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Black Cumin Pulp-Derived Activated Carbon: Synthesis, Characterization, and Pharmaceutical Soap Formulation

Çörek Otu Posasından Aktif Karbon Eldesi: Sentez, Karakterizasyon ve Farmasötik Sabun Formülasyonu

Besti Shahbazova[1], Sibel Kaymak[1], Betül Aydın[2], Nilüfer Vural[1,3*]

[1]Department of Biotherapeutic Product Research and Development, Ankara Yıldırım Beyazıt University, Ankara, Türkiye.
[2]Department of Biology, Gazi University, Ankara, Türkiye.
[3]Department of Food Processing, Ankara Yıldırım Beyazıt University, Ankara, Türkiye.

ABSTRACT

In this study, we explore the utilization of black cumin pulps for the synthesis of activated carbon and its subsequent application in pharmaceutical soap formulation. Activated carbon was produced from black cumin pulps using a carbonization process followed by activation with a suitable activating agent. The synthesized activated carbon was characterized using various analytical techniques including scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and Raman analysis. The results revealed the successful production of activated carbon with desirable properties for pharmaceutical applications. Subsequently, the activated carbon was integrated into soap formulations, and the resulting pharmaceutical soaps were evaluated for their antimicrobial efficacy and some chemical properties. The soap formulations exhibited effective antimicrobial activity against various microorganisms, including Candida albicans, while maintaining high skin compatibility. Moreover, the incorporation of activated carbon led to enhanced cleansing properties and biotherapeutic effects. Overall, this study highlights the potential of utilizing black cumin pulps for sustainable activated carbon production and their application in pharmaceutical soap development, contributing to both environmental and healthcare sectors.

Key Words
Black cumin pulps, activated carbon, pharmaceutical soap.

ÖZ

Bu çalışma, aktif karbon sentezi için çörek otu posasının kullanımnı ve sonrasında farmasötik sabun formülasyonunda uygulanmasını araştırmıştır. Aktif karbon, uygun bir aktifleştirici madde ile aktivasyonun ardından bir karbonizasyon prosesi kullanılarak çörek otu posalarından üretildi. Sentezlenen aktif karbon, taramalı elektron mikroskobu (SEM), Fourier dönüşümü kızılötesi spektroskopisi (FTIR) ve Raman analizi dahil olmak üzere çeşitli analitik teknikler kullanılarak karakterize edildi. Sonuçlar, farmasötik uygulamalar için arzu edilen özelliklere sahip aktif karbonun başarılı bir şekilde üretildiğini ortaya çıkardı. Sonrasında bu aktif karbon, sabun formülasyonlarına entegre edildi ve elde edilen farmasötik sabunlar, antimikrobiyal etkinlikleri ve bazı kimyasal özellikleri açısından değerlendirildi. Sabun formülasyonları, yüksek cilt uyumluluğunu korurken, Candida albicans dahil olmak üzere çeşitli mikroorganizmalara karşı etkili antimikrobiyal aktivite sergiledi. Ayrıca aktif karbonun eklenmesi, temizleme özelliklerinin ve biyoterapötik etkilerin artmasına yol açtı. Genel olarak bu çalışma, sürdürülebilir aktif karbon üretimi için çörek otu posası kullanma potansiyelini ve bunların farmasötik sabun geliştirmede uygulanmasını vurgulayarak hem çevre hem de sağlık sektörlerine katkıda bulunmaktadır.

Anahtar Kelimeler
Çörek out posası, aktif karbon, farmasötik sabun.

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Correspondence to: N. Vural, Department of Biotherapeutic Product Research and Development, Ankara Yıldırım Beyazıt University, Ankara, Turkey.
E-Mail: nilufervural@aybu.edu.tr

INTRODUCTION

Activated carbon is a carbon-based material possessing adsorbent properties, characterized by a large surface area, porous structure, and high surface reactivity [1]. Application range for activated carbon is quite extensive, including industrial wastewater treatment, air purification, development of various chemicals, food and pharmaceutical products, and electrochemical applications [2, 3]. Nowadays, activated carbon also has become one of the essential ingredients in the cosmetic industry due to its advanced material properties as well as its biotherapeutic characteristics. It has become a key component in cleansers such as soap, toothpaste, shampoo, and face masks [4]. In recent years, with the increasing awareness of waste recycling, research into alternative raw materials for activated carbon production has also emerged [5]. The criteria considered in the selection of the carbonaceous precursor material for activated carbon production include high carbon and low inorganic content, processability, and environmental impact [6]. The production strategies for activated carbon are diverse but generally obtained through the physical or chemical activation of the respective raw materials. In physical activation, the material is carbonized at high temperatures, whereas in chemical activation, active carbon is obtained by impregnating the raw material with different activating agents such as zinc chloride or oxide, aluminum chloride, and sodium hydroxide [1]. Among the biomass sources currently used in the production of activated carbon are wood, coconut, various food peels, and pulps of the different agricultural products such as citrus, flax and black cumin [7, 8]. Black cumin (*Nigella sativa* L.) is an annual herbaceous plant belonging to the Ranunculaceae family. Morphologically, it is a species with a variable height ranging from 20 to 50 cm, with branched, hairy, and sparse structure [9]. The flowering period is known to be in June and July. Its fruit is rich in seeds, which are also the most important and utilized part of the plant. It is cultivated in many countries, including Türkiye, and shows a wide distribution. Depending on the climate of the region where it is grown, there may be variations in the composition of black cumin seeds. However, they generally contain fixed oils (32-40%), carbohydrates (33.9%), proteins (16-19.9%), fibers (5.5%), minerals (1.79-3.44%), volatile oils (0.4-0.45%), amino acids, alkaloids, tannins, saponins, and other bioactive compounds [10]. The use of black seed due to its pharmaceutical effects dates back centuries. Black seed possesses various biotherapeutic properties such as antimicrobial, analgesic, anti-inflammatory, antiallergic, diuretic, antioxidant, antihypertensive, antidiabetic, anticancer, and immuno-regulating effects [11]. Among the methods for obtaining oil from black cumin seeds are the use of solvents, heat treatment, hydro-distillation, microwave extraction, and cold pressing [12]. While it is known that the amount of oil obtained from black cumin seed varies between 20 % and 30%, and the remaining 70-80% is pulp [13]. Considering that Türkiye produced 3603 tons of black seed in 2019, recycling these pulps is of great importance [14].

Within this scope, the main aim of the study is to evaluate the use of black cumin seed pulps in the production of activated carbon and to convert the obtained activated carbon into a high-value-added product. First, black cumin seed pulps were obtained and activated carbon samples were prepared using different zinc oxide impregnation ratios. Structural and morphological analyses of the activated carbon specimens were conducted. Subsequently, a soap containing activated carbon was produced, and its pharmaceutical effects were evaluated through in vitro antimicrobial tests.

MATERIALS and METHODS

Procurement and storage of black cumin seeds

The relevant samples of black cumin seed were obtained from Azerbaijan for use in the study. The samples, which generally exhibit a stable structure, were stored in closed containers in a cool area, away from moisture and direct light.

Extraction of oil from black cumin seeds

The oil from the black cumin seeds was obtained using a cold press extraction device at the research laboratories of Ankara Yıldırım Beyazit University, GETAT Researching Center, located at our Cubuk Campus. The remaining pulp was preserved for synthesis studies. Then, pulp was washed with hot and cold distilled water to eliminate impurities and other non-pulp materials. Subsequently, to remove the moisture content in the pulp samples, they were dried in an oven at 105°C overnight. The resulting dry samples were mechanically ground and stored in sealed containers, away from direct heat and moisture, for further studies.

Obtaining activated carbon from black cumin seed pulps

Obtained pulp samples were impregnated with ZnO at a 1:1 ratio. After the impregnation process, 10 ml of distilled water was added to the mixture samples and stirred at 72°C for 6 minutes. Once the mixture obtained a homogeneous consistency, oven drying was carried out at 120°C for 24 hours. Subsequently, the dried sample mixtures were carbonized in a muffle furnace at 500°C, 650°C, and 850°C for 2 hours. After carbonization, the samples were washed with 0.5 M HCl solution for 30 minutes and then washed with distilled water to neutralize the pH. Then, oven drying was conducted again at 110°C for 24 hours.

Characterization studies of black cumin pulp and activated carbon

SEM analysis

In order to perform detailed size analyses of the activated carbon and pulp samples obtained from black seeds, SEM images were obtained using the Hitachi SU5000 FE-SEM model device available at the Ankara Yildirim Beyazit University (AYBU) MERLAB center. Images obtained at different magnifications are presented in the results. Additionally, EDX analysis was performed during the SEM analysis.

FTIR analysis

The FT-IR spectra of different activated carbon samples and pulp samples were measured between wave numbers of 4000-400 cm^{-1} using the FTIR spectroscopy analysis device available at the Ankara Yildirim Beyazit University (AYBU) MERLAB center.

Raman analysis

For the investigation of the chemical bond structures and functional groups of the activated carbon and pulp samples, Raman (Raman Scattering Spectroscopy) analyses were conducted using the JASCO NRS4500 Confocal Microscope Raman Spectrometer available at the Ankara Yildirim Beyazit University (AYBU) MERLAB center. These analyses aimed to facilitate advanced structural analysis.

Pharmaceutical soap studies

Utilizing the obtained activated carbon, a biotherapeutic soap was developed by incorporating rosemary oil and green tea extract in addition to cumin seed oil to enhance synergistic efficacy. An initial experimental design study was conducted to determine the amounts of active ingredients, and the optimal ratios to produce one soap in the thesis study are presented in Table 1.

Subsequently, transparent solid soap base was melted, and activated carbon along with other ingredients were added. The mixture was then cooled and solidified in appropriate molds. The content of the soap base is as follows:

- Sorbitol, propylene glycol, PEG-400, glycerin, sodium laureth sulfate, lauric acid, stearic acid, sodium hydroxide, sodium chloride, citric acid, and triethanolamine.

Characterization studies of pharmaceutical soap

pH analysis

2 g of soap powder was weighed and dissolved in 10 mL of distilled water for pH measurements. Measurements were conducted using a pH meter. The obtained data were compared with standards and literature values. The measurement was conducted in triplicate.

Moisture analysis

For moisture determination, a CEM DT-616CT brand IR laser moisture and temperature meter were used. The relevant measurements were conducted in a closed environment, and with both laser and probe.

Antimicrobial analysis

Sterile surgical blade was used to randomly scratch from one side to take soap samples. 100 mg of soap sample was dissolved in 1 mL of sterile distilled water and used for antimicrobial activity analysis. Six bac-

Table 1. Pharmaceutical soap composition.

Ingredients	Amount
Activated Carbon	1 g
Nigella sativa oil	1 ml
Rosmarinus officinalis oil	0.5 ml
Green tea extract	0.5 g (decoction with 100 ml hot water)

terial and fungal test strains were used for antimicrobial activity analysis, included Gram-positive strains, Staphylococcus aureus subsp. aureus ATCC 25923, Enterococcus faecalis ATCC 51299 (Drug-resistant Enterococcus faecalis-DRE), Staphylococcus aureus subsp. aureus ATCC 43300 (Methicillin-resistant Staphylococcus aureus-MRSA); Gram-negative strains, Escherichia coli ATCC 35218, Pseudomonas aeruginosa ATCC 27853, and yeast strain Candida albicans ATCC 10231. Each bacterial and yeast culture were grown on Mueller Hinton Broth and Sabouraud Dextrose Broth at 37°C and 30°C, respectively. After incubation, organisms were suspended in 10 mL of physiological saline solution, and optical density readings were compared with 0.5 McFarland standard (1.5x108 colony-forming units/mL). The antimicrobial activity of the soap sample was determined using liquid microdilution analysis with sterile 96-well plates according to the CLSI reference methods M07-A9 for bacteria and M27-A for yeasts. Minimum inhibitory concentrations (MICs) of the soap sample were recorded as the lowest concentration at which no visible growth was observed in the microtitre plate wells. Minimum bactericidal concentration (MBC) and minimum fungicidal concentration (MFC) values were determined by subculturing from clear wells and spot inoculation onto an appropriate growth medium. Growth was recorded after incubation, and MBCs/MFCs were defined as the lowest concentrations that resulted in at least 99.9% killing compared to the initial viable counts. At least three replicates were performed for each assay.

RESULTS and DISCUSSION

Characterization of the activated carbon

Yield analysis

The yield of activated carbon was expressed as the ratio of the weight of the produced activated carbon to that of the initial material used. The yield of activated carbon production varied with different temperatures (500, 650, and 850°C) and amounts of pulp for the 1:1 impregnation ratio, resulting in yields of 24.9%, 33%, and 44.6%, respectively. Aljundi and Jarrah (2008) produced activated carbon from rice husk through chemical activation ($ZnCl_2$) and found a yield of 18% for a 3:1 impregnation ratio [15]. In addition, Sayın et al. (2016), determined that the carbonization temperature played a stronger role in the yield of activated carbon than the activation material [16]. The increase in yield was attributed to the selective separation of H and O rather than hydrocarbons or oxygenated organic compounds in the activation material [17]. The obtained yield values were quite high compared with those reported in the literature.

Moisture analysis

The moisture content obtained at varying temperatures (500, 650, and 850°C) was found to be 3.1%, 2.4%, and 1.89%, respectively. It is expected that as the temperature increases, there will be a decrease in the moisture content. Generally, the desired moisture content range in activated carbon would be low and should be kept below 50%, as reported [18].

High moisture can reduce the ability of carbon to effectively adsorb other substances by filling its pores with water molecules [19]. The results obtained emphasize low moisture and high yield in the activated carbon that will be obtained from black cumin seed pulp through the carbonization process at 850°C.

SEM results

SEM analysis is a characterization technique used to examine surface details and structures. SEM collects scattered electron signals from the surface by focusing an electron beam onto the sample and uses these signals to create an image, allowing for the observation of morphological and topographical details of the sample at high resolution [20]. First, in the pulp samples (Figure 1), it was observed that as the magnification increased, the edges of the material exhibited smooth and flat surface behavior. Considering the high-pressure technique used to obtain pulp from black cumin, which is output as a compressed layer, these structures demonstrate that the material surface is covered with a smooth, homogeneous, or thin layer. This indicates a certain thickness or density of the material on the sample surface; therefore, it interacts less when the electron beam strikes the sample surface [21]. At a magnification of 9.4 mm x 10.0k, it was determined that the bright areas of the measurement images are denser. These areas may indicate a higher content of active ingredients or a denser region on the sample surface. As electrons scatter more in dense materials and send more signals to the detector, causing glare, it is likely to obtain a bright appearance as we zoom in on the sample [22] and thought to be caused by nitrogen and phosphorus structures present in the pulp content. The average pore size was approximately 22 nm (Figure 2), indicating that the activated carbon exhibits a mesoporous structure. In a synthesis and characterization study of activated carbon from black cumin seeds activated with sulfuric acid, the pore

structures obtained were approximately 10 nm, and it was determined that the activated carbons exhibited the same mesoporous structure [23]. In another study, activated carbon obtained from pulps activated with potassium carbonate exhibited a mesoporous structure with an average pore size of approximately 20 nm [24].

EDX analysis

EDX analysis is a characterization technique used to determine the chemical structure of a sample and identify the elemental composition [25]. The results revealed an average carbon content of around 71%, followed by oxygen and nitrogen. Phosphorus was found to be the highest trace element present in the structure. The average nitrogen content of approximately 3.2% identified in pulp samples (Figure 3a) indicates the presence of organic chemicals within the residue [26]. In a study on the synthesis of activated carbon from black cumin seed pulps with $ZnCl_2$ impregnation, the nitrogen content was reported to be 5.18% [27]. Additionally, carbon content was found to be 50.35% and oxygen content was 37.09%. Compared to the study, our pulp sample exhibited a structure that is low in nitrogen and oxygen but quite high in carbon content. In another study on the synthesis of activated carbon from pulp with potassium carbonate impregnation, no nitrogen element was detected, but the residue exhibited a higher percentage of trace elements, including 1.47% phosphorus, 0.81% magnesium, 1.74% potassium, and 0.57% sulfur. Our pulp samples showed a denser and higher percentage of trace elements compared to the literature studies. The higher carbon content in our pulp samples compared to the literature studies can be directly attributed to the raw material. Both literature studies used pulp samples from Turkey, whereas the pulp in this study was obtained from Azerbaijani cumin seeds. It is possible to say that black cumin seeds from Azerbaijan yield a denser residue in terms of carbon, indicating that these samples may more suitable for activated carbon synthesis [28]. The result of the EDX analysis of activated carbon shows an increasing carbon percentage (%88), which is not surprising given the result obtained after the carbonization process (Figure 3b). The elemental percentages of activated carbon reported in the study of activated carbon prepared from black cumin pulp via $ZnCl_2$ impregnation were found to be 74.13% carbon, 3.91% nitrogen, and 19.73% oxygen. The average carbon content in our activated carbon, at 87.5%, is considerably higher than that reported in the literature. The fundamental indicator for understanding the production yield of activated carbon is its carbon content, and a high carbon level is expected [29].

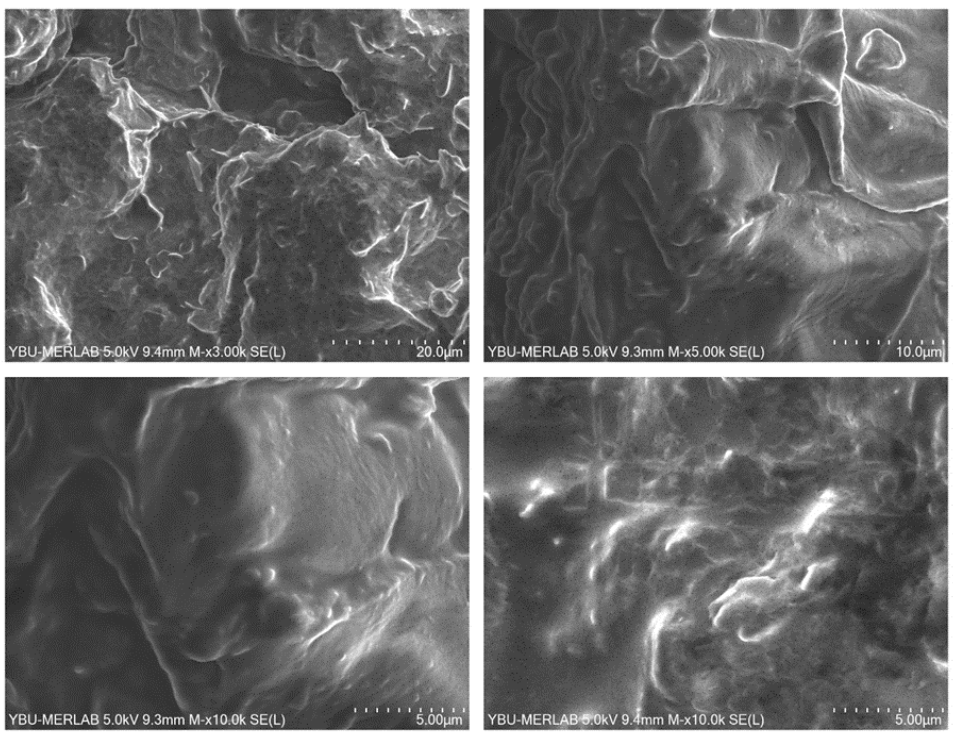

Figure 1. Black cumin seed pulp SEM analysis results.

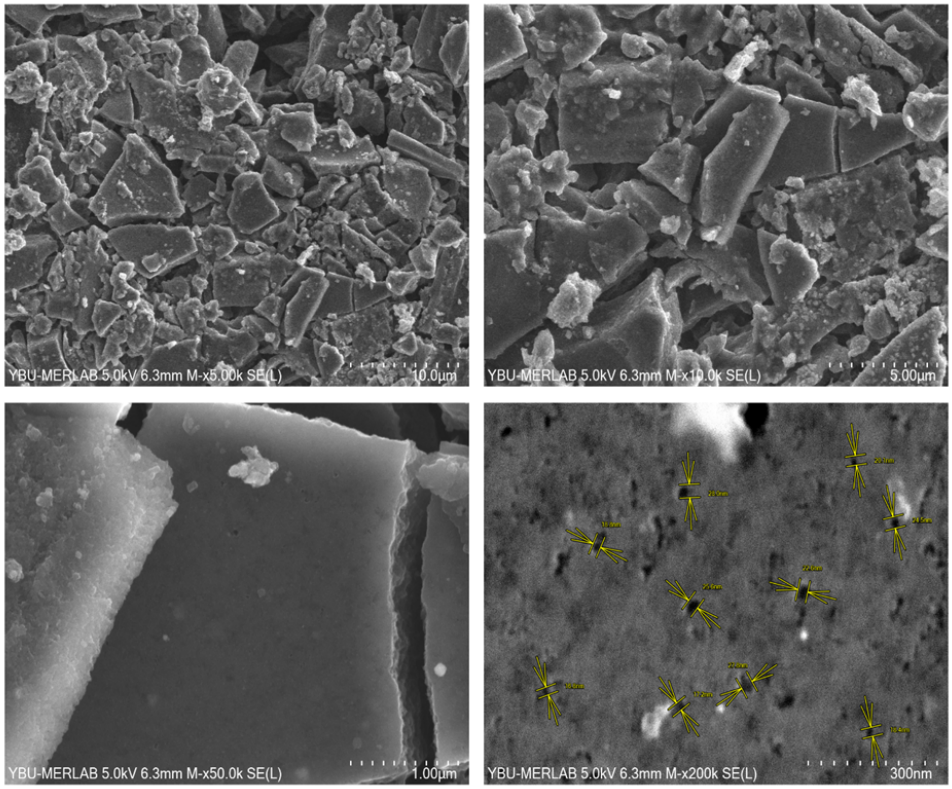

Figure 2. Activated carbon SEM analysis results.

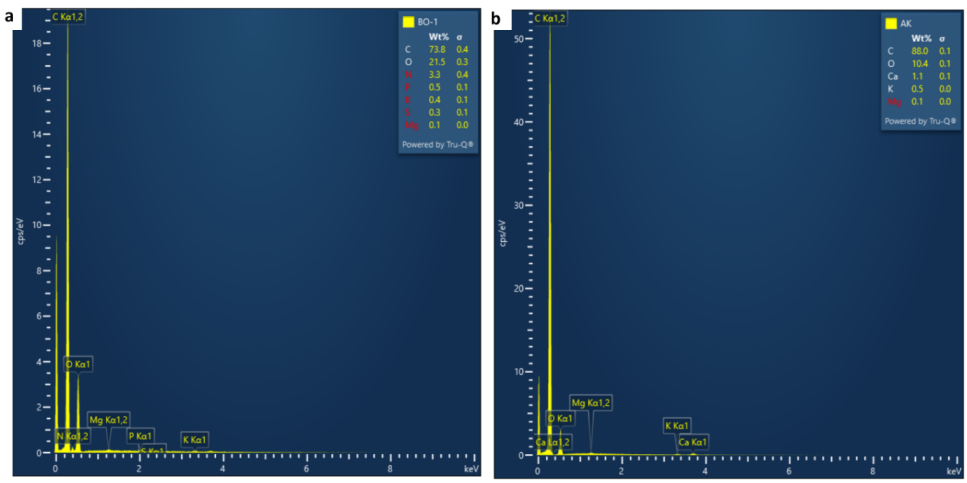

Figure 3. EDX analysis results (a) black cumin pulp and (b) activated carbon.

FTIR and Raman analysis

FT-IR analyses were conducted on the activated carbon and black cumin pulp samples. These analyses were performed to understand the molecular structures of the materials and provide information for potential applications. The obtained FTIR spectra, particularly reflecting the characteristic peaks of the carbon structure, have provided an important tool in determining the chemical composition of the material. The FTIR analysis result graphs for both samples are presented in Figure 4. The results obtained primarily indicate that the transmittance value of activated carbon is higher than that of black cumin pulps. This is believed to be due to the structural crystallinity and water content. The characteristic peaks for activated carbon were determined to be approximately 1000 cm^{-1} in intensity. Approximately at 996 cm^{-1}, there are vibrations of CH_3 and stretching motions of C-C [30]. Obtaining the maximum peak at this wavelength confirms the content when activated carbon consists of more than 80% carbon. The peak obtained just before 2500 cm^{-1} is interpreted as the reported C-O stretching band at 2361 cm^{-1} [31]. In the black cumin pulp, because of the abundance of organic components, the number of peaks obtained is higher than that of activated carbon. It is thought that the peak after 1500 cm^{-1} in the characteristic peaks of black cumin pulp could be the C-C stretching band at 1603 cm^{-1} [32]. Two peaks obtained just before 3000 cm^{-1} are interpreted as the C-H stretching at 2922 cm^{-1} [32] and the CH_3 stretching at 2873 cm^{-1} [30] based on literature information.

On the other hand, Raman analysis was performed to examine the molecular, crystal, and structural properties of the respective material samples. The obtained Raman spectrum results are presented in Figure 5. The obtained results indicate peaks at 1600 and 3000 cm^{-1} for black cumin pulp. The 1600 cm^{-1} peak is typically associated with the vibration of C=C (aromatic double bond) and can originate from vibrations of aromatic alkenes or protein amide bonds. Because the presence of organic compounds in black cumin pulp is known, the predominance of this aromatic interaction has been considered as a possible scenario [33]. Also the 3000 cm^{-1} peak generally arises from the asymmetric stretch modes of C-H (carbon-hydrogen) bonds [33]. This region is commonly observed in the Raman spectra of organic components; hence, no peaks were observed in the activated carbon Raman spectrum. The other peaks obtained at 1300 and 1600 cm^{-1} in activated carbon are reported in the literature as characteristic bands of activated carbon [34]. It is assumed that the other peaks are due to impurities.

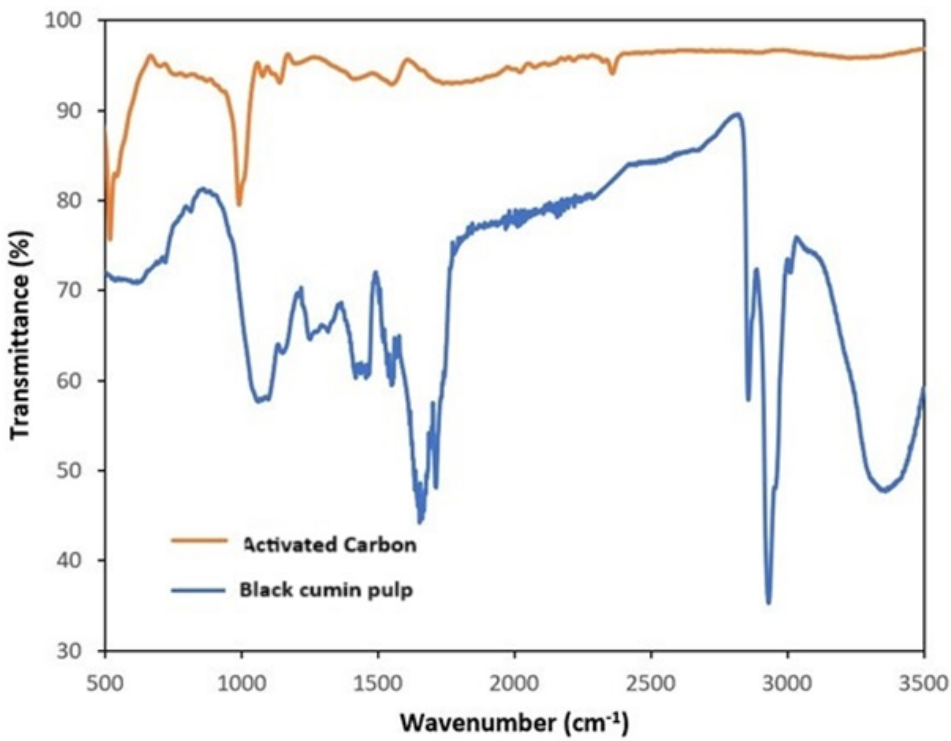

Figure 4. FTIR analysis results.

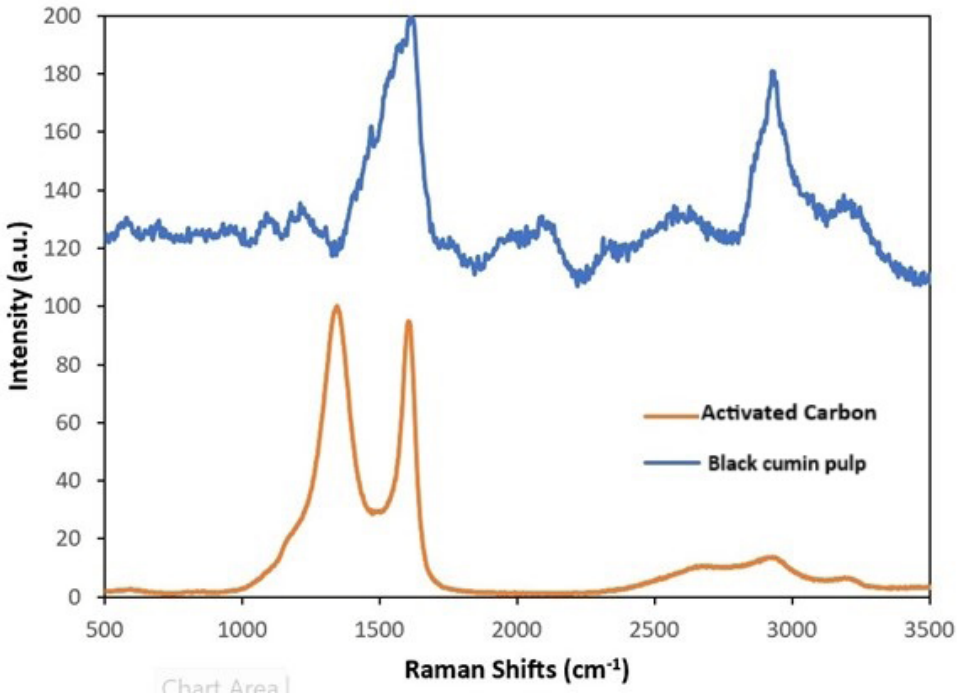

Figure 5. Raman analysis results.

Pharmaceutical soap characterization

pH analysis results

The pH measurement values obtained for the 4 different soap solutions prepared are provided in Figure 6. The calculated average pH value was found to be 8.67. Samples prepared with the same amount of soap and water but varying in quantity were preferred to minimize measurement errors. The pH results obtained from soap analysis provide information about skin compatibility. Considering that the average pH is around 8, this value can be considered slightly alkaline, and although the skin's natural pH is slightly acidic (around 4.7 to 5.75), the obtained pH value is considered tolerable [35]. It is known that the biotherapeutic soap that obtained, achieves a soothing balance with the oils used as active ingredients, enhancing its compatibility with the skin. Many commercial soaps on the market tend to be more alkaline, with pH levels between 9 and 10, or even higher. Our soap with a pH value of 8 balances effective cleansing with alkaline properties while maintaining high skin compatibility [36, 37].

Moisture analysis

Moisture analysis results conducted for the pharmaceutical soap are presented in Figure 7. Since the device has both laser and probe measurement capabilities, moisture analyses were conducted in two stages. The average moisture values obtained were found to be 41.8% for laser measurement and 47.9% for probe measurement.

Obtained moisture content of around 40% of the soap is also highly desirable. This moisture provides the material with a flexible structure, and the primary problem with soap and similar materials with moisture levels below a certain threshold is rapid drying and cracking [38]. It is believed that the average 40% moisture content in our soap formulation will play a significant role in providing an additional moisturizing layer during the cleansing process [39]. When soap interacts with water, it not only cleanses the skin but also provides moisture. This has the potential to provide benefits through a dual-action approach of cleansing and moisturizing, especially for individuals with dry or dehydrated skin.

Antimicrobial analysis

The results of pharmaceutical soap antimicrobial analysis performed on different strains are given in Table 2.

As shown in Table 2, the soap sample exhibited effective antimicrobial activity against the tested microorganisms at different concentrations. This activity was particularly pronounced in the *C. albicans* ATCC 10231 yeast strain, as measured by MIC and MFC values of 6.25 and 12.5 mg/mL, respectively. This indicates that the soap enhances its antimicrobial efficacy within spe-

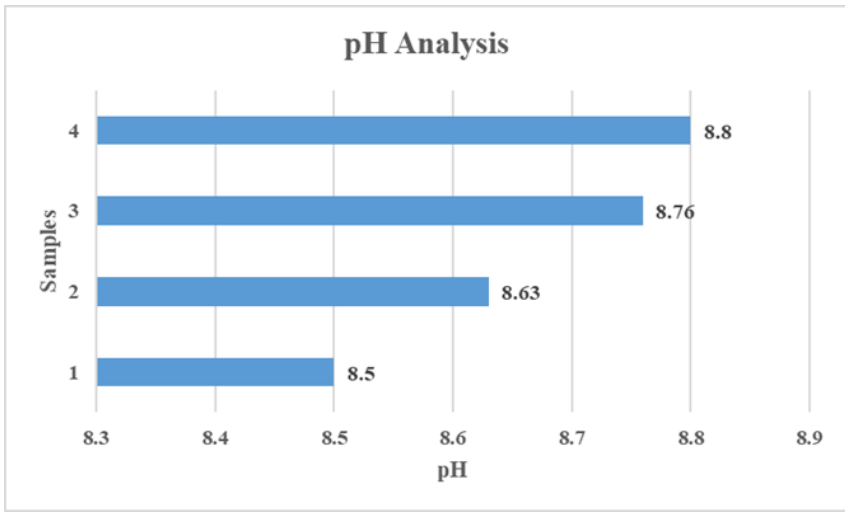

Figure 6. pH analysis of the soap samples.

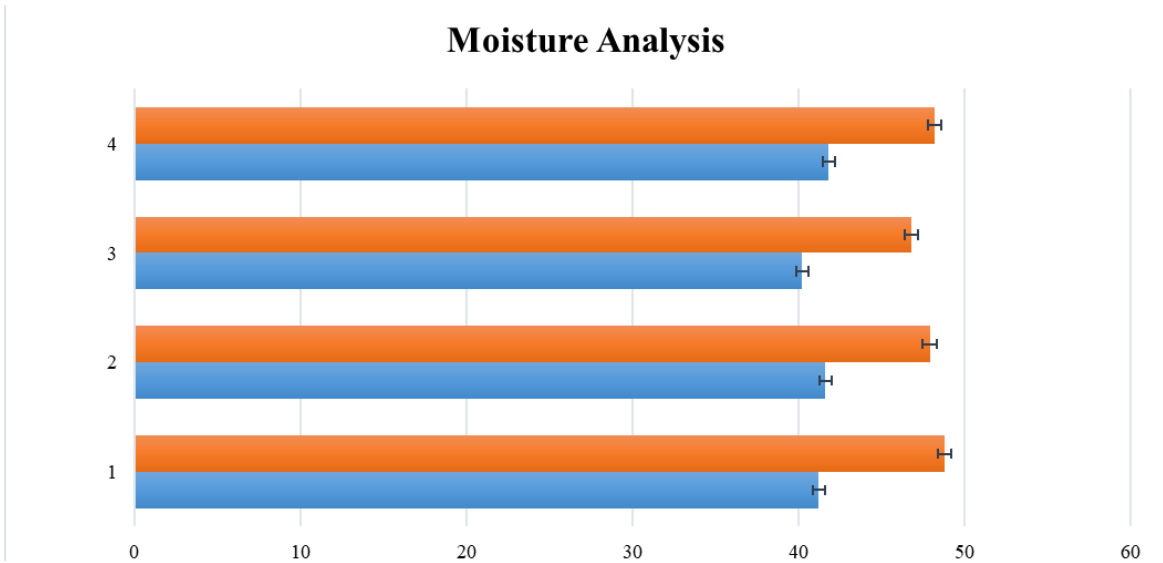

Figure 7. Moisture analysis of the soap samples with both laser and probe.

Table 2. Antimicrobial activity of the soap sample on test microorganisms.

Glycerol Concentrations (%)	mg/mL	Results
E. coli ATCC 35218	MIC	50
	MBC	50
S. aureus ATCC 25923	MIC	12.5
	MBC	50
P. aeruginosa ATCC 27853	MIC	>50
	MBC	>50
DRE ATCC 51299	MIC	50
	MBC	>50
MRSA ATCC 43300	MIC	12.5
	MBC	50
C. albicans ATCC 10231	MIC	6.25
	MFC	12.5

cific concentration ranges, particularly demonstrating significant antimicrobial activity against *C. albicans* yeast strains.

CONCLUSION

Despite well-documented ethnobotanical literature, there is limited scientific knowledge regarding the prevention of skin infections, skin compatibility, and other by-product efficacy of herbal soaps. As a result, the production of activated carbon from pulps and its incorporation into soap formulations represent an intersection between sustainable practices and phytochemical research. When integrated into soap formulations alongside other beneficial oils, activated carbon contributes to a multifaceted approach in cleansing and associated pharmaceutical processes. It is believed that the obtained soap will provide biotherapeutic effects and can be used in general cleansing processes.

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Conflıct of Interest

This article is derived from a Master's thesis dissertation entitled "Evaluation of the pharmaceutical product potential of black cumin pulp", conducted under the supervision of Assoc. Prof. Dr. Nilüfer VURAL. The authors have no conflicts of interest to declare.

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