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Use of Active Carbon Produced by Hydrothermal Method from Agricultural Waste in Methylene Blue Removal

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Abstract

In this study, the use of activated carbon produced from agricultural waste via the hydrothermal method for the removal of methylene blue from solution was examined. Pistachio roasting facility waste was selected as the agricultural waste. For activated carbon production, carbonized products were treated in a hydrothermal device in the presence of an activator for various durations. During processes conducted at a constant temperature, high pressure was achieved, allowing the activated carbon to attain a high adsorption capacity. The results showed that the sample with a KOH impregnation ratio of 1:1, treated in a hydrothermal device for 480 minutes at 160 °C, was able to remove methylene blue with a 99.85% extraction yield in a solution with a 350 ppm methylene blue concentration.

Keywords: Activated Carbon, Adsorption, Hydrothermal Carbonization, Methylene Blue, Organic Waste, Pistachio.

1. Introduction

Activated carbon (AC) is carbonaceous material with a highly developed internal surface area and porosity. The large surface area gives a high capacity for adsorbing chemicals from gases or liquids. The specific surface areas of ACs vary widely, with reported values ranging from 250 m^2/g to over 2000 m^2/g, making them versatile adsorbents with a wide range of applications [1]. However, production and regeneration of commercial activated carbons are still expensive and so the importance of activated carbon production by using low-cost raw materials and methods are still up to date [2].

Synthesis of activated carbon from waste biomass is of current interest towards sustainability. The properties of biomass derived activated carbon largely depends on the carbonization process [3]. Biomass being recyclable and abundantly present across the planet has been allotted numerous roles to play for sustainable development. In addition to being a food source and renewable raw material, it can be used for energy production, carbon sequestration and, as an essential element for the production of hydrochars and activated carbons [4]. In response to rising costs associated with waste disposal and increasing environmental demands, the sustainable conversion of wastes into useful products is becoming increasingly important [1].

The world consumption of activated carbons is steadily increasing and new applications are always emerging, particularly those concerning environmental pollution remediation, which should help to sustain demand for them. Important applications are related to their use in water treatment for the removal of flavor, color, odor and other undesirable organic impurities from water. Activated carbon is also used in industrial wastewater and gas treatment due to the necessity for environment protection and also for material recovery purposes. The food and pharmaceutical industries are also a major consumer of activated carbon. Activated carbon is defined as a carbonaceous material with a large internal surface area and highly developed porous structure resulting from the processing of raw materials under high temperature reactions. It is composed of 87% to 97% carbon but also contains other elements depending on the processing method used and raw material it is derived from. Activated carbon's porous structure allows it to adsorb materials from the liquid and gas phase [1]. Its pore volume typically ranges from 0.20 to 0.60 cm^3/g, and has been found to be as large as 1 cm^3/g. Its surface area ranges typically from 800 to 1500 m^2/g [2] but has been found to be in excess of 3,000 m^2/g.

Since the hetero atoms that may be present in the activated carbon structure cannot fully fill the strong valences surrounding the carbon atoms, they are bonded to the ends and corners of the crystal structure. If there is an incorrect arrangement of carbon atoms in the crystal lattice, these atoms react with oxygen, hydrogen and other atoms to reduce their energy. High-energy carbon atoms fill their valence by bonding to a neighboring simple crystal or by bonding to thermal decomposition products during carbonization [5-7].

Many studies can be found in the literature examining the production and characterization of activated carbon from a wide variety of carbon-containing materials and its potential to remove various organic and inorganic pollutants from aqueous solution. Hameed et al. in the study conducted on rattan sawdust by, the capacity of activated carbon was found to be 294.14 mg g^{-1} [8].

Duman et al. produced activated carbon from pine cones by chemical activation using H_3PO_4 and $ZnCl_2$ and after characterizing it, they reported the surface area as 1823 $m^2\ g^{-1}$ [9].

In the literature, sugar beet molasses [10], rice hulls [11], maize stalks [12], sunflower oil cake [13], peanut shells [14], green alga [15], date pits [16], lignite coal [17], bagasse, hard shells of apricot stones, almond, walnut and hazelnut shells [18] has been used for the production of activated carbon. It is possible to come across studies using organic materials.

The USA ranks first in world pistachio production with 523.900 tons in the 2023/24 production season. In the same production season, Iran follows the USA with 135.000 tons and Turkey with 119.355 tons. Turkey provides 12% of the world pistachio production [19].

In this study, the methylene blue adsorption ability of activated carbon produced by hydrothermal carbonization method was examined. It is aimed to evaluate these shells, which are left to the environment as waste after pistachio production, especially in the South Eastern Anatolia Region of Turkey.

2. Materials and Methods

2.1. Material

For the experiments, pistachio processing plant waste (PPPW) collected from the waste areas of three different pistachio processing facilities in Birecik district of Şanlıurfa were used. These wastes are materials that are not used in pistachio facilities and are generally thrown into the environment (Figure 1).

Figure 1. PPPW released into the environment after being processed in facilities

PPPW consists of the pistachio paddy located on the upper part of the pistachio fruit, known as the soft shell layer. It is a material consisting of pistachio paddy, pistachio resin and leaves.

Merck 1.05033.1000 cas number 85% purity potassium hydroxide, Merck 1.00317.2500 cas number 37% purity hydrochloric acid, Merck 1.00063.2511 cas number 100% purity acetic acid and Merck 106268.1000 cas number 99% purity sodium acetate were used in the experiments. Chem Bio, 61734 methylene blue with 99% purity was used as dye.

2.2. Equipment and Analysis

In the experiments, a domestic Vommak brand ball mill and a sieve manufactured by Yüksel Kaya Makina were used. Electro-Mag M5040P brand oven was used to evaporate the moisture in the PPPW.

For the hydrothermal treatment of PPPW samples, the Fytronix brand hydrothermal device shown in Figure 2 was used.

Figure 2. Hydrothermal device used in experiments

This device; it consists of a temperature and time controlled control panel, a hydrothermal reactor and a magnetic stirrer. The hydrothermal reactor is made of stainless steel. The interior of the reactor is designed to accommodate a 100 ml teflon container. There are two valves and a digital pressure gauge on the cover of the reactor to ensure gas inlet and outlet. The mixing process of the reactor is carried out by rotating the magnetic fish placed in the teflon container by the magnetic stirrer.

The activation process was carried out in a Nevola 160/7 brand muffle furnace with an 8-liter capacity, capable of reaching 1600 °C, and programmable in 30 different steps for time and temperature.

Shimadzu UVmini-1240 spectrophotometer device in the Metallurgical and Materials Engineering laboratories of Fırat University was used in the analyses. In the spectrophotometer with a single measuring cell, absorbance correction was made with distilled water (solvent) before the analyses. Thus, the absorbance of the solvent was subtracted during the analysis and the absorbance value of the sample was recorded directly.

Scanning electron microscope (SEM) analysis was carried out with Zeiss - EVO MA10 device in Fırat University Central Laboratory.

BET analyses were carried out using Micromeritics 3Flex device in Atatürk University DAYTAM laboratory and surface area and pore size analyser were determined. Adsorbed nitrogen gas with 99.999% purity was used in the measurements.

2.3. Method

In the literature, many studies have indicated that by increasing the surface area through grinding, higher efficiencies can be achieved in both activation and carbonization processes within shorter time periods. Additionally, grinding is necessary due to the small capacity of the HTC device’s chamber [20-22].

In addition, in a study by Şentorun-Shalaby et al. (2006) investigating the production of activated carbon from apricot pits of similar sizes, it was reported that smaller particles were more easily activated to produce activated carbons with relatively higher surface area [23].

In a study conducted by Yener et al. (2008), the use of commercially purchased granular and powdered activated carbons for the removal of methylene blue was investigated. This study found that the absorption capacity and rate were significantly higher in powdered activated carbons [24]. For these reasons, the raw material (PPPW) was ground before use.

PPPW samples were ground in a ball mill for 15 minutes, sieved through a 150 μm sieve and dried in an oven at 80 °C for 1440 minutes. Dried PPPW was placed in the teflon chamber of the hydrothermal device at the ratio of PPPW/Water = 1/10. It was processed in a hydrothermal device (HTC) at 160 ℃ for 240 min, 480 min and 720 min. During the operations carried out in the HTC chamber, the mixture was continuously stirred by a magnetic fish. During the process, it was observed that the pressure in the chamber varied between 6 and 7.2 bars. After the mixture taken out from the HTC chamber was filtered on filter paper, the solid material remaining on the upper part of the filter paper was called hydrochar. After the hydrochar was dried in the oven at 80 ℃ for 1440 minutes, mixtures were prepared for the activation process, with hydrochar/KOH=1/1 and hydrochar/KOH=1/0.5.

These mixtures were placed in a muffle furnace in steel crucibles with lids and the activation process of hydrochar was carried out by keeping them at temperatures of 600 ℃, 700 ℃, 800 ℃ and for 30 minutes, 60 minutes and 120 minutes for each temperature. The temperature of the muffle furnace was programmed to rise at 10 ℃ per minute. The product taken from the oven was placed in a beaker with a ratio of Carbonized Product / HCl solution = 1/10, and this beaker was swashed in a vortex device at the lowest speed (100 rpm) for 60 minutes. After the vortex process, the mixtures were filtered, and the sample remaining on the filter paper was washed repeatedly with pure water until pH = 6.5-7. The washed samples were dried in an oven at 80 ℃ for 1440 minutes and stored in zip lock bags for analysis.

The adsorption capacities of activated carbon samples produced from PPPW were determined using methylene blue (MB). For this purpose, 1000 mg/L MB stock solution was prepared. The stock MB solution was diluted to a concentration of 350 mg/L using 0.03 M acetic acid - 0.07 M sodium acetate solution (pH = 4.85). 50 ml of MB solutions together with 0.05 g adsorbent (activated carbon) were transferred to 250 ml conical flasks, and shaken at 150 rpm for 24 hours. Sodium acetate-acetic acid buffer solution, containing the same amount of adsorbent but without MB, was used as a blank solution. At the end of 24 hours, the samples taken from the shaker were filtered on blue banded filter paper, and the MB concentrations remaining in the filtered solution were determined by analysing them with a UV spectrophotometer at a wavelength of 664 nm after diluting with sodium acetate-acetic acid buffer solution at the appropriate ratio. To determine the amount of MB in absorbance values, the calibration curve created from the absorbance values of standard MB solutions at a concentration between 2-10 mg/L was used. The amount of MB adsorbed per unit amount of adsorbent at equilibrium (q_e, mg/g) was calculated using equation

(2.1.) based on the MB concentration values of the solutions before (c_0, mg/L) and after (c_e) adsorption [25].

$$q_e = \frac{(C_0 - C_e)V}{M_S} \qquad \textbf{(2.1.)}$$

Here,
V: MB solution volume (L),
M_S: indicates the amount of adsorbent (g) used in adsorption.

Then, according to the Langmuir adsorption equation (2.2.) given below for each concentration, the calculated c_e/q_e values against the equilibrium concentration values (c_e) were plotted and Langmuir adsorption capacities (q_m, mg/g) were calculated from the slope of the obtained line. Thus, the MB adsorption capacities of each carbonized product were determined.

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{bq_m} \qquad \textbf{(2.2.)}$$

Here,
b is the constant related to the adsorption energy.

After the process in HTC, the experimental conditions and sample codes of activated carbon production carried out with an activator in the furnace are given in Table 1.

Table 1. Experimental conditions of carbonization processes

Hidrochar/KOH= 1/1 (N Series)				
		HTC Time (160 °C)		
Muffle Furnace		***240 min***	***480 min***	***720 min***
600 °C	30 min.	N1	N2	N3
	60 min.	N4	N5	N6
	120 min.	N7	N8	N9
700 °C	30 min.	N10	N11	N12
	60 min.	N13	N14	N15
	120 min.	N16	N17	N18
800 °C	30 min.	N19	N20	N21
	60 min.	N22	N23	N24
	120 min.	N25	N26	N27
Hidrochar/KOH= 1/0.5 (A Series)				
		HTC Time (160 °C)		
Muffle Furnace		***240 min***	***480 min***	***720 min***
600 °C	30 min.	A1	A2	A3
	60 min.	A4	A5	A6
	120 min.	A7	A8	A9
700 °C	30 min.	A10	A11	A12
	60 min.	A13	A14	A15
	120 min.	A16	A17	A18
800 °C	30 min.	A19	A20	A21
	60 min.	A22	A23	A24
	120 min.	A25	A26	A27

3. Results and Discussion

The samples, which were processed for 240, 480 and 720 minutes in the HTC device, were then activated for different periods of time in the muffle furnace at 600 ºC and the MB removal efficiencies were examined (Figure 3 (a) and (b)).

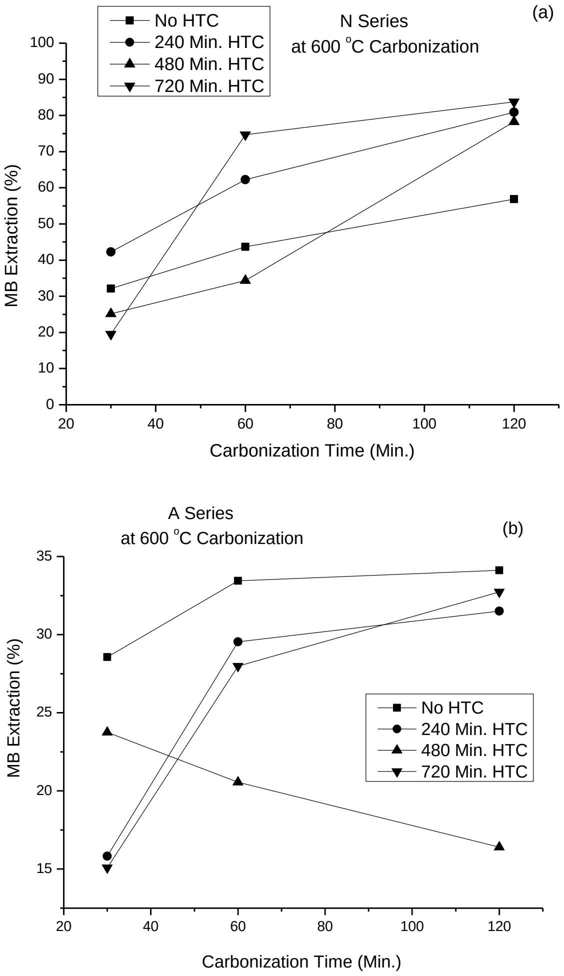

Figure 3. Methylene blue adsorption capacities of samples activated for 30, 60 and 120 min at 600 ºC, **(a)** N Series: Hydrochar/KOH=1/1 **(b)** A Series: Hydrochar/KOH=1/0.5

While the MB extraction yield of the activated carbon obtained from the sample carbonized for 120 minutes at 600 ºC with Hydrochar/KOH=1/1 (N Series) without treatment in the HTC device was 56.83%, after being processed in the HTC for 720 minutes, the sample which was activated at the same conditions of carbonization, the extraction yield of the was calculated as 83.75%.

As a result of examining the graphs, it was observed that the best performance was obtained with the sample coded N20, which was subjected to the process in the HTC device for 480 minutes, then prepared with a Hydrochar/KOH ratio of 1/1, and subsequently activated in a muffle furnace at 800 °C for 30 minutes. The MB removal yield of this sample was calculated as 99.85%.

Figure 4 (a) shows the image of the solution containing 350 mg/L methylene blue, and Figure 4 (b) shows the image of this solution after being treated with the N20 coded sample for 24 hours.

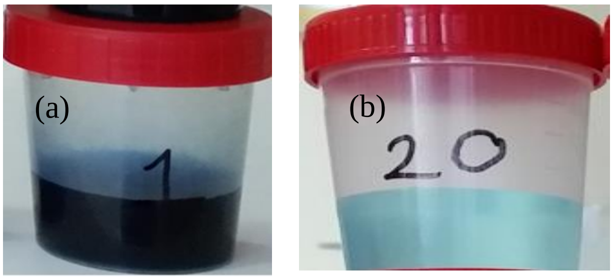

Figure 4. a) 350 mg/L MB solution **b)** the solution in which was removed MB with the sample of N20

The time for adsorption to reach equilibrium is known as the contact time. If the contact between the adsorbent and the adsorbed is achieved by shaking or mixing, the adsorption rate also increases due to the increase in the number of intermolecular collisions. With the mixing and shaking processes, the number of collisions of the molecules increased, and the MB removal yield in the solution reached up to 99.85%.

The SEM image of the sample coded N20 is given in Figure 5. When Figure 5 is examined, the honeycomb structure, which is the distinctive feature of activated carbon, is clearly seen in this sample.

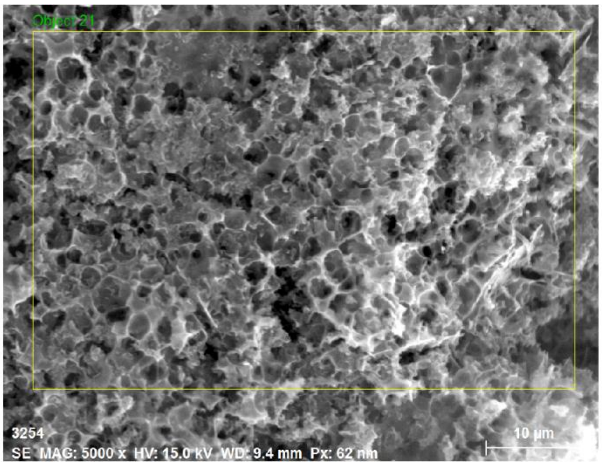

Figure 5. SEM image (5000X) of the N20 coded sample

According to SEM images, it can be said that the activation method applied with KOH is effective in pore formation. First, dehydration reactions took place within the PPPW, which was taken from the hydrothermal device and mixed with potassium hydroxide and placed in the oven. Potassium hydroxide acted as a catalyst and accelerated the degradation of lignin and hemicellulose molecules that form the cell wall. Cellulose molecules, which consist of carbon, hydrogen and oxygen, which are resistant to chemical degradation, have undergone thermal degradation during the carbonization stage.

Surface area measurements of carbonized products obtained by hydrothermal carbonization were carried out with the Surface Area Measurement Device (BET). Accordingly, the highest BET surface area was determined as 1,094.34 m²/g in sample A27, while the surface area of sample number N20, which had the highest methylene blue extraction efficiency, was determined as 1,035.74 m²/g.

In the literature, it is stated that the impregnation ratio strongly influences the properties of activated carbon, and selecting the appropriate impregnation ratio is crucial for producing activated carbons with the highest surface area and pore volume. It has been noted that the composition of lignocellulosic materials affects the structure of activated carbons in terms of porosity, and if they contain a relatively high amount of lignin, the BET surface area of the activated carbons will increase [13].

In commercial products, the BET surface area values are stated to be ≥800 m²/g for Merck [26], <1000 m²/g for APC Pure [27], and between 500-1000 m²/g for Sigma Aldrich [28]. In the literature, the BET surface area values for activated carbon samples produced from various organic sources are reported as ranging from 1368.5 to 792.2 m²/g for sugar beet molasses [10], 1684 m²/g for maize stalks [12], 726 m²/g for peanut shells [14], 1000 m²/g for Tunçbilek lignite coal [17], and 1387.30 m²/g for apricot stones [18].

4. Conclusion

In this study, activated carbon was produced by hydrothermal method using pistachio processing plant waste (PPPW), which are grown abundantly in our country, especially in the eastern and South Eastern Anatolia regions, and which cause environmental pollution after harvesting. For activated carbon production, carbonized products were processed in a hydrothermal device (HTC) in the presence of an activator for various periods of time. In the processes carried out at constant temperature, high pressure values were reached, enabling activated carbon to reach a high adsorption capacity. As a result of the studies, it was determined that the sample held in the HTC at 160 °C for 480 minutes, then impregnated with KOH at a 1:1 ratio, and subsequently activated in a muffle furnace at 800 °C for 30 minutes, was able to remove MB from a solution with a concentration of 350 ppm with a 99.85% extraction efficiency.

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Author’s Contributions

Mustafa BOYRAZLI: Supervised manuscript writing, performed the result analysis.

Elif ARANCI ÖZTÜRK: Drafted and wrote the manuscript, assisted in analytical analysis on the structure.

Emrah ÇELİK: Supervised the experiment’s progress, helped in manuscript preparation.

Mehmet Ali YASLI: The experimenter is a graduate student.

Ethics

There are no ethical issues after the publication of this manuscript.

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