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RESEARCH ARTICLE

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Activated carbon production from black cumin pulp, walnut shell, almond kernel shell and apricot kernel shell

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Abstract

Black cumin pulp, walnut shells, almond kernel shells and apricot kernel shells are plant residues from agricultural crops. Millions of tonnes of such biomass rich in carbon content are increasing every year in the world. In this study, the above-mentioned plant residues were collected and activated carbons were produced from these residues to be used in cyanide gold solution. The plant residues were subjected to carbonisation and then activation processes at different times and temperatures. As a result of carbonisation processes, products containing 68.80% C for black cumin pulp, 90.76% C for walnut shell, 89.10% C for almond kernel and 92.61% C for apricot kernel were obtained. BET and SEM analyses of these materials, which have sufficient carbon content for activated carbon production, were performed. As a result of BET analyses, it was determined that the surface area of activated carbon produced from apricot kernel shell increased up to 984 m²/g. SEM analyses showed that the activated carbon produced from almond shells had optimum porosity.

Keywords: Black cumin pulp; biomass; apricot kernel shell; almond shell; walnut shell; carbonisation; activation:

1. Introduction

In recent years, the rapid depletion of natural resources worldwide, the increase in environmental and atmospheric pollution and the recovery of depleted resources are becoming increasingly difficult. In order for humans and other

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living creatures to survive in a healthy way, various solution proposals are being developed, and in this direction, it is aimed to popularise the understanding of 'environmental sustainability'.

Many issues from environmentally friendly products to renewable energy sources have gained importance within the framework of sustainability. Various adsorbent materials can be used to reduce water pollution and pollutants generated in industrial plants at the source. In this context, materials such as minerals, resins and activated carbon with adsorption capacity play an important role in controlling environmental pollution.

Activated carbon is a mainly amorphous solid with an extremely large internal surface area and a large pore volume [1]. Activated carbon is produced from materials containing high amounts of carbon and its composition usually contains 87-97% carbon. The remaining part may contain elements such as hydrogen, oxygen, sulphur and nitrogen. In addition, depending on the raw materials used and other chemicals added during the production process, activated carbon is known to contain different elements [2]. Activated carbons have the capacity to adsorb a wide range of substances and can act as adsorbents by attracting molecules to their inner surfaces. Generally, the pore volume of activated carbons is greater than 0.2 ml/g [3]. Activated carbons are widely used in many sectors such as medicine, environment, chemistry, energy, metallurgy, metallurgy, textile and food in processes such as separation, purification, removal and recovery. An important advantage of activated carbon is that it can be produced from various biomass wastes with high carbon content and low inorganic matter content and can be reused through regeneration [4].

Today, many agricultural residues can be used for activated carbon production. For example biomasses such as palm fibres [5], tea plant waste, almond shells [6], hazelnut and walnut shell [7], from the outer green shell of pistachios [8], sugar beet pulp [9], sunflower seed shells and trays [10] and pistachio shells [11] can be shown.

In order to develop lower cost activated carbons, there are many researches on cheap and easily available materials. Thanks to their high surface areas, microporous structures and surface chemical properties, activated carbons stand out as effective potential adsorbents for the removal of heavy metal ions in industrial wastewater. Activated carbon produced from apricot kernel, an agricultural waste, can be used as an effective adsorbent for the removal of heavy metal ions from aqueous solutions [12].

In this study, the carbonisation of black cumin pulp, almond shell, walnut shell and apricot kernel shells, which are biomasses to be used for gold adsorption in cyanide gold solutions, was carried out, the weight losses of the samples after carbonisation were examined and these materials were evaluated as raw materials for activated carbon production. The obtained samples were subjected to SEM tests to determine their morphological properties and BET tests to analyse their surface properties.

2. Experimental Studies

2.1. Material-Method

In this study, various biomasses such as black cumin pulp, walnut shell, almond kernel shell and apricot kernel shell were used (Figure 2.1). Carbonisation and activation processes were carried out in Nüve brand MF 201 model and Nevola brand muffle furnace. In the experiments, Merck brand potassium hydroxide (KOH) with CAS number 1.05033.1000 was used as activating agent, and in order to remove potassium from the environment, washing with Merck brand hydrochloric acid (HCl) with CAS number 1.00317.2500 was applied, followed by rinsing with pure water. The obtained samples were analyzed by SEM and BET and the results are shown in Table 6 and Figure 4.

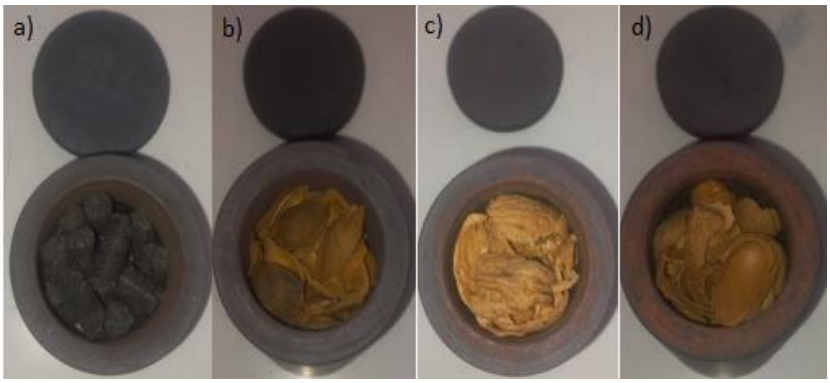


Fig. 1. Materials used in the carbonisation process: a- black cumin pulp, b- apricot kernel shell, c- walnut shell, d- almond shell.

Carbon (C) and sulphur (S) analyses of the obtained samples were carried out in LeCo CS 230 device before carbonisation and activation processes and the analysis results are given in Table 1.

Table 1. Crude C-S analysis results of almond shell, apricot kernel shell, walnut shell, black cumin pulp

Materials	Elements, %	
	C	S
Almond	46.4	0.001
Apricot	48.5	0.00131
Walnut	47.5	0.021
Black Cumin Pulp	----	----

Raw black cumin pulp showed a negative effect during the C and S analyses and the analyses could not be completed.

2.1.1. Carbonisation

The samples were carbonised in the muffle furnace at 600 °C, 700 °C and 800 °C for 2, 4 and 8 hours respectively. The samples in the crucibles were kept in the furnace without opening the lids to prevent contact with oxygen and allowed to cool down to room temperature. As a result of carbonisation processes, a weight loss of approximately 74-77.4% was observed in apricot kernel shell, 75.2-77.1% in almond kernel shell, 73.4-76.9% in walnut shell and 74.75-78.7% in nigella pulp. This weight loss indicates that impurities other than carbon in the structure were removed and a carbon-dominated material was obtained. Figure 2 details the weight loss of the materials during the carbonisation process.

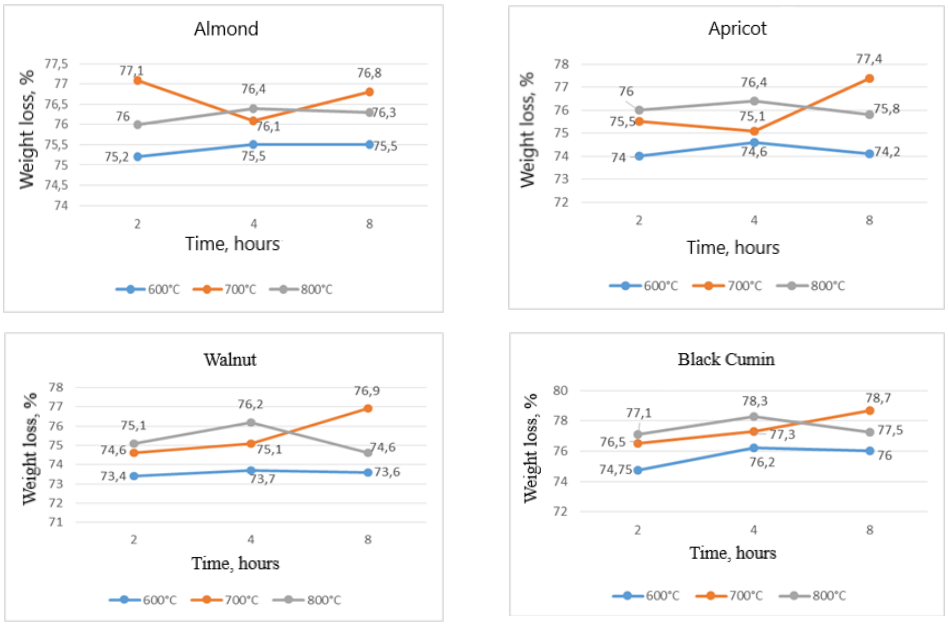


Fig. 2. Weight losses occurring in the structure of materials

After the carbonisation process was completed, the samples were removed from the furnace, weighed and stored in ziplock bags. Then, analyses were carried out to determine the carbon and sulphur contents.

When the analysis results of the carbonised products were examined, it was found that the carbon content of the almond shell treated at 800 °C for 4 hours was 89.1% and the sulphur content was 0.013% and these data are presented in Table 2.

Table 2. C-S analysis results of carbonised almond kernel shell

Material	Carbonisation temperature (°C)	Carbonisation time (hours)	Elements, %	
			C	S
Almonds	600	2	87.21	0.002
		4	88.23	0.015
		8	88.08	0.022
	700	2	87.8	0.04
		4	88.31	0.025
		8	88.4	0.016
	800	2	88.3	0.012
		4	89.1	0.013
		8	88.53	0.004

As a result of the carbonisation of apricot kernel shell, it was determined that the carbon content of the sample treated at 800 °C for 4 hours increased to 92.61% and the sulphur content decreased to 0.01%, and these results are presented in Table 3. These values represent the highest carbon yields reported for activated carbons derived from almond shells.

Table 3. C-S analysis results of carbonised apricot kernels

Material	Carbonisation temperature (°C)	Carbonisation time (hours)	Elements, %	
			C	S
Apricot	600	2	90.905	0.002
		4	91.91	0.022
		8	86.92	0.006
	700	2	91.44	0.016
		4	92.25	0.017
		8	87.35	0.013
	800	2	91.82	0.006
		4	92.61	0.01
		8	88.43	0.017

After the carbonisation process, the carbon content of walnut shells, which was 47.5% in raw form, increased to 90.76% in the sample treated at 800 °C for 8 hours. The sulphur content of the same sample was determined as 0.073% (Table 4).

Table 4. C-S analysis results of carbonised walnut shell

Material	Carbonisation temperature (°C)	Carbonisation time (hours)	Elements, %	
			C	S
Walnut	600	2	88.1	0.09
		4	88.25	0.035
		8	88.3	0.062
	700	2	88.94	0.079
		4	89.32	0.057
		8	89.3	0.087
	800	2	89.3	0.086
		4	90.72	0.086
		8	90.76	0.073

After the carbonisation of black cumin pulp, it was determined that the highest carbon content reached 68.8% in the sample treated at 800 °C for 2 hours. However, the sulphur content of this sample was quite high compared to the others and was determined as 0.37%. It is also shown in Table 5 that a decrease in carbon content was observed with increasing processing time.

Table 5. C-S analysis results of carbonised black cumin pulp

Material	Carbonisation temperature (°C)	Carbonisation time (hours)	Elements, %	
			C	S
Black Cumin Pulp	600	2	67.28	0.026
		4	65.21	0.101
		8	61.14	0.097
	700	2	68.2	0.345
		4	65.78	0.274
		8	62.52	0.35
	800	2	68.8	0.37
		4	66.46	0.43
		8	63.75	0.33

2.1.2. Activation

The activation studies were carried out on the samples obtained under the conditions yielding the highest carbon

content, as specified in the 'Carbonization' section. After the carbonization process, the carbonized product was crushed and sieved and the samples of -2;+1 mm size were subjected to activation process.

The carbonized products obtained from apricot kernel shell, walnut shell, almond kernel shell and black cumin pulp were activated by mixing with KOH at a ratio of 1/0.5 and 1/1. The activation process was carried out in a muffle furnace at 600 °C for 4 hours. The activated samples were cooled in the oven in a controlled manner to prevent contact with air.

Then 500 mL HCl solution was prepared per 50 g of material to remove KOH from the samples. This solution was prepared to contain 208 mL of 37% HCl (Merck) and the remaining volume was completed with distilled water. In order to bring the pH level to the desired range, the samples were subjected to sequential washing with distilled water and the washing process was continued using filter paper until the pH value reached 5.5-6. After the washing process was completed, the samples were subjected to drying and kept in an oven at 80 °C for 24 hours. Figure 3 shows the flow chart of the activated carbon production process.

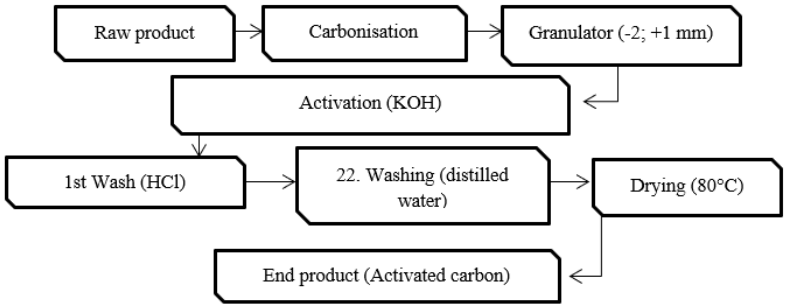


Fig. 3. Activated carbon production flow chart

Activated carbons were subjected to BET and SEM analyses. The results of BET analysis to determine the surface area of the activated carbon obtained as a result of activation processes and the description of the coded samples are presented in Table 6.

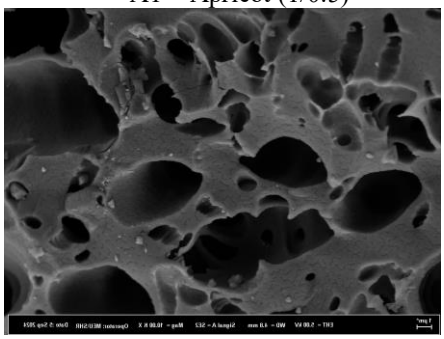
The production conditions of the activated carbons sent to BET analysis were determined as 600°C temperature and 4 hours as standard.

Table 6. BET surface area test results of the samples

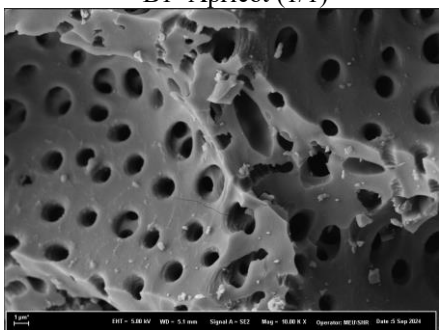
Code of the material	Name of the material	Activator Ratio (Carbonized product / KOH)	Activation time, (h).	BET surface area (m2/g)
A1	Apricot	1/0.5	4	526
A2	Walnut	1/0.5	4	520
A3	Almond	1/0.5	4	274
A4	Black Cumin Pulp	1/0.5	4	109
B1	Apricot	1/1	4	984
B2	Walnut	1/1	4	712
B3	Almond	1/1	4	688
B4	Black Cumin Pulp	1/1	4	514

It was determined that the surface area of the activated carbon coded B1 produced from apricot kernel shell activated at 600 °C for 4 hours by mixing with KOH at a ratio of 1/1 had the highest surface area with 984 m^2/g compared to the other samples. SEM analysis images of the samples and the amount of activator ratio in the coded samples are shown in Figure 4.

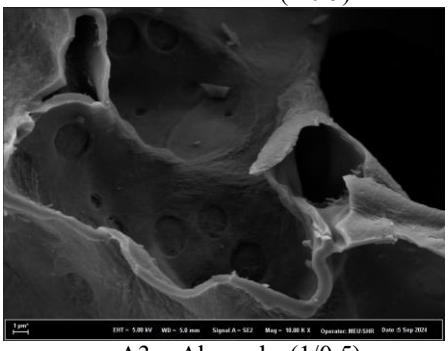
A1 – Apricot (1/0.5)



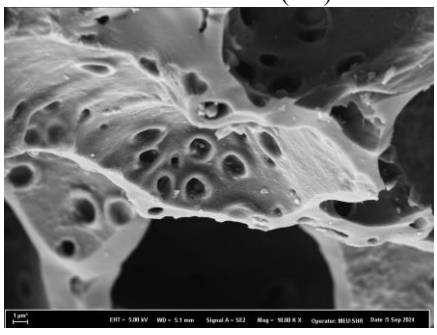
B1- Apricot (1/1)



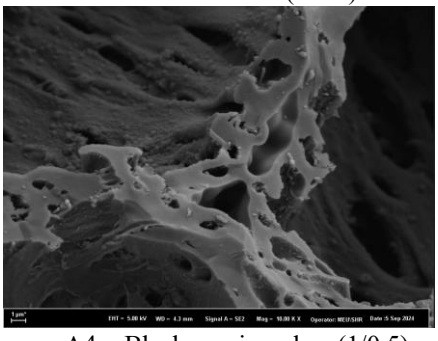
A2 – Walnut (1/0.5)



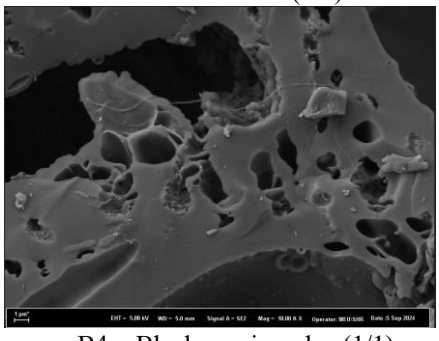
B2 – Walnut (1/1)



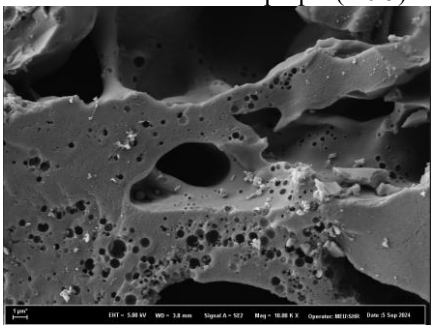
A3 – Almonds (1/0.5)



B3 – Almonds (1/1)



A4 – Black cumin pulps (1/0.5)



B4 – Black cumin pulps (1/1)

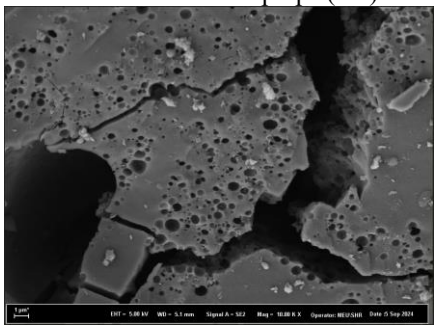


Fig. 4. SEM images of specimens magnified up to 1 µm, 10.00 K X.

3. Results

In this study, it was aimed to obtain high carbon content as a result of carbonisation processes of almond shell, apricot kernel shell, walnut shell and nigella pulp known as biomass wastes and to produce activated carbon from these products. BET and SEM analyses of activated carbons produced as a result of carbonisation and activation processes were carried out and the following results were obtained:

As a result of the carbonisation processes performed on almond shells, while the carbon content of the raw material of almond shells was 46.4%, the sample with the highest increase in carbon content after the carbonisation processes was the sample treated at 800 °C for 4 hours and the carbon content increased up to 89.1%. The sulphur content of the same sample was 0.013%. The BET surface area of activated carbon produced from almond shell increased to 274 m^2/g with the use of 1/0.5 activator ratio and to 688 m^2/g with the use of 1/1 activator ratio.

As a result of the carbonisation processes performed on apricot kernel shells, it was observed that the carbon content of the raw material was 48.5%, while the carbon content increased to 92.61% in the sample treated at 800 °C for 4 hours as a result of carbonisation processes. The sulphur content of the same sample decreased by 0.01%. The BET surface area of activated carbon produced from apricot kernel shell increased to 526 m^2/g with the use of 1/0.5 activator and to 984 m^2/g with the use of 1/1 activator.

While the carbon content of the walnut shells was 47.5% in the raw state, it was observed that the carbon content increased up to 90.76% in the sample treated at 800 °C for 8 hours as a result of the carbonisation processes. The sulphur content of the same sample was determined as 0.073%. The BET surface area of the activated carbon produced from walnut shell increased to 520 m^2/g with the use of 1/0.5 activator and 712 m^2/g with the use of 1/1 activator.

While the carbon content was 44.7% in the raw material of black cumin pulp, it was determined that the highest carbon content was obtained from the sample treated at 800 °C for 2 hours after carbonisation process and the carbon content could reach up to 68.8%. The sulphur content of the same sample was determined as 0.37%. The BET surface area of activated carbon produced from black cumin pulp increased to 109 m^2/g with the use of 1/0.5 activator ratio and to 514 m^2/g with the use of 1/1 activator ratio.

As a result of the carbonisation processes, it was observed that the best carbonisation process occurred in apricot kernel shells and the lowest carbonisation process occurred in black cumin pulp. In line with the analyses performed as a result of activation processes, SEM analyses showed that the best porosity occurred in activated carbons produced from almond shells. According to BET surface area analyses, the highest surface area values were obtained in activated carbons produced from apricot kernel shell. In these samples, it was determined that 526 m^2/g surface area was reached with the use of 1/0.5 activator ratio and 984 m^2/g surface area was reached with the use of 1/1 activator ratio.

In conclusion, the study demonstrated that biomass waste can be effectively utilized as a carbon source and holds significant potential as a precursor for the production of high-value activated carbon.

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Author Contributions

Kamil Mammadov: Research, Writing - review and editing, **Salih Aydogan:** Research, Writing – review and editing.

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