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INTERNATIONAL ADVANCED RESEARCHES and ENGINEERING JOURNAL International Open Access

Volume 07 Issue 01 April, 2023

Journal homepage: www.dergipark.org.tr/en/pub/iarej

Research Article

Production of activated carbon from the waste paper by chemical activation method

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ARTICLE INFO	ABSTRACT
Article history: Received 21 December 2022 Accepted 24 March 2023 Published 15 April 2023 <i>Keywords:</i> Activated carbon Chemical activation Recycling Waste paper	The cellulose and paper industry accounts for a large part of the circular economy. The need for activated carbons is gradually increasing, especially in the environmental and energy fields. In this study, the production of activated carbon from waste papers was carried out with the help of the chemical activation method and activation agents (phosphoric acid and zinc chloride). The parameters used in the experiments and analyzed were kept constant for all activated carbons. The density values of activated carbon were analyzed more than once in each sample with a helium-gas pycnometer device. Fourier transform infrared spectroscopy (FT-IR) was used to detect functional groups in the structure of activated carbon, and a field emission scanning electron microscope (FE-SEM) was used to study surface properties and porosity development. The distributions of activated carbons and their elemental analysis were examined by energy dispersive spectrometry (EDS) and Mapping analyses. When the results obtained from the activated carbon, it was observed that the waste paper had a better surface and pore structure than commercial activated carbon for the production of activated carbon, and the activation process was successfully performed

1. Introduction

Conventional activated carbons are produced from organic and inorganic substances with high carbon content, such as wood, peat, bituminous coal, and anthracite [1]. The increase in the current production of activated carbon due to the increasing demand for adsorbent materials to solve environmental problems has pushed the industry and research groups to look for alternative ways in which different methods and techniques are used. Currently, the use of many agricultural and industrial wastes (energy crops, agricultural biomass residues, forest residues, and foodbased waste) as a renewable raw material source for producing activated carbons has paved the way for the production of low-cost and larger amounts of activated carbon, and many scientific studies have been conducted on this topic [2-4].

Activated carbon is a term used to refer to well-built porous structures and carbon-rich materials. Activated carbon is a versatile material with a surface area containing high and well-organized macro, meso, and micropores, includes a wide variety of chemical functional groups and can find different application areas for itself [5].Physical and chemical activation methods are used to improve the pore structure in carbon. Physical activation methods are an older method compared to the chemical activation method and it is widely used in the activated carbon production. Oxygen and hydrogen are completely removed from the material used in the production of activated carbon, and the main skeleton is formed. Then, with the use of water vapor or CO₂ gas or both as an activator agent for the activation process at a temperature of 800-1000 °C, the activation process is realized and activated carbon is produced [6]. However, the limited extent of pore development, not very large surface area, low micropores volume, the need for high temperatures for production, low adsorption rate, and relatively low activation rate are the main disadvantages of the physical activation method [7].

Many researchers around the world are performing studies on the chemical activation method to produce activated carbon. Chemical activation is often preferred because it allows the production of activated carbon with

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higher carbon content by using lower heating temperatures and faster reaction times [8].Chemicals such as potassium hydroxide, potassium carbonate, phosphoric acid, sulfuric acid, zinc chloride, hydrochloric acid, pyrophosphoric acid, nitric acid, sodium hydroxide, potassium carbonate, sodium chloride, ammonium chloride, ferric chloride, sodium carbonate, and hydrogen peroxide are used as activating agents [4,9-19]. Alkalis such as potassium hydroxide (KOH) are usually realized with a large impregnation rate [20,21] between 400~1000 °C and are very effective in forming micropores [22]. Considering the environmental effects, energy cost, and efficiency, phosphoric acid (H_3PO_4) carbon is remarkable, and its use in large-scale activated carbon production has steadily increased in recent years [23]. Phosphoric acid (H₃PO₄) can occur at low temperatures of 400~600 °C with a low impregnation rate and consists mainly of mesoporous with a surface area of about 1000~1500 m²·g-1 BET[24,25]. As a catalyst, phosphoric acid (H₃PO₄) promotes bond fragmentation reactions. It facilitates cross-linking by forming a connecting layer, such as phosphate and polyphosphate esters, that can maintain the condensation and internal pore structure through the cycle and thus prevent excessive combustion on carbon activation [26]. In the literature, some studies in which activated carbon was produced using different materials are as follows.

Usmani et al. reported that a product with a surface area of 942 m².g⁻¹ was obtained from lignite coal with a high sulfur content using a chemical activation method and a zinc chloride activation agent at a temperature of 650 °C and after one-hour activation [27]. In the study conducted by Liu et al., they activated hemp waste with zinc chloride and determined the surface area of the activated carbon they obtained to be 1100 m².g⁻¹ [28].

In a study conducted by Zhang and his colleagues, the effects of physical activation of activated carbon obtained from bamboo on pore structure and surface chemistry were studied, and it was concluded that the activation conditions significantly affected the surface area development, porosity, efficiency, and combustion of activated carbon [29].

In the study conducted by Fernandez et al., it was found that the surface area of activated carbon produced from the orange peel as a result of activation with phosphoric acid developed up to 1090 m².gr⁻¹. It was stated that the obtained activated carbon was successfully used in the adsorption of waste dyes in water [30]. In another study, the micro-porosity of the activated carbon produced from the sawdust of a rubber tree was improved by chemical activation with potassium hydroxide. The surface area of the produced activated carbon was found to be 1491 m².gr⁻¹ [31]. Today, sustainability is gaining importance with the developing technology. The cellulose and paper industry also forms a large part of the circular economy, and the need for activated carbon, especially in the environmental and energy fields, is increasing day by day. The purpose of this study was to use waste paper for the production of activated carbon and to evaluate the effects of experimental parameters and chemical activation agents (ZnCl₂ and H₃PO₄) used in the production of activated carbon.

2. Material and Method

Used writing paper waste utilized in this study. During the activation process, zinc chloride (ZnCl₂) and phosphoric acid (H₃PO₄) were used, while hydrochloric acid (HCl) and potassium hydroxide (KOH) were used to wash the activated carbon. All the chemicals used in this study were of analytical purity, and the production of activated carbon from the waste paper was carried out with the help of phosphoric acid and salt (zinc chloride) activation agents (Figure 1). The ZnCl₂, H₃PO₄, HCl, and KOH materials used in the study were obtained from Bereket Chemical Company (Bereket Chemical Medical Technical Trade and Industry Ltd. Co.).

Production of activated carbon with phosphoric acid:

3 kg of waste paper in small pieces was mixed by adding 1.5 liters of phosphoric acid (50% wt.) and 3 liters of pure water. In order for the waste paper to react with phosphoric acid, it was treated at 110°C for two hours. Then, the mixture was dried at 80°C oven for 24 hours. The dried material was subjected to carbonization for 1.5 hours at 600°C under argon gas (50 milliliters/min) for the activation process, and then cooled at room temperature. It was washed with 0.5 M KOH and 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 ready for use [30]

Production of activated carbon with zinc chloride:

3 kg of waste paper in small pieces was kneaded by adding 1.5 kg of zinc chloride and 3 liters of pure water until it became a dough. In order for the raw material to react with zinc chloride, it was treated for 24 hours at room temperature. Then, the mixture was dried by keeping it in the oven at 80°C for 24 hours. After the dried material was subjected to carbonization process for 1.5 hours at 600 °C under argon gas (50 mL/min) for the activation process, it was cooled at room temperature.

2.1 Characterization

FE-SEM images of activated carbons produced by the chemical activation method were obtained by FT-IR spectroscopy. To determine the density of the activated carbons produced, a pycnometer (Micromeritics Accupyc II 1340) and a 10 cm³ sample cup were used.



Figure 1. Production of activated carbon from waste paper

The sample was carefully prepared and loaded into the sample cup within the Micromeritics instrument. The density value was then obtained using the Accupyc II 1340 Pyc software. Helium gas was preferred because it is an ineffective gas that penetrates tiny sub-micron pores up to 0.3 nm in size [32]. A field emission scanning electron microscope (FE-SEM) (Hitachi-SU 1510) was used to examine the surface structure and pore development morphologies of activated carbons. In order to improve the quality of conductivity, an iridium (Ir) coating process with a thickness of 5 nm was applied to the surfaces of activated carbons. The operating voltage of the microscope was determined as 20 kV, and microstructure images of activated carbons were examined. For the purpose of characterizing the chemical structure of the resulting activated carbon, Fourier transform infrared spectroscopy (FT-IR) analyses were performed to determine the functional groups in the structure of activated carbon. The FT-IR spectra were determined with the FT-IR (Thermo Scientific - Nicolet iS20) device in the range of 4000-400 cm⁻¹. As a result, detailed work was carried out at each stage of the experiments to improve the micro-porosity and surface area of the activated carbon produced from waste paper during the experimental process. After the carbonized materials were washed with 0.5 M HCl solution, the activated carbon was washed with hot deionized water until the pH value was 6-6.5. Then, it was dried at 100°C for 6 hours and made ready for use by grinding [33].

3. Results and Discussion

FTIR, density, FE-SEM, EDS, and Mapping analyses of the produced activated carbon were performed and compared both with commercial activated carbon and among themselves. The parameters used in the experiments and analyzes were kept constant for all activated carbons.

3.1 FTIR Analysis

FT-IR analyses were performed to determine the functional groups in the structure of activated carbon produced as a result of the chemical activation of waste paper with $ZnCl_2$ and H_3PO_4 agents. The FT-IR spectrum of activated carbons produced by the chemical activation method is given in Figure 2.

When the FT-IR spectra in Figure 2 are examined, the presence of the OH functional group connected by peak hydrogen bonds at 3670 and 3375 cm⁻¹ wavelengths is seen[34]. Peaks observed around 2970-2976 cm⁻¹ indicate the aliphatic C-H functional group, while peaks formed at wavelengths of 2310-2320 cm⁻¹ indicate the C=C functional group in alkyne groups [35]. The peaks around the wavelengths of 1615-1515 cm⁻¹ are associated with C=O or C=C functional groups [36]. The peak in the range of 1200-1500 cm-1 refers to the C-O groups [37]. The sharp peak formed around 1060-1040 cm⁻¹ is associated with the C-O-C group [38]. Peaks smaller than 1000 cm⁻¹ refer to functional groups originating from the aromatic ring [34]. On the other hand, the peaks in the range of 1127-973 cm⁻¹ bands are the P=O and P=O-OH groups, which occur due to phosphoric acid [39].



Figure 2. FT-IR spectrum of activated carbons

According to the spectrum analysis, in common with all three samples, in the wavelength range of 400-4000 cm⁻¹, the strongest adsorption band in the WPA, WPS, and O codes is in the wavelength range of 3482-3456 cm⁻ ¹ and corresponds to the -OH functional group. It is known that in nature, hydroxyl and carboxyl groups make the surface of the activated carbon acidic [40]. Therefore, the adsorption of activated carbon and the permeability percentage are affected. In the study, it is seen that in the zinc chloride (WPS) activation performed at a wavelength of 3451 cm⁻¹, the percentage of permeability decreases significantly compared to phosphoric acid (WPA) and ready-made (O) activated carbon. Peaks in the range of 1586-1405 cm⁻¹ band indicate the C-O group, while peaks in the 1082-1014 cm⁻¹ wavelength range indicate the P=O and P=O-OH groups. Finally, the 630-544 cm⁻¹ band refers to the C-C and C-O aromatic functional groups. It is observed that in the content of the prepared activated carbons, there is not only carbon but also other hetero atoms such as hydrogen, oxygen, nitrogen, and phosphorus.

3.2 Density Analysis

Table 1 shows the densities of activated carbon obtained from waste paper and commercial activated carbon. In the analysis results, the WPS density increases to 1.824 gr/cm³ and the WPA density increases to 1.653 gr/cm³. The increase in the actual density values measured by the helium gas pycnometer is related to the formation of a denser array due to the high stability of the cross-links of the molecules. It is also known that as the temperature increases and as planar clusters of aromatic rings are stacked on top of each other, heavier structures of irregular carbons are formed, and therefore the density increases [41].

3.3 FE-SEM Analysis

Scanning electron microscopy studies were conducted to examine the surface properties and porosity development of activated carbons produced using different activation agents. In order to make an assessment, FE-SEM images (taken at a size of 10k) of commercial activated carbon and activated carbon produced with different agents were used. When the FE-SEM images of commercial active (O) carbon in Figure 3a are examined, it is observed that it has a heterogeneous cavity structure and small-sized pits surrounding these cavities.

Activated carbons	Density(g/cm ³)
0	2.14
WPS	1.82
WPA	1.65

When the FE-SEM images of WPA-coded activated carbon produced by chemical activation of waste paper with phosphoric acid are examined in Figure 3b, it is observed that pores that are in different sizes and like ellipse shape are lined up on the outer surface of the activated carbon which is not entirely round. When carefully examined, Figure 3b evokes a honeycomb. When compared with commercial activated carbon, it is seen that hierarchical-porous activated carbons with a high specific surface area are obtained. It is known that in studies where phosphoric acid is used as an activation agent at the stage of activated carbon production, the surface width distribution shows more heterogeneous characteristics compared to others [42]. For activated carbons, the pore structure, size, and distribution are important for characterizing the heterogeneity of materials. The heterogeneity characteristic is closely related to the balanced distributions of a rigid internal structure model and gives important clues about the used material [43]. It is possible to say that these pores are useful for any adsorption process because the large-sized pores serve as feeder pores to the lower-sized meso and micropores [44]. In many studies, it has been stated that activation temperature, pressure, and duration are important. It is emphasized that the specified parameters affect the pore volume, product efficiency, and surface volume of activated carbon, and the appropriate temperature is 600 °C [45,46].

When the FE-SEM images of WPS-coded activated carbon produced as a result of the chemical activation of zinc chloride (ZnCl₂) activation agent with waste paper are examined (Figure 3c), it is seen that there are irregular cavities and large and small amorphous pores on the outer surface of the activated carbon. Compared to commercial activated carbon, it is seen in Figure 3c that the pore width is deeper and has a cavity structure. Some studies in the literature have also revealed that the production of activated carbon with agricultural waste material and ZnCl2 activation agent increases the pore width [47]. ZnCl2 were used as an activation agent, the expanded since outward expansion pores and decomposition occurred on the activated carbon surface over time [48]. In another study, Gonzalez-Serrano et al. reported that they obtained a large surface area in the production of activated carbon from the chemical activation of Kraft lignin with zinc chloride, and they noted that the activated carbons produced were usually microporous [49]. Recently, activated carbons with a good pore volume and the desired surface area have been produced from many agricultural wastes and lignocellulosic materials. Experimental studies have shown that similar situations occur in terms of pore and surface area in the production of activated carbon from waste paper, and the use of zinc chloride as an activation agent has an effect on the activation process of waste paper.

3.4 EDS Analysis

Within the scope of the study, the activated carbons obtained as a result of treatment of waste paper with H_3PO_4 and $ZnCl_2$ activation agents and commercially purchased activated carbon were compared, and in order to make characterization, elemental analysis and distributions of activated carbons were studied. Figure 4 shows the Mapping and EDS analyses.



Figure 3. FE-SEM images of (a) Commercial activated carbon, (b) WPA and (c) WPS activated carbons



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

The results of EDS elemental analysis performed on commercial activated carbon samples (Figure 4b) showed that the activated carbon produced from coconut shell included 72.5 wt% carbon concentration (C), 18.1% oxygen (O), 5.4% Silicon (Si), and 1.8% aluminum (Al). The distributions of the elements are observed in the FE-SEM Mapping images. In addition, it was found that there was 2.2% iron (Fe) element in commercial activated carbon, and this element was not homogeneously distributed as seen in FE-SEM images and Mapping elemental analyses (Figure 4g). In the study conducted by Mirshafiee et al., based on EDS analysis, it was reported that 73% C and 25% O were found in activated carbon produced from coconut shells. In addition, in the same study, it was found that elements, except for the Fe element, were distributed homogeneously in SEM-Mapping images [50]. On the other hand, it has been stated that Si and Al, found in commercial activated carbon, are also found in the stem and fruit peels of tropical tree species such as coconut [51].Figure 5 shows the EDS and Mapping analysis results of activated carbon produced from waste paper by using phosphoric acid (H_3PO_4) activation agent.

As seen in Figure 5a and 5b, it was observed that carbon (C) (70%), oxygen (O) (21.8%), Phosphorus (P), and Calcium (Ca) elements were present respectively in the activated carbon produced by phosphoric acid activation. On the other hand, the homogeneous distribution of elements C, O, and P in the material is seen in Figure 5c, Figure 5d, and Figure 5e, respectively. In the literature, in the EDS analysis of the activated carbon produced from eucalyptus waste [52] and kraft lignin [53] by using phosphoric acid activation agents, 75% - 82 C, and 11% - 20 O, and 3-7% P element were identified. In the current study, FE-SEM images and Mapping analyses also showed a homogeneous distribution of all elements on the surface of activated carbon. In addition, the presence of Ca element in the activated carbon produced within the scope of the study (Figure 5f) was due to the use of calcium carbonate as a filler during production paper [54].



Figure 5. FE-SEM Mapping and EDS analyses of the activated carbon produced from waste paper by using phosphoric acid (H₃PO₄) activation agent

In the study conducted by Manandhar et al., EDS analysis determined that the Ca element was found in the activated carbon at a rate of 2.91% [55]. Because calcium carbonate is resistant to high temperatures [56], the presence of 2.1% Ca element in the activated carbon produced in the current study shows that it is consistent with the literature.

Figure 6 shows the EDS and Mapping analyses of activated carbon produced from waste paper using zinc chloride (ZnCl₂) as an activation agent.

As seen in Figures 6a and 6b, in activated carbon produced from waste paper by using zinc chloride as an activation agent, 65% carbon (C), 15.6% zinc (Zn), 12.8% oxygen (O), 5.5% chlorine (Cl), and 1.2% calcium (Ca) elements were detected. The FE-SEM images and Mapping elemental analyses revealed that all elements, except for the Ca element, were distributed homogeneously in the produced activated carbon (Figure 6g). It has been stated that 72.11% C and 22.06% O were

found in office waste papers [55]. It has been claimed that the decrease in the element O, especially after the production of activated carbon, is due to the removal of the element O from the environment under high temperatures [57]. A study stated that there was O element at a rate of 6.62% in activated carbon produced from waste paper at a temperature of 850° C by using ZnCl₂ as an activation agent [58].

In addition, it is seen in Figures 6d and 6f that there are Zn and Cl elements in the environment due to the ZnCl₂ activation agent used. In the literature, it has been observed that activated carbons produced from different raw materials by using the ZnCl₂ activation agent have similar results [59,60]. In their study, using FE-SEM images and Mapping elemental analysis, Fardim and Holmbom showed that the element Ca was distributed heterogeneously in paper [61]. Also, another study found that the element Ca deteriorates at a temperature of 710-720 °C [62].



Figure 6. FE-SEM Mapping and EDS analyses of activated carbon produced from waste paper by using zinc chloride (ZnCl₂) as activation agent

4. Conclusion

Recycling waste paper is an important contribution to protecting the environment and increasing the diversity of raw materials used, particularly in the paper industry. However, In Turkey, the rate of recycling waste paper is low because the waste paper often contains impurities and unwanted materials, which make it more difficult and costly to recycle. Moreover, high-cost facilities are required for recycling waste paper. Therefore, it is necessary to evaluate waste paper in different areas like activated carbon production. In this study, activated carbon produced from waste paper was successfully produced with chemical activation agents (ZnCl₂ and H₃PO₄. The porous structures of the activated carbons produced in the FE-SEM images were determined. Activated carbons produced with phosphoric acid were found to have low density due to higher oxygen content as a result of EDS analysis. In the Mapping analyses of the activated carbons (WPA and WPS) produced within the scope of the study, Ca elements were found at rates of 2.1% and 1.2%, respectively. The use of calcium carbonate as a filler during paper production explains the presence of the Ca element. When the commercial activated carbon was compared with WPS and WPA activated carbons, it was seen that successful results were obtained in terms of pore size, distribution, and other analyzes of the produced activated carbons. Based on the results of the study, it can be concluded that the production of activated carbon from waste paper will be a good source of recycling for sustainability as well as an important material for the national economy.

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

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