Determination of Copper Content

Copper concentrations were determined spectrophotometrically at 447 nm using a UV/Vis spectrophotometer (Logen Scientific), following complexation with sodium diethyldithiocarbamate. The standard method used 2 mL of sample and 1 mL of 0.2% (w/v) reagent solution [15, 16].

Adsorption Tests

The adsorption experiments were conducted using 0.1 L of cachaça and 1.0 g of activated carbon with different particle sizes under magnetic stirring at room temperature (25 ± 1 °C) for 1 h. The copper content was analyzed at different contact times (3, 5, 10, 15, 30, and 60 min). The adsorption capacity at equilibrium (q_e) (mg/g) was determined by the equation (Eq. 4):

$$q_e = \frac{C_o - C_e}{m} * V \tag{4}$$

where C_0 and C_e re the initial and final concentrations (mg/L), respectively, m is the mass of adsorbent (g), and V is the volume of the solution (L).

The experimental results were adjusted to the pseudo-first-order model. In this model, the sorption rate is proportional to the solute concentration [17]. The experimental results were correlated with the following equation (Eq. 5):

$$q_t = q_e * \left(1 - e^{-k*t}\right) \tag{5}$$

where q_e and q_t are the quantities of adsorbed copper at equilibrium and at time t, and k is the Lagergren adsorption rate constant.

Additionally, the pseudo-second-order equation (Eq. 6) was applied, which is generally used for adsorption systems where the chemisorption mechanism takes place [18–20]:

$$q_t = \left(q_e^2 * k * t\right) / (1 + q_e * k * t) \tag{6}$$

where k is the pseudo-second-order constant, q_e is the quantity of copper at equilibrium, and q_t is the quantity of copper adsorbed at time t.

The pseudo-first-order model assumes that the adsorption rate is proportional to the difference between the amount of copper adsorbed on the carbon at time t and the amount of copper adsorbed at equilibrium, while the pseudo-second-order model considers that the adsorption rate is proportional to the square of this difference.

The parameters of the models were determined through nonlinear regression using Statistic 7.0 software (StatSoft, USA). The quality of the fit was verified according to the coefficient of determination (R^2) and root mean square error (RMSE) of the experimental data to Eq. 7:

$$RMSE = \sqrt{\sum \left(\frac{Obs - Pred}{n}\right)^2} \tag{7}$$

where *Obs* is the observed value, *Pred* is the predicted value, and n is the number of data points.

This equation calculates the root mean square error, which is a measure of the differences between values predicted by a model and the actual observed values. Lower values of RMSE indicate better model fit to the data.

Statistical Analysis

All assays (activation and adsorption) were employed at a standard deviation of 5%, and the data obtained were analyzed using on Statistica 7.0 software (StatSoft, Tulsa, USA), using ANOVA analysis of variance, and submitted to Tukey's test, considering the significance level of 95%.

Results and Discussion

Characterization of Pecan Nutshells

Table 1 presents the mass, moisture content, and particle size distribution for the three samples of pecan nutshells used in this study.

The data presented in Table 1 delineated the physical characteristics of three distinct samples of pecan nutshells. Each sample was numerically identified (1, 2, and 3), and various parameters such as raw nutshell mass, moisture content, major diameter, minor diameter, and Sauter mean diameter were measured.

Regarding mass and moisture content, a noticeable variation among the samples is observed. Sample 1 exhibited the highest raw nutshell mass, at 103.80 g, followed by sample 2, with 33.10 g, and sample 3, with 27.30 g. Correspondingly, the moisture content showed a similar trend, with sample 1 exhibiting the lowest moisture content ($10.8 \pm 0.5\%$), followed by sample 2 ($13.5 \pm 0.7\%$) and sample 3 ($13.8 \pm 0.7\%$).

The particle sizes (Table 1) provide details about the major and minor diameters, measured in micrometers (μm). Sample 1 has the largest particle size, with a diameter of 1000 μm. On the other hand, sample 3 exhibits the smallest diameter, with ≤53 μm.

Additionally, the Sauter mean diameter, which offers insights into the average particle size considering surface area, volume, and surface-to-volume ratio, is provided for both samples. Sample 1 has a Sauter mean diameter of 925 μm, while sample 2 has a value of 390 μm. Considering the implications for adsorption efficiency, particle size and surface area play fundamental roles. Furthermore, moisture content can influence adsorption properties, as excess moisture can decrease the available surface area for adsorption.

Overall, this data is important for quality control and standardization in activated carbon production. Ensuring consistency in particle size and moisture content is imperative to guarantee the reproducibility and efficacy of activated carbon, especially in adsorption applications such as the removal of copper from artisanal cachaça.

Characterization of Activated Carbon

After pyrolysis, washing, and drying, the final activated carbon masses obtained from samples 1, 2, and 3 were 37.14 g, 8.41 g, and 4.07 g, respectively. These values represent the actual recovered mass of activated carbon derived from the initial raw nutshells reported in Table 1. Due to the small yield of activated carbon from sample 3, which would be insufficient for adsorption tests, this sample was disregarded in subsequent experiments.

The moisture content of activated samples 1 and 2 was found to be 16.4% and 21.8%, respectively, indicating the granulometric interference since there was a greater removal of water in the smaller diameter grain. Regarding the degree of carbonization, the results were

Table 1 Physical properties of Pecan nut shell samples

Sample	Raw nutshell mass (g)	Moisture (%)	Largest diameter (μm)	Smallest diameter (μm)	Sauter mean diameter (μm)
1	103.80	10.8 ± 0.5	1000	850	925
2	33.10	13.5 ± 0.7	425	355	390
3	27.30	13.8 ± 0.7	53	bottom	≤53

78.12% and 47.34% for samples 1 and 2, respectively, highlighting a higher amount of fixed carbon in the structure of the former, considered of higher purity, given that these are intrinsic characteristics. This difference between them was due to the amount of mass disposed in the crucibles as well as the differential value of the surface area.

Regarding yield, they achieved significantly different values, with sample 1 standing out at 36.85%, i.e., 16.76% more than sample 2. Regarding the iodine index, sample 2 stood out with a value of 973.3 mg/g, almost twice the 571.18 mg/g observed for sample 1. This trend was linked to the smaller grain size, which provides a greater area per unit mass and consequently highlights higher adsorption efficiency.

The iodine index, usually presented in mg/g, is an important tool in evaluating the quality and adsorption capacity of activated carbon. Its simplicity and speed of analysis make it important in this context. Furthermore, it provides insights into the amount of micropores in activated carbon, indicating its effectiveness as an adsorbent. Micropores, formed during the activation process, are crucial for the high surface area of activated carbon. A high iodine index suggests a higher degree of activation, typically ranging between 500 and 1200 mg/g, corresponding to a surface area between 900 and 1100 m^2/g [21].

Although advanced surface characterization techniques such as BET surface area measurements or SEM imaging were not available for this study, the iodine number was adopted as a reliable indicator of the adsorbent's microporosity and surface activity. Previous studies have shown a strong correlation between iodine number and BET surface area in lignocellulosic-based activated carbons, where values above 900 mg/g typically correspond to surface areas greater than 900 m^2/g. Therefore, the observed iodine numbers (571.18–973.3 mg/g) strongly suggest that the synthesized carbons possess well-developed microstructures with significant adsorption potential.

Similar results were recently reported by Zhang et al. [22], who obtained a high fixed carbon content (70.1%) and well-developed pore structure from pecan nutshell biochar. Zou et al. [23] also reported a BET surface area of 1554 m^2/g and total pore volume of 0.744 cm^3/g for activated carbon synthesized from pecan shells using a self-activation process. These findings are consistent with the present study, confirming that pecan shell–based activated carbons exhibit a well-developed microporous structure and high adsorption potential.

Copper Concentration in Cachaça

The concentration of copper in the cachaça was found to be 11.30 mg/L, exceeding the limit allowed by legislation. According to the Normative Instruction No. 13, dated June 29, 2005, from the Ministry of Agriculture, Livestock, and Supply (MAPA) of Brazil, the maximum permitted limit of copper in cachaça is 5.0 mg/L [24].

Activated Carbon Efficiency

Table 2 presents the parameters used to describe the kinetics of adsorption processes of pseudo-first-order and pseudo-second-order models. q_e represents the equilibrium adsorption capacity (mg/g), k is the rate constant (min^{-1}), R^2 is the coefficient of determination, and RMSE is the root mean square error, which measures the accuracy of the model predictions.

As we can observe in Table 2, in both models, the experimental data were well correlated with the models. The parameters of the adsorption model play a crucial role in understand-

Table 2 Kinetic model parameters for copper adsorption

Model	Sample	q_e (mg/g)	k (min^{-1})	R^2	RMSE
Pseudo-first order	**1**	0.701	0.1094	96.89	0.044
	2	0.997	0.0991	96.70	0.066
Pseudo-second order	**1**	0.849	0.1333	96.14	0.050
	2	1.220	0.0819	95.84	0.074

ing the adsorption process for the evaluated samples. For the pseudo-first-order model, we observed that sample 1 exhibited an equilibrium adsorption capacity (q_e) of 0.701 mg/g, with a rate constant (k) of 0.1094 min^{-1}. Meanwhile, sample 2 recorded a q_e of 0.997 mg/g and k of 0.0991 min^{-1}. These values indicate the maximum adsorption capacity achieved by the samples and the speed at which equilibrium is reached, respectively. On the other hand, in the pseudo-second-order model, we noticed that sample 1 reached a q_e of 0.849 mg/g, with k of 0.1333 min^{-1}. Sample 2 exhibited a q_e of 1.220 mg/g and k of 0.0.0819 min^{-1}. These results suggest a slower relationship between adsorption and solute concentration for both samples, as indicated by the lower rate constants.

When evaluating the coefficients of determination (R^2) and the root mean square error (RMSE), we observed that both models fit well to the experimental data, with R^2 values above 95%. However, the lower RMSE for the pseudo-first-order model indicates better accuracy of predictions of this model compared to the pseudo-second-order model for both samples. These results suggest that the pseudo-first-order model may be more appropriate for describing the adsorption process for the evaluated samples since it provides more accurate predictions and has a more direct interpretation of parameters in terms of adsorption kinetics. However, it is important to consider that the choice of the appropriate model may vary according to the specific characteristics of the adsorption system under study.

Although adsorption kinetics were modeled using classical approaches, the consistency of the parameters obtained, together with the strong agreement with values reported in recent literature employing advanced surface analyses (e.g., BET, EDX), confirms the robustness of the present results. These findings indicate that even with conventional characterization methods, the adsorption behavior can be accurately interpreted and correlated with surface structure and functional groups described in more instrumentally intensive studies.

Similar adsorption mechanisms have been reported by Yang et al. [25], who observed that pecan shell biochar follows pseudo-second-order kinetics, governed primarily by chemisorption and π–π interactions. This behavior is consistent with the kinetic results obtained in the present study for copper adsorption, suggesting that surface functional groups and the π-conjugated structure play key roles in the adsorption mechanism.

Figure 2 presents the adsorption kinetics of copper on activated carbon (experimental data and models) for samples 1 and 2. The experimental data for both samples were fitted to the pseudo-first-order and pseudo-second-order kinetic models. The adsorption of copper on activated carbon can occur through different mechanisms, such as physical (or porous) adsorption and chemical (or chemisorption) adsorption. In physical adsorption, copper ions are adsorbed on the surface sites of activated carbon by Van der Waals forces or dipole-dipole interactions, while in chemical adsorption, reactions occur between copper ions and functional groups present on the surface of activated carbon.

In the initial stages (at 10 min) of copper adsorption on activated carbon (Fig. 2), the kinetics are often dominated by rapid processes of physical adsorption and surface diffusion. During this phase, copper ions rapidly accumulate on the surface of activated carbon

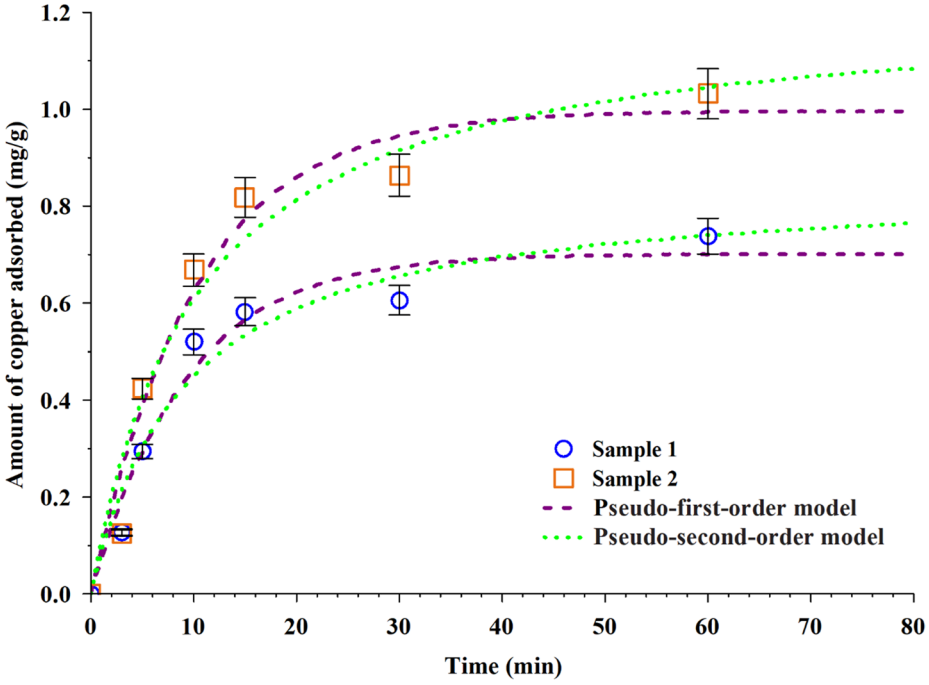


Fig. 2 Amount of copper adsorbed (mg/g) in the cachaça

through physical interactions such as Van der Waals forces, hydrogen bonds, and dipole-dipole interactions. This process is favored by the high availability of adsorption sites on the surface of activated carbon and the rapid transport rate of copper ions at the solid-liquid interface. At this initial stage, copper adsorption may follow pseudo-first-order kinetics, where the adsorption rate is proportional to the concentration of unadsorbed copper in the solution. The pseudo-first-order model is commonly applied in systems where initial adsorption is fast and dominated by the availability of adsorption sites on the adsorbent surface. However, it is important to note that the adsorption kinetics at initial times can be affected by various factors, such as the initial concentration of copper in the solution, the surface area and porosity of the activated carbon, temperature, and solution pH, among others.

Regarding the time required to reach adsorption equilibrium, both samples had a time of 60 min (Fig. 2). However, since sample 2 has a larger surface area, it was more efficient in removing copper from the cachaça. Evaluating the current legislation in terms of copper concentration in cachaça, it is noted that both samples were in compliance with the allowed limits.

The study conducted by Barbosa et al. [26] examined the use of coconut fiber as an adsorbent to remove copper from cachaça. They observed that the adsorption reaction reached equilibrium in 120 min. The maximum adsorption capacity recorded was 1.38 mg/g, indicating that each gram of coconut fiber could adsorb up to 1.38 milligrams of copper. After the adsorption process, the copper levels in the cachaça decreased from 8.57 mg/L to 4.97 mg/L.

In another study conducted by Honorato et al. [27], the removal of copper ions from alambique cachaça through adsorption using a variety of biosorbents was examined. These included sugarcane bagasse, okara seeds, and moringa oleifera, as well as three distinct types of zeolites and two types of commercial charcoal. The results revealed that thermally treated sugarcane bagasse was able to completely eliminate the copper present in the cachaça sample, while other low-cost natural biosorbents achieved removals around 50%, as seen in moringa oleifera and okara seeds. Kinetic analysis indicated that a pseudo-second-order model was appropriate to describe the adsorption process, with equilibrium times of 15 h for thermally treated sugarcane bagasse, *okara*, and *moringa oleifera* seeds used as adsorbents. Furthermore, the maximum adsorption capacity was determined to be 17.00 mg of Cu/g for thermally treated sugarcane bagasse, 0.77 mg of Cu/g for *moringa oleifera* seeds, and 5.33 mg of Cu/g for *okara*.

In a percentage analysis of copper removal from cachaça (Fig. 3), the sample 2 was deemed to be more relevant, as it showed greater efficiency from 5 min of adsorption onwards, since, at the end of the reaction (60 min), it was able to remove more than 90% of the initial copper quantity, leaving an extremely low residual copper content (0.97 mg/L).

In addition to the chemical safety aspects, it is important to consider the potential sensory implications of applying activated carbon in distilled beverages. Parameters such as odor, color, and taste are critical for consumer acceptance and commercial applicability. Although these aspects were not evaluated in the present study, previous research with other plant-derived adsorbents suggests that changes in sensory attributes may occur depending on

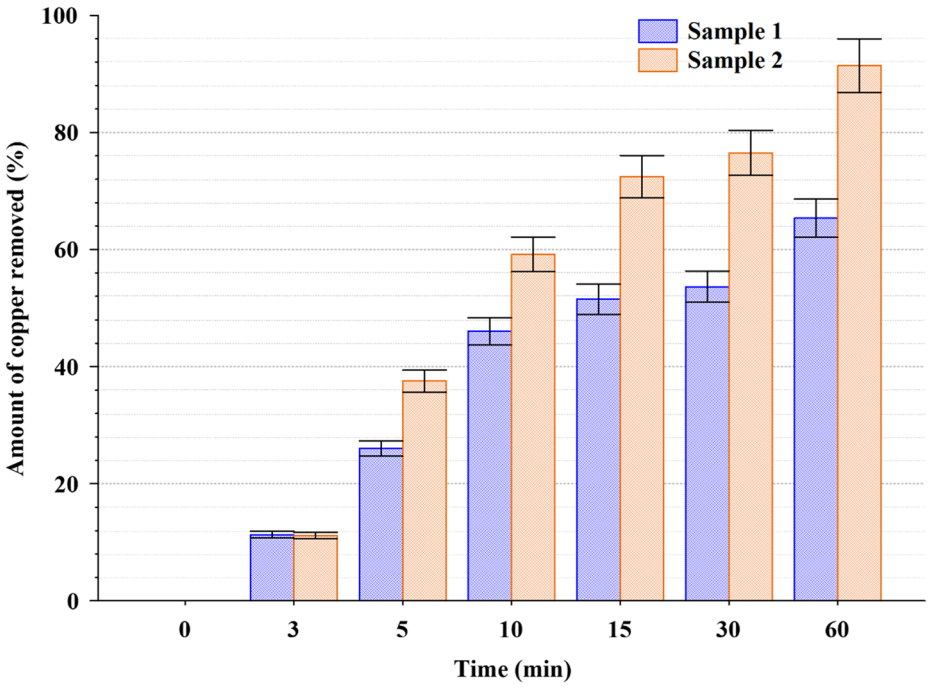


Fig. 3 Amount of copper removed (%) in the cachaça

the dosage applied and the contact time with the beverage. Therefore, future work should include a systematic sensory analysis to ensure that the treatment maintains the desirable characteristics of artisanal cachaça while effectively reducing copper content.

Over the years, several research studies have explored the use of pecan nutshells in the production of activated carbon and its application as an adsorbent in different contexts. Shawabkeha and Abu-Nameh [28] investigated the adsorption capacity of phenol and methylene blue by activated carbon derived from pecan nutshells. They observed a high removal capacity for methylene blue, reaching 410 mg/g at pH 9 and a solution concentration of 35 mg/L, while the adsorption of phenol was moderate, reaching 18 mg/g at pH 11 at the same solution concentration.

Klasson et al. [29] evaluated the adsorption of copper (II) by activated carbons derived from pecan nutshells. Their results highlighted the importance of oxygen content during the activation of pecan nutshells impregnated with phosphoric acid, influencing the adsorption capacity of copper (II) ions in aqueous solutions.

Niandou et al. [30] demonstrated the effectiveness of activated carbons derived from pecan nutshells in removing inorganic contaminants, such as Cu^{2+} and NO^{3-}, and organic contaminants, such as atrazine and metolachlor, from contaminated waters.

Durán-Jiménez et al. [31] developed oxygenated carbonaceous adsorbents from pecan nutshells for the removal of Pb^{2+} in aqueous solutions, achieving an excellent Pb^{2+} sorption capacity of 261 mg/g. Torres-Pérez et al. [32] investigated the use of a sustainable adsorbent obtained from pecan nutshells for the removal of the dye tartrazine, demonstrating its effectiveness in removing this dye, which is classified as carcinogenic.

Kaveeshwar et al. [33] synthesized activated carbon derived from pecan nutshells for the removal of iron (II) from fracking wastewater, achieving a maximum adsorption capacity of 41.66 mg/g at an iron (II) concentration of 55 mg/L and pH 3. They also applied the pseudo-second-order model to describe the iron adsorption. In another work, Valenzuela-Padilla and Torres-Pérez [34] studied the removal of azo dyes (A5 and R40) with activated carbon from pecan nutshells in aqueous media.

Limitations

This study was conducted using accessible analytical techniques to ensure feasibility for small-scale laboratories and distilleries. However, advanced characterization methods such as BET surface area analysis, SEM/EDX imaging, and FTIR spectroscopy would provide additional insights into the textural and chemical characteristics of the activated carbon. The integration of these analyses could further correlate the pore structure and surface functionalities with adsorption performance in greater detail. Despite these limitations, the present methodology demonstrates that reliable and reproducible results can be achieved using conventional and cost-effective approaches, which is essential for practical and scalable applications.

Preliminary Cost Analysis

In addition to the adsorption efficiency, a preliminary cost estimation was performed to evaluate the economic feasibility of the proposed adsorbent. Considering the use of pecan nutshells as an agricultural residue with negligible cost, the main expenses are related to

chemical reagents ($ZnCl_2$ and HCl) and energy consumption during drying and carbonization. Based on current market prices, the estimated production cost of pecan shell–derived activated carbon is in the range of US$ 3–5 per kg. This value is substantially lower than that of most commercial activated carbons (US$ 10–20 per kg), reinforcing the low-cost and sustainable character of the proposed adsorbent.

Thus, the study and application of pecan nutshells in the production of activated carbon and their subsequent use as adsorbents in various contexts have shown promising and versatile results. The reviewed studies highlighted the effectiveness of these materials in removing a variety of contaminants, both inorganic and organic, from contaminated waters and aqueous solutions. From the removal of metal ions such as copper and lead to the elimination of dyes and organic compounds such as atrazine and phenol, pecan nutshells have proven to be a viable and sustainable source of activated carbon with high adsorption potential.

The significant adsorption capacity of these materials, combined with their abundant availability and low production cost, suggests that the use of pecan nutshells in the manufacture of activated carbon may represent an attractive and environmentally sustainable alternative for the remediation of contaminated waters and treatment of industrial effluents. Furthermore, the studies also emphasize the importance of considering factors such as activation method and process conditions to optimize the performance of the adsorbent in specific applications.

Thus, the removal of copper from cachaça is of utmost importance. Excessive copper consumption can lead to a condition known as “copper toxicity,” which can have several adverse health effects. Highlighting some health problems associated with excessive copper consumption: gastrointestinal problems, liver damage, neurotoxicity, kidney failure, hemolytic anemia, and cardiovascular damage [35–37]. It is important to note that the effects of copper toxicity can vary depending on the dose and duration of exposure. Additionally, individual susceptibility can influence how the body responds to excess copper.

Copper contamination in alcoholic beverages besides cachaça can occur in other distilled beverages that are produced using copper equipment, such as stills. However, it is important to note that copper contamination may be minimal. In addition to cachaça, some distilled beverages that may be produced in copper stills include whiskey, gin, tequila, brandy, and rum. The presence of copper in these beverages is generally not considered harmful to health, as long as the stills are manufactured and operated in accordance with appropriate safety and quality standards.

In summary, the results of this research highlight that pecan nutshells transformed into activated carbon have the ability to remove copper from cachaça and other alcoholic beverages. Furthermore, they present promising potential to play a relevant role in reducing environmental pollution and preserving water resources. This underscores the importance of continuing to investigate and improve these materials as sustainable and effective alternatives for environmental remediation.

The consistency between the present findings and recent reports [22]– [23, 25] reinforces the high potential of pecan nutshell–derived activated carbon as a sustainable material. Its physicochemical features, such as large specific surface area and hierarchical pore structure, make it suitable not only for adsorption of metal ions but also for advanced applications such as energy storage.

Conclusion

The results of this study demonstrated that the use of pecan nutshells for the production of activated charcoal is a viable and efficient alternative for copper removal in artisanal cachaça. Among the evaluated samples, Sample 2, with an average particle diameter of 390 μm, exhibited the best adsorptive performance, standing out due to its high iodine index (973.3 mg/g), indicative of greater microporosity and specific surface area. This sample was able to remove over 90% of the copper present in the cachaça within 60 min, reducing the concentration from 11.30 mg/L to 0.97 mg/L, well below the limits established by Brazilian legislation (5.0 mg/L for the domestic market).

Kinetic modeling indicated a better fit to the pseudo-first-order model, with determination coefficients above 96%, suggesting that adsorption occurred predominantly through physical mechanisms. Although Sample 1 showed a higher carbonization yield and lower moisture content, its adsorptive efficiency was lower due to its smaller surface area (iodine index of 571.18 mg/g).

Therefore, the obtained data reinforce that activated charcoal derived from pecan nutshells, especially in smaller particle sizes, is a promising material for the removal of heavy metals in artisanal alcoholic beverages. In addition to meeting regulatory requirements, the proposed application offers a sustainable, low-cost solution with the potential for valorizing agro-industrial waste.

Although advanced analytical tools were not employed, the consistency of the experimental findings with recently published studies using BET, SEM, and EDX analyses reinforces the validity of the present results. The observed adsorption behavior, iodine index, and kinetic parameters align well with those reported for pecan shell–based activated carbons characterized by more sophisticated techniques.

In addition to its high adsorption efficiency, the material can be produced at an estimated cost of US$ 3–5 per kg, which is considerably lower than commercial activated carbons, further supporting its practical applicability.

Acknowledgements This study was financed in part by the URI Erechim.

Authors' Contributions E. Otowicz and T. Polon participated in designing and performing experiments, processing and interpreting data, as well as in preparation, writing and revising the manuscript. B. Fischer: was instrumental in the processing and interpretation of data, as well as in the preparation, writing, and revision of the manuscript. R. M Dallago: participated in designing and performing experiments, and processing and interpreting data. A. Junges played a key role in supervision, the processing and interpretation of data, and was also heavily involved in the preparation, writing, and revision of the manuscript.

Funding The Article Processing Charge (APC) for the publication of this research was funded by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) (ROR identifier: 00x0ma614). Not applicable.

Data Availability Data supporting the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Ethical Approval Not applicable.

Conflict of Interest The authors declare no conflict of interest.

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