

Adsorption of Gold from Cyanide-Bearing Solutions onto Activated Carbon Derived from Black Cumin Pulp

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ABSTRACT

In this study, activated carbon was synthesized from black cumin pulp as a biomass precursor via chemical activation and its capacity to adsorb gold from cyanide-containing solutions was systematically investigated. The carbonization process was conducted by subjecting the precursor material to thermal treatment at 600°C, 700°C and 800°C for durations of 2, 4 and 8 hours. The maximum mass loss observed during carbonization was determined to be 78.7%. Chemical activation was carried out using potassium hydroxide (KOH) at impregnation ratios of 1:1 and 1:0.5. Various activation conditions were evaluated to identify the optimal parameters for maximizing adsorption performance. The physicochemical properties of the produced activated carbon were characterized using Brunauer-Emmett-Teller (BET) surface area analysis, scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR) and atomic absorption spectroscopy (AAS). BET results indicated that the sample activated at a 1:0.5 ratio exhibited a surface area of 109 m²/g, whereas the sample activated at a 1:1 ratio reached 514 m²/g. SEM analysis confirmed the presence of a well-developed porous structure. FTIR analysis was employed to identify the chemical composition and functional groups of the material. According to AAS measurements, 0.5 g of activated carbon achieved 77.96% gold adsorption from the cyanide solution within 25 minutes. Overall, the findings demonstrate that activated carbon derived from black cumin pulp possesses a highly porous structure, a considerable surface area and effective adsorption performance. These results suggest that black cumin pulp is a promising and viable raw material for the production of activated carbon.

Keywords: Adsorption; Gold; Activated carbon; Black cumin pulp; BET; SEM; FTIR; AAS

Introduction

Activated carbon is a porous material with a high carbon content, characterized by a network of pores at the molecular scale. Owing to this structure, it is widely employed in the separation and purification of water, air and gas mixtures. Its highly developed porosity renders activated carbon a unique material, as these pores provide an extensive surface area that enables significant adsorption capacity. The fundamental mechanism of adsorption is primarily governed by interactions between adsorbate molecules and the pore surfaces through Van der Waals forces¹. Depending on the intended application, activated carbon can be produced in powdered, granular or pelleted forms, allowing its use across a broad range of processes. It is extensively utilized as an effective adsorbent in various industrial sectors-including food processing, healthcare, chemical production and hydrometallurgy-for the removal of impurities and the separation of components². Activated carbons can be produced via physical or chemical activation methods and considerable research has been devoted to developing more cost-effective production techniques³. Typically, raw materials with high carbon content are preferred for this purpose. Agricultural by-products, in particular, are regarded as valuable precursors due to their low cost, widespread availability, carbon-rich composition and environmentally sustainable nature⁴. Owing to its versatile production methods and wide range of applications, activated carbon is also extensively employed in hydrometallurgical operations. Its high adsorption capacity enables effective interaction with metal ions in solution, making it a critical component in modern industrial processes. In gold recovery, the Carbon-In-Pulp (CIP) method has been widely and effectively applied for many years⁵. Within the gold-cyanide hydrometallurgical system, gold forms a stable cyanide complex in solution, which is subsequently extracted and adsorbed onto the surface of activated carbon, resulting in the accumulation of gold ions on the carbon matrix⁶. Globally, activated carbons derived from coconut shells are among the most commonly used materials in gold recovery processes⁷.

However, in recent years, increasing attention has been directed toward reducing production costs and utilizing locally available biomass resources for activated carbon synthesis. Various studies have explored alternative precursors such as tea processing waste⁸, almond shells⁹, walnut shells¹⁰, olive stones¹¹, date pits¹² and pine nut shells¹³. The present study aims to evaluate black cumin pulp as a low-cost biomass precursor and to produce activated carbon with a high surface area and well-developed porosity via chemical activation. In addition, the adsorption performance of the synthesized activated carbon for gold recovery from cyanide-containing solutions is systematically investigated.

Materials and Methods

In this study, defatted black cumin pulp was selected as the precursor material for activated carbon production. Experimental procedures were carried out using a Nuve MF 201 model muffle furnace and a Nevola ashing furnace. Elemental analyses for carbon (C) and sulfur (S) were performed using a LeCo CS 230 analyzer. In addition, potassium hydroxide (KOH), hydrochloric acid (HCl) and an Atomic Absorption Spectroscopy (AAS) instrument were utilized throughout the study.

Carbonization of black cumin pulp

The black cumin pulp was first accurately weighed using a precision balance and then placed into specially designed crucibles with lids. For the carbonization process, the crucibles were transferred into an ashing furnace capable of maintaining a controlled atmosphere. The samples were subjected to thermal treatment at predetermined temperatures of 600°C, 700°C and 800°C for durations of 2, 4 and 8 hours, respectively. Upon completion of the carbonization process, the mass loss of the samples was determined (**Figure 1**).

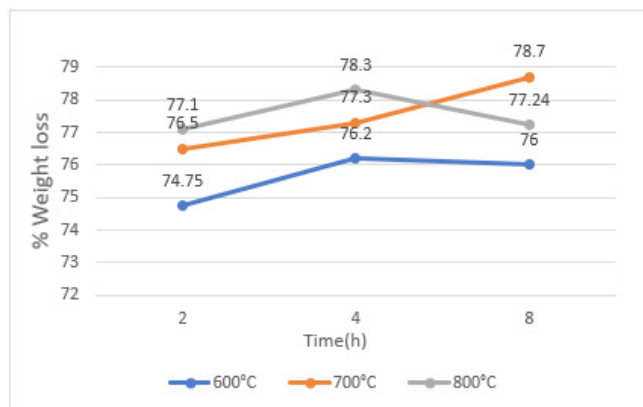


Figure 1: Mass Loss of the Sample After Carbonization.

Following the completion of the carbonization process, elemental analyses for carbon (C) and sulfur (S) were conducted. The results indicated that the highest carbon content, 68.8%, was obtained from the sample treated at 800°C for 2 hours. However, a decrease in carbon content was observed with increasing processing time. This trend is presented in detail in (**Table 1**).

Table 1: Carbon (C) and Sulfur (S) Contents (%) of Black Cumin Pulp After Carbonization Processes.

Material	Carbonization Temperature (°C)	Carbonization 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

Analysis indicates that the carbon content decreases during the carbonization process in correlation with increasing temperature and duration. Based on these results, the optimum temperature and time for the activation studies were determined and the procedures were carried out at 600 °C for 4 hours.

Activation of black cumin pulp

The carbonized black cumin pulp was subjected to the activation process within a covered steel crucible. Potassium hydroxide (KOH) was utilized as the activating agent. The activation process was conducted by mixing the carbonized black cumin pulp with KOH at ratios of 1:0.5 and 1:1 (**Figure 2**). This procedure was performed in a muffle furnace at 600 °C for a duration of 4 hours.

Following the completion of the activation process, the samples were cooled within the furnace in a controlled manner to minimize atmospheric contact. Subsequently, to ensure the complete removal of KOH from the samples, a hydrochloric acid (HCl) solution was prepared at a ratio of 500 mL per 50 grams of material. The solution was adjusted to contain 208 mL of 37% concentrated HCl (2.10 M HCl) (Merck), with the remaining volume supplemented with distilled water. As a result of this process, the KOH present in the medium was entirely removed and the pH value was reduced to zero. To bring the pH level to the desired range, the samples were sequentially washed with distilled water; the rinsing process using filter paper was continued until the pH value reached a level of 5.5–6.



Figure 2: Mixture of black cummin pulp and KOH prior to activation.

Results and Discussion

BET analysis of activated carbon

Upon examining the BET surface area of the samples activated via the chemical activation method after 4 hours of treatment, it was determined that a higher surface area was achieved at a 1:1 ratio, depending on the amount of KOH (**Table 2**).

Table 2: BET surface area analysis results of activated carbons produced from black cummin pulp.

Sample Name	Activant ratio	Activation time, (h)	BET surface area (m ² /g)
Black Cummin Pulp	1/0.5	4	109
	1/1		514

As shown in (**Figure 3**), a distinct difference between the adsorption and desorption curves of the activated carbon is observed. This situation indicates that capillary condensation is effective within the pore structure. At low relative pressure values (p/p_0), adsorption increases rapidly, signaling the presence of a limited amount of micropores in the structure. In the medium relative pressure range, the increase in adsorption is more gradual and steadier, with the isotherm exhibiting a more stable behavior in this region. At high p/p_0 values, a sudden rise in the adsorbed amount is observed, suggesting that the capillary

condensation mechanism within the mesopores is predominant. The adsorption capacity of the activated carbon reached levels of 3.2–3.3 mmol/g with the use of a 1:0.5 ratio of activator (KOH). This level indicates that the activated carbon possesses a moderate surface area and a micro-mesoporous structure.

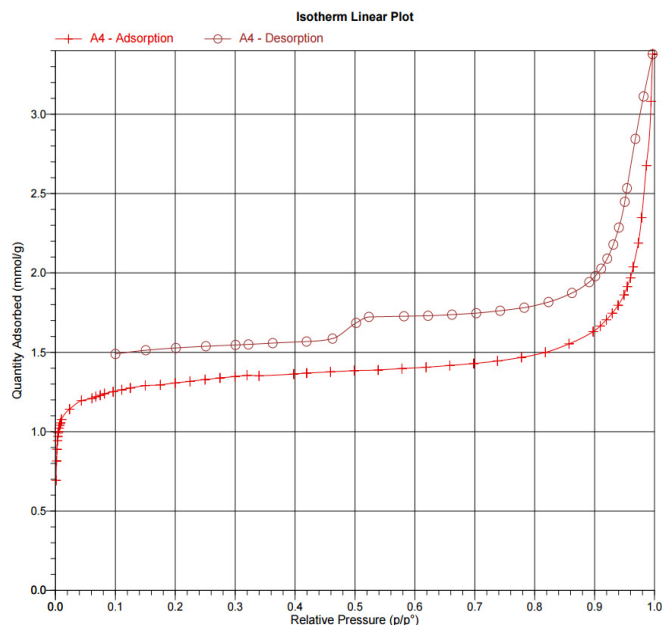


Figure 3: Linear isotherm plot of the activated carbon (KOH ratio 1:0.5).

When examining the plot of the activated carbon sample with an activator ratio of 1:1, it is observed that it possesses a higher adsorption capacity compared to the activated carbon produced using a 1:0.5 KOH ratio. The rapid increase in the amount adsorbed at low relative pressure values indicates that the micropores of this activated carbon are highly developed. At high p/p_0 values, more intense capillary condensation in the mesopores is observed, reaching an adsorption rate of approximately 7.4–7.5 mmol/g. This explains that, compared to the other sample, it has a much higher adsorption capacity and a larger surface area (**Figure 4**).

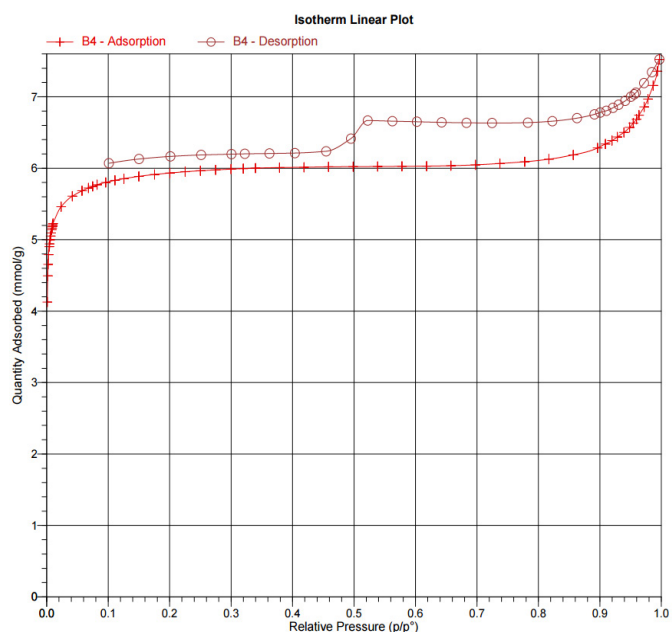


Figure 4: Linear isotherm plot of the activated carbon (KOH ratio 1:1)

SEM analysis of activated carbon

Pores are also visible in the scanning electron microscope (SEM) images of the activated carbon produced from black cumin pulp. BET analysis demonstrated that the activated carbon obtained with a 1:1 activator ratio possesses a surface area of 514 m²/g and a significantly larger pore volume compared to the other sample (**Table 3**). The incomplete removal of inorganic substances inherent in the black cumin during the production phase of the activated carbon results in a brittle material structure at the connecting edges of the porous framework. This prevents the formation of new pores within the existing pore structure, as observed in the SEM analyses presented in (**Figures 5,6**).

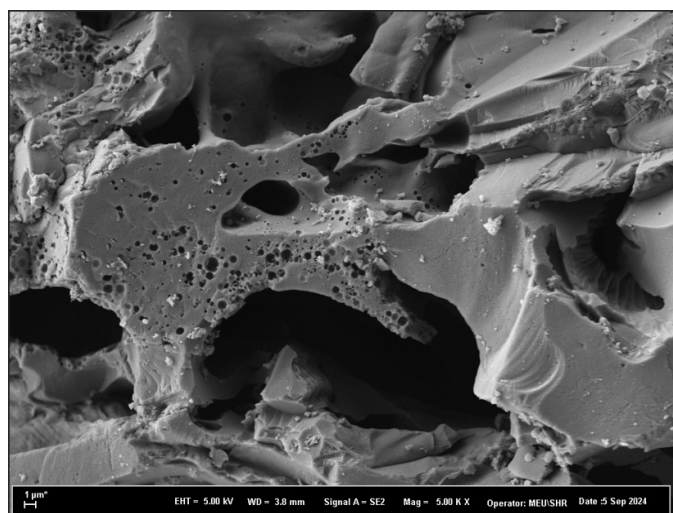


Figure 5: SEM micrograph of activated carbon - KOH ratio 1:0.5 (5.00 K X magnification).

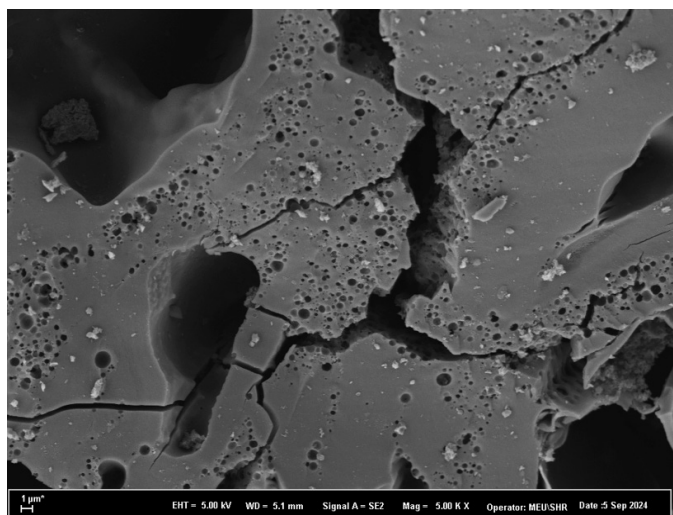


Figure 6: SEM micrograph of activated carbon - KOH ratio 1:1 (5.00 K X magnification).

FTIR spectroscopic analysis of activated carbon

This indicates the presence of functional groups containing carboxyl, alkyne/nitrile and C–O on the surface of the activated carbon. These structures contribute to the adsorption capacity by enhancing the chemical interaction of the surface. Furthermore, the presence of aromatic structures suggests that the carbon skeleton is well-developed.

The activated carbon sample possesses various functional groups, such as thiols (–SH), carboxylic acids, nitriles, alkynes, metal-carbonyls and halogenated compounds. These groups can

influence the chemical reactivity and adsorption capacity of the activated carbon. Atmospheric CO₂ and halogenated compounds may originate from environmental contaminants¹⁴.

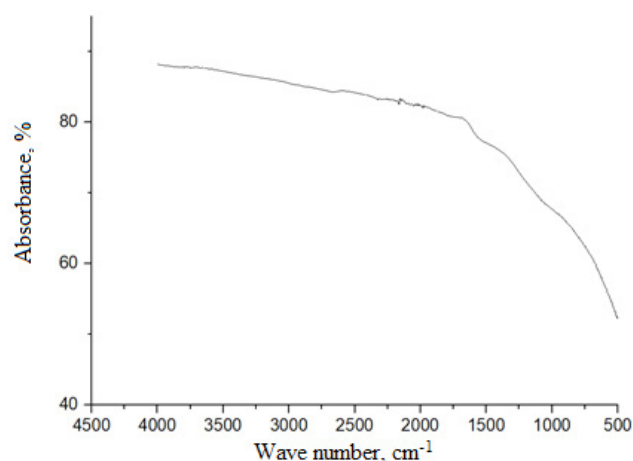


Figure 7: FTIR analysis of activated carbon (KOH ratio 1:0.5).

Table 4: Specific wavenumbers and functional groups of activated carbon (KOH ratio 1:0.5).

Wave number (cm ⁻¹)	Functional groups
2655	Acidic O–H stretching vibration
2161	C≡C (alkyne) or C≡N (nitrile) triple bond vibration
1979	Combination bands or overtones; sometimes in metal-carbonyl compounds
1547	N–H bending or C=C (aromatic ring-internal stretching)
1059	C–O stress vibration

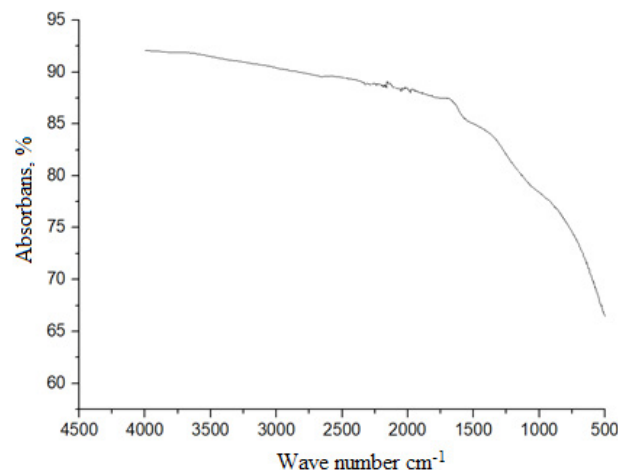


Figure 8: FTIR analysis of activated carbon (KOH ratio 1:1)

Adsorption of activated carbon

The activated carbons produced from black cumin pulp were utilized as adsorbents in a cyanide-bearing gold solution. Within the scope of the experiments, samples weighing 0.1, 0.2, 0.3, 0.4 and 0.5 g were taken from each specimen and added to a 100 mL NaCN solution containing 2.07 mg/L Au. Throughout the adsorption process, the samples were manually stirred for specified contact durations to ensure homogeneous contact with the solution.

Experimental studies were conducted for each sample at durations of 1, 2, 4, 8, 15 and 25 minutes at a constant temperature of 25 °C. At the end of each specified interval, a 5 mL aliquot was taken from the solution and subjected to Atomic Absorption

Spectroscopy (AAS) analysis to determine the change in gold concentration; the analysis results are presented in (Table 6).

Table 5: Some wave numbers and functional groups of activated carbon. Activator (KOH) ratio 1/1.

Dalga sayısı (cm ⁻¹)	Fonksiyonel gruplar
2653	–SH (thiol) or carboxylic acid Usually appears weak; may belong to carboxylic acid dimers or, rarely, thiol groups..
2318	CO ₂ (carbon dioxide) Absorption of carbon dioxide from the atmosphere; it is mostly an environmental contaminant
2162	C≡N (nitrile) or C≡C (alkyne) This is the characteristic sharp peak of nitrile groups; rarely, terminal alkynes also give a peak in this region.
1980	Metal-carbonyl (M-CO) overtone May belong to metal-carbonyl complexes or overtones in aromatic systems.
577	C–Cl, C–Br (halogenated compound) Vibrations of C–halogen bonds are observed in this region, especially in chlorinated or brominated compounds..

In the subsequent stage of the study, the optimum amount of activated carbon was determined. To investigate the effect of temperature on the adsorption process, the adsorption tests were repeated at 35 °C and 45 °C while maintaining the same sample amounts and experimental parameters (Table 7).

Table 6: Results of Au adsorption tests on activated carbons produced from black cumin pulp.

	Amount of black cumin pulp				
	0.1 g	0.2 g	0.3 g	0.4 g	0.5 g
Time, min.	Adsorption efficiency, %				
1	0	12.26	24.47	27.83	28.10
2	13.98	27.70	34.16	29.56	37.21
4	17.04	22.61	78.36	43.32	44.20
8	24.20	26.50	61.11	56.24	63.36
15	45.40	66.46	77.21	66.90	83.50
25	55.44	61.06	74.12	77.39	77.96

Table 7: Results of Au adsorption tests on activated carbons produced from black cumin pulp at different temperatures.

Temperature			
35 °C		45°C	
Time, min.	Adsorption efficiency	Sample quantity	
1	34.56	35.54	0.4 g
2	41.27	47.50	0.4 g
4	41.19	52.8	0.4 g
8	51.31	73.76	0.4 g
15	52.00	74.37	0.4 g
25	56.43	80.76	0.4 g

Conclusions

The linear isotherm plots reveal that both samples possess a porous structure; however, the second sample exhibits superior adsorption behavior compared to the first. It is observed that the second sample fulfills the requirements of high-quality activated carbon more effectively, both in terms of the amount adsorbed and the density of micro pore capillary condensation.

The activated carbon produced from black cumin pulp demonstrates remarkably rapid adsorption kinetics. At ambient temperature, the activated carbon reached an adsorption efficiency of 78.36% within a 4-minute agitation period, whereas this efficiency increased to 80.76% at 45 °C under 25-minute agitation conditions.

According to the FTIR analysis results, both samples generally exhibit C≡N (nitrile) or C≡C (alkyne) triple bond stretching vibrations at the 2161–2162 cm⁻¹ peaks, indicating the presence of carbon-based bonding environments.

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