

## Green Synthesis of Cosmetic Soaps Obtained from Laurel Nobilis

Hatice KARAER YAĞMUR<sup>1\*</sup>, Kübra BAYKARA<sup>1</sup>, Seyithan SÖNMEZ<sup>1</sup>

<sup>1</sup> Dicle University, Faculty of Science, Department of Chemistry, Diyarbakir, Türkiye

(ORCID: [0000-0002-3728-1825](https://orcid.org/0000-0002-3728-1825)) (ORCID: [0009-0005-7889-634X](https://orcid.org/0009-0005-7889-634X)) (ORCID: [0009-0003-1249-7640](https://orcid.org/0009-0003-1249-7640))



**Keywords:** Laurel nobilis oil, activated carbon, soap, saponification value, peroxide value.

### Abstract

In this study, Laurel nobilis oil was obtained from Laurel nobilis fruits. Qualitative analysis of Laurel fixed oil was performed by GC-MS. Additionally, saponification value (SV), acid value (AV), free fatty acid value (% FFA) and peroxide value (PV) values of bay Laurel oil were determined as 140.0 mg KOH/g, 26.5 mg KOH/g, 13.25 mg KOH/g, and 10.28 meq O<sub>2</sub>/kg, respectively. Activated carbon was obtained as a result of carbonization of the Laurel fruit seeds, which were waste as a result of oil extraction, at 500 °C. Activated carbon was characterized by FT-IR, BET, SEM and TGA. Soap is made of different fatty acid salts, either sodium or potassium. The obtained Laurel fruit oil and activated carbon were used in the preparation of solid soaps with peeling effect by the cold process method. pH, foam test, total alkaline test, and total fatty matter determination (TFM) of the prepared soaps were determined. Thus, the synthesis of activated carbon with peeling effect, which has good cleaning and foaming properties, was carried out.

### 1. Introduction

Natural cosmetics with bioactive phytochemical substances have a significant pharmacological impact and aesthetic value while posing less of a risk to the environment and users. In addition to other negative effects, many artificial and synthetic ingredients in skin and hair cosmetics typically cause dry skin, irritation, and damaged skin. The primary byproduct of the chemical interaction between lye solution (sodium hydroxide) and triglyceride, or fixed oil from seeds, is soap. The method is known as saponification (Scheme 1) [1]. Soaps are chemical mixtures of Na<sup>+</sup> or K<sup>+</sup> ions with fatty acids. The components of fats and oils utilized in the production of soap are called fatty acids. They are divided into two groups: saturated and unsaturated. However, the most prevalent unsaturated fatty acids are oleic and linoleic acids, while the most prevalent saturated fatty acids are palmitic and stearic acids [2]. The types of oils used, the degree of saponification, the age of the soaps, and the

strength and purity of the alkali all affect the chemical properties of soaps. Total fatty acids (TFM), pH, free alkali, and foam stability are examples of these physico-chemical properties [3].

Laurel nobilis L. (Lauraceae), also referred to as sweet bay, bay laurel, roman laurel, or real laurel, is an evergreen tree that may grow up to 10 meters in height. In Mediterranean countries including Turkey, Algeria, Morocco, Portugal, Spain, and Italy, bay laurel is farmed economically for its aromatic leaves and oils [4]. In addition to its special aroma, it is utilized globally as a medicine. Certain elements of this plant, like its organic acids and essential oils, have demonstrated potent antibacterial properties [5]. The industrially significant plant L. nobilis is utilized in medications, food products, and cosmetics. The food sector uses a lot of dried leaves and essential oils to season meat items, soups, and fish [6]. Of the fixed oil in bay laurel trees (Figure 1), 24–30% is found in their fruits (Figure 1). Typically, fixed oil made from the

\*Corresponding author: [profhatice23@hotmail.com](mailto:profhatice23@hotmail.com)

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fruit's kernel has a high lauryl acid content. About 70% of the entire fruit is made up of the fruit's kernel. [4]. The fruits' volatile and fixed oils are mostly utilized in the production of soap. Laurel nobilis L. essential oil is widely produced and used at home in Hatay (a province of Turkey). Laurel nobilis L. Trees (Figure 1) can be found growing wild in Antakya, Defne, Yayladagi, and Samandagi (districts of Hatay). There are differences in the climate and geography of these three towns.

Natural soaps are becoming more and more popular as consumers start to explore alternatives to commercial soaps with all of their ingredients. Therefore, the tendency towards activated carbon soaps has increased. The phrase "activated carbon" refers to a group of highly porous carbonaceous compounds. Because of their large surface area, activated carbons are powerful adsorbents. Due to their lower cost and renewable nature, agricultural wastes are regarded as a very important feedstock for the synthesis of activated carbon [7].

Because of its high surface area and pores, activated charcoal has gained popularity as an ingredient in cosmetic products. It can pull bacteria and pollutants from the skin by absorbing substances through these pores. This makes it possible for it to absorb chemicals and pollutants off the skin's surface, allowing it to be rinsed or peeled off to remove any clogged pores and detoxify the skin. Activated charcoal, which has the function of adsorbing oil, dark spots and pollutants that stick to our skin, is used as an additive in cosmetic products. It is utilized in a variety of beauty products, including face masks, cleansers, and even soaps, because of its adsorbing qualities [8].

In our country, bay leaves are generally used as spices in food. In the literature, bay leaves and activated carbon produced from these leaves have been used in adsorption studies. Bay oil obtained from bay fruits is generally used in soap making. As a result of Laurel oil production, tons of Laurel fruit seeds are in waste condition and do not contribute to our

country's economy. On the contrary, they cause environmental pollution. Activated carbon is an expensive product. In this study, it was aimed to obtain oil from Laurel fruits and to produce activated carbon from waste seeds. The obtained activated carbon was used as an additive in the production of Laurel soap. Thus, it was aimed at produce a high value cosmetic product from a waste material. No study was found in the literature on obtaining such a product from bay laurel fruits for cosmetic purposes. It is thought that this study will contribute to the literature in this respect.

## 2. Material and Method

### 2.1. Materials

Laurel nobilis fruits were supplied from the Defne district of Hatay (in November 2023). Sodium hydroxide (NaOH), potassium hydroxide (KOH), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, %98), phenolphthalein (C<sub>20</sub>H<sub>14</sub>O<sub>4</sub>, 3,3-bis(4-hydroxyphenyl)-1(3H)-isobenzofuranone) and hydrochloric acid (HCl, %37.5) were supplied from Merck.

### 2.2. Obtaining Laurel nobilis oil from Laurel nobilis fruits

Laurel nobilis fruit oil was obtained according to the literature [9]. Hot water flotation is a traditional method for extracting Laurel oil in Hatay. First of all, bay fruits were washed with pure water to remove dust. 200 g of L. nobilis fruit was taken into a 500 mL beaker. Distilled water was added to cover it. The mixtures were boiled at 80-90°C for 5 hours. After cooling, the upper oil layer was separated and extracted with hexane. Approximately 100 mL of oil was obtained from approximately half a kilo of bay fruit. L. nobilis oil was weighed and stored at 4°C for use. The Laurel oil obtained was characterized by GC-MS (Figure 4).



**Figure 1.** Laurel nobilis tree and Laurel nobilis fruit.

### 2.3. Determination of physicochemical parameters of Laurel oil

#### 2.3.1. Saponification value of oil (SV)

2 g of the oil sample was added to a vial containing 30 mL of ethanolic KOH/NaOH and then refluxed for 30 minutes to ensure complete dissolution of the sample. After the sample cooled, 1 mL phenolphthalein was added and titrated with 0.5 M HCl until it gave a pink color [1], [2].

$$SV = \frac{56.1 * M(V2 - V1)}{W} = \frac{mgKOH}{g} \text{ or } \frac{mgNaOH}{g} \quad (1)$$

where W=weight of sample(g); V1= Volume of hydrochloric acid used in test; V2= Volume of hydrochloric acid used in the blank; M=Molarity of hydrochloric acid; Molecular weight of KOH (g/mole) = 56.1

#### 2.3.2. Acid value (A.V.)

1 g of oil was taken into a conical flask containing 25 mL of methanol, and 3 drops of phenolphthalein indicator were added. The mixture was heated in a water bath for 5 minutes and titrated with 0.1 M KOH until a pink color appeared. The acid value was calculated using the following equation [1].

$$AV = \frac{VKOH * M * 56.1 \left( \frac{g}{mol} \right)}{W} = mg \frac{KOH}{g} \quad (2)$$

M: molarity of KOH, W: weight of sample (g)

#### 2.3.3. Determination of Free Fatty Acids (FFA)

The FFA value of the oil was calculated according to the literature [10].

$$\text{Free Fatty Acids (FFA)} = \text{Acid Value (AV)} / 2 \quad (3)$$

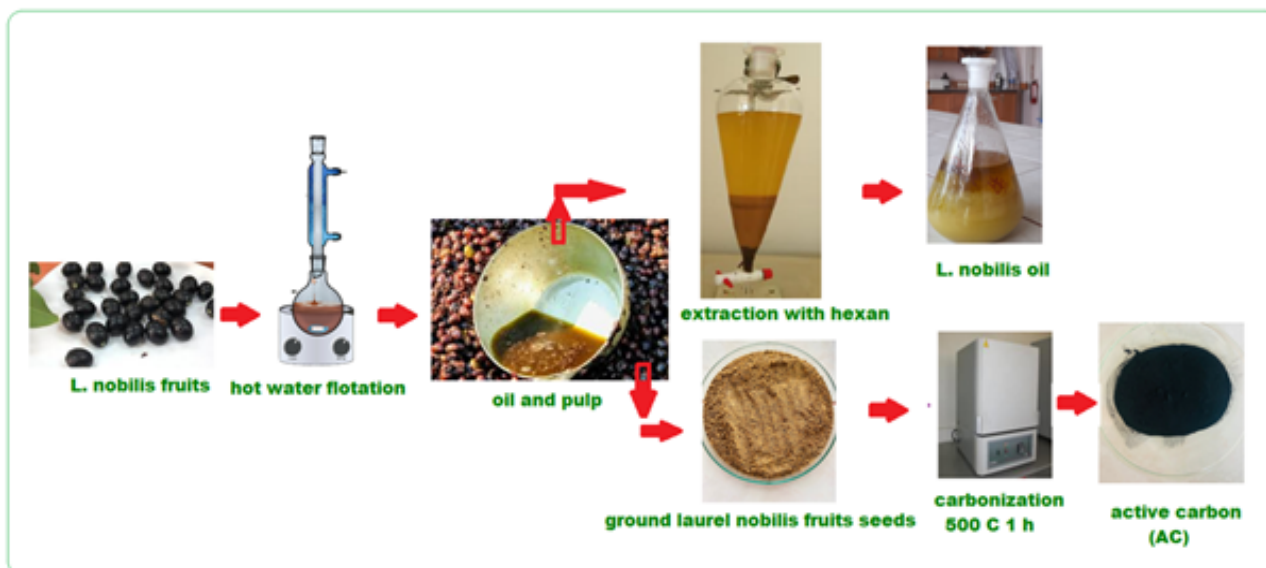
FFA of Laurel nobilis oil: 13.25 mg KOH/g

#### 2.3.4. Peroxide value (PV)

The PV value of the oil was calculated according to the literature [1].

### 2.4. Synthesis of activated carbon from Laurel nobilis fruit seeds

L. nobilis fruits were used as a starting material in experimental studies to produce activated carbon. After obtaining bay oil from bay fruits by hydroflotation, bay fruit seeds were separated from the pulp, washed with distilled water and dried in an oven at 105 °C for 48 hours. After the drying process, bay fruit seeds were ground in a grinder and stored in a desiccator to be used. The carbonization process is carried out by placing the 7 cm diameter and 100 cm long stainless steel tube reactor in the furnace (Protherm PTF 12/20/400) and passing nitrogen (N<sub>2</sub>) gas at a flow rate of 150 mL/min for 1 hour at 500 °C at a speed of 15 °C/min. Heating was provided. After the carbonized sample was cooled to room conditions under a nitrogen atmosphere, HCl (0.1 M) was refluxed for 1 hour to purify the sample from impurities and reduce the ash content. Then, the sample was filtered and washed with hot distilled water until it did not react with chloride. A Chloride test (1%) was performed with AgNO<sub>3</sub> [11]. Experimental stages of obtaining Laurel oil and activated carbon from Laurel fruits are shown in Figure 2. The resulting activated carbon was coded as AC (Figure 2).



**Figure 2.** Obtaining bay oil and activated carbon from bay fruits

## 2.5.Characterization

The fatty acid composition of the *Laurel nobilis* was analyzed using a Rtx-5MS (30 m x 0.25 mm inner diameter x 0.25  $\mu\text{m}$  film thickness) column on a Shimadzu GC-MS/QP2010. Carrier gas is helium (2.32 mL/min.), split 1. Ion source temperature was applied as 300  $^{\circ}\text{C}$  for qualitative analyses. It was diluted with methanol for qualitative analysis of the oil. The Brunauer-Emmett-Teller (BET) surface analyzer (a TriStar 3000 surface analyzer.) was used to measure the surface area and pore size distribution of the AC at a temperature of -196  $^{\circ}\text{C}$  and  $\text{N}_2$  gas. Conversely, using an FTIR spectrometer (Model 2000, Perkin-Elmer, USA), Fourier transform infrared (FTIR) spectra were acquired throughout the range of 4000-400  $\text{cm}^{-1}$ . TG measurement of activated carbon was performed between 20 and 1000  $^{\circ}\text{C}$  (200 mL  $\text{min}^{-1}$ , in  $\text{N}_2$  atmosphere 10  $^{\circ}\text{C min}^{-1}$ , Perkin Elmer Diamond Thermal Analysis).

## 2.6. Preparation of Soap

One of the most significant components of fats, fatty acids (FAs) and free fatty acids (FFAs) are non-volatile aliphatic monocarboxylic molecules that can also be used as a soap base. One of the most often used for personal hygiene is soap. It is created when FA triglycerides undergo the saponification reaction (hydrolysis) with a strong base (often potassium or sodium hydroxides), resulting in the production of glycerol and soap (potassium or sodium salts of FAs). Although the primary ingredient in soap is salts of FAs, it also contains glycerol, FFAs, and unsaponifiable

matter, which includes sterols, higher aliphatic alcohols, pigments, hydrocarbons, and other substances. Free-form FFAs also have a significant impact on the quality of soap. FFA-enriched soap has higher fattening and moisturizing qualities, as well as improved foaming. These attributes serve as markers of high-quality soap [12].

Using a cold procedure, the extracted oil was saponified. Soaps were prepared using the soap formulations in Table 1. The saponification value of olive oil and coconut oil was determined according to previous studies [13]. The main ingredient of the formula consists of the following components as a percentage by mass: 200 g of olive oil, 200 g of coconut oil, 200 g of laurel oil and 37% of the total oil amount consists of distilled water. Activated carbon (1-10%) can be used as an additive [14]. In our study, 10% of the total amount of oil was used. In active carbon soap studies, a teaspoon of active carbon was generally used. If the amount of oil used is not too much, using more than a teaspoon of activated carbon will result in gray foam [15]. Figure 3 shows the preparation of the soap. After following conventional technique, a thick, semi-solid mass of soap was obtained and allowed to cool and set for six weeks. The saponified product's pH, foaming capacity, hardness, cleaning efficacy, and total alkalinity were characterized.

**Table 1.** Formulation of soap

	Saponification Value (SAP)( gr NaOH/gr)	activated carbon free soap	with activated carbon soap
		Amount of oil(g)	Amount of oil(g)
L.nobilisoil	0.140	200 g	200 g
Olive oil	0.135	200 gr	200 gr
Coconut oil	0.183	200 gr	200 gr
Amount of water		%37 (a according to the total amount of fat )	%37 (a according to the total amount of fat )
Activated carbon		-	6 gr
Colorant		Not used	
Soap fragrance		Since Laurel oil is scented, no essence was used.	

**2.7. Determination of physiochemical parameters of soaps**

**2.7.1. Determination of pH of soaps**

pH values of soaps were determined using a pH meter according to the literature. 1 g of soap with and without activated carbon was weighed separately. The soaps were dissolved in 10 mL of distilled water. Make up to 100 mL with distilled water to obtain a 1% homogeneous soap solution.

**2.7.2. Foaming ability tests**

Each of the soaps, 0.2 g, was placed in a 100 mL conical flask, and 10 mL of pure water was added. The mixture was shaken vigorously for 2 minutes to form foam. After shaking the mixture, it was left for 10 minutes. Foam height was measured.

**2.7.3.Total Alkaline**

100 mL of ethanol and 5 mL of 1 N H<sub>2</sub>SO<sub>4</sub> solution were added to 10 g of soap. The mixture was heated until the soap sample was completely dissolved and then titrated with 1 N NaOH using phenolphthalein. Total alkali was obtained by the following formula:

$$\%Total\ alkali = \left( \frac{V_{acid} - V_{base}}{m} \right) \times 3.1 \quad (4)$$

m: mass of soap

**2.7.4. Total Fatty Matter Determination (TFM)**

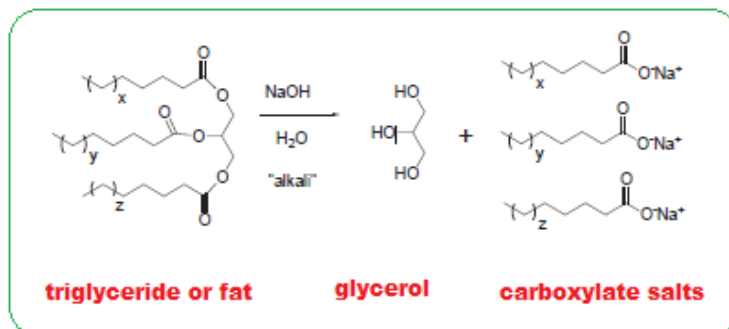
10 g of the finished soap was weighed, 150 mL of distilled water was added, and it was heated slightly in a water bath. The soap was dissolved in 20 mL of 15% H<sub>2</sub>SO<sub>4</sub> by heating until a clear solution was obtained. The fatty acids on the surface of the resulting solution were solidified by adding 7 g of beeswax and reheated. The apparatus was allowed to cool to form a solid cake-like mold. The oily patch on the surface was removed and allowed to dry for several days and weighed to obtain the total oily matter using the following formula (5):

$$\%TFM = \left( \frac{A - X}{w} \right) \times 100 \quad (5)$$

A: mass of wax+oil

X: mass of wax

W: mass of soap



**Scheme 1.** Saponification Reaction



Figure 3. Preparation of soap

### 3. Results and Discussion

Saponification value (SV) can be expressed as a measurement of the alkali-reactive groups in fats and oil or as the milligrams of KOH/NaOH required to saponify one gram of oil. Additionally, the SV shows the oil's average molecular weight. In other words, SV describes the molecular weights of the fatty acids in oil, which are inversely related to the fatty acids' average molecular weights or chain lengths. It has been stated that this is one of the key factors in the industrial methods used to make soap. According to studies, oils with high SVs are good raw materials for the soap industry, but oils with low SVs are not acceptable for use in the soap industry. The saponification value of Laurel nobilis oil was calculated as 0.1925 g KOH/g (192.5 mg KOH/g) or 0.140 g NaOH/g (140.0 mg NaOH/g).

These values are above the specification range of (175 mg KOH/g-187 mg KOH/g) recommended for oils by ASTM (the American Society for Testing and Materials). Vermaak et al., state that high SV indicates the predominance of low molecular weight fatty acids [16]. Even if the SV obtained is greater than the ASTM standard values, the values from this study are lower than those from the most common oils, such as palm oil (200 mg KOH/g), and coconut oil (257 mg KOH/g) reported by Nchimbi [2].

The acid value (AV) of Laurel nobilis oil was calculated as 0.0264 g KOH/g or 26.5 mg KOH/g. For oils and fats, AV is a standard parameter used in specification and quality control. It also serves as a physicochemical property indicator for oil, indicating its age, quality, edibility, and suitability for usage in various sectors. There could be a greater quantity of FFA in the oil, which would explain the increased AVs seen. These raise the possibility of oxidative deterioration, which lowers the material's suitability for industrial uses. Therefore, in order to be suitable for industrial uses such as soap production, the resulting oil must be refined to lower acidity to the prescribed requirements [2]. The acidity of an oil indicates how much lipase action has broken down the glycerides. Heat and light typically speed up the breakdown process. Amino acids, free fatty acids, and acid phosphates are the acids that are often produced. Compared to other forms of acids, free fatty acids are generated more quickly. The oils' low acid levels also suggest that they might be kept in storage for a long period without degrading [17].

FFA of Laurel nobilis oil: 0.01325 g KOH/g (13.25 mg KOH/g). Fatty acids, or FFAs, are created in any stage of the process by hydrolytic reactions from the triglycerides of oils. One of the most often used metrics to evaluate the quality of oil during production, storage, and marketing is its FFA content. It is also used to categorize the oils.

It has been stated that a high percentage of FFAs in oils indicates low quality; this could be the consequence of impurity contamination, which could cause the ester linkage to hydrolyze, raising the percentage of FFAs. On the other hand, low FFA levels indicate high quality and prevent the oil from going rancid or smelling bad, which makes it more suitable for usage in the soap manufacturing sector [2].

The peroxide value (PV) of Laurel nobilis oil was calculated as 10.28 meq O<sub>2</sub>/kg. The peroxide value (PV) is one of the most commonly assessed quality metrics during the processing, storage, and marketing of oil. It is typically is an important indicator of the degree of oxidation of fatty acids [18], [19]. The conventional method for determining PV is based on the reaction between potassium iodide and hydroperoxides found in oils, which causes iodide oxidation to produce iodine [20]. The highest level of peroxide value (PV) permitted for edible oils and cold press oils in accordance with Iranian national standards can be divided into four ranges:  $0 < PV \leq 1$ ,  $1 < PV \leq 2$ ,  $2 < PV \leq 5$ , and  $5 < PV \leq 10$  meq/kg for edible oils at the time of production, free at the port of shipment, and consumable limit, in that order. Additionally, the maximum PV levels for refined and cold press oils are  $PV < 10$  and  $PV \leq 15$  meq/Kg, respectively, according to the Codex standards [21].

The indicator of oil's oxidative rancidity is the peroxide value. When an enzyme or specific chemical substances are present, unsaturated fatty acids' double bonds might become more oxidatively rancid. The release of short chain carboxylic acids is what gives rancidity its characteristic flavor and odor. A greater rate of rancidity is correlated with elevated peroxide levels. The oils' low peroxide values suggest that, at room temperature, oxidative rancidity will not affect them as much [17]. Antioxidants added to oils may result in low peroxide levels. Antioxidants can be added so as to prevent rancidity and increase shelf-life.

The chromatogram of GC-MS analysis and shows fatty acid compositions of Laurel nobilis oil

were given in Figure 4. 26 components of the total oil content, were identified by GC-MS analysis of the hydrodistilled fixed oil of the fruits. 1.8-cineole was identified as the main component. According to the literature, it has been determined that the main component of Laurel oil obtained from Turkey is 1.8-cineole [22].

Soap was characterized according to the literature [1]. The pH values of soaps with and without activated carbon were determined as 8.75 and 8.85, respectively. pH is an important parameter that indicates whether the soap is alkaline or acidic, and soap that is too alkaline can irritate the skin [23]. Literature has shown that soaps will not harm skin or fabric when the standard pH value of soap is between 7 and 10 [22]. Additionally, the natural acidic state of the skin can be neutralized by high alkaline soap [25].

The foam test was performed according to the literature [1]. While it was 5.3 cm for soap with activated carbon, it was measured as 4.8 cm for soap without activated carbon.

Total alkaline value was calculated according to the literature [26]. The % total alkali values were defined as 0.31 and 0.25 for activated carbon and without activated carbon respectively. Total alkaline content is one of the parameters that determine the corrosiveness of any soap [27]. According to the Bureau of Indian Standards (BIS), the alkaline content of quality soaps should be less than 5%, while according to ISO, quality soaps should contain only less than 2% alkali [27, 28].

Total Fatty Matter Determination (TFM) was determined according to the literature [3]. The %TFM values were defined as 73.23 and 72.52 for activated carbon and without activated carbon respectively. A lower TFM value is generally associated with hardness and lower quality. In some European countries, soaps with a minimum of 75% TFM are considered Grade 1. If the TFM value is 65%, this is considered a Grade 2. Soap with a higher TFM gives more suds, lasts longer, and most importantly, cleans your skin better and more gently. Grade 3 quality soap must have at least 60% TFM [28].

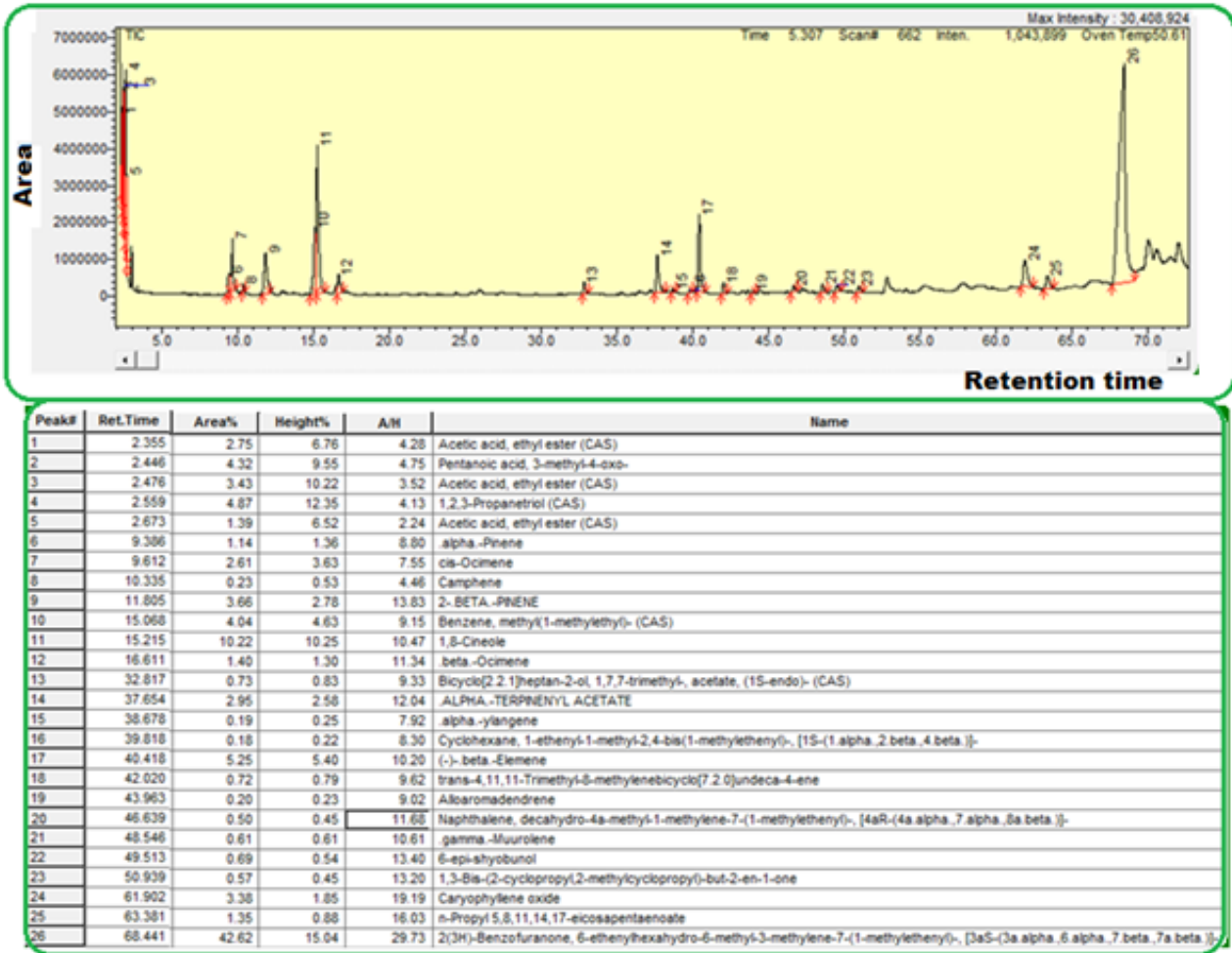


Figure 4. GC-MS chromatogram of Laurel nobilis oil and fatty acid compositions of Laurel nobilis oil.

The FT-IR method, the primary uses of which are illustrated in Figure 5, was utilized to characterize the surface functional groups. The C = C stretching of aromatic rings is responsible for the bands with centers of 2320 and 2103  $\text{cm}^{-1}$ , respectively [29]. The bands within the range of 1800–1500  $\text{cm}^{-1}$  correspond to the C=O stretching vibrations of the lignin's keto-carbonyl groups and the C=C of its aromatic rings (1660 and 1545

$\text{cm}^{-1}$ ). Alcohol, carboxyl, and phenol all have C–O stretching vibrations, which are responsible for the bands between 1300 and 1000  $\text{cm}^{-1}$ . The existence of aromatic benzene rings is indicated by the bands about 950, 747, and 613  $\text{cm}^{-1}$ . These bands are attributed to the aromatic C–H out-of-plane bending [30], [31]. The absorption peak is at 700-400  $\text{cm}^{-1}$  for the C-C stretching vibration. [32].



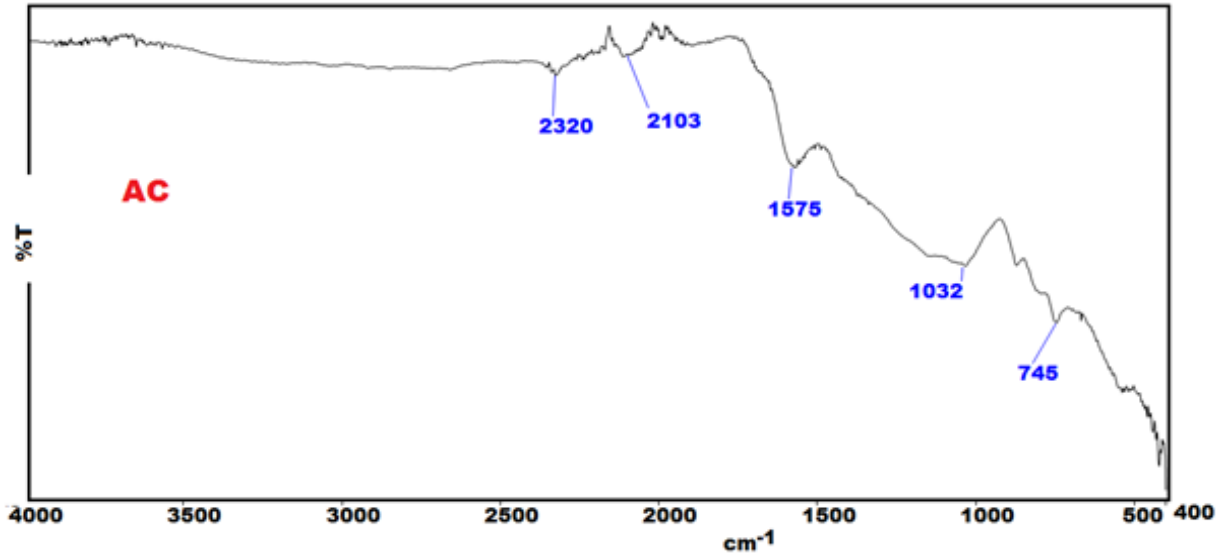


Figure 5. FT-IR spectrum of AC

The nitrogen adsorption-desorption isotherm of the activated carbon was measured in order to determine their physical characteristics, including specific surface area and total pore volume. The BET surface area was determined using the Brunauer-Emmett-Teller (BET) equation. The specific BET surface area and average pore diameter of the obtained activated carbon were determined as 783.58 m<sup>2</sup>/g and 3.58 nm. Also, the total pore volume is 0.70 cm<sup>3</sup> g<sup>-1</sup>. According to IUPAC, pores are divided into 3; micropore (<2 nm), mesopore (2-50 nm), and macropore (>50 nm) [30]. Table 2, which displays the BET surface areas of the AC activated at 500 °C, demonstrates the large surface area of the activated charcoal. The majority of the activated carbon generated in this work has mesoporous pores. The activated carbon adsorption and desorption isotherm generated at 500 °C was displayed in Figure 6.

The TGA curve of the resulting AC is given in Figure 7. Initial decomposition temperature (Ton) 276 °C. While the temperature at which 5% mass loss occurred was 481 °C, the remaining mass loss at 1000 °C was determined as 70.92.

The SEM image of AC was given in Figure 8. When you look at the images, you can see a non-flat, heterogeneous structure consisting of different shapes.

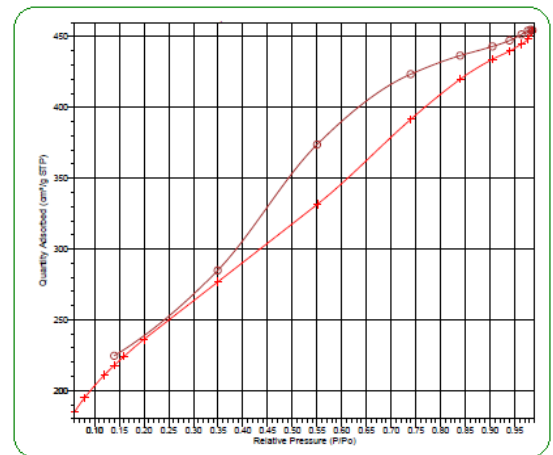


Figure 6. Adsorption–desorption isotherm of N<sub>2</sub> at 77 K of the activated carbon at 500°C.

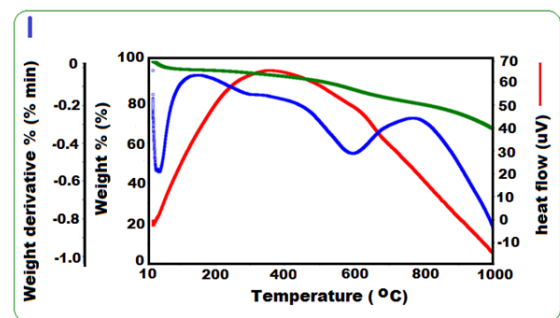


Figure 7. TGA analysis of AC

Table 2. BET results of activated carbon

	SBET (m <sup>2</sup> /g)	micro volume (V <sub>micro</sub> ) (cm <sup>3</sup> g <sup>-1</sup> )	meso volume (V <sub>meso</sub> ) (cm <sup>3</sup> g <sup>-1</sup> )	total volume (V <sub>t</sub> ) (cm <sup>3</sup> g <sup>-1</sup> )	Average pore diameter (nm)
AC	783.58	0.039	0.661	0.70	3.58

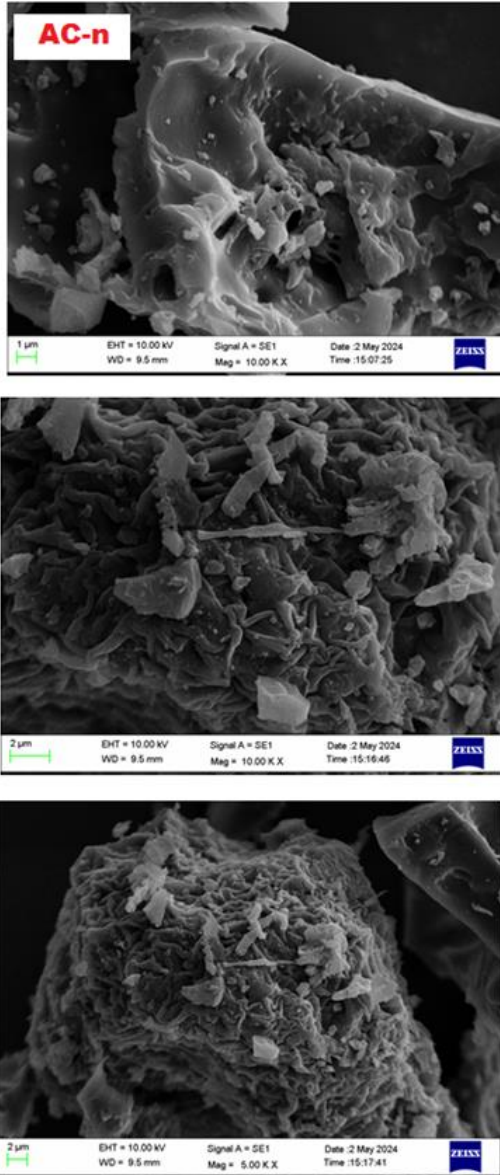


Figure 8. SEM images of AC

#### 4. Conclusion and suggestions

*L. nobilis* oil was obtained from Laurel fruits brought from Hatay's Defne district in November 2023 by the hydroflotation method. Activated carbon was obtained from Laurel fruit seeds, which were waste as a result of oil extraction. The obtained activated carbon was characterized by FTIR, TGA and BET. Solid soap was obtained by the cold process method using oil obtained from bay fruits and activated carbon. Physicochemical parameters of the soap were determined. Activated carbon soaps with peeling effect and good foaming and cleaning properties were obtained.

#### Contributions of the authors

Hatice Karaer Yağmur: Methodology, Formal analysis, visualization, data curation, writing & editing and resources.

Kübra Baykara and Seyithan Sönmez: Investigation and resources.

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#### Conflict of Interest Statement

There is no conflict of interest between the authors.

#### Statement of Research and Publication Ethics

The study is complied with research and publication ethics.

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