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Activated Carbon Pruduction and Characterization Studies from Cane (*Phragmites australis*) by Microvave Assisted Pyrolsis Process

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Abstract: Activated carbon is an adsorption material with a large internal surface area and pore volume, which is obtained by the activation of the increasingly high content of carbon containing materials. Active carbon is used in the removal of organic pollutants and taste and odor disorders from household and industrial wastewater, as well as many areas such as various gas phase applications as well as the purification of chemical products. The cane grows spontaneously in lakes all of them in western Anatolia. Due to its easy accessibility and abundance, the cane is very economical to use it in production of activated carbon. The aim of this work is to obtain activated carbon from the cane, which as a cheap and an abundant raw material, by means of the quick microwave oven. Activated carbon production was carried out in two stages. In the first step sawdust of cane was activated with o-phosphoric acid solutions (30%, 40% and 50%). The second stage is carbonization in a microwave oven. Carbonization processes of 20 and 30 minutes were carried out at 300 and 450 watt microwave power levels. Methylene blue aqueous solutions were used for performance tests of the obtained activated carbon samples. BET surface area measurement, SEM photographs and FT-IR analyzes were used to determine the physical properties. It was concluded that 20 minutes of carbonization at the 300 watt microwave power level was sufficient for the studies. BET surface area of the best powder active carbon sample which is obtained in optimum conditions has been specified as 1096,9 m^2/g.

Keywords: Activated Carbon, Cane, Adsorption, Color Removing

1. Introduction

Activated carbon can be defined as a highly improved adsorbent material with internal surface area and pore volume by the activation process applied to substances with high carbon content. It has a wide range of application areas such as food, pharmaceutical, chemical, petroleum, mineral, nuclear, automobile, waste gas and water cleaning [1, 2]. Activated carbon can be produced by chemical and physical activation. Chemical activation is the combination of activation and carbonization processes with the help of the appropriate chemical ($ZnCl_2$, NaOH, LiOH, H_2SO_4, H_3PO_4, KOH) [3-6]. In previous studies, the usability of many raw materials such as olive corn [7], hazelnut shell [8], cotton and corn stalk [9], grape pulp [10] and pumpkin seed [11], which are high in cellulose ratio, have been investigated in our country. In this study, the production of activated carbon from the cane *(phragmites australis)* growing around Afyonkarahisar/Bolvadin Eber Lake in Turkey was investigated. The dry straws collected from the perimeter of the lake were first separated from their leaves and turned into sawdust (< 0.5 cm). Activation was performed with H_3PO_4 as the second step. In the third and last stage, carbonization process was realized in microwave oven at different furnace power levels and durations. In order to determine the properties of the activated carbons, BET surface area, SEM photographs were taken and elemental analysis was performed with SEM-EDX. In this way, the porosity ratio and pore widths of the surface areas were determined. XRD analyzes were also performed.

2. Experimental Studies

2.1. Raw Material Preparation and Pre-Tests

As a raw material, dried straws were obtained from Bolvadin Eber Lake in its natural state. The reeds were spread in the laboratory and dried at room temperature for 1 week. The leaves of the reed were peeled and turned into sawdust (< 0.5 cm). Specimens taken from the sawdust; ash determination, determination of volatile matter, moisture determination and constant carbon determination were performed. The fixed carbon value was calculated based on the results of ash and volatile matter.

2.2. Preparation of Activated Carbon Samples

Activation of the Straw: Activation of the straws with H_3PO_4 was performed. For this; 250 mL of 30% o-Phosphoric acid was added to 100 grams of reed sawdust. The reed sawdust was mixed until they absorbed the solution. After the absorption was complete, the beakers were covered with aluminum foil and kept in the oven at 105°C for 6 hours. The oven was then turned off and allowed to cool down. This mixture was taken into a glass jar and the sample was purged with nitrogen gas and the stock sample was prepared by closing the mouth. Similarly, H_3PO_4 solutions in the concentrations of 40%, 50% and 60% were also used. Thus the reed shavings were activated at four different concentrations. The specimens were left in this dark environment for two weeks.

Microwave Baking Carbonization Processes: 20 gram sample was taken from the stock sample with 30% o-Phosphoric acid activated. In the microwave oven, carbonization was carried out by heating in 300 watts for 20 minutes under nitrogen gas. When the process ended, the sample in the crucible was kept in the microwave oven until it reached room temperature with nitrogen gas. Samples taken from the oven were ground in porcelain mortar and activated carbons were prepared. These processes were repeated for 30 minutes pyrolysis time. Similarly, studies were repeated for H_3PO_4 solutions at concentrations of 40%, 50% and 60%. In addition, the pyrolysis processes for 300 watts for 20 and 30 minutes were also repeated for 450 watts. Thus, four different acid concentrations, two different furnace power and two different time calcined in a total of 16 different activated carbon samples were obtained.

The activated carbons were washed 5 times with 50 mL of distilled water in the magnetic stirrer and completely dried for 2 hours at 105°C in the oven. The dried samples are put into closed vials and ready for analysis. The BET surface areas were measured to determine the properties of the activated carbons. The higher the internal surface area and pore volume of the activated carbons are so high activity. Because it shows so much adsorbent property. In addition, by taking into account the concentration of each acid, the total morphology of eight samples with high surface areas was also examined. Elemental analysis was also performed with SEM-EDX. In this way, the porosity ratio and pore widths of the surface areas were determined. XRD analyzes were also performed.

3. Results and Discussion

3.1. BET Surface Area Analysis Results

The surface area measurements were made by BET analyzes to investigate the physical properties of the activated carbon samples. The internal surface areas of the samples obtained at the end of the studies are given in Table 1. As it can be seen from the table, it can be concluded that the acid concentration for activation is 30-40% and the heating process for 20-30 minutes at 300 W for carbonization is sufficient. The best results are also marked in red in the table below.

Table 1. *Results of BET surface areas of activated carbon samples.*

% H_3PO_4	Power (Watt)	Carbonization Time (min.)	Surface Area (m^2/g)
30	300	20	873,82
30	300	30	1056,73
30	450	20	841,77
30	450	30	1054,19
40	300	20	1096,90
40	300	30	738,89
40	450	20	957,16
40	450	30	839,92
50	300	20	981,16
50	300	30	946,54
50	450	20	872,27
50	450	30	859,78
60	300	20	844,37
60	300	30	943,19
60	450	20	922,10
60	450	30	1055,07

3.2. SEM Analysis Results

Microstructure and porosity were taken from the samples with the highest surface area from the samples produced with o-Phosphoric acid and rapid microwave-assisted pyrolysis system with Scanning Electron Microscope (SEM). Figure 1 shows 30% H3PO4 concentration of 300 W of 30 minutes carbonization of the sample at 200 magnification. Figure 2 shows 40% H_3PO_4 concentration of 300 W of 20 minutes carbonization of the sample at 200 magnification.

In addition, SEM-EDX analysis was performed from four different samples and elemental analysis results were determined. SEM-EDX analysis of a sample selected in Figure 3 and the results of elemental analysis in Table 2 are shown. When the results are examined, it is seen that the sample contains a small amount of Phosphorus due to the phosphoric acid used and the small amount of silicon which is due to the impurities adhering to the natural environment.

Table 2. *Resuls of SEM-EDX analysis of activated carbon obtained after 20 minutes at 300 W for 40% H_3PO_4 concentration.*

Element	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [wt.%]
Carbon	44990	51.09	51.09	59.73
Oxygen	8916	42.58	42.58	37.37
Silicon	2766	0.58	0.58	0.29
Phosphorus	27214	5.76	5.76	2.61
Total				100.0%

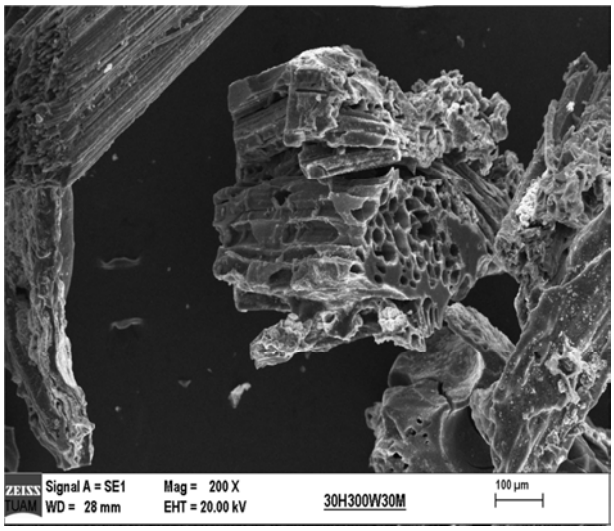

Figure 1. *SEM photo of active carbon at 200 magnification at a concentration of 30% H_3PO_4 after 30 minutes at 300 W.*

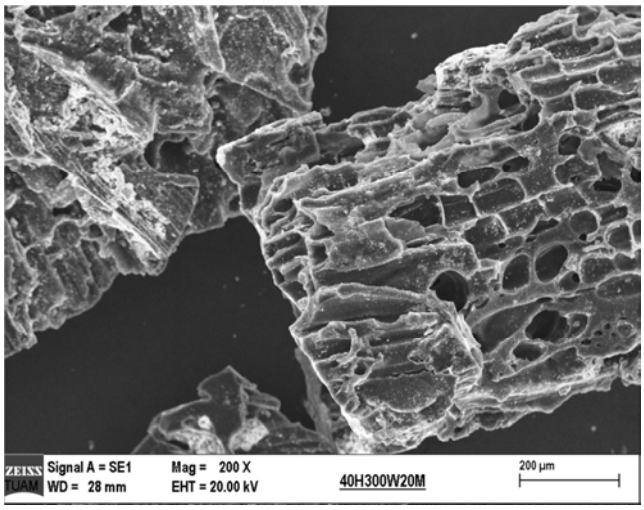

Figure 2. *SEM photo of active carbon at 200 magnification at a concentration of 40% H_3PO_4 after 20 minutes at 300 W.*

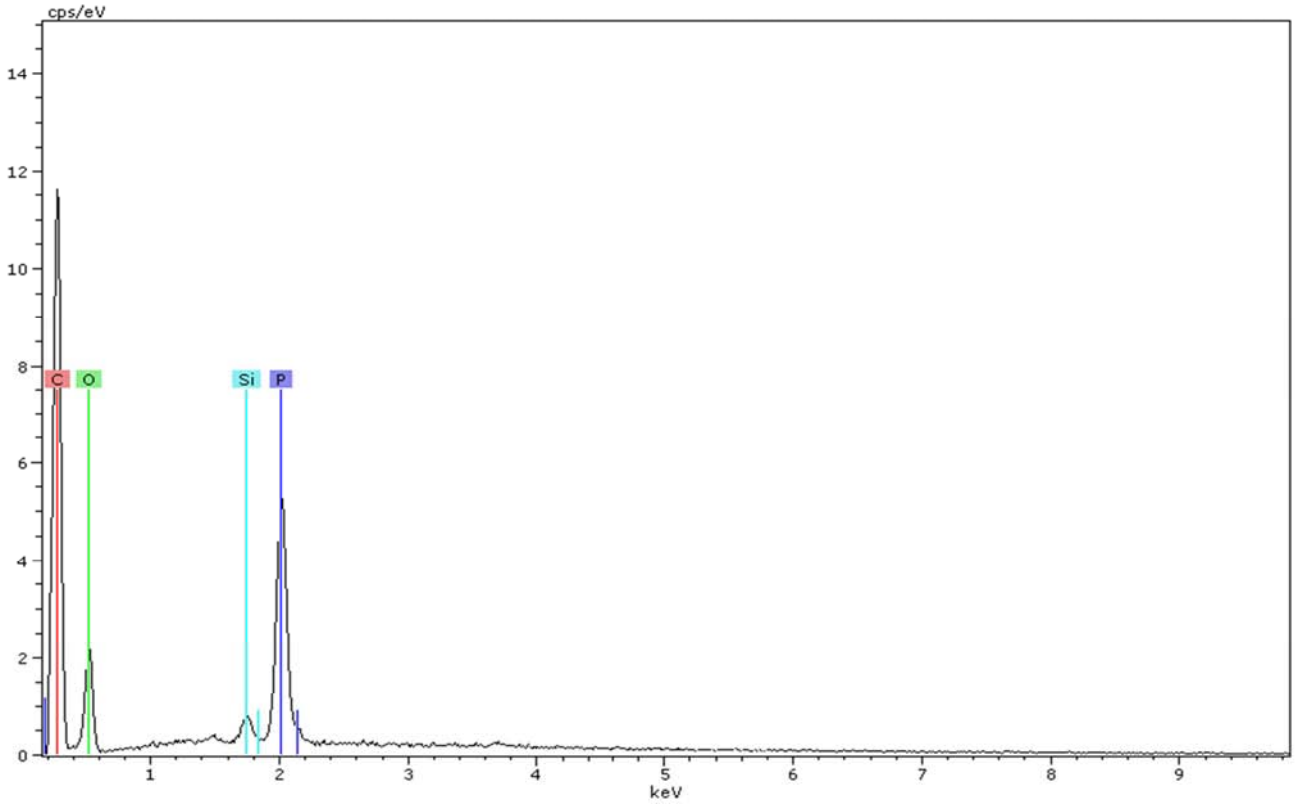

Figure 3. *SEM-EDX analysis of activated carbon obtained after 20 minutes at 300 W for 40% H_3PO_4 concentration.*

3.3. XRD Analysis Results

XRD analyzes of two samples with the highest surface area were also performed. When the XRD diffractograms of the samples are examined, it is understood that a complete crystal structure is not formed, ie the active carbon samples are amorphous. This situation is already expected for activated carbons and is parallel to the literature. Figure 4 shows the XRD analysis diffractogram of activated carbon obtained after 20 minutes at 300 W at 40% H3PO4 concentration.

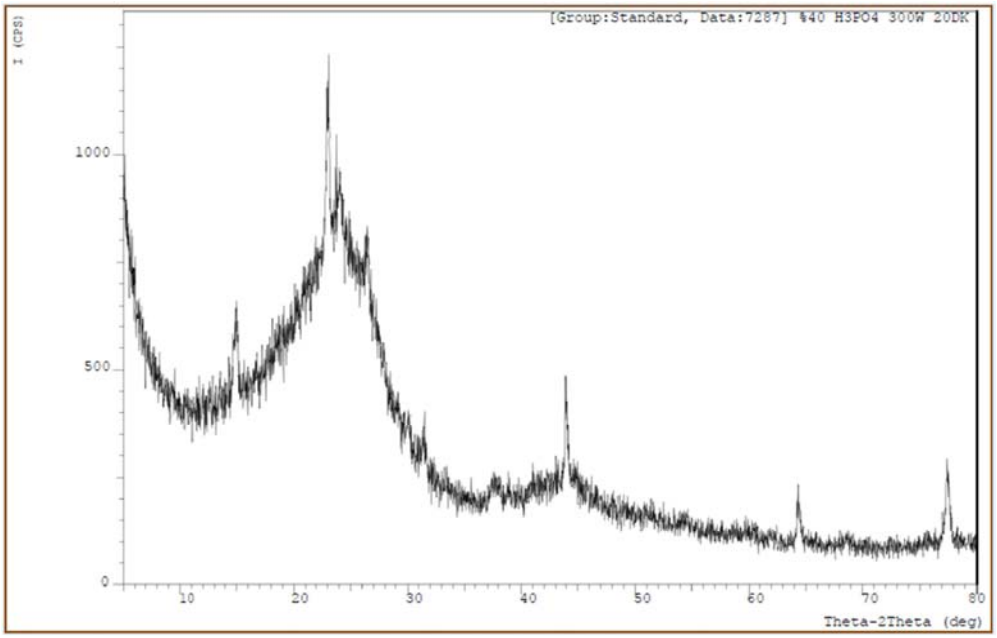

Figure 4. *XRD analysis diffractogram of activated carbon obtained after 20 minutes at 300 W for 40% H3PO4 concentration.*

4. Conclusion

According to the results obtained from the studies, it was determined that the production of activated carbon by rapid microwave assisted pyrolysis process from reed (Phragmites australis) is possible and the surface area of the obtained samples is quite high.

When SEM microstructure pictures of the samples are examined, it can be said that the general pore structure is spongy. When all the results are evaluated together; carbonization by rapid microwave-assisted pyrolysis from cane samples activated with acidic activation agent; It was found that activated carbon could be produced with high energy saving in a shorter time than conventional method. Thanks to this method, it is possible to use cane which are cheap and environment friendly biomass as adsorbent in adsorption processes.

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