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Production and characterization of activated carbon from Black Poplar (*Populus Nigra*) wood waste with different chemical activation methods

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ABSTRACT

In this study, the producibility of activated carbon from wood waste by using the chemical activation method was investigated and the produced activated carbon was compared with commercial activated carbon. Activated carbon was produced from black poplar wood waste using zinc chloride and phosphoric acid. The density values of the produced activated carbons were determined by the picometer method. Field Emission Scanning Electron Microscopy (FESEM) was used to analyze the microstructure and perform the elemental mapping. To determine the chemical content of activated carbon, it was also characterized by Fourier-transform infrared spectroscopy (FTIR) and energy dispersion spectroscopy (EDS). Based on the density and FE-SEM results, it was determined that the produced activated carbon had a lower density and porous structure. In addition, EDS analysis showed that the activated carbon produced from black poplar wood waste was purer than commercial activated carbon.

1. Introduction

Materials obtained by increasing the internal surface area and pore volume through activation of organic and inorganic substances, the majority of which consist of carbon, by chemical and physical methods are called activated carbon [1]. This type of carbon can be distinguished from elemental carbon by the fact that its outer and inner surfaces have oxygenated functional groups. In the high and environmental area, it is microporous, and the total pore volume is greater than 0.2 mL/g, and the BET (Brunauer–Emmett–Teller) surface area is wider than 400 m^2. The pore diameter varies between 3 Å and a few 1000 Å [1,2].

Activated carbon is mostly used in water treatment to adsorb organic compounds [3], oils [4], and toxic substances [5]. It is often applied due to its high surface area and adsorption capacity. The increasing demand for water treatment all over the world due to the exhausting natural resources [6–8] and the increasing amount of pollutants lead to the activated carbon sector growth [9]. Moreover, strict rules and regulations related to the treatment of industrial wastewater promote the further production of activated carbon [10]. In the food and beverage sectors, activated carbon is also used to remove pollutants such as heavy metals, odor and color-giving substances, amino acids, and toxic components [11]. Due to the fact that customers pay more attention to food safety and quality, the consumption of activated carbon is increasing day by day [12]. Furthermore, activated carbon has industrial uses for removal CO_2 [13–15], recovering different solvents [16,17], and controlling mercury vapor emissions [18,19] and waste gases [20,21]. The activated carbons are utilized in the manufacture of the special chemicals, metal recovery [22], and energy storage [23]. The current increasing energy demand has led to an increase in battery consumption, which has led to an improvement in the manufacture of energy storage devices [24–26]. As a result of the emergence of new usage areas of activated carbon every year and the corresponding increase in production capacity, the global activated carbon market size reached $2.96 billion in 2020. The market is expected to grow from $3.12 billion in 2021 to $4.50 billion in 2028 [27].

It is known that there are different manufacturing methods in activated carbon production; the most commonly used of these methods are physical and chemical activation methods. Physical activation consists of two complementary stages as degradation of organic

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substances and activation of the carbonized structure. In the first place, hydrogen and oxygen are removed from the raw material and the main skeleton is formed. In the second stage, the activation process is carried out by using water vapor or CO_2 gas at a temperature of 800-1000 °C or by using both gases, and activated carbon is produced [28]. Because the efficiency of chemical activation is higher and it is produced at low temperatures, it is preferred more compared to physical activation.

The surface areas of the activated carbons obtained as a result of chemical activation are larger and the pore volumes are bigger. Depending on the chemical activation agent used, the chemical and physical properties of the resulting activated carbon may vary. Activated carbon is obtained by bringing the starting material to the appropriate dimensions and reacting with the chemical substance at 400-1000°C, or by reacting the carbonized starting material with the chemical substance. When the literature is examined, it is seen that activation agents with different properties are used in this method. Some of them are boric acid, calcium hydroxide, calcium chloride, phosphoric acid, sulfuric acid, zinc chloride, iron (III) chloride, potassium carbonate, potassium hydroxide, manganese (II) chloride, nitric acid, sodium chloride, and sodium sulphate [29,30]. Some studies related to this issue in the literature are as follows.

In a study conducted by Hajati et al. [31], activated carbon was produced using the chemical activation method. As an activation agent, they used nitric acid. They conducted investigations on the surface characterization and adsorption of the obtained activated carbon. They observed that the obtained activated carbon expanded the surface area and increased the absorption rate [31].

In a study conducted on eucalyptus wood waste, KOH and NaOH were used as activation agents and the obtained activated carbon was compared with activated carbon obtained from lignite. Adsorption capacity and porosity distribution of activated carbons obtained by the chemical activation method were investigated. Based on the data obtained, scholars reported that the microporosity distribution of activated carbon produced by the NaOH activation agent was larger than that of KOH. They also observed that its adsorption capacity was higher than that of lignite [32].

Acharya et al. successfully produced activated carbon from the activation of tamarind wood with zinc chloride. They investigated the usability of activated carbon in the treatment of wastewater (removal of lead) and in agricultural fields. They emphasized that the surface area of the produced activated carbon enlarged, the lead contained in the wastewater could be adsorbed, and it was a cheaper method compared to other methods [33].

The aim of this study is to investigate the physical and chemical properties of activated carbons obtained from black polar wood waste by using two different methods with $ZnCl_2$ and H_3PO_4 chemical activation agents. In addition, today, it is aimed to increase a sustainable economy and environmental awareness and to obtain products with a higher added value.

2. Material and Method

The black poplar (Populus nigra) wood waste used in this study was obtained in the form of powder and free of charge from an enterprise located in Konya Seydişehir Carpenters Industry. Commercial activated carbon was purchased from Aromel Chemical (Konya, Turkey) in order to compare it with the activated carbon which produced in this study. Zinc chloride ($ZnCl_2$) and phosphoric acid (H_3PO_4) were used in the activation process, while hydrochloric acid (HCl) and potassium hydroxide (KOH) were used during the washing of the produced activated carbon. $ZnCl_2$, H_3PO_4, HCl and KOH were of analytical purity, and they were obtained from Merk (Darmstadt, Germany) or Fluka (Jul, Switzerland) companies.

In this study, activated carbon production was carried out using black poplar wood (Populus nigra) with the help of acid (phosphoric acid) and salt (zinc chloride) activation agents (Figure 1).

2.1 Production of Activated Carbon with Phosphoric Acid

3 kg of black poplar wood waste in powder form was mixed by adding 3 liters of phosphoric acid (50% wt) and 3 liters of pure water. In order to react wood waste with phosphoric acid, it was treated at 110°C for two hours. Then the mixture was dried by waiting in the drying-oven at 80°C for 24 hours. For the activation process, the dried material was cooled at room temperature after keeping it for 1.5 hours at 600° under argon gas (50 milliliters/min). It was washed first with 0.5 M KOH, then with hot deionized water until the pH value was 6-6.5. After the washed activated carbon was dried at 100°C for 6 hours, it was ground and become ready for use [34].

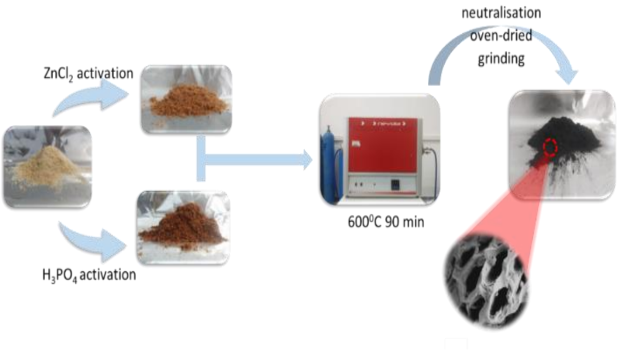

Figure 1. Production of activated carbon from black poplar

2.2 Production of Activated Carbon with Zinc Chloride

For the production of activated carbon with zinc chloride, 3 kg of powdered black poplar wood waste was made into a dough by adding 1.5 kg of zinc chloride and 3.75 liters of purified water and kneaded. In order for the raw material to react with zinc chloride, it was treated at room temperature for 24 hours. Then the mixture was dried by keeping it in the drying-oven at 80°C for 24 hours. The dried material was kept for 1.5 hours at 600° under argon gas (50 milliliters/min) for the activation process and then cooled at room temperature. It was first washed with 0.5 M HCl, then with hot deionized water until the pH value was 6-6.5. The washed activated carbon was dried at 100°C for 6 hours and then ground and become ready for use [35].

2.3 Characterization

The densities of the produced activated carbons were determined by helium pycnometer (Micromeritics–Accupyc2 1340). On the other hand, the morphologies of activated carbons were examined using Field Emission Scanning Electron Microscopy (FE-SEM) (ZEISS GeminiSEM 500). To increase their conductivity, activated carbons were coated with iridium with a thickness of 5 nm. During the examination of the microstructure images, the operating voltage of the microscope was selected as 2.00 kV. In addition, the functional groups of the activated carbons and their changes during the exposure process were determined by identifying the absorbance values whose FTIR (Thermo Scientific - Nicolet iS20) spectrums were determined at the range of 400 – 4000 cm^{-1} and the groups corresponding to these in the literature.

3. Result and Discussion

The FTIR spectra of commercial activated carbon (O) and zinc chloride (WWS) and phosphoric acid (WWA) based activated carbons between 400 and 4000 cm^{-1} are shown in Figure 2.

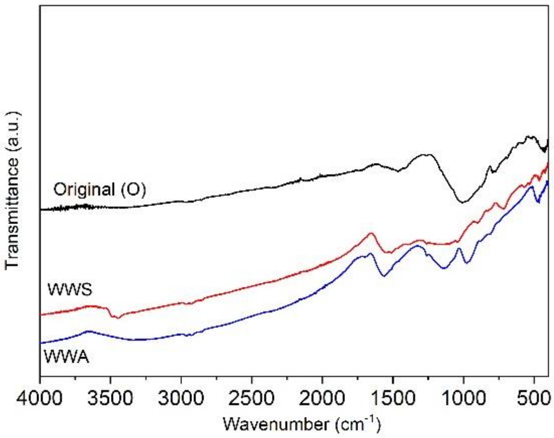

Figure 2. FT-IR results of commercial and produced activated carbon

The spectra of commercial activated carbon (O) samples displayed the following band: 1460 cm^{-1} could be assigned to C–H group. The presence of bands in the 1012 cm^{-1} wave number ranges could be due to C–O stretching vibrations. When the activated carbon peaks produced by zinc chloride activation (WWS) are examined, the peak at 3480 cm^{-1} shows the -OH peak and the peak at 1543 cm^{-1} shows the presence of carboxylic acid and/or lactone groups. When the peaks of activated carbon produced by phosphoric acid activation (WWA) are examined, it is seen that the peaks observed in the range of 3340 cm^{-1} bands are hydroxyl (O-H) groups. In addition, the peak observed in the 1400 cm^{-1} band indicates the presence of phenol in activated carbon. Peaks seen in the range of 2855-3000 cm^{-1} bands indicate the presence of aliphatic structures. These peaks were observed in activated carbon samples produced by both activations. Peaks seen in the band range of 1550-1800 cm^{-1} indicate a C=O bond structure; ketones, aldehydes, esters, and carboxylic acids are organic groups with this type of bond structure. Peaks were observed in this band range in the activated carbon samples produced by both activation agents. In particular, the peak seen in the 1600 cm^{-1} band indicates the presence of aromatic structures (C=C) and it was observed in both samples. The peaks observed in the band range of 950-1300 cm^{-1} indicate the presence of C-O, P=O, C-O-P, and P=O-OH groups. In both samples, there are peaks in this band range, but it is rather difficult to determine to which group these peaks belong. In the literature, it has been stated that the 1216-1196 cm^{-1} band corresponds to C-O, the 1085-1240 cm^{-1} band corresponds to P=O, and the 1180 cm^{-1} peak corresponds to P=O-OH groups [36,37]. Especially in activated carbon samples produced by chemical activation, peaks in the band range of 1080-1185 cm^{-1} are more evident. The reason for this is that there are P=O and P=O-OH groups due to the phosphoric acid used for activation [38].

Although activated carbons are known as materials with a large specific surface area and high porosity, it seems that many commercial activated carbons do not fully fit this definition [39]. In Figure 3 (a, b), FE-SEM images of commercial activated carbon taken with different magnifications are given. When the images were examined, it was seen that there were heterogeneously distributed pits on the surface of activated carbons rather than pores. In particular, in the FE-SEM images taken with 10 k magnification, the presence of small pores surrounding the pits is observed. Figure 3 (c, d) shows the surface physical morphology of WWA-encoded activated carbon obtained from wood waste by the chemical activation method using phosphoric acid and the FE-SEM images of the obtained activated carbon samples. When the images are examined, it is possible to say that compared to the commercial activated carbon with O code, pores are formed more clearly, and the pore volume is larger. Moreover, it is clearly seen that compared to HA activated carbon, it is distributed not in the form of dimples, but in hollow spherical pores form and more homogeneously. In addition, it was found that the specified activated carbon had smooth walls and identified edges, and there was a distance between the gaps. It was also observed that the gaps were of different sizes. This situation suggests that the structure of wood waste is reorganized during activation [34,40]. FE-SEM images of WWS-coded activated carbon obtained from wood waste by chemical activation method using Zinc chloride ($ZnCl_2$) are given in Figure 3 (e, f).

When the FE-SEM images were examined, the formations of pore morphologies were detected relatively clearly compared to commercial activated carbon. Especially in the FE-SEM examination including high magnification, it was observed that the pores occurred in a larger number and almost all of them appeared in the ellipse shape morphology. It is thought that the $ZnCl_2$ chemical impregnation method leads to carbonization of the carbon skeleton after activation by causing the decomposition of cellulosic material and increases pore formation by undergoing aromatization. The formation of gaps was also caused by the evaporation of zinc chloride during the activation, and as a result of the evaporation and separation of zinc chloride from the places it originally occupied, the number of gaps increased [41]. In this case, it can be clearly seen that the shape morphologies of activated carbons produced by the chemical activation method are more efficient and in desirable pore morphologies compared to commercial materials. Also, it was observed that the activated carbons obtained using the activation mechanism have different pore sizes and shapes depending on the activation agents [42]. In the study, to be able to compare activated Mapping and EDS analyses.

Mapping and EDS elemental analysis results of the commercial activated carbon (O) which have C, O, Si, Al and Fe elements were given in Figure 4a and Figure 4b. It was found that all elements, except Fe, were distributed homogeneously. As can be seen both in the EDS results (Figure 4b) and in the Mapping images (Figure 4c and Figure 4d), since activated carbons are mostly produced from organic raw materials or wastes, the intensive presence of C and O is an expected result [10]. In a study conducted by Park et al. (2020), it was reported that activated carbon produced from coconut fiber has C and O contents, similar to our study [43].

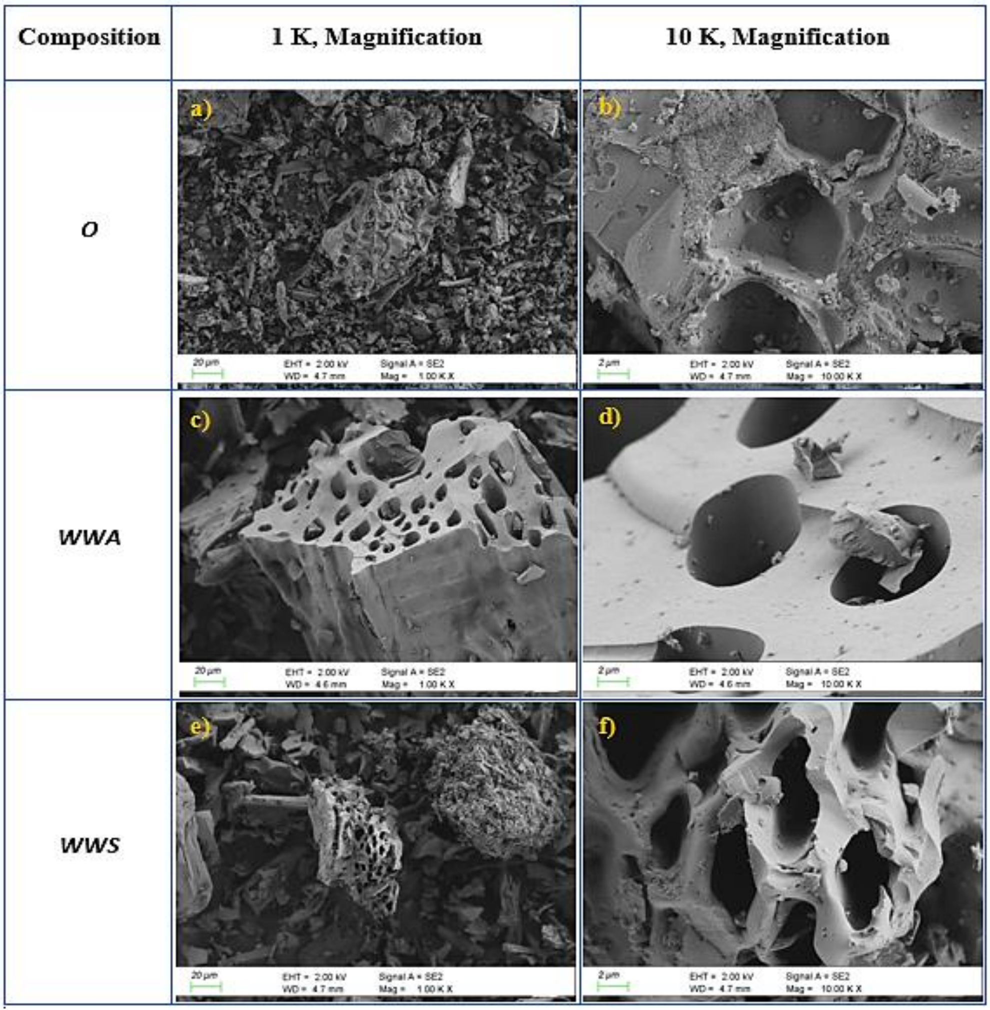

Figure 3. FE-SEM images for different type of activated carbons

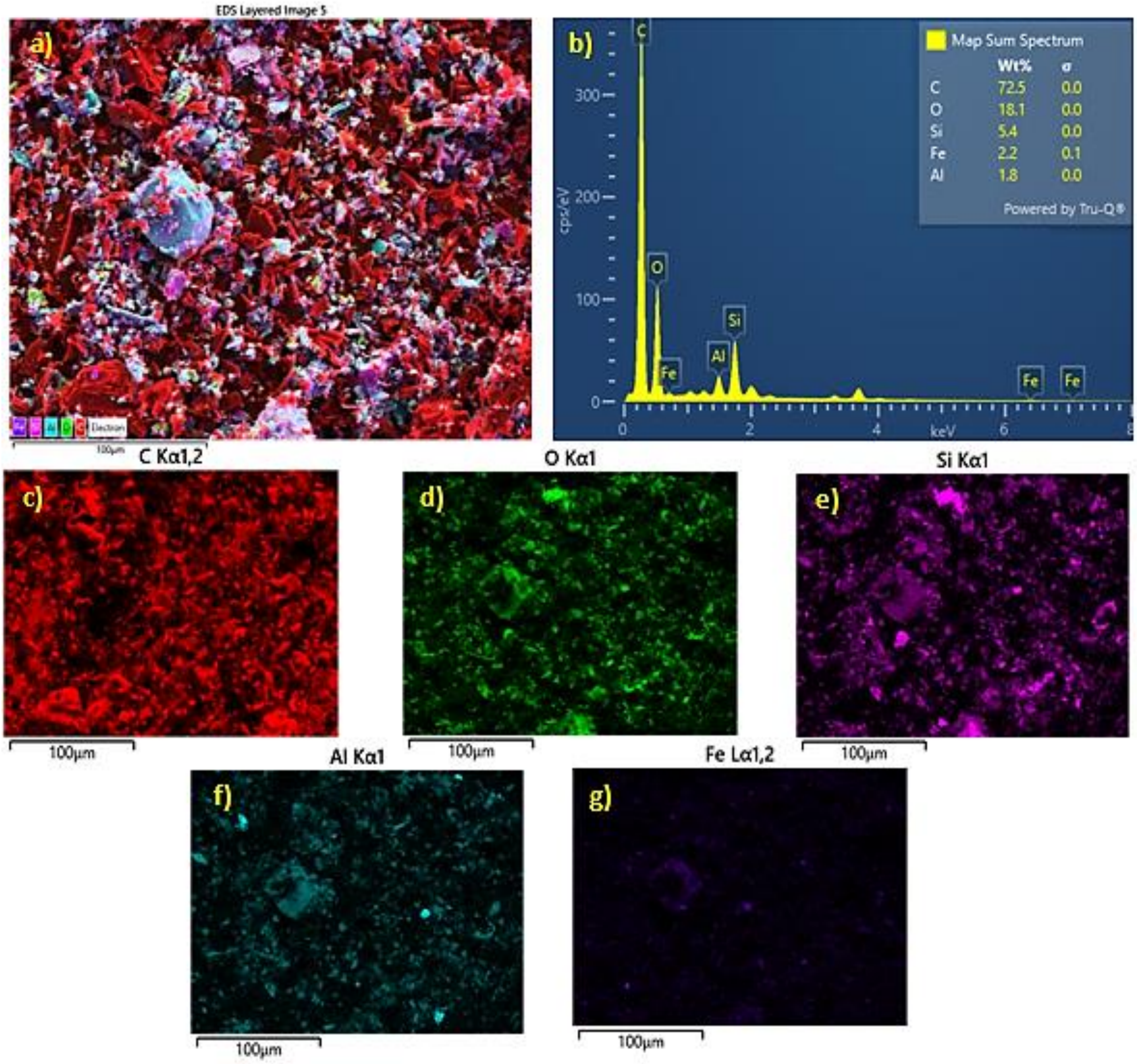

Figure 4. FE-SEM Mapping and EDS analyses of commercial activated carbon

In this study, the Al and Si elements found in commercial activated carbon produced from coconut fiber are homogeneously distributed as seen in Figure 4e and Figure 4f. Monteiro et al. (2005) revealed the presence of Al and Si elements in coconut fibers by EDS analysis [44]. In addition, the Fe element, whose distribution is seen in Figure 4g, is present at a rate of 2.2% in commercial activated carbon. It is believed that the element Fe is an impurity that occurs during production or arises from the environment in which production is carried out.

Figure 5 shows the EDS and Mapping analyses of activated carbon produced from black poplar wood waste with a zinc chloride activation agent.

As can be seen in Figure 5a and Figure 5b, activated carbon produced from black poplar wood waste contains C, O, Zn, and Cl elements. It was determined that all the elements contained in activated carbon were distributed homogeneously. Since black poplar wood contains about 49% C and 42% O [45], the fact that C and O elements were found intensively after pyrolysis was an expected result as can be seen both in the EDS results (Figure 5b) and in the Mapping images (Figures 5c, d). On the other hand, due to the activation process carried out at a high temperature, the amount of C in activated carbon increased and the amount of O decreased. In the study conducted by Fernandez et al. (2014), it was determined that the used raw material contained 43% C and 50.2% O elements before the production of activated carbon, while it contained 82.5% C and 14.1% O after the production of activated carbon [34]. In addition, it was observed that there were Zn and Cl elements in the environment due to the $ZnCl_2$ activation agent used (Figures 5e and f). In the literature, activated carbons produced from different raw materials with the $ZnCl_2$ activation agent also have similar results [35,46]. Figure 6 shows the EDS and Mapping analyses of activated carbon produced from black poplar wood waste with the phosphoric acid activation agent.

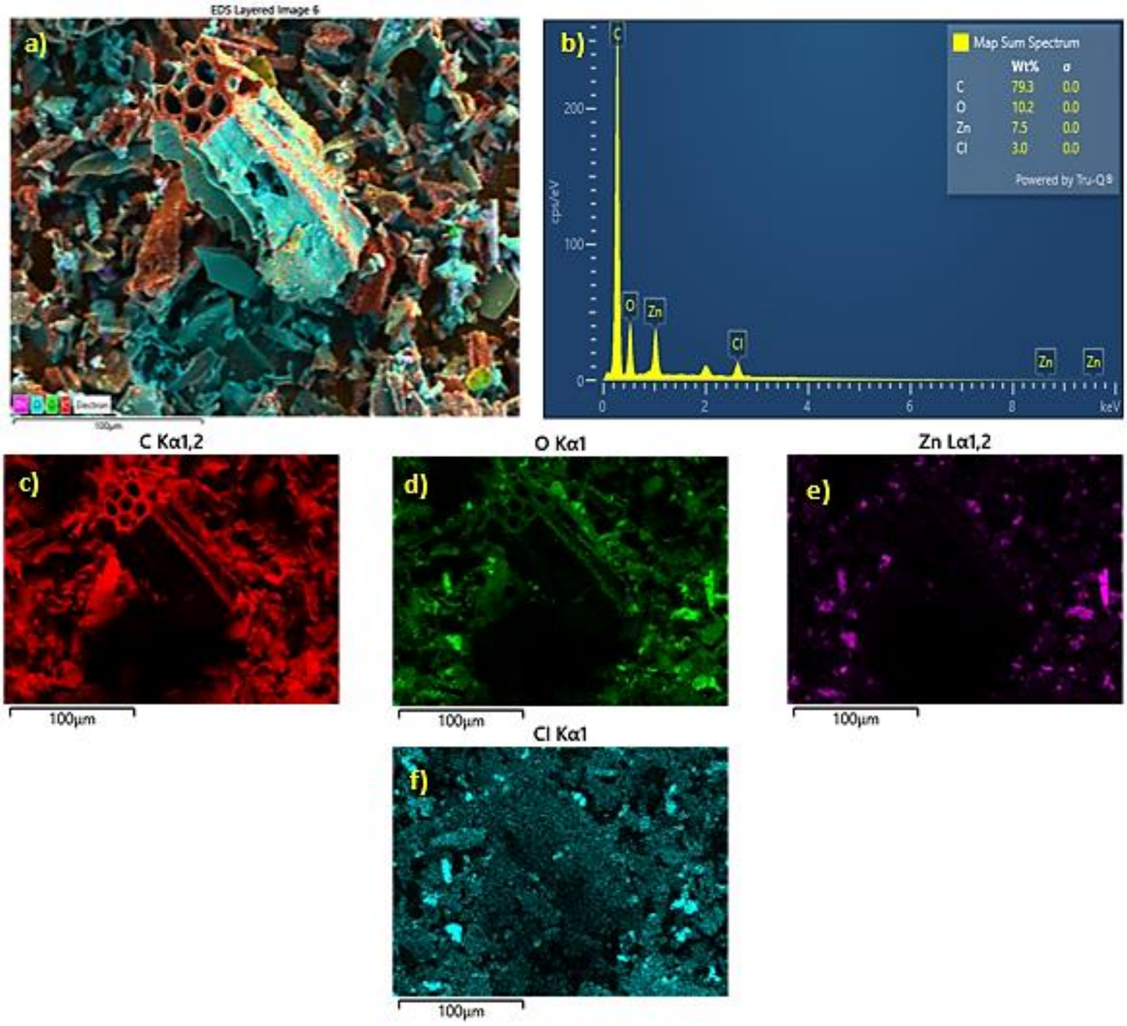

Figure 5. FE-SEM Mapping and EDS analyses of black poplar activated carbon produced with $ZnCl_2$

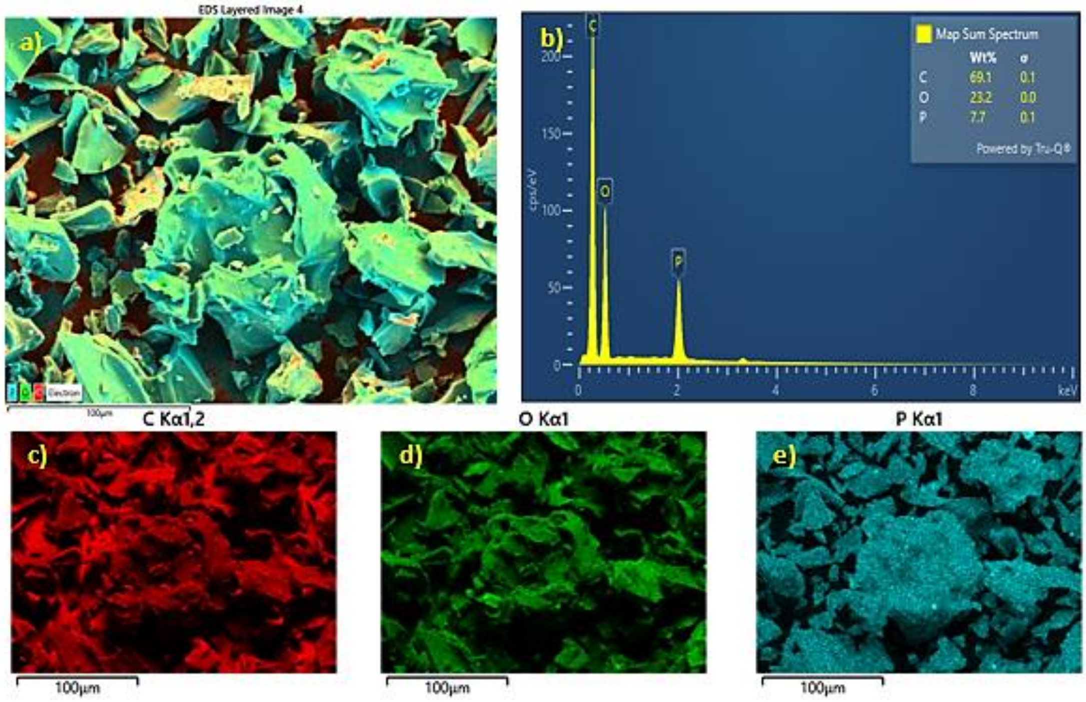

Figure 6. FE-SEM Mapping and EDS analyses of the activated carbon produced from black poplar wood waste with H_3PO_4

Table 1. Densities of activated carbons

Activated Carbons	Density (g/cm^3)
O	2.14
WWA	1.37
WWS	2.08

As can be seen in Figures 6a and 6b, there are C, O, and P elements in the activated carbon produced from black poplar wood waste. As mentioned above, it was found that the activated carbon produced as a result of pyrolysis of black poplar wood waste contains 69.1% C and 23.2% O. In addition, the distribution of C and O elements homogeneously in the material is seen in Figure 6c and Figure 6d. In the study conducted by Danish et al. (2013), as a result of EDS analysis, it was determined that the activated carbon produced from the Acacia mangium tree by using phosphoric acid activation contained 76.22% C, 17.8% oxygen, and 5.98% P [47]. On the other hand, it is seen in Figure 6e that the P element in the produced activated carbon is homogeneously distributed and the PO_4^3 ions are successfully modified to the activated carbon surface [48]. Since phosphoric acid is used as a chemical activation agent in the production of activated carbon, it is observed that carbon contains the P element. In their studies where they used phosphoric acid in the production of activated carbon, Vázquez et al. (2012) detected the presence of the P element in EDS and Mapping analyses [49].

Table 1 shows the densities of activated carbon obtained from black poplar wood waste and commercial activated carbon. The density of HA-coded activated carbon is 2.14 g/cm^3, while the densities of activated carbons obtained in salt (WWS) and acid (WWA) media are 2.09 and 1.37 g/cm^3, respectively. The slightly increased surface area and pore volume are mainly due to the formation of micropores by the breakdown of oxygen-enriched WWA activated carbon [50]. The EDS analysis results of activated carbons confirm this situation. Whereas WWA activated carbon has 23.2% O, WWS activated carbon has 10.2% O. In general, the large pore volume contradicts with the high density. The well-developed porous structure of activated carbons usually implies a very low material density [51]. When the FE-SEM images are examined, it is possible to say that some large-sized pores are formed as a result of inhomogeneous distributions of loose carbon precursors, and this also causes a low density.

4. Conclusions

In this study, it was seen that activated carbon could be produced from black poplar wood waste with the chemical activation method by using phosphoric acid and zinc chloride activation agents. FTIR analyses performed at absorbance values determined in the range of 400-4000 cm^{-1} showed that activated carbons were successfully synthesized. When the FE-SEM images were examined, it was seen that the density of activated carbon obtained by using phosphoric acid is higher than that of other activated carbons used in the study. The fact that this condition reduces the density was also confirmed by the results of density experiments. The EDS results showed that commercial activated carbon is not pure, that is, there are different elements in it. On the other hand, it was found that the obtained activated carbons (WWA and WWS) were pure, no other elements were found in them, and the main skeleton was formed correctly. As a result, it was understood from the experiments and analyses that activated carbon production was carried out successfully.

Declaration

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The author(s) also declared that this article is original, was prepared in accordance with international publication and research ethics, and ethical committee permission or any special permission is not required.

Author Contributions

M. E. Ergun developed the methodology. S. Bulbul performed the analysis. M. E. Ergun and S. Bulbul wrote the manuscript and proofread the manuscript together.

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