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STUDY OF THE STRUCTURE AND SURFACE MORPHOLOGY OF ACTIVATED CARBON FROM BLACK CUMIN SEED MEAL (*Nigella sativa* L.)

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ABSTRACT

The present study explores the potential of black cumin seed meal (Nigella sativa L.) (a by-product of industrial oil extraction) to produce activated carbon. The meal was carbonized at 500°C and chemically activated with 85 % H_3PO_4 at 500°C for 1 h. The obtained materials were characterized by Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), and elemental analysis (C, H and N). FT-IR spectra revealed the formation of a carbonized structure with surface oxygenated and phosphate functional groups. SEM micrographs provided a visual indication of a developed surface morphology with finely distributed pores; however, this observation does not substitute for a quantitative pore structure analysis such as N_2-sorption/BET. The obtained results confirm that black cumin seed meal can be used as a promising material for activated carbon production under phosphoric acid activation.

Keywords: activated carbon, black cumin seed meal, carbonization process, structural and surface characteristics.

INTRODUCTION

Black cumin (*Nigella Sativa L.*) is an annual plant of the Ranunculaceae family. It is cultivated in various parts of the world, especially in the Mediterranean, Eastern Europe, Western Asia and the Middle East [1 - 4]. Seeds of black cumin contain 37.7 % oil, 2.5 % unsaponifiable compounds, 0.9 % phospholipids, 0.2 % sterols and 177.1 mg kg^{-1} tocopherols [5]. Also they content 25 - 40 % carbohydrates, up to about 4 % ash and a minimal amount of moisture. Black cumin oil has a wide range of applications in medicine and for various therapies. When extracting the oil from the seeds of the plant, a large amount of residue is obtained, which is usually utilized in animal husbandry and agriculture [6].

Activated carbon (AC) belongs to the group of carbon materials with a developed internal and surface porous structure. The surface morphology and functional groups, as well as the type, shape and size of the pores, incl. chemical properties of AC depend on the starting material and the conditions of preparation. The ability to regulate the porous structure makes activated carbons a preferred material in various fields: a widely used sorbent for wastewater and air purification; in medical practice; for gas separation; electrode materials in electrochemical devices; for soil improvement and recently for energy and gas storage [7, 8].

Commercial activated carbons are typically produced from high-carbon feedstocks, such as wood, peat, bituminous coal, and anthracite, which are expensive, non-renewable, and of limited availability [9, 10].

The expensive traditional sources to produce activated carbon have necessitated the search for alternative, cheaper and environmentally friendly raw materials. Black cumin seed meal, which is a residual product after oil extraction, is rich in cellulose and hemicelluloses, making it suitable to produce activated carbon [7, 11]. Furthermore, the use of waste materials as a raw material helps to reduce the environmental burden (footprint) and improves the economic efficiency of the process [12]. Activated carbons are obtained through well-established steps of carbonization of the initial material and physical or chemical activation to increase the specific surface area and set the desired chemical properties of the surface [10, 13].

The aim of this work is to investigate the possibility of obtaining activated carbon from black cumin meal and determine some of its characteristics.

EXPERIMENTAL

Preparation of the initial material and production of activated carbon

After the extraction of black cumin oil, the residual seed meal was obtained as an industrial by-product in the form of extrudate, which was dried at 105°C. Since the material still contained residual oil, it was ground, homogenized, and subjected to an additional Soxhlet extraction with petroleum ether (p.a., Valerus, Bulgaria) at approximately 70°C to remove the remaining lipids. The defatted meal was then dried and stored under ambient conditions for further use.

Activated carbon was produced from black cumin seed meal through a carbonization-activation process. Approximately 30 g of the meal was pyrolyzed at 500°C for 1 h. The carbonized material was then impregnated with phosphoric acid (85 % H_3PO_4) in a 1:2 (biomass : H_3PO_4) ratio. After impregnation, 10 cm^3 of distilled water was added to the mixture under stirring. The impregnated sample was dried in a vacuum oven at 180°C for 4 h and subsequently activated in a tubular furnace at 500°C for 1 h under vacuum. The activated product was washed repeatedly with distilled water until neutral pH and dried again at 110°C for 12 h. The activation temperature of 500°C was experimentally determined as optimal for the formation of a stable and well-developed porous structure [14].

In this work, results are shown for the following materials used in the experimental work process (Table 1).

Characterization of the obtained materials

The determination on some of characteristics of the studied materials was carried out using the following techniques and methods:

Fourier transform infrared spectroscopy (FT-IR) to determine the surface functional groups was performed on a Varian 660 FT-IR Spectrometer (Agilent Technologies, Inc, USA), with the Resolution Pro® software product. The IR spectra were recorded in the mid - IR region (4000 - 400 cm^{-1}) in transmission mode, resolution 4 cm^{-1} from tablets with spectrally pure KBr.

Elemental analysis for determination of the percentage of C, N and H was performed with a Euro EA 3000 apparatus (Euro Vector Sp A, Italy). Samples with a mass of 1.5 mg were burned at 980°C in an oxygen atmosphere. The resulting reaction mixture of N_2, CO_2 and H_2O gases was passed through a chemical/absorption zone, and the individual components of the mixture were separated with a gas chromatography column with TCD detection. The standard curve was made with acetanilide CAS: (CPA Chem) 103 - 84 - 4 (C_8H_9NO) with the computer product Callidus.

Scanning electron microscopy - energy-dispersive X-ray spectroscopy (SEM-EDS) analysis to determine the surface morphology and porous structure was performed with SEM Zeiss EVO 10 (Carl Zeiss Microscopy GmbH, Germany), image processing was performed with SmartSem Version 7.05 with Service pack 3 software. Surface elemental composition was determined with EDS attachment Oxford Instruments, Xplore 30 (Halifax Rd, UK) with EDS software: AztecOne, resolution of 5.9 KeV: 129 eV. Nitrogen 5.0

Table 1. Samples used in the experiments.

Type of the sample	Designation
Initial black cumin meal (extrudate)	1
Defatted black cumin meal after additional extraction	2
Carbonized sample 1 (without additional extraction)	3 - 1
Carbonized sample 2 (after additional extraction)	3 - 2
H_3PO_4-activated sample 3-1	4 - 1
H_3PO_4-activated sample 3-2	4 - 2

(≥ 99.999 %), compressed gas, ADR 2, 1A, CAS № 7727 - 37 - 9, EC № 231 - 783 - 9 (Messer, Bulgaria) was used for the camera.

RESULTS AND DISCUSSION

Fourier transform infrared spectroscopy (FT-IR)

Fig. 1 presents the comparative FT-IR spectra of the starting (1) and extracted cumin meal (2) and of the activated carbons obtained from it by carbonization (3 - 1 and 3 - 2) and activation with phosphoric acid (4 - 1 and 4 - 2). The obtained spectra reveal the functional groups on the surface of the porous material which are largely due to both the cumin waste used for the preparation of the activated carbon and the activating agent used.

The FT-IR spectrum of the initial cumin meal (sample 1, Fig. 1) recorded the presence of oxygen-containing and amide groups from the starting material and residues of the essential oil from the processed cumin seeds. The broad absorption band at ~ 3300 cm^{-1} corresponds to the stretching vibrations of -OH groups, which overlap with the stretching vibrations of the amide groups (-NH_2) in the seed residues. The absorption bands at 2924 and 2853 cm^{-1} correspond to the stretching vibrations of -CH_3 and -CH_2 groups. The band at 1711 cm^{-1} reflects the stretching vibrations of the -C=O group, and the absorption bands at 1645 and 1539 cm^{-1} correspond to the vibrations of Amide I and Amide II from the protein structure of the cumin meal. The band at 1456 cm^{-1} is attributed to the deformation vibrations of -CH groups and at 1416 cm^{-1} to -COOH, which overlaps with the deformation vibrations of -OH groups. The absorption peak at 1248 cm^{-1} is associated with stretching vibrations of the -C-O bonds. The band at 1061 cm^{-1} is caused by stretching vibrations of the C-O-C bond [4, 16] and at 720 cm^{-1} reflects the deformation vibrations of -CH groups [15].

After the re-extraction of the black cumin seed meal in the FT-IR spectrum of the residue (sample 2, Fig. 1) an increase in the intensity of the broad band at ~ 3300 cm^{-1} for hydroxyl groups and the bands at 1652 and 1539 cm^{-1} for Amide I and Amide II is observed, and the band of the carboxyl group at 1711 cm^{-1} is hinted at as a shoulder in the spectrum. The absorption also increases at ~ 1054 cm^{-1}, which indicates an additional degree of purification of the material structure. The strong broadening of the band of hydroxyl groups is probably also due to absorbed moisture in and on the surface of the residue of the meal after the extraction of the residual oil, since it was not subjected to drying before taking the spectrum. The absorption bands in the spectra indicate the presence of many oxygen-containing groups and amide groups on the surface of the extracted cumin seed meal (2).

After the carbonization of the cumin meal (samples 3 - 1 and 3 - 2, Fig. 1), it is seen that the intensity of the absorption bands of -OH and -CH groups in the high frequency region decreases strongly, and the band of the amide group is not registered. In the spectra of the carbonized and activated samples, a broad band appears

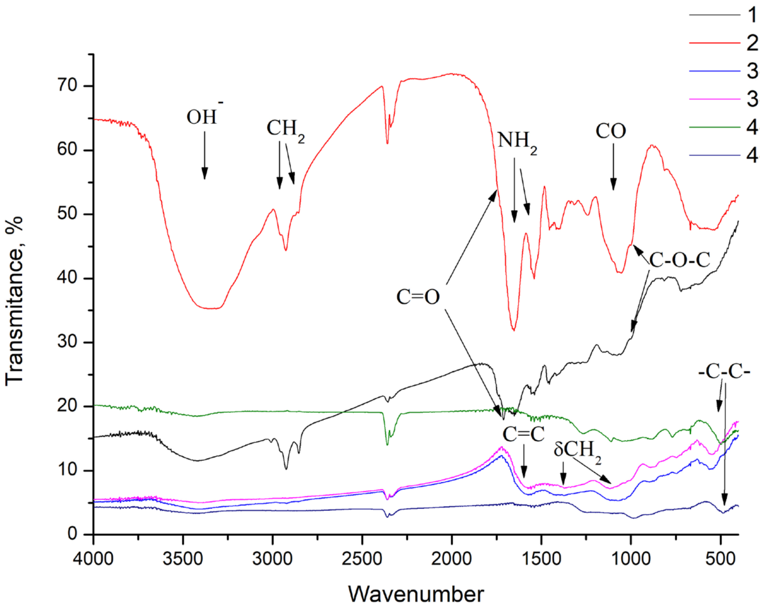

Fig. 1. Comparative FT-IR spectra of raw cumin seed meal (1), defatted cumin meal after re-extraction (2), carbonized cumin meal (3 - 1), carbonized defatted cumin meal (3 - 2), and the corresponding activated samples obtained after phosphoric acid activation (4 - 1 and 4 - 2)

at ~ 1570 cm^{-1}, which corresponds to sp^2 hybridized C=C bonds of the carbon structure [10, 23]. This is an indicator of the carbonization of the cumin meal and the reorganization of the surface functional groups. The bands of the stretching vibrations of the -CO groups in the interval 1110 - 1030 cm^{-1} are also observed, but the absorption peak is quite broadened [16]. Weak bands are also recorded in the region of the deformation vibrations of -CH groups ~ 1372 cm^{-1} and 900 - 700 cm^{-1}, and the more pronounced band at ~ 550 cm^{-1} is due to skeletal -C-C - vibrations of the formed carbon structure [8, 17, 18].

In the FT-IR spectra of the samples obtained after activation with H_3PO_4 (samples 4-1 and 4-2, Fig. 1) the absorption bands of the amide group are absent, and the bands for -CO groups at ~ 1269, 1110 and 1046 cm^{-1} are weakly implied in sample 4 - 1, but are not registered in the spectrum of sample 4 - 2.

This region (1269 - 1046 cm^{-1}), initially attributed to C=O and C-O-C stretching vibrations typical of lignocellulosic residues, may also introduce phosphorus-containing functional groups, whose P=O, P-O-C or P-O-P vibrations can overlap with the same spectral range [15, 16]. The broad and low-intensity character of these bands suggests superposition of carbon-oxygen and phosphorus-oxygen modes, which, together with the amorphous structure of the material, results in diffuse absorption features rather than distinct phosphate peaks. The presence of phosphorus-containing species is independently confirmed by SEM-EDS analysis, which shows about 36 wt. % P in both activated samples, consistent with the chemical interaction of phosphoric acid with the carbon matrix. In the spectra of both samples, deformation vibrations of -CH groups below 1000 cm^{-1} are registered.

In general, the spectra of the samples obtained from carbonized and activated with H_3PO_4 black cumin seed meal (samples 4 - 1 and 4 - 2) are typical IR spectra of functionalized carbon. The differences reported in the spectra after carbonization and activation of the samples are due to the nature of the activator, the activation conditions and the nature of the interactions between the carbonized mass and the activator [8].

Scanning electron microscopy - energy-dispersive X-ray spectroscopy (SEM-EDS)

The morphology and type of the porous structure, as well as the elemental composition of the obtained activated carbons were investigated by SEM-EDS (Fig. 2 - 7).

The SEM-EDS image of the initial cumin meal (sample 1, Fig. 2) shows an irregular surface morphology with isolated pores. The elemental composition clearly shows the presence of various elements in the sample, which are indicative of the biological origin of the initial material and the presence of residual oil. High carbon content (61 %) is reported, while the oxygen content (21.30 %) confirms the presence of many surface oxygen-containing groups.

Fig. 3 shows that after the additional extraction of the residual oil from the cumin meal (sample 2) the inhomogeneity of the sample surface deepens, with some of the pores expanding and growing into cracks. The extraction of the oil from the biomass leads to the destruction of the cell wall and the formation of the observed texture and differences in morphology [20]. The elemental composition of the sample shows a high carbon content. The oxygen content on the surface also remains unchanged (21.27 %), and many of the accompanying elements (Na, Mg, Si, P, S, Cl, Fe, Br) are removed from the structure of the cumin residue.

After carbonization of the initial sample (sample 3-1, Fig. 4), the formation of an inhomogeneous structure with an uneven distribution of small pores of different shapes and isolated larger voids on the surface is noted. In addition to the high carbon content (55.23 %), an increase in the oxygen content to 42 % is noted, most likely due to the change in the type and shape of the pores, revealing a larger number of oxygen-containing groups than the internal structure.

In sample 3 - 2 (Fig. 5) a clearly formed carbon structure is observed, characteristic of the sp^2

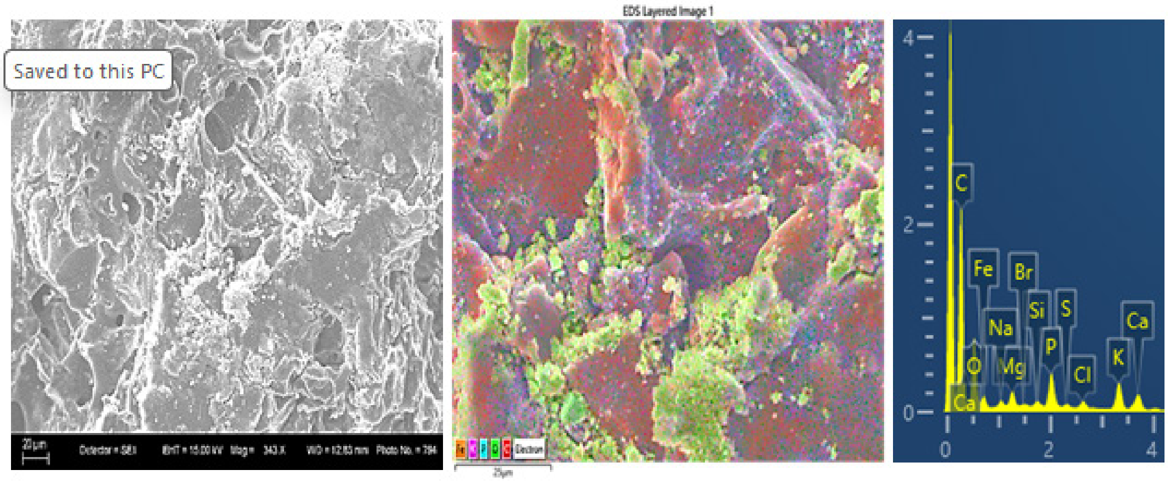

Fig. 2. SEM-EDS micrograph and surface elemental composition of the initial black cumin seed meal (sample 1).

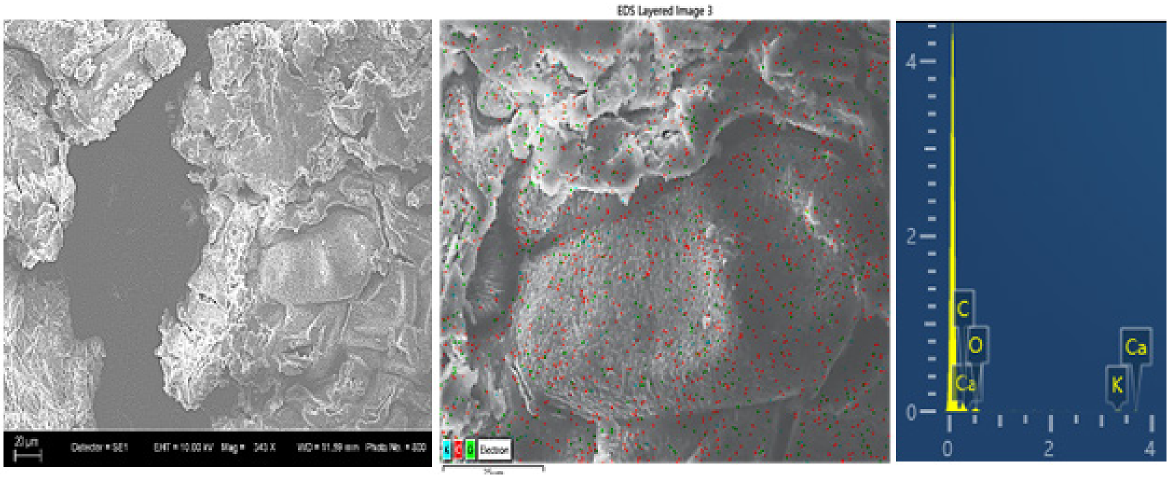

Fig. 3. SEM-EDS micrograph and surface elemental composition of the defatted black cumin seed meal after re-extraction with petroleum ether (sample 2).

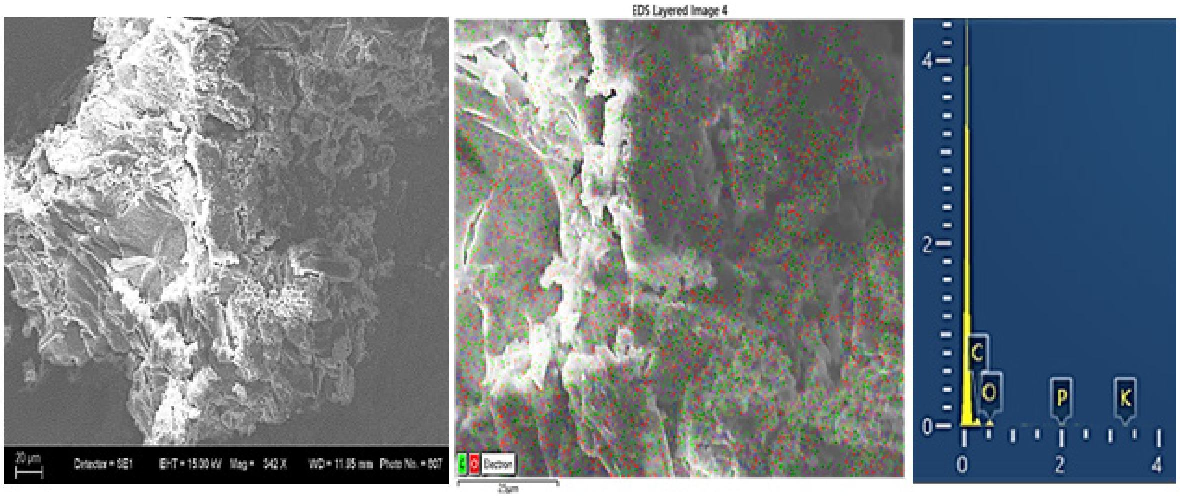

Fig. 4. SEM-EDS micrograph and surface elemental composition of carbonized black cumin seed meal without additional extraction (sample 3 - 1).

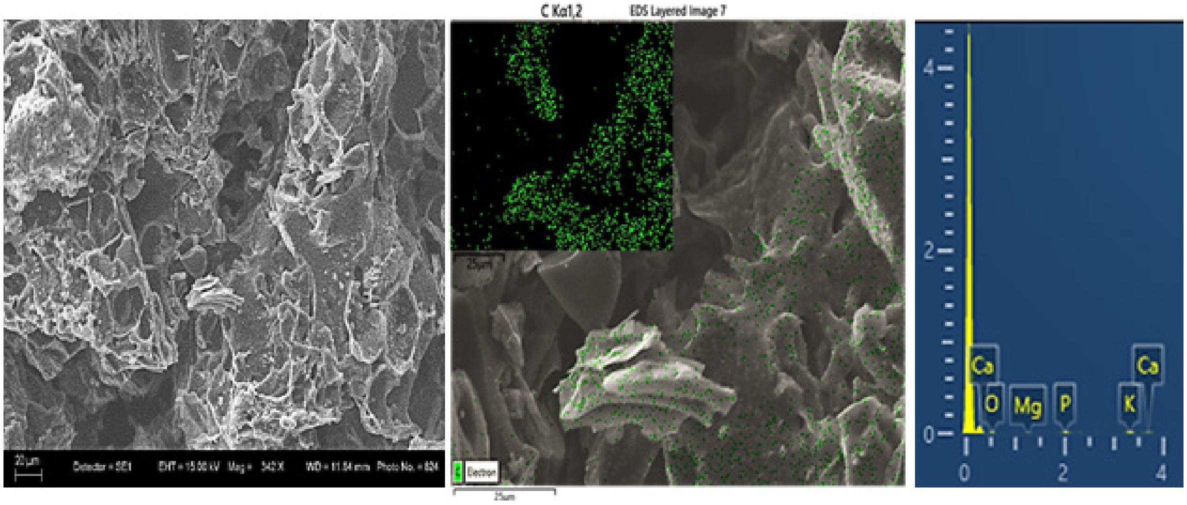

Fig. 5. SEM-EDS micrograph and surface elemental composition of carbonized black cumin seed meal after additional extraction (sample 3 - 2).

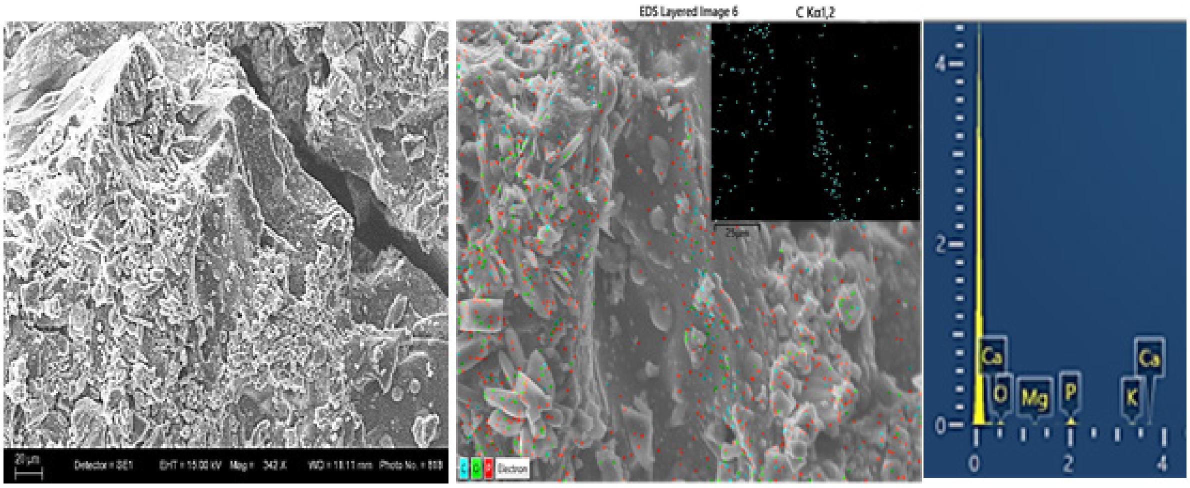

Fig. 6. SEM-EDS micrograph and surface elemental composition of the activated carbon obtained from sample 3-1 after H_3PO_4 activation (sample 4 - 1).

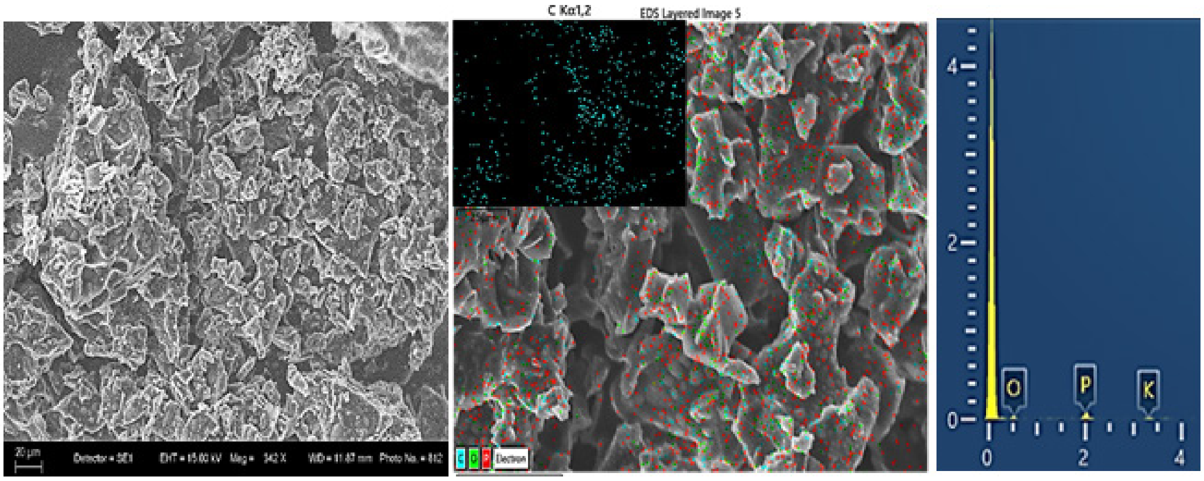

Fig. 7. SEM-EDS micrograph and surface elemental composition of the activated carbon obtained from sample 3 - 2 after H_3PO_4 activation (sample 4 - 2).

configuration of carbon atoms [10]. The distribution of pores on the surface is relatively uniform and visually an approximately equal average size is observed. EDS analysis shows a high content of carbon and oxygen, and a moderate presence of phosphorus (14.17 %) in the structure of the sample.

SEM images of samples 4 - 1 and 4 - 2 activated with H_3PO_4 (Fig. 6 and 7) present visually porous surfaces with small pores, more evenly distributed in sample 4 - 2. In this case, the interaction of H_3PO_4 with the carbonized samples probably contributes to the formation of a microporous structure with a relatively uniform distribution of pores. This is due to the ability of phosphoric acid to degrade and restructure the material at a very fine level. It penetrates the structure and causes swelling of the cell walls, which expands the already existing pores and creates new micropores [9, 21, 22]. During the thermal treatment (at temperatures of 500°C), phosphoric acid catalyses the decomposition of organic compounds and contributes to the formation of a stable porous structure. The acid also acts as a dehydrating agent, causing the formation of cross-linking in the carbon matrix.

EDS analysis of the samples shows that the structure is markedly carbon-rich and high in oxygen, indicating the presence of oxygen-containing, likely phosphate groups on the surface. The increase in phosphorus content from 14.17 % in the carbonized sample 3 - 2 to significant amounts of ~ 36.5 % in both activated samples suggests that phosphoric acid is actively involved in the formation and modification of the structure, integrating at a fine level into the developed surface of the samples [8, 9, 21].

The appearance of phosphorus in the carbonized sample (3 - 2) may be attributed either to trace phosphorus species naturally present in the cumin meal or to partial cross-contamination during carbonization. In contrast, the markedly higher P content in the activated samples (4 - 1 and 4 - 2, ~ 36 wt. %) reflects the chemical incorporation of phosphate groups into the carbon matrix during activation with H_3PO_4. The activated materials were washed several times with distilled water, yet a certain fraction of phosphate species may remain bound to the surface as stable P-O-C or polyphosphate structures. As SEM-EDS measures surface rather than bulk composition, relatively high P levels (~ 36 wt. %) are reasonable and confirm the presence of surface-bound phosphate functionalities.

The micrographs (Figs. 2 - 7) revealed noticeable differences in the texture and compactness of the carbon materials obtained at the different processing stages.

SEM-EDS analyses of the studied samples visually present the formation of a microporous structure with a uniform distribution of pores on the surface of the samples.

The obtained SEM-EDS results agree well with the applied activation methodology and support the conclusions drawn from FT-IR spectroscopy.

Table 2. Elemental composition (C, N, H) of samples used in the experimental works.

	Content of, %		
Sample	Nitrogen (N)	Carbon (C)	Hydrogen (H)
1	11.72	45.64	4.74
2	4.68	42.83	4.46
3-1	11.44	42.80	4.40
3-2	11.27	41.79	4.29
4-1	12.25	48.55	4.95
4-2	11.66	45.21	4.53

Elemental analysis

The results of the elemental analysis for determining the carbon, nitrogen and hydrogen content in the differently treated cumin meal are presented in Table 2. The table confirms that after the carbonization and activation of the waste from the processing of cumin seeds, activated carbons with approximately the same carbon content are obtained for all samples. The highest carbon content is in sample 4-1, activated with H_3PO_4 (~ 49 %). The carbon content in sample 4-2 (45 %) also remains high, which is close to the carbon content of the starting meal (1→ 45.64 %). The established values for the carbon content in the carbonized samples 3-1 and 3-2 are ~ 43 and 42 %, respectively. The nitrogen and hydrogen content remains approximately constant for all samples.

The relatively moderate carbon content (~ 42 - 49 %) is attributed to the incorporation of oxygen- and phosphorus-containing species during chemical activation with H_3PO_4. This is consistent with the formation of phosphate functionalities within the carbon matrix, which increase surface oxygen and phosphorus but decrease the relative carbon percentage. Similar values have been reported for phosphate-activated lignocellulosic carbons, where the residual phosphorus contributes to both acidity and surface functionality [22, 23].

The obtained results follow the trends of the results obtained from FT-IR and SEM-EDS analyses. It should be noted that the higher values for carbon obtained with EDS are due to the measurement in limited areas of the surface and do not truly reflect the overall composition of the samples.

CONCLUSIONS

The applied sequence of carbonization and chemical activation at 500°C produced carbon materials with distinct morphological development and the presence of oxygen- and phosphorus-containing functional groups. The SEM observations provided qualitative evidence of pore formation and surface texturing, while the high phosphorus content detected by EDS reflects the chemical incorporation of phosphate species during activation. Although quantitative pore structure parameters (e.g., BET surface area) were not determined, the obtained results indicate that phosphoric acid activation contributes to the structural stabilization and surface functionalization of the carbon matrix. All these results demonstrate that black cumin seed meal, can serve as a suitable material for the preparation of activated carbon through phosphoric acid activation.

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Authors' contributions

M.N.-S., H.G.: Conceptualization and writing of original draft; M.M., M.N.-S., L.M., K.R., G.G.: Methodology and experimental work; M.N.-S., L.M., M.M.: Writing - review and editing.

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