

Evaluation of Eucalyptus Root Wood in Activated Carbon Production with Two-Stage Production Method

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Abstract – In this study, lignocellulosic wastes were utilized in activated carbon production. The root wastes remaining on the soil surface after cutting of eucalyptus trees (*Eucalyptus grandis*) grown in Karabucak region, Mersin, Turkey were evaluated as lignocellulosic raw materials. For this purpose, eucalyptus root wood was ground and sized, and wood flours with a size between 20-40 mesh were used in the production of activated carbon. Activated carbon productions were carried out in two stages. For this, the root wood flours were firstly carbonized in a closed autoclave at 200°C and 225°C for 24 hours in the hydrothermal stage by utilizing the subcritical point of water. Afterward, activated carbons were produced in the pyrolysis stage at 400°C, 500°C, 600°C and 700°C for waiting 30 minutes in nitrogen environment. The yield calculations, scanning electron microscopy (SEM), Brunauer-Emmett-Teller (BET), and elemental analyses of the activated carbon samples were performed. According to the findings, the surface area of the activated carbons produced at 700 °C using the two-step process was obtained as 267.08 m²/g. According to the BET analysis results, it was understood that activated carbon samples generally exhibited mesoporous properties. According to the SEM images obtained, it was understood that the activated carbons had a partially porous structure. Pyrolysis process at low temperatures ensured higher O/C and H/C ratios and more functional groups in the structure of activated carbon. However, as the temperature increased, the hydrogen and oxygen ratios in the structure of activated carbon decreased. As a result, it is understood that activated carbons with intermediate level surface area and oxidized functional groups can be produced by using two-stage hydrothermal and pyrolysis methods of eucalyptus root woods.

Keywords – Hydrothermal Carbonization, Pyrolysis, Eucalyptus Root Wood

Okalıptus Kök Odununun İki Aşamalı Üretim Yöntemi ile Aktif Karbon Üretiminde Değerlendirilmesi

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Araştırma Makalesi

Öz – Bu çalışmada, lignoselülozik atıklar aktif karbon üretiminde kullanılmıştır. Lignoselülozik atık olarak Mersin Karabucak bölgesinde yetiştirilen okalıptüs ağaçlarının (*Eucalyptus grandis*) kesim sonrasında toprak yüzeyinde kalan kök atıklarının değerlendirilmesi amaçlanmıştır. Bu amaçla okalıptüs ağacının kök odunu öğütüldükten sonra boyutlandırılmış ve 20-40 mesh boyut arasında kalan odun unları aktif karbon üretiminde kullanılmıştır. Aktif karbon üretimi iki aşamada gerçekleştirilmiştir. Bunun için kök odunu ilk olarak kapalı bir otoklavda 200°C ve 225°C'de 24 saat boyunca suyun kritik noktasından yararlanılarak hidrotermal olarak karbonize edilmiştir. Daha sonra piroliz aşamasında 400°C, 500°C, 600°C ve 700°C'de 30 dakika süreyle azot ortamında bekletilerek aktif karbonlar üretilmiştir. Aktif karbon numunelerinin verim hesaplamaları, taramalı elektron mikroskobu (SEM), Brunauer-Emmett-Teller (BET) ve elementel analizleri yapılarak karakterizasyon işlemi gerçekleştirilmiştir. Elde edilen bulgulara göre, iki aşamalı proses kullanılarak 700 °C'de üretilen aktif karbonların en yüksek yüzey alanı 267,08 m²/g olarak hesaplanmıştır. BET analiz sonuçlarına göre aktif karbon örneklerinin genelde mezogözenek özelliği gösterdiği anlaşılmıştır. Elde edilen SEM görüntülerine göre aktif karbonların kısmen gözenekli bir yapıya sahip olduğu anlaşılmıştır. Düşük sıcaklıklardaki piroliz işlemi aktif karbonun yapısında O/C ve H/C oranlarının ve fonksiyonel grupların daha fazla olmasını sağlamıştır. Ancak sıcaklık yükseldikçe aktif karbonun yapısındaki hidrojen ve oksijen oranları azalmıştır. Sonuç olarak okalıptüs kök odunlarından iki aşamalı hidrotermal ve piroliz yöntemleri kullanılarak orta düzey yüzey alanına ve oksitlenmiş fonksiyonel gruplara sahip olan aktif karbonların üretilebileceği anlaşılmıştır.

Anahtar Kelimeler – Hidrotermal karbonizasyon, Piroliz, Okalıptüs kökü odunu

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1. Introduction

Activated carbons (ACs) are produced from many plant sources today. In addition to wood wastes, many wastes such as various fruit peels and lignocellulosic factory wastes are evaluated. Especially wastes with high carbon content and amount are preferred. Nowadays, as a result of industrialization, it has become indispensable to use ACs with very high pore properties and adsorption properties in order to prevent the spread of various pollutants in the environment (Chattopadhyaya et al 2006, Adinata et al 2007). As the area of use expands, the properties of ACs are also improved. Today, ACs with rich pore structures and absorption properties are gaining importance. Eucalyptus tree is grown in our country in wetlands, swampy areas and in the Mediterranean region where temperate climate prevails. There is an artificial forest of eucalyptus trees in Karabucak region, Mersin, Turkey (Ayata 2008). In the literature, the cellulose content of Eucalyptus grandis stem and root wood was determined as 49.8% and 47.9%, respectively. Likewise, when we look at the amount of lignin in stem and root wood, this ratio was determined as 25.1% and 34.8%, respectively (Tutus 2008). Here, it is understood that root wood has higher lignin content and lower cellulose content than the stem wood (Xiao et al 2019). ACs were especially used to remove pollutants that cause bad smell, turbidity and taste in wastewater of factory wastes and domestic wastes. The use of ACs with high pore properties in this fields is extremely important for the removal of organic and inorganic substances from the industrial wastewater. Activated carbon (AC), notable for its high adsorption capacity, is one of the most important carbon-based functional materials used today in a wide range of applications, including biomedical applications, filtration processes, environmental treatment technologies, energy storage systems, and catalytic carrier materials (Chung 2019, Tsai and Jiang 2023). They can be used as absorbents in many areas such as recovery of solvent compounds in wastewater for reuse, cleaning of air in the industrial areas dealing with food, cosmetics, chemistry, and cleaning of vehicle exhausts (Samdan 2013). When the usage area of AC is examined, it gives better results in absorbing harmful chemicals in the purification of domestic and industrial wastewater due to the high pore volume increasing the surface area (Yang 2003). The free electrons in carbon structure have a positive effect on the adsorbing properties of AC. ACs generally contain elements bonded with oxygen and hydrogen. These bonding free elements occur when the carbonization process can be realized at the maximum level in the AC structure and these electrons continue to bond during the activation process. Therefore, the presence of oxygen, hydrogen and mineralized structures in the structure of AC enriches the activity of the structures (Cheremisinoff and Ellerbusch 1978). AC production is generally realized by applying different methods with physical and chemical activation processes. Therefore, different processes can be applied to the lignocellulosic material depending on the method before the carbonization process (Orkun 2011). When acidic and basic activation chemicals are used in the pyrolysis process, the structure of lignocellulosic substances can be very different. During the activation process, this causes many differences in the carbon structure (Yalcin and Arol 1993, Bansal and Goyal 2005).

In this study, the eucalyptus root woods were firstly carbonized in a closed autoclave at 200°C and 225°C for 24 hours in the hydrothermal stage by utilizing the subcritical point of water. Afterwards, ACs were produced at 400°C, 500°C, 600°C and 700°C in a nitrogen environment for 30 minutes in the pyrolysis stage. Yield calculations, scanning electron microscopy (SEM), Brunauer-Emmett-Teller (BET) and elemental analysis of the obtained AC samples were performed.

2. Material and Methods

2.1. Material

In this study, the root wood of Eucalyptus grandis trees left in the artificial eucalyptus forest in Tarsus-Karabucak region, Mersin, Turkey was used as lignocellulosic material for the production of activated carbon. Eucalyptus root wood was passed through grinding mills and turned into sawdust. Pure water was used in the hydrothermal stage and neutralization stage.

2.2. Method

Activated carbon productions were carried out in two stages. The root wood of *Eucalyptus grandis* was ground in a grinder and then classified as mesh. Afterward, the root wood particles in the size range of 20-40 mesh were used in the production of AC. First, the root wood flours were subjected to hydrothermal treatment by utilizing the subcritical property of water in a closed autoclave. The samples prepared at this stage were kept in pure water at 200 °C and 225 °C for 24 hours in a closed autoclave. According to the literature, the amount of lignin in eucalyptus root wood is higher than in other parts of the tree. In the hydrothermal carbonization method, it has been stated that the use of high temperatures in the carbonization process of woods with high lignin content generally gives better results (Jain et al. 2016). The carbonized samples removed after the hydrothermal process were filtered and washed repeatedly with pure water and subjected to neutralization process. The produced AC were repeatedly washed with pure water during the neutralization process to bring its pH value to 7. Afterwards, they were dried in an oven at 103±2°C for 24 hours. The second stage (pyrolysis process) was started for the dried carbonized samples. Table 1 shows the temperatures of the experiments performed in the first and second stages.

Table 1

The temperature conditions of the samples during hydrothermal carbonization and pyrolysis temperature

	Samples	Hydrothermal Temperature (°C)	Pyrolysis Temperature (°C)
1	S225/400	225	400
2	S225/500	225	500
3	S225/600	225	600
4	S225/700	225	700
5	S200/400	200	400
6	S200/500	200	500
7	S200/600	200	600
8	S200/700	200	700

The pyrolysis reactor was purchased from Proterm Furnaces. For the pyrolysis process, 2.5 g. of the samples were weighed and taken into the pyrolysis reactor. The lids of the reactor were tightly in closed environment was continuously supplied with nitrogen gas (N₂) at a flow rate of 0.5 l/h. When samples into the pyrolysis reactor reached the desired temperature, they were kept for 30 m. and then allowed to cool in the nitrogen environment in the reactor. The samples removed from the reactor were washed with pure water on filter paper until the pH was 6-7. After washing, the AC samples were placed in an oven for drying at 85°C for 12 h. After drying, the AC samples were and stored for use in the characterization stages.

2.3. Yield Calculation Analysis

In the two-stage AC production process, yield calculations were made by proportioning the amount of dry AC obtained after both stages according to the amount of material initially entering the reactors. The yield calculations of the samples were calculated according to equation 1 (Karapınar, 2018).

$$\text{Yield (\%w)} = \frac{\text{Obtained Dry Activated carbon (g)}}{\text{Dry matter used initially (g)}} \times 100 \quad (1)$$

2.4. BET (Brunauer-Emmett-Teller) determination of surface areas

BET analyses of AC samples were carried out using the Micromeritics Tristar II 320 device in the laboratories of Kahramanmaraş Sutçu Imam University Applied Sciences Center (KSU-USKIM). Before the measurements, the samples were degassed under vacuum for 1 hour at 110 °C. In determining the pore properties of AC samples, accelerated surface area and porosimetry system (ASAP 2020; Micromeritics Co., 2020), which is widely used for the determination of surface area and porosity, was used. This method is based on nitrogen

adsorption-desorption isotherms. The obtained isotherm findings were evaluated according to the IUPAC classification (Thommes et al 2015). In addition, qualitative information about the pore structure of AC was obtained. Different methods were used in the quantitative calculations of micro, meso and total pore volumes. While the total pore volume was determined by Gurvich's rule or the Dubinin-Raduskevich equation; Micropore volumes were determined by the t-method (Sj and Sing 1982, Rouquerol et al 1987, Rodriguez-Reinoso et al 1987).

2.5. SEM Analysis (Scanning Electron Microscopy)

SEM images were obtained using a Zeiss EVO LS10/Bruker scanning electron microscope (SEM) at the KSU USKİM laboratories. The device operated at an accelerating voltage of 15.00 kV, and images were recorded at 500X, 750X, 1.00 KX, and 3.00 KX scales.

2.6. Elemental Analysis of Samples

In order to determine the elements C (carbon), O (oxygen), H (hydrogen) and N (nitrogen) in the structure of ACs obtained from eucalyptus root woods, the elemental analyses were performed with Eltra CS580 brand device in KSU USKİM laboratories. In the process of elemental analysis, approximately 2 mg of the samples were taken and the amount of elements in the structure of AC was determined by burning at 950-1000 °C temperature ranges.

3. Results and Discussion

3.1. Yield Calculation Analysis Results

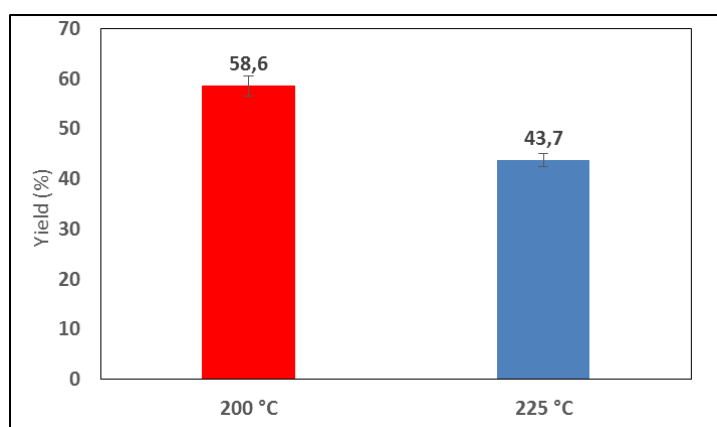


Figure 1. Solid product yields of the hydrothermal carbonization process

The yield values as a result of the hydrothermal carbonization process, which is the first stage of eucalyptus root wood, are shown in Figure 1. According to the findings, eucalyptus root wood samples carbonized by hydrothermal method at 200°C yielded 58.6% carbonized solid product. Similarly, 43.7% carbonized solid product yield was obtained from eucalyptus root wood samples carbonized by hydrothermal method at 225°C. As shown in Figure 1, the yield is higher at lower temperatures. Hydrochar yield increases with a short residence time but decreases with a longer residence time. Under the same hydrothermal temperature conditions, a longer residence time can increase hydrochar polymerization, resulting in more carbon fractions being released into the water and a lower mass yield. A longer residence time increases the intensity of the hydrothermal reaction and increases intermediate product polymerization, ultimately leading to secondary polymerization of dissolved and degraded substances in the water product and the formation of an aromatic hydrocarbon structure. It has been observed that as the temperature of the hydrothermal process increases, product yield decreases and the aromatic and hydrocarbon fractions released into the water increase significantly (Titirici et al 2008, Yang et al 2023).

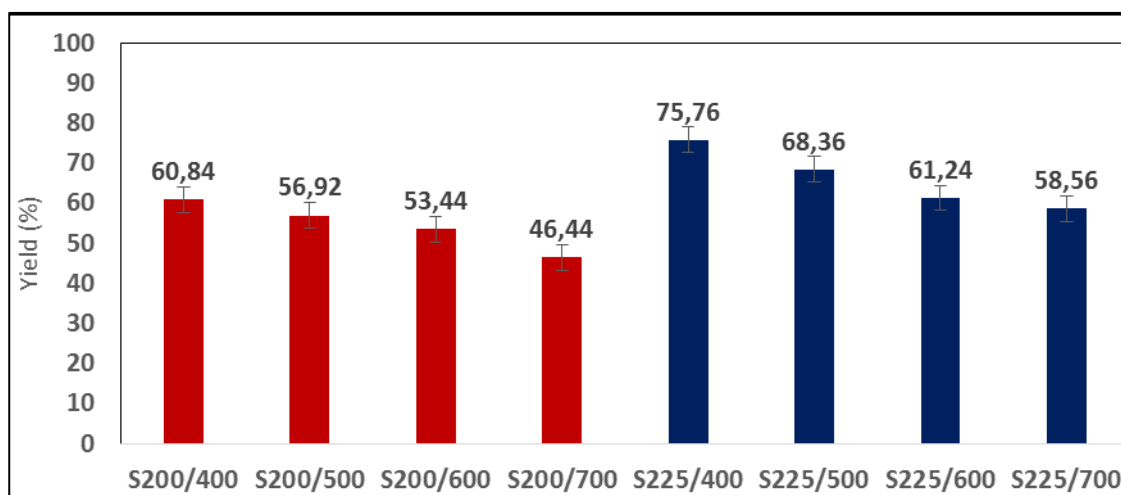


Figure 2. Solid product yields of samples

It is understood that AC efficiency decreases as the temperature increases. Hydrothermal usually takes place in the subcritical range of water in the range of 150-260°C. In the hydrothermal process, most of the hemicellulose and lignin structures in the biomass structure break down. The degraded fractions can be converted into oligomer and monomeric sugars, furfural or various organic acids in water. Apart from these fractions, they also form solid carbon structures (Moniz at al. 2013, Fang et al. 2008). In the study for the carbonization process, the temperature was used high due to the high amount of lignin. Many studies have shown that the efficiency decreases as the temperature increases in the hydrothermal carbonization process. However, in two-stage production, if high temperature is applied in the first stage, the yield is higher when the pyrolysis stage is started (Rajasekaran and Raghavan 2020). In a study, solid product yields were higher due to the lower amount of biomass and components degraded in the hydrothermal carbonization process carried out by applying lower temperatures in the hydrothermal process. It was observed that the solid product yields of the samples treated at 225°C were higher. Hydrothermal carbonization processes are carried out by applying low temperatures, the amount of degraded biomass and components will be lower and solid product yields are higher (Basu 2010).

3.2. BET Analysis Results

Table 2

BET Analysis Findings of Activated Carbon

	¹ BET (cm ² /g)	² APW (nm)	³ MQA (cm ³ /g STP)	⁴ V _t (cm ³ /g)	⁵ V _{mi} (cm ³ /g)	⁶ V _{me} (cm ³ /g)	⁷ V _{ma} (cm ³ /g)
S225/400	8.848	10.84	17.47	0.024	0	0.016	0.007
S225/500	9.36	11.41	20.07	0.026	0	0.001	0.025
S225/600	264.08	2.21	97.52	0.146	0.081	0.053	0.0128
S225/700	237.78	1.75	68.89	0.104	0.098	0.006	0
S200/400	5.75	11.61	11.76	0.016	0	0.016	0
S200/500	8.07	10.91	15.77	0.022	0.014	0.008	0
S200/600	59.38	3.37	34.72	0.050	0.016	0.033	0
S200/700	246.79	2.11	88.51	0.130	0.087	0.043	0

¹BET surface area, ²Adsorption average pore width nanometer, ³maximum quantity adsorption cubic centimeters per gram at Standard Temperature and Pressure, ⁴Total pore volume, ⁵Micro pore volume, ⁶Meso pore volume, ⁷Macro pore volume

The BET analysis results of the AC samples are presented in Table 2. According to the findings, samples treated with hydrothermal pretreatment at 225°C generally had higher BET surface areas. The average pore

sizes decreased with increasing temperature during the pyrolysis phase. In comparing nitrogen gas absorption values, the use of higher temperatures during the pyrolysis phase resulted in higher absorption values. It is clearly evident that the ACs produced in the study generally exhibit mesoporous properties. However, the S200/500, S200/600, and S200/700 AC samples also exhibited microporous properties. The formations of micropores in the structure of AC were thought to be due to the use of lower temperatures in the hydrothermal phase.

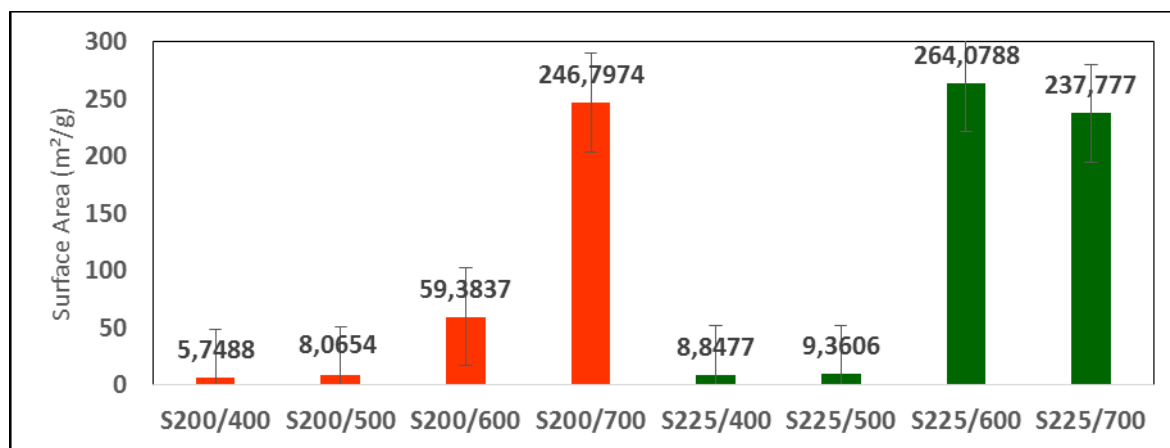


Figure 3. BET analysis results of samples

As can be seen from Figure 3, when the BET surface area results are analyzed, it is understood that the hydrothermal treatment performed at 225°C yielded higher surface area results than the hydrothermal treatment performed at 200 °C. It was observed that 264.07 m²/g result was obtained in the S225/600 sample. It was concluded that the lowest BET surface area result was 5.7 m²/g in the S200/400 sample. In addition, in the studies carried out, the formation of surface areas and porous structures due to evaporation as the temperature increases during carbonization processes with chemical activation has been observed as a result of evaporation (Deng et al, 2010). However, considering the studies, it has been observed that the porous structures of the samples are not always macro-sized, and although the pores do not form on the outer surface of the pores, they can form on the inner surfaces to a significant extent (Orkun 2011).

3.3. SEM Analysis Results

The SEM images below show a comparison of AC produced at the same pyrolysis temperature but different hydrothermal temperatures. When the SEM images in Figure 4 are compared, it is seen that pores are more pronounced when higher hydrothermal temperatures are used. When the SEM images in Figure 5 are compared, similar images are observed. When the SEM images in Figure 6 are compared, it is understood that the samples subjected to hydrothermal treatment at 225 °C experienced fragmentation, which positively affected the pore structure. However, it is understood that the pore structures of the samples subjected to hydrothermal treatment at 200 °C and pyrolysis at 600 °C were different. When the SEM images in Figure 7 are compared, it is understood that the pore structure is quite good in both samples and that they have similar properties. In addition, the similar results in the total surface area of the samples in the BET analysis support the SEM images. Pore size is very important in the AC samples produced. It is concluded that if the pore size and also the surface porosity structure of the AC samples are high, the surface areas of the samples obtained high. It is thought that the more the amount of surface area, the more adsorption capacity (Ucer Ozasik 2002).

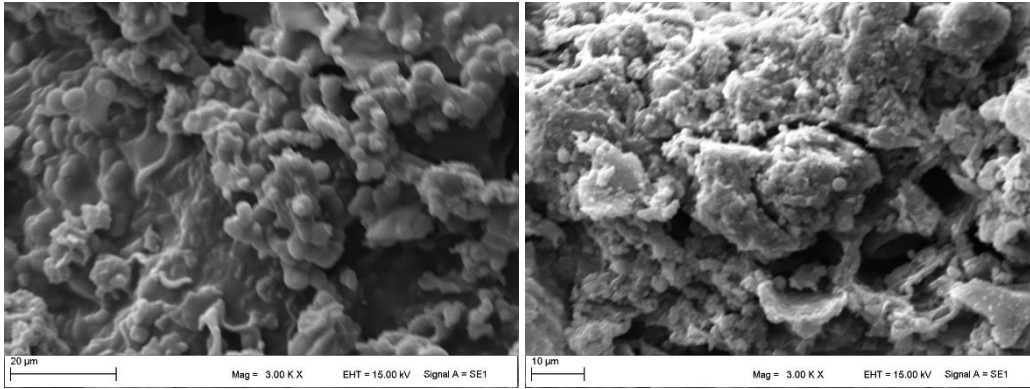


Figure 4. SEM image of samples S225/400 and S200/400 respectively

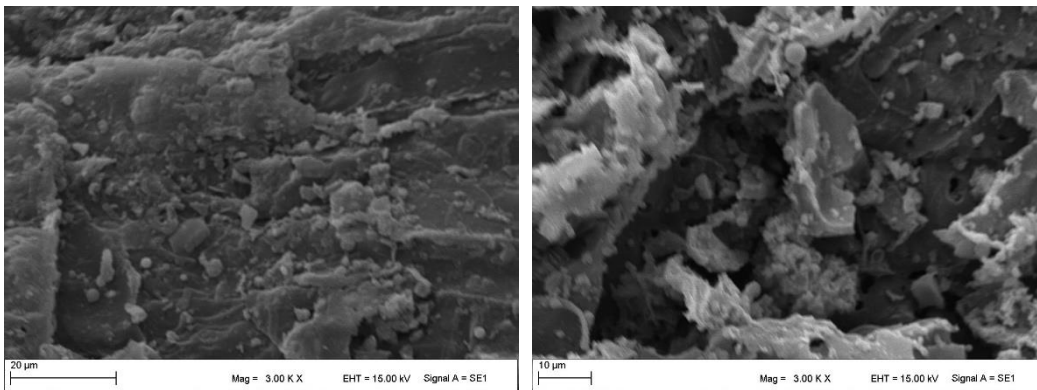


Figure 5. SEM image of samples S225/500 and S200/500 respectively

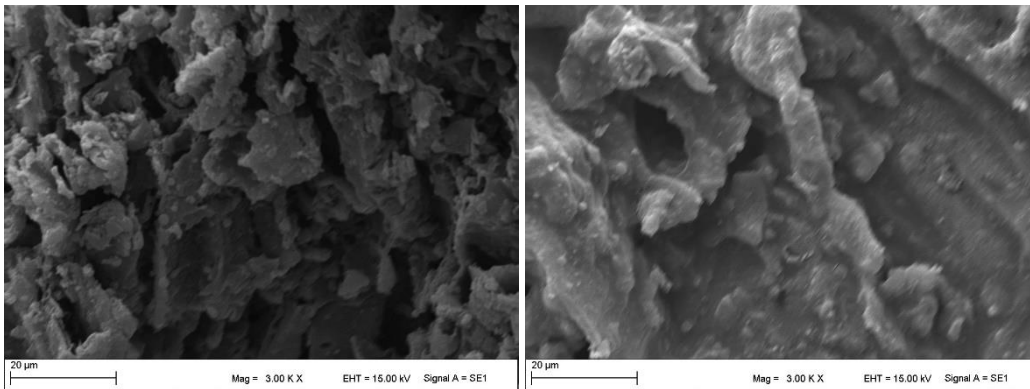


Figure 6. SEM image of samples S225/600 and S200/600 respectively

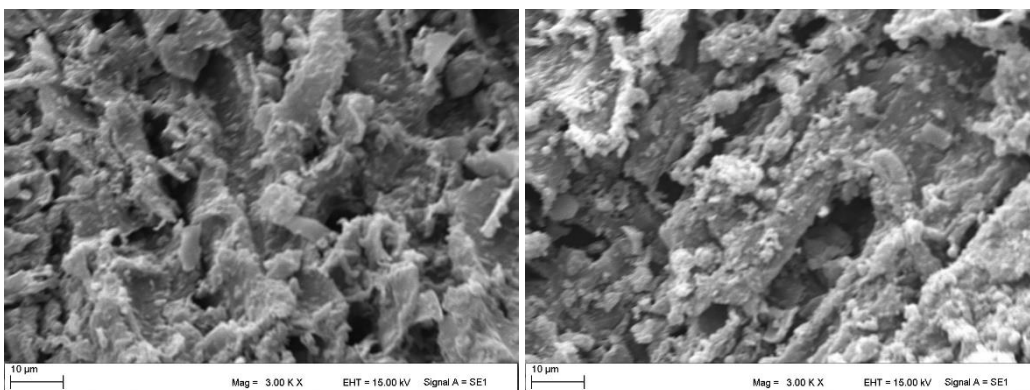


Figure 7. SEM image of samples S225/700 and S200/700 respectively

When all the SEM images given are examined, it is understood that the pore structures of the samples produced at the same hydrothermal temperatures increase and change with the increase of the pyrolysis temperatures. However, a layered and heterogeneous surface structure was observed. However, it is thought that the porous structures are not in the macro size range and that the porous structures are not formed on the outer surface but on the inner surfaces at significant dimensions (Orkun 2011). According to SEM images, it was observed that after hydrothermal pretreatment and pyrolysis, the hard and durable structure of AC decreased and more fragmentation occurred as a result of thermal decomposition.

3.4. Elemental Analysis Results of Activated Carbone

The findings of the elemental analysis performed to determine the elements C (carbon), O (oxygen), H (hydrogen) and N (nitrogen) in the structure of the samples whose activation processes were completed are shown in Table 3.

Table 3
Elemental Analysis Results of Eucalyptus Root Wood

Sample Code	C (%)	H (%)	N (%)	O (%)	H/C Molar ratio	O/C Molar ratio
S225/400	57,90	3,16	0,89	38,05	0,65	0,49
S225/500	65,50	2,83	0,93	30,74	0,51	0,35
S225/600	68,20	2,16	1,25	28,39	0,38	0,31
S225/700	76,70	1,75	1,97	19,58	0,27	0,19
S200/400	61,80	3,29	2,26	32,65	0,63	0,39
S200/500	68,30	2,99	2,55	26,16	0,52	0,28
S200/600	70,50	2,27	2,68	24,55	0,38	0,26
S200/700	56,00	2,20	0,49	41,31	0,47	0,55

Although there are aromatic constructions in the structure of AC, there are also oxidized functional groups. The higher the amount of C (carbon), the more aromatic structures increase. However, high amounts of O (oxygen), H (hydrogen), N (nitrogen) results in a high amount of oxidized functional groups in the structure of AC. The pore structure and oxidized functional groups of activated carbons vary depending on where and for what purpose they are to be used. The high amount of oxidized functional groups is thought to increase the adsorption capacity of the produced AC samples (Pradhan and Sandle 1999, Li et al 2014). When we examine the studies, while the amount of C (carbon) increased with the increase in temperature, the amount of O (oxygen) decreased. With the decreases in O/C and H/C ratios, C(carbon) increased, leading to an increase in aromatic structures in the environment and the formation of more regular structures (Karapınar 2018). Along with the increase in the regular structures, the decrease in the amount of H/C and the increase in the amount of C (carbon) lead to an increase in graphite structures (Koseoglu 2005). However, C (carbon) structures are thought to be an important factor in the formation of aromatic structures and the increase in regular structures (Karapınar 2018). When we look at the temperatures applied to the samples produced in two stages, hydrothermal carbonization and pyrolysis, it is seen in the graphs that the increase in the amount of C (carbon) and the decrease in the amounts of H (hydrogen), O (oxygen) and N (nitrogen) with the increase in temperature is a major factor. When increasing from 200°C to 225°C and then pyrolysis temperatures were applied, a decrease in functional groups was observed in AC samples, while an increase in aromatic structures was observed. Many studies have shown that the high number of functional oxidized groups in the structure of AC is very important in improving its absorption properties. (Jain et al 2016, De la Torre-Miranda et al 2023).

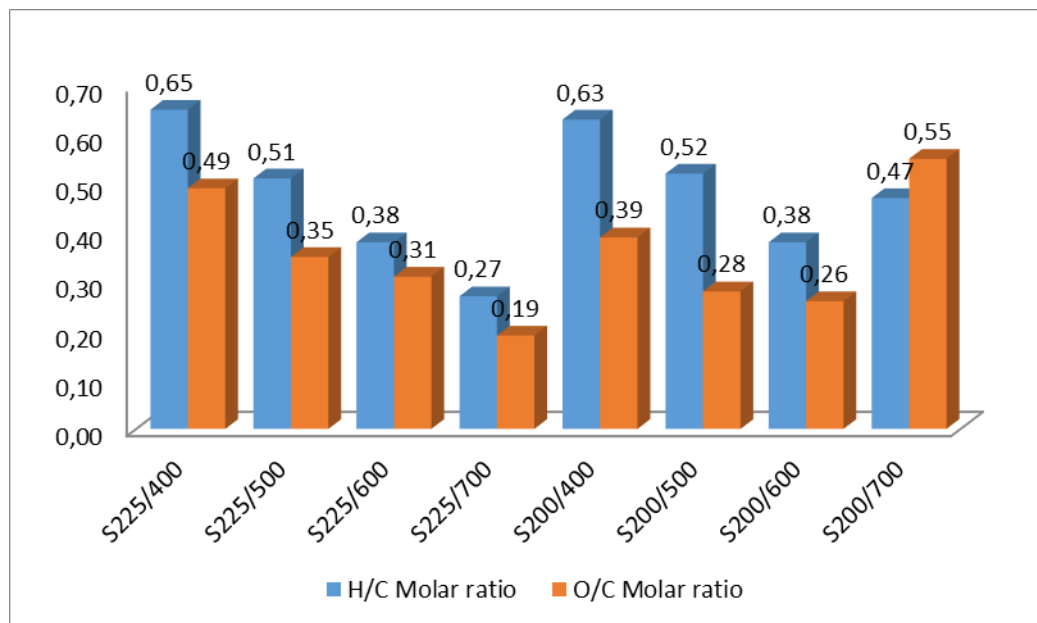


Figure 8. Elemental H/C and O/C content analysis results of pure treated samples

4. Conclusion

According to the analysis results, when the solid product yields of hydrothermal samples from eucalyptus root wood samples were examined, the highest value was 58.6% carbonized solid product yield at 200°C. When the BET surface area results were examined, it was concluded that the AC samples with the highest surface area were in the S225/600 sample with 264 m²/g, while the lowest BET surface area results were in the S200/400 sample with 5.7 m²/g. According to the BET analysis results, it was understood that ACs generally have mesoporous properties. When the SEM images were examined, the use of high temperatures in the pyrolysis stage caused an increase in the porous structure. When the elemental analysis results of the produced AC samples were examined, it was seen that the highest C (carbon) content belonged to the S225/700 AC sample, reaching 76.70%. It was also observed that the S225/700 sample had the lowest O (oxygen) level, determined as 19.58%. The O (oxygen), H (hydrogen), and N (nitrogen) values in the AC structure decreased as the C (carbon) ratio increased at higher temperatures. As a result of the findings, it was understood that eucalyptus root wood can be used in the production of AC. Additionally, new studies can be conducted on eucalyptus root wood in the AC production.

Conflicts of Interest

The authors declare no conflict of interest.

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