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PRODUCTION OF CARBON-BASED ADSORBENTS FROM WALNUT SHELLS AND THEIR PHYSICOCHEMICAL PROPERTIES

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Abstract
This study presents the production of carbon-based adsorbents from walnut shells through thermal pyrolysis and steam activation. The process was conducted in an oxygen-free environment at temperatures between 300 and 800°C to form a porous carbon structure. The influence of temperature and duration on yield, moisture, and ash content was analysed. Experimental results indicated that the optimal carbonisation temperature was 500°C, producing activated carbon with high adsorption capacity. Elemental and X-ray analyses confirmed the presence of calcite, microcline, and quartz minerals in the ash composition. The obtained adsorbents demonstrated favourable physicochemical and sorption properties, making them suitable for industrial wastewater purification and environmental protection applications. The research provides a cost-effective and eco-friendly approach to converting local agricultural and food waste into valuable adsorptive materials.
Keywords: Activated carbon; Walnut shell; Thermal pyrolysis; Steam activation; Carbonisation; Physicochemical properties; Adsorption; Waste valorisation

Introduction

The increasing generation of agricultural and food waste has created an urgent need for sustainable recycling methods that contribute to both environmental protection and resource efficiency. Among various waste-to-resource technologies, the conversion of lignocellulosic biomass into carbon-based adsorbents has gained significant attention due to its potential applications in wastewater treatment, gas purification, and catalysis. Walnut shells, which are abundant and inexpensive agricultural by-products, possess a high carbon content and a dense lignin–cellulose structure, making them a promising precursor for the production of activated carbon materials.

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Thermal pyrolysis and subsequent steam activation are among the most effective techniques for producing porous carbon materials with high surface area and strong adsorption capacity. During these processes, biomass is subjected to heat treatment in an inert atmosphere, resulting in carbonisation and the development of a porous microstructure. The physicochemical properties of the obtained adsorbents depend on the process temperature, residence time, and activation conditions.

This study focuses on obtaining carbon-based adsorbents from walnut shells using thermal pyrolysis under an argon atmosphere and investigating the influence of temperature and duration on yield, moisture, and ash content. The structural and compositional characteristics of the resulting materials were analysed using X-ray diffraction and elemental analysis. The research aims to optimise processing conditions for producing high-quality activated carbons suitable for environmental purification and industrial applications.

Materials and methods

Thermal pyrolysis and steam activation methods play a crucial role in the production of carbon-based adsorbents from local food and agricultural waste. In this study, the initial stages of pyrolysis and carbonisation were carried out using walnut shells as a raw material to obtain activated carbons with high adsorption capacity. The process involved thermal treatment of biomass under atmospheric pressure at temperatures ranging from 300 to 800°C in an oxygen-free environment. Such conditions promote the formation of a porous carbon structure, which significantly enhances the adsorption properties of the final product.

The parameters of the process, experimental setup, and the effect of temperature on the physicochemical properties of the obtained carbons were investigated in detail. The methodology provides insight into the optimal conditions and mechanisms required to convert biomass-derived food waste into high-value adsorptive materials.

Figure 1 presents a schematic block diagram illustrating the physical and chemical stages of the activated carbon production process from walnut shells. Among the carbonised samples produced at different temperatures, the one obtained at 500°C was selected for further activation based on benzene vapour adsorption results. The activation was first carried out by the physical method using steam.

Natural coals generally have a complex chemical composition containing not only carbon but also trace elements such as gold, aluminium, uranium, and germanium [1, pp. 75–76].

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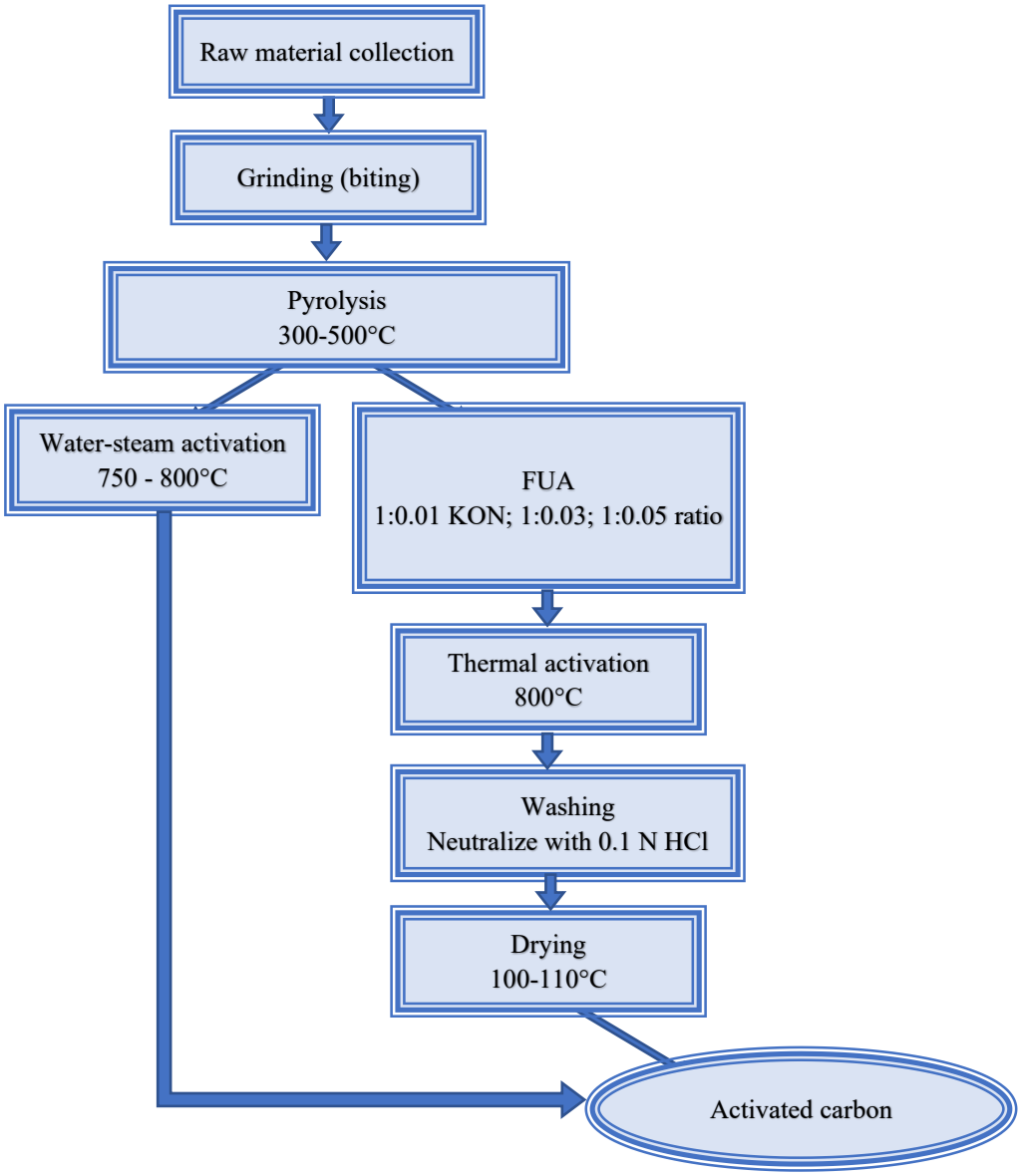

Figure 1. Block diagram of the process for obtaining activated carbon from walnut shells.

The technology for producing clean and cost-effective activated carbon adsorbents from plant-based raw materials such as coconut shells, peach pits, and other agricultural residues has been described in previous studies [2, p. 306]. Elemental analysis was performed to examine the morphology and structure of the obtained carbon materials, while the sorption and operational characteristics of the resulting adsorbents were also investigated.

Laboratory experiments were carried out using a stainless-steel pyrolysis reactor designed for thermal treatment, with a height of 1 m and a diameter of 0.055 m (Figure 1). The pyrolysis process was conducted under an argon atmosphere with a controlled gas flow rate of 40 ± 1 mL/s.

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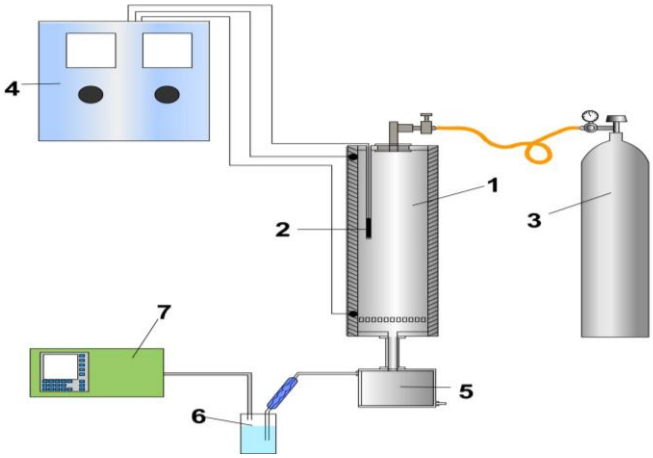

Figure 2. Laboratory experimental pyrolysis setup.
1 – Pyrolysis furnace; 2 – Thermocouple; 3 – Argon (nitrogen) inlet; 4 – Electronic control unit; 5 – Tar collector; 6 – Condensate collector; 7 – Gas analyser.

The operating principle of the experimental setup is as follows. Initially, 500 g of dry raw material was placed on a metal mesh fixed to the internal supporting frame of the pyrolysis furnace (1). The furnace lid was then tightly sealed using a reinforced plug to ensure complete insulation. Argon gas was introduced through a flow meter and pressure control valve to create an inert atmosphere inside the reactor. Subsequently, the thermocouple (2) for temperature monitoring was inserted into a designated section of the furnace, and the power supply (4) was switched on.

A slow heating rate was selected for the activation process, and pyrolysis was initiated at a heating rate of 5°C per minute. The initial temperature was 24°C, with a current of 30 V, which was sufficient to raise the temperature to 300°C. As the temperature increased by every 100°C increment, argon gas was supplied to maintain atmospheric stability. For each experimental run in the range of 400°C to 800 ± 20°C, the heating intensity, final temperature, reaction time, and yield of the expected product were carefully monitored.

During pyrolysis, volatile and liquid by-products, mainly tars, were released through special exhaust outlets of the reactor and collected in the tar trap (5). The volatile gases and condensates exiting the tar trap were collected in the condensate trap (6) and subsequently analysed using a gas analyser (7) [102, pp. 1–5].

The influence of temperature and duration on the physicochemical and adsorption characteristics of the activated carbonised materials was investigated. It was observed that increasing the temperature from 300°C to 800°C gradually decreased the yield. This behaviour is attributed to the decomposition of carbon monoxide and carbon dioxide and partial oxidation of the carbonised biomass. The moderate reduction in yield indicates that the process remains economically feasible for the production of activated adsorbents.Images of the carbonised carbon-based adsorbents are presented in Figure 3.

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A) B) C)

A – Walnut shell; B – Crushed sample; C – Biomass-derived

Figure 3. Images of carbon-based adsorbents obtained from walnut shells charcoal

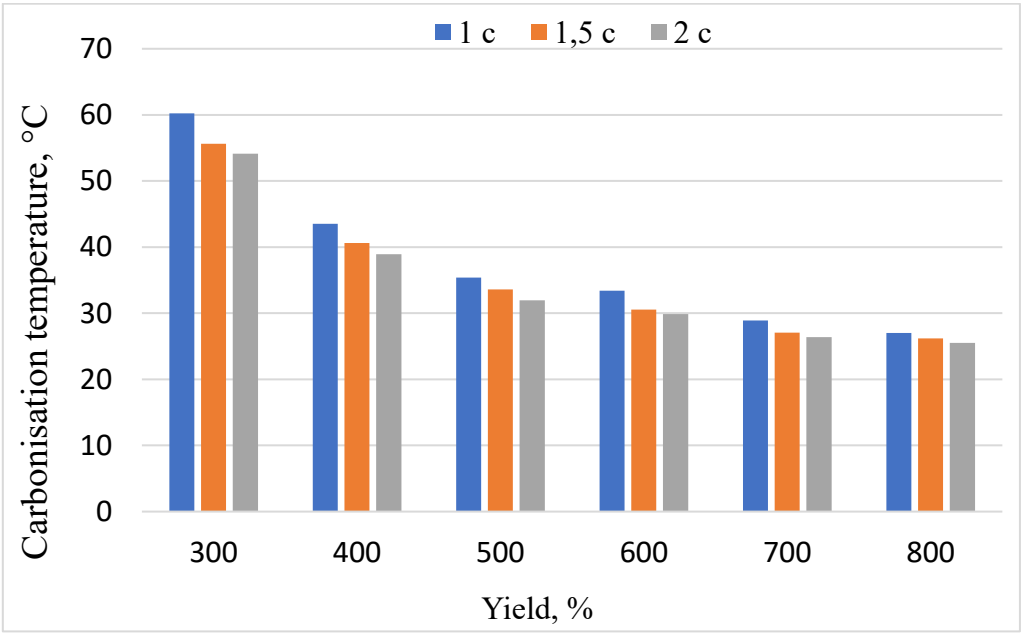

Figure 4. Effect of temperature on yield during the carbonisation process.

From the diagram, the blue column representing the sample carbonised for one hour demonstrates the lowest mass loss compared to the others. This indicates that a duration of one hour is insufficient to obtain the expected carbonised product. The brown (1.5 h) and grey (2 h) columns display nearly similar values, suggesting that a carbonisation duration of 1.5 hours is the most appropriate for walnut shell feedstock. This duration provides sufficient time for the decomposition and removal of organic components within the shell structure.

The moisture content (W_a, %) of the thermally pyrolysed samples was determined using an MA 210.R analytical device. For this purpose, 1 g of each carbonised sample obtained at various temperatures, with particle sizes smaller than 0.2 mm, was weighed into pre-dried glass containers using an analytical balance. Each sample was dried at a temperature of 105–110°C for three hours. Subsequently, the crucibles containing the coal samples were placed in a desiccator, and after 30 minutes they were reweighed using the analytical balance.

The moisture content was calculated using the following formula (%):

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$$W = \frac{m_1 - m_2}{m_2 - m_3};$$

Where: m_1 — mass of the glass container with the carbonised sample before drying, g; m_2 — mass of the glass container with the carbonised sample after drying, g; m_3 — mass of the empty dried glass container, g [103, p. 3].

The ash content ($A_{(c)}$, %) was determined in accordance with GOST 11022-95 [4]. Ash composition is one of the most important indicators for all types of carbon adsorbents. Crucibles selected for the analysis were first brought to constant mass. For this purpose, they were heated in a muffle furnace at (815 ± 10) °C, maintained for 15 minutes, then switched off and cooled. After cooling, the crucibles were placed in a desiccator.

Analytical samples of the carbonised material were ground to a particle size passing through a 200 μm (0.2 mm) sieve. Before starting the analysis, each sample was thoroughly mixed mechanically for 1 minute. Crucibles previously brought to constant mass in the muffle furnace were weighed using an analytical balance, the crucible mass was recorded, then the test sample was placed inside and re-weighed. Two crucibles were used for each sample, and the difference between parallel measurements was kept within the permissible limits.

The crucibles containing the samples were gradually heated in the muffle furnace to (800 ± 10) °C and held at this temperature for 1.5 hours until constant weight was achieved [105, pp. 136–140]. After cooling, the samples were re-weighed, and the ash content $A_{(c)}$ (%) was calculated according to the following formula:

$$Ac = \frac{m*100*100}{m_1\left(100 - x_1\right)}H$$

where: m — mass of the residue, g; m_1 — mass of the test sample taken for analysis, g; x_1 — moisture content of the test sample, %.

To determine the elemental composition and concentration of components in the ash, **X**-ray diffraction (XRD) analysis was carried out.

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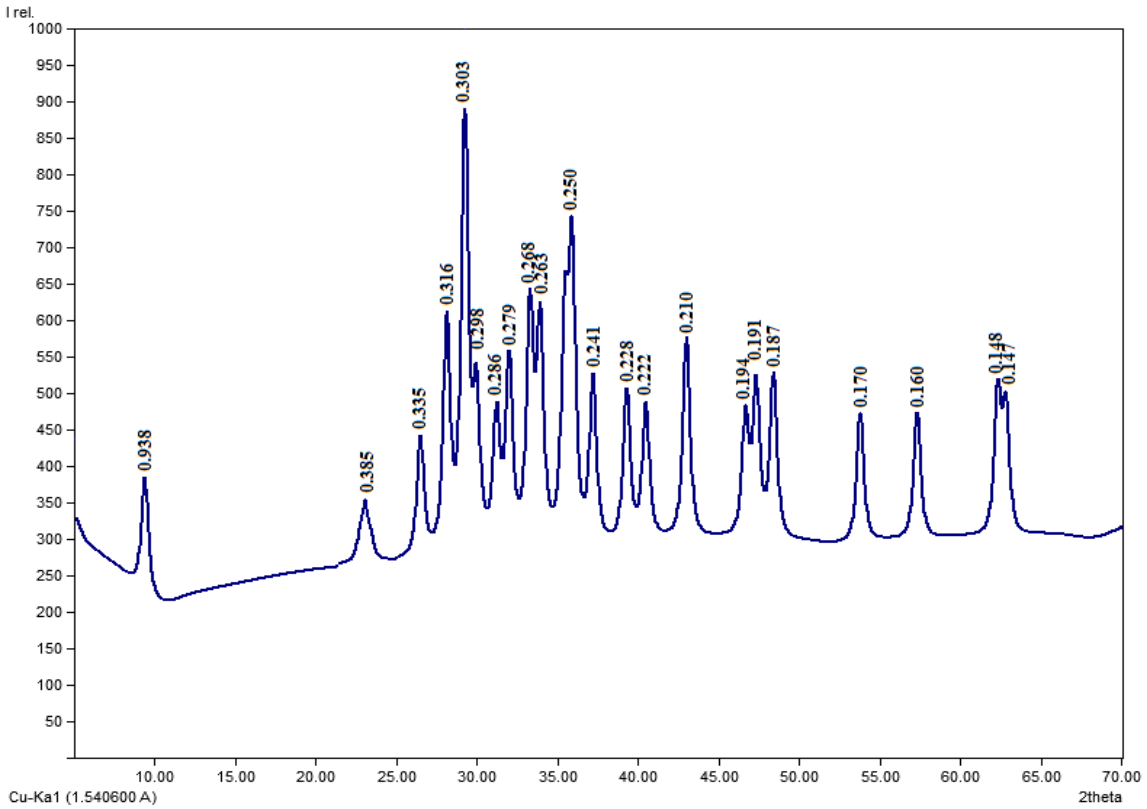

Figure 5. X-ray diffraction (XRD) pattern of walnut shell ash.

The X-ray diffractogram of the ash obtained from walnut shells revealed diffraction maxima characteristic of several minerals, primarily **calcite** (d = 0.385, 0.303, 0.286, 0.250, 0.228, 0.210, 0.194, 0.191, 0.187, 0.160, 0.148, 0.147 nm), microcline (d = 0.316, 0.298 nm), muscovite (d = 0.957, 0.277, 0.241, 0.170, 0.147 nm), and a smaller quantity of quartz (d = 0.335, 0.228, 0.210 nm).

The diffractogram indicates that the diffraction maxima and peak intensities corresponding to calcite are significantly higher than those of the other minerals, suggesting its quantitative predominance in the ash composition.

Within the framework of this research, the transformations and characteristics associated with the carbonisation process were analysed in detail.

Table 1. Effect of carbonisation temperature on mass loss (pyrolysis duration: 1.5 hours).

T, °C	Product yield, %			Moisture content, %	Ash content, %
	Coal	Tar	Gaseous		
300	55,61	11,2	10,7	3,772	1,21
400	55,61	15,4	43,97	3,561	1,32
500	33,61	21,8	44,59	3,475	1,34

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600	30,57	22,3	44,59	2,902	1,45
700	27,07	12,4	44,59	2,896	1,58
800	26,21	10,7	63,09	2,702	1,91

The results of the study showed that the release of tar and gaseous compounds from the coal was at its maximum within the temperature range of 500–600°C, while a decrease was observed at higher temperatures. These findings indicate that controlling the duration and temperature of activation allows optimisation of the physicochemical and adsorption properties of the desired activated carbon. This also provides a basis for selecting suitable physical and thermochemical processing conditions during the study.

The scientific findings presented in this work suggest that the obtained adsorbents can be effectively applied in the purification of industrial and petroleum wastewater. Moreover, the proposed method offers a foundation for developing environmentally friendly technologies for the utilisation and recycling of organic waste materials.

Conclusions

This research demonstrated that walnut shells can serve as an efficient and low-cost raw material for producing carbon-based adsorbents through thermal pyrolysis and steam activation. The optimal carbonisation temperature was determined to be 500–600°C, at which the release of volatile tar and gaseous compounds was most intense, forming a highly porous carbon structure. The obtained activated carbons exhibited favourable physicochemical and adsorption characteristics suitable for wastewater purification. X-ray analysis confirmed calcite as the predominant mineral phase in the ash composition. The findings indicate that the developed method not only provides a sustainable approach to converting agricultural waste into valuable adsorbent materials but also contributes to environmentally safe and resource-efficient technologies for waste management.

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