EFFECT OF IMPREGNATING AGENTS ON THE POROSITY AND SORPTION CAPACITY OF CARBON BLOCKS

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Abstract

This study presents the results of thermochemical synthesis and functional modification of carbon–silicon sorbents derived from rice husks, intended for use as filtering materials with antiseptic properties. Carbonization was carried out in an inert gas atmosphere at temperatures ranging from 600 to 900 °C, followed by activation in a flow of carbon dioxide or water vapor. The resulting porous structures were characterized by a high specific surface area (up to 550 m²/g) and a considerable micropore volume. Molecular iodine, chlorhexidine gluconate, and tannin were employed as impregnating agents. Experimental data confirmed the ability of the sorbent to absorb iodine vapors from both gaseous and liquid phases, with a saturation capacity of up to 259 mg/g. The influence of impregnation on the textural properties of the material was investigated: an increase in specific surface area and pore volume was observed upon the introduction of active components. BET analysis demonstrated a total micropore surface area of up to 551 m²/g, confirming the efficiency of the modification for air purification purposes. The developed filtering monoblocks are promising for application in air disinfection systems within enclosed and sanitation-sensitive environments.

Keywords

Carbon–silicon sorbents, carbonization, activation, iodine, antiseptic activity, porous structure, filtering materials, rice husk, adsorption

1. Introduction

In the modern world, where air quality is becoming increasingly problematic due to urban development, industrial emissions, and other factors, ventilation systems play a key role in ensuring clean and healthy air. Atmospheric pollution poses serious threats to human health and contributes to the emergence of various respiratory diseases.

Carbon–silicon filters represent a class of materials capable not only of capturing the finest particulate pollutants but also of effectively neutralizing bacteria and viruses owing to their antibacterial properties. Bactericidal carbon–silicon filters hold the potential to revolutionize ventilation systems by providing not only clean but also safe air for residents and workers in indoor environments.

Contaminated air contains a wide range of solid particles, harmful chemical gases, as well as bacteria, viruses, and other hazardous impurities. The impact of these pollutants on human health and ecosystems has been extensively studied, with evidence confirming their contribution to numerous diseases and increased premature mortality rates. Atmospheric air may contain a wide spectrum of hazardous gaseous impurities, including sulfur and nitrogen compounds (such as sulfur dioxide, SO_2; sulfate anions, SO_4^{2-}; nitrate anions, NO_3^-), carbon oxides (e.g., carbon monoxide, CO), formaldehyde (HCHO), and various volatile organic compounds (VOCs). In recent years, growing concern has also been raised about the spread of bacteria and viruses transmitted through airborne pathways. Consequently, the development of effective and safe air filters has become increasingly urgent [1–4].

Carbon–silicon filters are mainly produced from rice husk. To date, the production of sorbents from rice-processing waste has been developed primarily within the framework of husk utilization. Since rice floral bracts (husks) and straw contain significant amounts of amorphous silicon dioxide, these residues can serve as sources for various silicon-containing sorbents (such as diatomites, tripoli, and opoka) or for pyrogenic silica. According to the literature [5], defatted rice bran is capable of removing chromium, copper, and zinc ions from solutions, whereas rice husk can remove strontium, cadmium, nickel, lead, zinc, chromium, cobalt, and aluminum ions.

Sorbents derived from rice husk possess a unique ability to effectively adsorb radionuclides and heavy metals. In [6], sorption of rare earth metals from model solutions was also tested. In recent years, sorbents obtained from agricultural waste after physico-chemical processing have been proposed for use as oil sorbents. In particular, rice husk has attracted considerable attention as an oil sorbent [7]. The effectiveness of sorbents already developed from plant-based raw materials has proven to be fairly high.

Gas filtration and ventilation systems [8] represent an innovative solution in the field of exhaust gas treatment, ensuring efficient purification of air from harmful substances and pollutants. These systems are equipped with specialized filters designed to capture and neutralize various types of gases, including toxic and odorous components, thereby making the air safer and more suitable for breathing.

2. Methods and Materials

The synthesis process begins with carbonization, i.e., the thermal decomposition of the raw material (pyrolysis) at 600–900 °C in an inert argon atmosphere. This stage aims to remove volatile components, increase the carbon and silicon content, and form a material with a well-developed porous structure and high specific surface area. Subsequently, activation of the carbon material is carried out using oxidizing gases such as carbon dioxide or water vapor at temperatures ranging from 600 to 1000 °C. This process facilitates the removal of amorphous carbon and enhances the porous structure, resulting in improved physicochemical properties.

Carbonization and activation were performed in a muffle furnace at temperatures from 300 to 900 °C for 30–120 minutes in a carbon dioxide atmosphere without air access. *Figure 1* presents the experimental setup with a capacity of 5 kg/h, providing sequential moisture removal, depolymerization, and partial decomposition of the raw material within the temperature range of 650–800 °C. At temperatures up to 270 °C, dehydration occurs, while within 600–850 °C, processes of decarboxylation and the formation of pyrolysis tars are observed, accompanied by dehydration and decarbonylation [9–10].

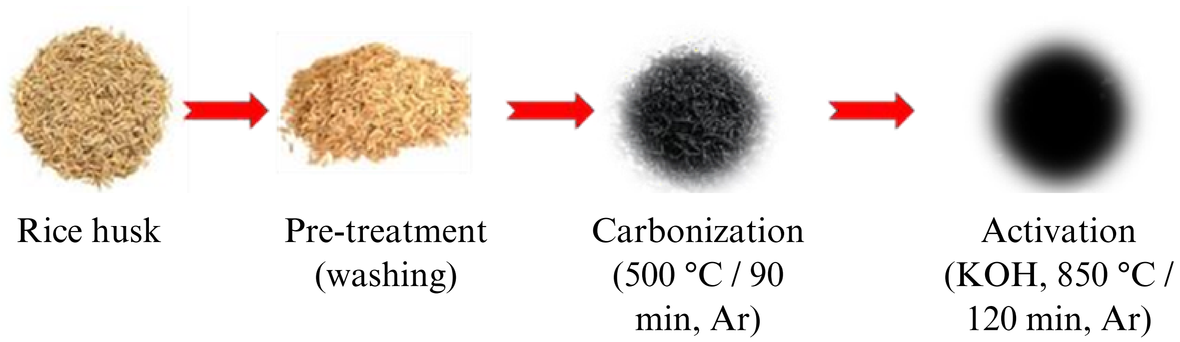

Figure 1. Schematic of the thermochemical process for obtaining carbon materials from rice husk

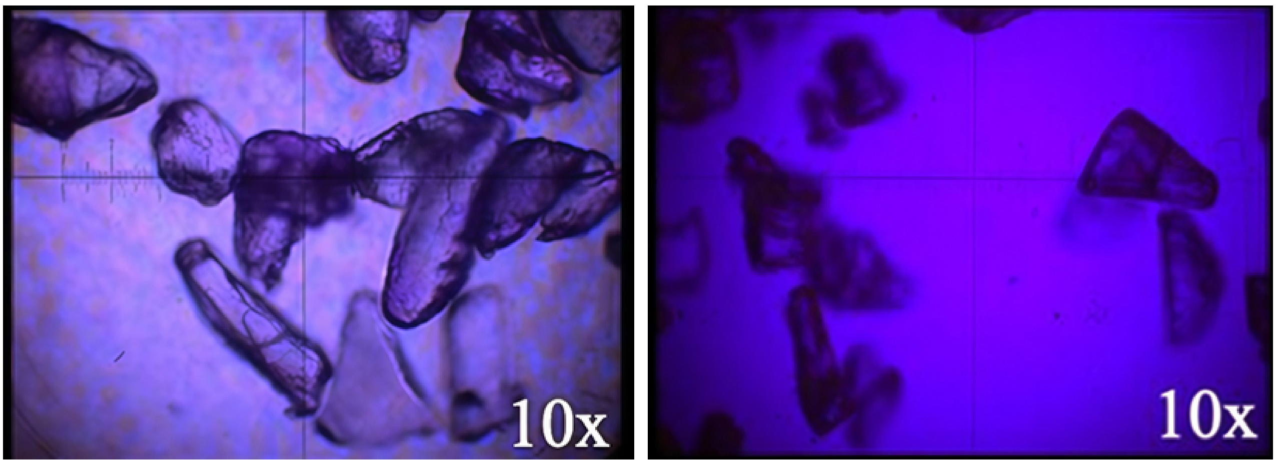

Figure 2. Optical microscopy results at 10× magnification

The schematic of the thermochemical production of carbon materials from rice husk is shown in *Figure 1* [11] and reflects the sequence of the main process stages. The raw material — rice husk — is preliminarily treated with a potassium hydroxide solution to remove mineral impurities, followed by dehydration in a fume hood. Subsequently, carbonization is carried out in a muffle furnace, leading to the formation of a porous carbon structure.

The study of the microstructural surface of carbonized rice husk makes it possible to determine its functional properties when used in pyrotechnic compositions. One of the most effective methods for analyzing surface structure is optical microscopy (OM). This method allows for quantitative morphological analysis and the measurement of linear parameters of the surface microstructure of solid materials.

The structure of the carbonized material was examined using optical microscopy at ×10 magnification. The micrographs revealed a heterogeneous surface with elements of a porous structure. Areas with varying carbon material density were observed, indicating incomplete carbonization in certain zones. Optical analysis also identified microcracks and channel-like formations, which are characteristic of plant-derived carbons.

3. Experimental Section

3.1 Impregnating Agents in Carbon Blocks

The range of substances with antiseptic properties that can be employed as impregnating agents is quite broad. However, their intrinsic characteristics impose limitations on their use in air purification applications. One of the most widely used substances in practice is iodine. Iodine and its compounds have been applied as antiseptics for more than 150 years and are well-established agents. An iodine solution is rightly considered one of the most common antiseptics in everyday life. Nevertheless, the use of iodine-based preparations for air disinfection has not been reported in the literature.

The problem of air purification can be addressed by passing an airflow through channel filters impregnated with iodine. Activated carbon can serve as a suitable material for the fabrication of such filters, as its pores are capable of retaining solutions of antiseptic agents.
Preliminary studies revealed bactericidal activity in samples of activated carbon with different iodine adsorption levels (*Table 1*).

Table 1. Iodine sorption from the gas phase at 25 °C

Initial mass of sorbent, g	Mass of sorbent + I_2, g					
	10 days	20 days	30 days	40 days	50 days	60 days
10,03	15,6	17,5	18,85	19,44	19,44	19,53
10	15,56	17,54	18,91	19,33	19,43	19,52
10,05	15,49	17,56	18,93	19,35	19,45	19,49

To investigate the feasibility of using channel filters made of carbon–silicon carbonized rice husk for air disinfection, the sorption of iodine vapors from the gas phase was studied.

The experiment was based on the sublimation properties of iodine. At 25 °C, the vapor pressure of iodine above its crystals in a closed volume is 41.2 Pa. Sorption of iodine vapors, carried out in a hermetically sealed desiccator, was performed on a carbon sorbent in the form of a multichannel monoblock. The sorbent exhibited a developed surface area ranging from 250 to 550 m²/g. Sorption was continued until complete saturation of the sorbent with molecular iodine (*Figure 3*).

Figure 3 shows the dependence of sorbent saturation with iodine on time, obtained at a temperature of 25 °C and an iodine vapor pressure of 41.2 Pa. Saturation is expressed in milligrams of adsorbed iodine per gram of sorbent.

As can be seen from *Figure 3*, complete saturation of the sorbent is achieved after 400 hours (16–17 days) and amounts to 259 mg/g.

To determine the maximum sorption capacity of the sorbent under static conditions, a Petri dish containing iodine crystals was placed in a tightly sealed desiccator. Alongside, a glass beaker containing a sorbent sample of fixed mass was also placed inside the desiccator.

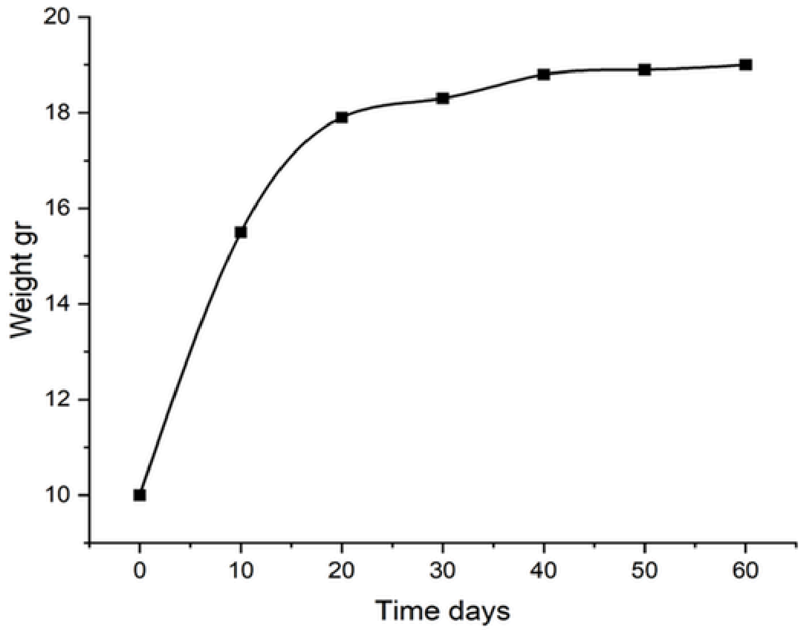

Figure 3. Dependence of sorbent saturation with iodine on time

The sorption of molecular iodine from aqueous solutions was also studied. This investigation was carried out since impregnation of the block material with aqueous iodine solutions was considered as an alternative approach. The solubility of iodine in water at 25 °C is 340 mg/L. To examine the sorption activity of the sorbent with respect to molecular iodine dissolved in water, a series of standard iodine solutions with concentrations ranging from 50 to 340 mg/L was prepared. The concentrations of the prepared solutions were verified by titration with 0.01 M sodium thiosulfate solution in the presence of starch as an indicator.

Subsequently, 100 mL of each solution was transferred into 250 mL conical flasks, brought into contact with 1 g of sorbent, and after sorption equilibrium was established, the residual iodine content in the solutions was determined by titration under the same conditions. Based on the results obtained, the mass of adsorbed iodine and the degree of its removal from the solutions were calculated.

To determine the maximum sorption capacity of the aluminosilicate sorbent with respect to molecular iodine, potassium iodide solutions were used, in which the solubility of iodine is significantly higher. Sorption was carried out from solutions with different iodine significantly higher. Sorption was carried out from solutions with different iodine concentrations.

Based on the titration data obtained, the adsorption of iodine per 1 g of sorbent was calculated, and the Langmuir adsorption isotherm was derived as the dependence of adsorption on the equilibrium iodine concentration in the solution after sorption equilibrium was established (*Figure 4*).

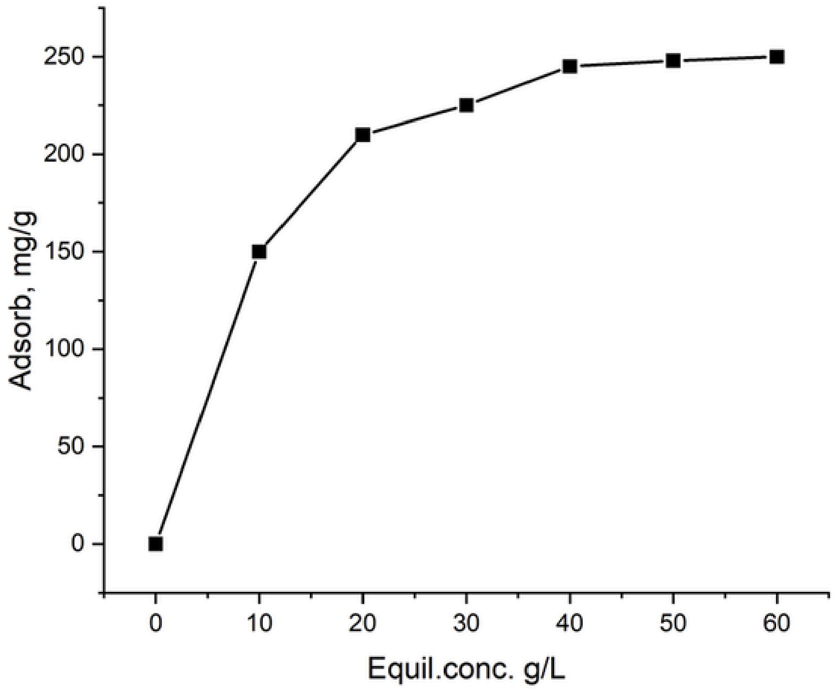

Figure 4. Iodine adsorption isotherm from aqueous solutions

Chlorhexidine gluconate solution and tannin powder were selected as impregnating agents. The effect of the carbonization process on the specific surface area and specific pore volume of the initial sorbent was investigated. It was established that carbonization enables the production of stronger carbon sorbents with a high specific surface area.

Measurements of the specific surface area and pore size of impregnated carbon–silicon sorbents were carried out. The study of the influence of impregnating agents on the sorption characteristics of the carbon–silicon sorbent showed that increasing the concentration of chlorhexidine and tannin led to an increase in both the specific surface area and the specific pore volume of the sorbents. It was established that impregnation of the carbon sorbent resulted in an increase in specific surface area from 320 m^2/g to 350 m^2/g and in specific pore volume from 0.1256 cm^3/g to 0.1399 cm^3/g.

To obtain information about the porous structure of the filter block material, the method of low-temperature nitrogen adsorption was employed. The study was carried out on a 3H-2000PS instrument after preliminary conditioning of the samples at 200 °C under a residual pressure of less than 0.001 mmHg. Nitrogen adsorption isotherms were measured at 77 K within a relative pressure range of 0.005 to 0.991, followed by standard analysis using the Brunauer–Emmett–Teller (BET) method. The parameters determined included the total pore volume (V_Σ), micropore volume ($V\mu$), and average pore diameter (Davg, accounting for both micro- and mesopores). The capability of this method to determine pore size distribution is limited to the micro- and mesopore regions. The measurement error for this method is estimated at 10%. The total surface area of micro- and mesopores was found to be 551 m^2/g.

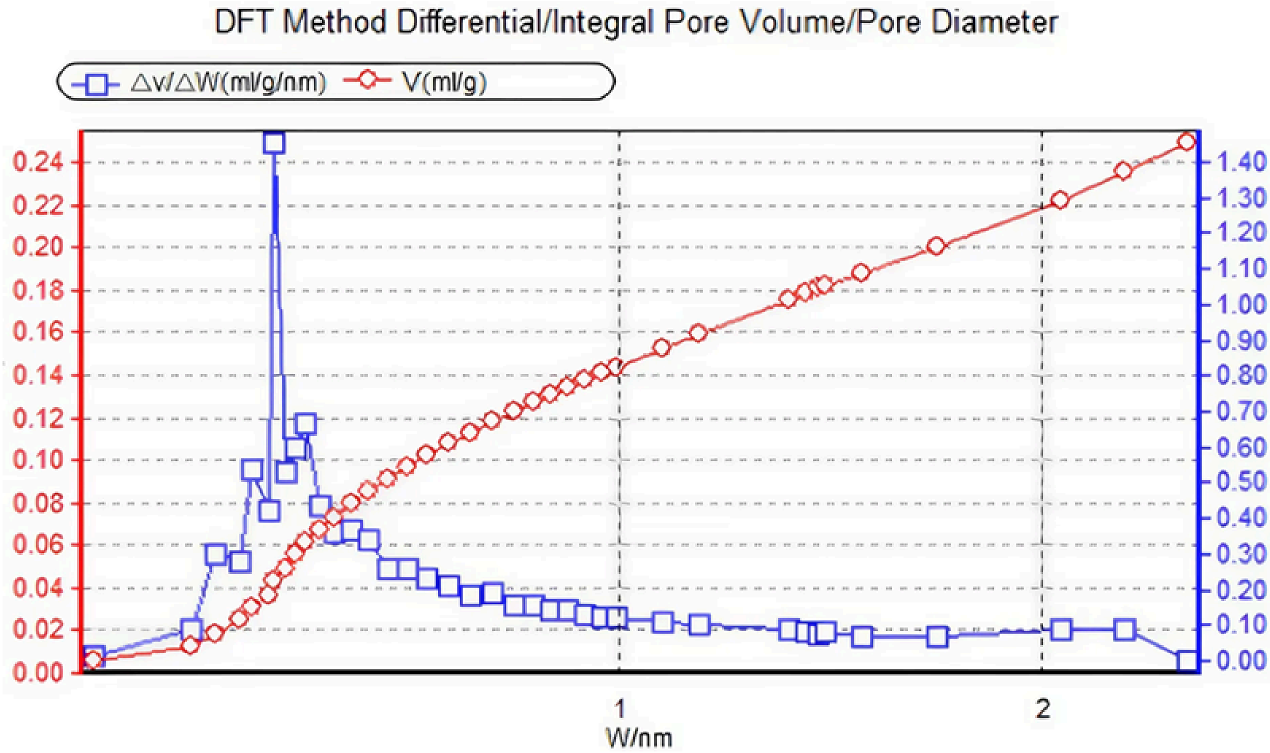

Figure 5. Adsorption isotherm of the monoblock sample obtained by the differential–integral method

According to the measurement results, the micropore volume (≤1.69 nm) was 0.2007 mL/g. The total pore volume (≤2.54 nm) was 0.2492 mL/g. The volume of all pores, including macropores, was measured using the water impregnation method. Although this method is not considered standard, it most closely corresponds to the conditions of the intended application of the filters. The prepared filters were weighed, immersed in water, and then subjected to vacuum to ensure complete impregnation. The total pore volume was determined to be 0.545 mL/g. Thus, upon impregnation, the filter is capable of retaining an amount of dissolved impregnating agent equivalent to its own mass.

4. Conclusion

As a result of the present work, a technology for producing carbon–silicon sorbents from rice husk with subsequent impregnation by antiseptic agents has been developed and tested. It was established that the combination of carbonization and activation processes enables the formation of a material with a high specific surface area and a well-developed porous structure, suitable for capturing and retaining antiseptic substances. Impregnation of the sorbents with iodine, chlorhexidine, and tannin led to an increase in sorption capacity and improvement of the textural characteristics of the material.

The fabricated filtering monoblocks demonstrated a high capacity for absorbing iodine vapors from both gaseous and liquid phases, confirming their potential for application in air purification systems with antiseptic properties. The results presented here may be utilized in the development of effective filtering elements for medical, sanitary, and military applications.

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