



A new method on producing high surface area activated carbon: The effect of salt on the surface area and the pore size distribution of activated carbon prepared from pistachio shell

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ABSTRACT

Activated carbons having high surface area were produced from pistachio shells that were chemically activated using zinc chloride at room temperature and then activated in carbon dioxide gas that was saturated in water vapor. The surface area and pore structure of activated carbon produced from pistachio shells were investigated depending on the amount of chemical activating reagent, activation temperature, duration time in the reagent solution and the kinds of activation reagent. In this study, a method, which provides activated carbons having the highest surface area was developed. As a result, the activated carbon produced from pistachio shells treating with 40% ZnCl₂ then with 40% HCl in room temperature and then activated at 900 °C for 90 min has 3256 m²/g of BET surface area and 3822 m²/g of DR surface area and 1.36 cc/g of DR micropore volume. It was found that the activated carbon produced from pistachio shells treated 40% of sodium chloride and activated at 900 °C for 90 min has 3895 m²/g of BET surface area and 5235 m²/g of DR surface area and 1.86 cc/g of DR micropore volume.

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1. Introduction

Activated carbon (AC) is an adsorbent produced from a variety of carbonaceous source materials. Having high surface area and porosity, it is used as an adsorbent for removal of organic compounds from air and water streams. They are also used as catalysts and catalyst supports in the catalytic processes. Therefore, high surface area and porosity are very important for AC quality because the removal of large amounts of chemical compounds from gas or liquid streams on ACs. Adsorption via carbon is an expensive process. Therefore production of low cost AC has been developed. There are many studies on ACs done with a variety of shells and agricultural wastes [1–14]. The reason of choice of them for this aim is their abundance and availability.

The qualities and characteristics of ACs depend not only on the physical and chemical properties of the initial material but also on the activation methods used. Generally, physical and chemical methods are used to prepare AC having high surface area. In the physical activation, a raw material is firstly carbonized and then

activated by steam or carbon dioxide (CO₂). Lua and Yang [15] carried out studies on the effect of physical activation on the characterization of AC prepared from pistachio-nut shells. In the chemical activation, a raw material is impregnated with an activating reagent and then heat-treated in inert atmosphere. Chemical activation with zinc chloride (ZnCl₂) was studied by many researchers on different preparation conditions [16,17].

However, Hourieh et al. [18] studied the effect of ZnCl₂ concentration on the properties of AC using a raw material like pistachio-nut shell. Activation temperature, duration time in activating reagent solution, the amount and the type of activating reagent, activation time are important parameters that determine the quality of AC.

In this study, ACs were produced from pistachio shells which were chemically activated by using ZnCl₂ and then physically activated by carbon dioxide passed through water. The gas mixture was used as an inert atmosphere during activation as it is believed that CO₂ activation mainly causes the creation of microporosity while steam activation increases the development of mesoporosity to a higher extent. As a result, ACs with the higher surface area were produced from pistachio shells prepared under the condition of 40% salt solution impregnation and then activated at 900 °C for 90 min.

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2. Experiment

2.1. Sample preparation

Firstly, pistachio shells were picked up on the field of Gaziantep city of Turkey. The soft part of shell was removed and the hard shell was crushed into big pieces. Then they were washed with hot distilled water until it becomes colorless and then dried at room temperature. ZnCl_2 , sodium hydroxide (NaOH), hydrochloric acid (HCl) and salt (NaCl) were used for chemical activation. In the activation procedure; 17 g of washed pistachio shells were put into the baker and the effect of different duration times (1 day and 1 week) were examined. Before heating begun, air in reactor was purged by CO_2 that was passed through water. At all of experiments the flow rate of inert gas and heating rate were fixed at $100 \text{ cm}^3/\text{min}$ and at $10^\circ\text{C}/\text{min}$ during carbonization, respectively. At the end of the activation process, the flow of inert gas was not stopped and the obtained product was cooled by the flowing inert gas, since carbon reacts with oxygen in air at high temperature resulting in formation of CO_2 . This may cause losing carbon in the structure. The product was washed by pure water and then dried in an oven at 80°C over a day. In order to investigate the effect of activation temperature, the pistachio shells obtained from one of the series that held in solution more than 1 day were activated at 800°C and the others at 900°C . It is thought that the chemical activation which is applied with different activation reagents might be increasing the surface area of AC. So, chemical activation was carried out in two stages, the first stage includes the impregnation of 40% ZnCl_2 solution and the second stage 40% solution of another activating reagent. Also, in order to investigate the effect of acid and base as other activating reagents at the second stage, HCl as acid and NaOH as base, were used. On the other hand, salt impregnation was carried out at only one stage.

2.2. Sample characterization

Characterization of obtained ACs was carried out instrumentally. Its adsorption characterizations were determined by nitrogen adsorption at -196°C with surface area and pore size analyzer (NOVA 4000e-Quanta chrome instruments). Before analysis, all of the obtained ACs were evacuated under vacuum 180°C over night in order to clean all the pores. Their surface areas were calculated from the isotherms by the Brunauer–Emmett–Teller (BET) equation [19]. The cross sectional area of a nitrogen molecule is assumed to be 0.162 nm^2 . Their micropore volumes were calculated by using Dubinin–Radushkevich (DR) equation [20]. The pore size distribution was determined by using Barrett–Joyner–Halenda (BJH) model [21] and Horvath–Kawazoe (HK) method [22].

Fourier transforms infrared (FT-IR) spectroscopy spectra were obtained using a spectroscope (PERKIN-Elmer Spectrum RXI FT-IR Spectrometer) at 4-cm^{-1} resolution. Pressed potassium bromide KBr pellets at a sample/KBr weight ratio of 1:100 were scanned and recorded between 4000 and 450 cm^{-1} . Scanning electron microscope (Zeiss EVO 50 Model) was carried out to show the pore structure of obtained ACs.

3. Results and discussion

3.1. Effects of the amount of activating reagent

In this part of the study, solutions which involved different percentages of ZnCl_2 (10, 20, 40 and 50) were prepared. 17 g pistachio shells were immersed into each solution and the shells held in the solutions for 1 day, then they were activated at 900°C for 45 min. Fig. 1 shows the isotherms of nitrogen adsorption at -196°C for obtained ACs.

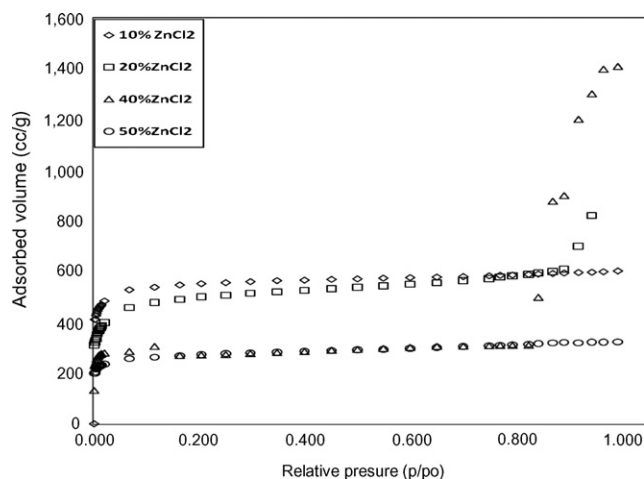


Fig. 1. Effect of the amount of activating reagent on the adsorption isotherm of the AC. (Act. temp., 900°C ; act. time, 45 min; duration time in solution, 1 day.)

As seen in the figure, monolayer adsorption is observed through the pores of AC obtained from 10% and 50% ZnCl_2 solutions. This behavior of the isotherms point to Type I isotherm which indicates microporous material coated monolayer with respect to IUPAC classification. If there is a little increase in adsorbed gas volume in plateau in Type I, the formation plateau at moderate pressure can also be acceptable such as the multilayer adsorption of the pores, e.g. the isotherm pre-treated with 10% ZnCl_2 solution. A capillary condensation and multilayer adsorption were observed on the pores of AC impregnated from 40% ZnCl_2 and 20% ZnCl_2 solutions. This behavior presents Type IV isotherms which indicate a mixture of micro- and mesoporous materials. The isotherm IV means that relative lower pressures micropores are filled by adsorbate gas, the knee formation means monolayer coated micropores; at moderate pressures observed plateau means multilayer adsorption of the pores, and increasing of adsorbed volume at high relative pressure means capillary condensation in the mesopores. These characteristic observations indicate the development of micro- and mesopore cities for the AC produced from pistachio shells by these conditions. The ACs from 10% ZnCl_2 more micropores are available in its structure solution had more surface area than the others as. Fig. 2 shows the change of BET surface and micropore volume of obtained ACs from different ZnCl_2 concentrations.

According to the results in the figure, BET surface areas and micropore volumes of produced carbon by pre-treating

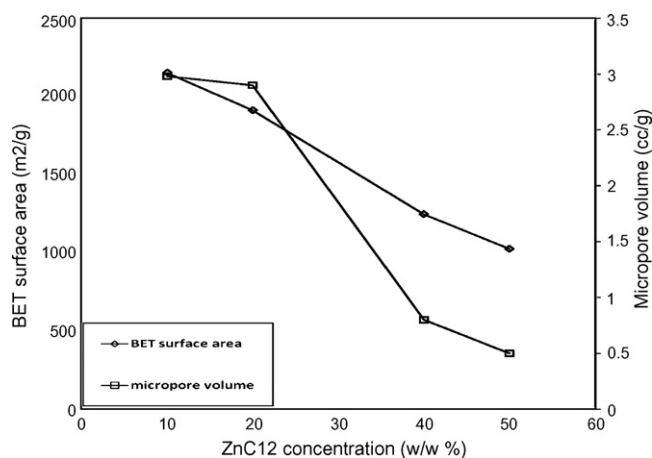


Fig. 2. The effect of ZnCl_2 ratio on BET surface area and micropore volume of AC. (Act. temp., 900°C ; act. time, 45 min; duration time in solution, 1 day.)

Table 1

The volume and surface area of pores belonged to AC obtained from different conditions.

Conditions ZnCl ₂ conc. (mol/L)	Pore temperature (°C)/duration time	Pore surface volume at 0.96 nm (cc/g)	Pore area at 5 nm (m ² /g)	Volume at 5 nm (cc/g)
10	900 °C/1 day	0.845	46.24	0.05
20		0.7561	47.44	0.06
40		0.4821	103.47	0.15
50		0.4163	12.49	0.03
10	800 °C/1 day	0.7406	55.5	0.07
20		0.7239	30.31	0.04
40		0.4097	89.65	0.11
50		0.3755	20.5	0.038
10	900 °C/1 week	0.71	62.75	0.07
20		0.6038	162.37	0.22
40		0.2526	84.73	0.12
50		0.1945	14.26	0.02
HCl	900 °C/1 day	0.6724	353.25	0.45
NaOH		1.302	370.52	0.5
NaCl		1.5844	703.3	0.93
Commercial AC		0.7694	107	0.16
Raw pistachio shell		0.2424	16.84	0.02

with the 10%, 20%, 40% and 50% ZnCl₂ are 2151 m²/g–2.98 cc/g; 1911 m²/g–2.9 cc/g; 1247 m²/g–0.8 cc/g and 1025 m²/g–0.5 cc/g, respectively. As the ZnCl₂ ratio was increased, the surface area and micropore volume were decreased. The dehydration agent-ZnCl₂ was used excessively, a destructive result can be observed in micropore structure as expected. Mesopore structure developed. When the values calculated by using software of the analyzer are investigated, this effect can be easily seen. These values obtained in different conditions, the volume of pores having 0.96 nm of diameter and surface area and volume of pores having about 5 nm of diameter, were given in Table 1.

According to the results in the table, while the amount of ZnCl₂ was increased, there is a decreasing of micropore volume (at range of 0.01–0.96 nm). In spite of this result, at the range of 1–5 nm ACs obtained from 40% ZnCl₂ solution depending on temperature have a pore surface area which is larger and a pore volume which is bigger than that in 10% ZnCl₂. Increase of ZnCl₂ impregnation increases releasing of volatiles presented on raw pistachio shells structure. This is the reason of increase in the widening of the pore; micropores formed are converted to mesopores and even macropores. This mechanism of pore widening had also been reported by other researchers [16,23]. But at conditions depending on time the increasing of activating reagent may cause the destruction of pores that can be widened after the treatments. This effect was observed at obtained AC from 50% ZnCl₂. As a result of this the surface area and volume of pores having 5 nm of diameter belonged to the AC were 14.26 m²/g and 0.02 cc/g, respectively. Naturally, when the BET surface area of the AC was 1025 m²/g at 1 day of duration time, it was 510 m²/g. It is thought that this result supports the destruction.

It can be seen their isotherm graph that obtained AC from 40% ZnCl₂ solution adsorbed most gas volume. But this AC has the BET surface area of 1247 m²/g, whose value is lower than that of 10% ZnCl₂ solution. The reason of this behavior is due to their micropore volumes. The micropore volume of obtained AC from 40% ZnCl₂ solution is 0.8 cc/g (on the basis of DR method) and its mesopore volume is 1.7 cc/g (on the basis of BJH method). The micropore volume of obtained AC from 10% ZnCl₂ solution has 2.98 cc/g and its mesopore volume is 0.83 cc/g. It is believed that the surface area is sum of micro- and mesopore volume. But micropore volume is very important for surface area. The isotherms of ACs support this result.

3.2. Effects of the activation temperature and duration time in the activating reagent solution

Experiments in this part were carried out to see the effects of the activation temperature and duration time in the activating reagent solution on BET surface area and the development of porous structure. For the examination of the activation temperature conditions mentioned above were the same but only the activation temperature was changed to 800 °C. Isotherms of obtained AC were given in Fig. 3.

As seen in Fig. 3, the isotherms illustrate Type IV mesoporous development but they also show microporous destruction, the less micropore volume obtained in comparison with Fig. 1. The isotherm belonged to 10% ZnCl₂ is related Type IV. At 20% ZnCl₂, the mesoporosity was developed more than the one at 10% ZnCl₂, but its BET surface area was less than one at 10% ZnCl₂. Micropore adsorptions which were obtained from ACs prepared from 40% ZnCl₂ and 50% ZnCl₂ solutions were less in comparison with one prepared from 10% ZnCl₂ solution. But, there are the capillary condensation and multilayer adsorption on the pore of AC prepared from 50% ZnCl₂.

If we look at Table 1, it is seen that BET surface and micropore volumes of ACs were decreased by decreased carbonization temperature. But mesopore formation was limitedly promoted by temperature decreasing. Because of the ZnCl₂ concentration was

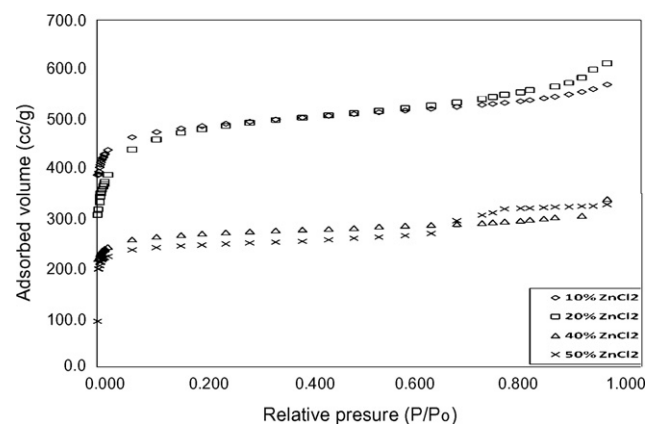


Fig. 3. Effect of activation temperature on the adsorption isotherm of the AC. (Act. temp., 800 °C; act. time, 45 min; duration time in solution 1 day.)

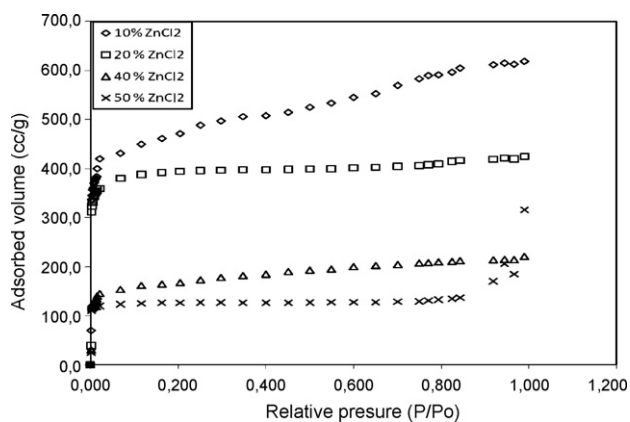


Fig. 4. Effect of duration time on the adsorption isotherm of the AC. (Act. temp., 900 °C; act. time, 45 min; duration time in solution, 1 week.)

increased, the radius of formed pore did not increase but the volume and the surface area of pores increased. The pore volume and the pore surface area of obtained ACs at 1–5 nm range are 0.07 cc/g and 55.5 m²/g at 10% ZnCl₂; 0.04 cc/g and 20.5 m²/g at 50% ZnCl₂, respectively.

In order to see the effect of duration time in the activating reagent solution on BET surface area and development of porous structure, other conditions were kept the same but duration time was changed to from 1 day to week. Isotherms of obtained ACs are given in Fig. 4.

As seen from Fig. 4, there is the capillary condensation and multi layer adsorption on the pores of AC obtained from 50% ZnCl₂, while multi layer adsorption was observed on the other ACs. As multi layer adsorption and capillary condensation formed on mesopores, it can be said that mesoporous structure was developed. In the meantime, micropore volume decreased due to that widened micropores as carbon was evaporated by forming CO₂, which also decreased BET surface area.

The effect of activation temperature on BET surface area and micropore volume which is calculated from isotherms of obtained ACs in this experimental set versus ZnCl₂ concentration and ACs was given in Fig. 5.

As seen in Fig. 5, as ZnCl₂ concentration increased the BET surface area and micropore volume decreased. This result was expected because of decrease in the activation temperature. At the same time, mesopore structures developed more in comparison with 900 °C while their micropore volumes at 10% and 20% ZnCl₂ concentrations decreased more.

Also, as the amount of ZnCl₂ increased in a week duration time and low activation temperature decreased BET surface area and micropore volume of obtained ACs more than 1 day duration. These results were expected due to the raw material impregnated with ZnCl₂ solution for a long time. The destructive effect of ZnCl₂ on microporous structure was observed more at 20% ZnCl₂ solution. Hence, provided that the other conditions remained same, BET surface area would be 2151 m²/g at 10% ZnCl₂ when exposed solution for one day, 1751 m²/g for 1 week. For 1 day and 1 week contacting these values were 1911 m²/g and 1552 m²/g at 20% ZnCl₂; 1247 m²/g and 624 m²/g at 40% ZnCl₂ and 1025 m²/g and 510 m²/g at 50% ZnCl₂. These decreases were increased by ZnCl₂ concentration.

Briefly, the radii of ACs were widened and their micropore volumes were decreased by decreased activation temperature from 900 °C to 800 °C. In the case of 40% at 800 °C activation temperature, micropore volume was higher but its mesopore volume was lower than one at 900 °C. In the case of 1 week duration time, widening radius was available at 40% and mesopore volume was low which is

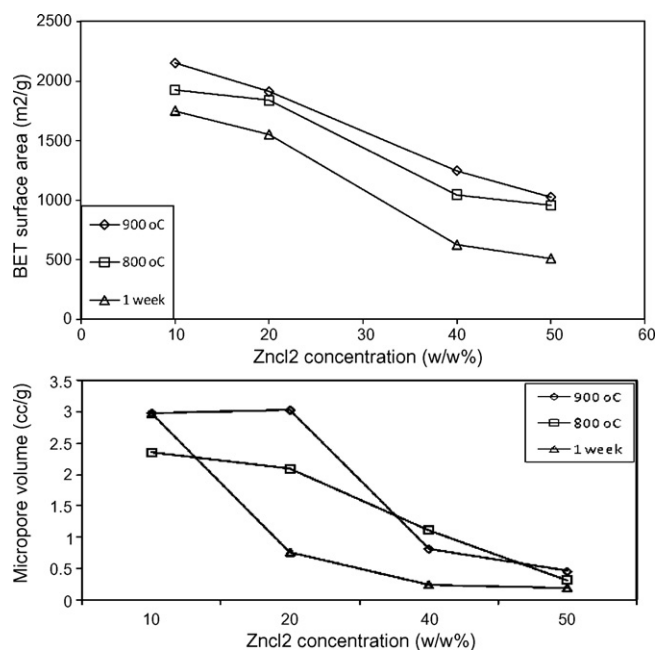


Fig. 5. The comparison of the effect of activation temperature, waiting time of ZnCl₂ solution on BET face area of ACs prepared different ratio of ZnCl₂.

in contrast with the micropore volume that did not comparatively change at 10%. Its mesopore volume was considerably low.

As a result of, not only activation temperature affected the BET surface area of and micropore volume of each ACs but also longer duration time affected each of them. On the other hand when microporous decreased, mesoporous developed limitedly.

3.3. Effects of the acid, base and salt as activating reagents at second chemical activation stage on surface area and pore development

To investigate the double effect of acid and base as activating reagents together with ZnCl₂, chemical activation was carried out in two stages. The first stage involved impregnation of 40% ZnCl₂ solution in raw pistachio shells. They were impregnated the reagent for 1 day. After the shells were filtrated, the half of the shells were put into 40% of HCl solution and the other half of the shells were put into 40% of NaOH solution. Both samples were let to stay into the reacting agent solutions at second stage for 1 day, too. Activation temperature was 900 °C and activation time was 90 min. Salt has irritating and dehydrating features because of its containing ions. Therefore it was expected that activated carbon having high porous structure can be obtained by treating with salt solution. In order to compare the results, raw pistachio shells were also activated under the same conditions as obtained ACs. The isotherms of nitrogen adsorption at –196 °C for the chemically ACs prepared at different conditions were shown in Fig. 6.

In the figure, condition 1 represents obtained AC prepared from 40% ZnCl₂ solution, stayed for 1 day into the solution and carbonized at 900 °C for 45 min; condition 2 represents obtained AC prepared from 40% ZnCl₂ solution stayed for 1 day into the solution and carbonized at 800 °C for 45 min; condition 3 represents obtained AC prepared from 40% ZnCl₂ solution stayed for 1 week into the solution and carbonized at 900 °C for 45 min; condition 4 represents obtained AC prepared from 40% ZnCl₂–40% NaOH solution stayed for 1 day into the each solution activated at 900 °C for 90 min; condition 5 represents obtained AC prepared from 40% ZnCl₂–40% HCl solution stayed for 1 day into the each solution activated at 900 °C for 90 min; condition 6 represents obtained raw

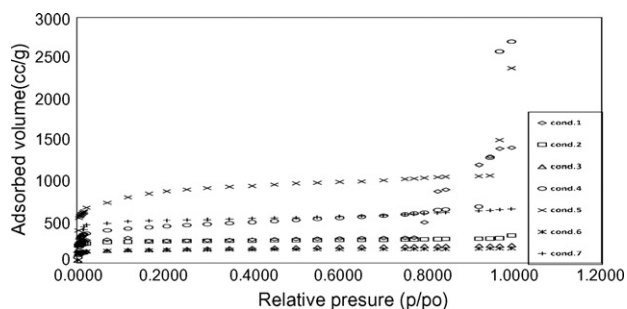


Fig. 6. The comparison of the adsorption isotherm of the AC prepared under different condition.

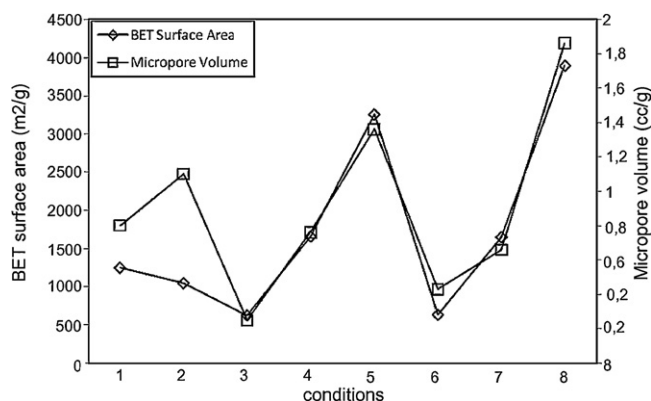


Fig. 7. The comparison of the BET surface area and micropore volume of obtained AC and raw pistachio shell and commercial AC.

pistachio shells activated at 900 °C for 90 min; condition 7 represents commercial AC; condition 8 represents obtained AC prepared from 40% NaCl solution stayed for 1 day into the solution and then activated at 900 °C for 90 min.

The general trends of the isotherms of ACs prepared from conditions 2, 3, 6 and 7 were similar which has available knee formation at a very low relative pressure which means all micropores were filled. Under conditions 4, 5 and 8 obtained ACs adsorbed more gas compared to the ones of under conditions 2, 3, 6 and 7 due to capillary condensation. The obtained BET surface area from isotherms and micropore volume from DR method versus ZnCl₂ concentration method were shown in Fig. 7.

It can be seen that AC prepared from condition 8 has the highest BET surface area and micropore volume. As expected the cause of this result is the irritating and dehydrating features of salt.

According to the results, the impregnation of different ions (e.g. Na⁺, OH⁻, H⁺, Cl⁻) affected BET surface area and micro- and mesopores formation. Table 2 shows comparatively DR micropore surface area, DR micropore volume, DR pore radius, BJH pore volume of each obtained ACs from different conditions together with the used methods for calculation.

As followed from Table 2, BJH pore volumes of ACs impregnated by 40% ZnCl₂ solution (conditions 1–3) decreased by decreasing activation temperature and duration time in the activating reagent solution. On the other hand, at impregnation NaOH, condition 4,

especially, BJH pore volume increased. It was 3.51 cc/g. Its micropore volume was only 0.77 cc/g due to that available micropores on structure was damaged in this condition. So, its BET surface area was 1657 m²/g. AC that produced by condition 5, at impregnation HCl, had BET surface area of 3256 m²/g. At the same time, the AC had micropore volume of 1.36 cc/g and mesopore volume of 2.29 cc/g.

3.4. Microstructure of obtained ACs

The microstructures of the ACs prepared from conditions 1, 5 and 8 were shown in Fig. 8. AC prepared from condition 1 had less BET surface area and pore volume than ones from conditions 5 and 8. The cause of this can be related to the fact that its pores did not open. This can be attributed to melting observed on the surface of the AC. Evidence of insufficient opening on pores can be clearly seen from the scanning electron micrograph of the AC in Fig. 8(a). Applied processes on pistachio shells at condition 5 caused pores to open due to a great part of the volatiles originally available in pistachio shell have evolved. As can be clearly seen from Fig. 8(b), obtained AC at condition 5 has quite good and clear pore structure. Small pores, transitional pores, and large pores with different shapes could also be clearly identified from this micrograph, which is responsible for the higher BET surface area and pore volume (depended on DR method and BJH method). The impregnation with HCl of pistachio shells increased the effect of ZnCl₂. This coupled effect helped the formation and the opening of pores. Fig. 8(c and d) illustrated the micrographs of AC prepared from condition 8. It may be think that formed particles on and through the pores by impregnating salt can be responsible higher surface area and more pore volume.

3.5. Surface chemistry of obtained ACs

As known, the H element and OH groups are the main components of lignocellulosic materials whose family includes pistachio shells. It is already reported that the main oxygen groups present in pistachio-nut shells are the carbonyl groups, ethers, esters, alcohols, and phenol groups [15]. It was thought that the bonds on the structure of the impregnated shell would be rearranged during activation at a high temperature. It is possible that the groups on the structure of AC were decomposed and their by-products as volatile materials were released during impregnation of HCl and heat treatment. So, the spectrum of the AC with HCl solution after impregnation with ZnCl₂ at 900 °C was shown in Fig. 9.

The spectra display following bands:

- 3343 cm⁻¹: O–H stretching vibrations in alcohols and phenols (3600–3200 cm⁻¹), 2877 cm⁻¹: C–H stretching vibrations in alkane or alkene groups (3000–2850 cm⁻¹),
- 2271 cm⁻¹: C=C stretching vibrations in alkyne groups (2260–2100 cm⁻¹ variable, not present in symmetrical alkynes),
- 1717 and 1654 cm⁻¹: C=O stretching vibrations in carbonyl groups in amid (1820–1650 cm⁻¹),
- 1559, 1508 and 1458 cm⁻¹: C=C stretching vibrations in aromatic ring (1600–1400 cm⁻¹),
- 1166 cm⁻¹, 1118 cm⁻¹ and 1065 cm⁻¹: C–O stretching vibrations in alcohols, phenols or ether or ester groups (1050–1150 cm⁻¹).

Table 2

The properties of obtained ACs from different conditions.

Condition	Cond. 1	Cond. 2	Cond. 3	Cond. 4	Cond. 5	Cond. 6	Cond. 7	Cond. 8
DR average pore width (nm)	0.603	1.759	0.831	1.148	1.191	1.436	0.868	1.146
BJH pore volume (cc/g)	1.7	0.12	0.09	3.51	2.29	0.03	0.25	4.59
DR micr. surface area (m ² /g)	1306	314	696	2154	3822	1200	1849	5235
DR micr. volume (cc/g)	0.8	1.11	0.25	0.77	1.36	0.43	0.66	1.86

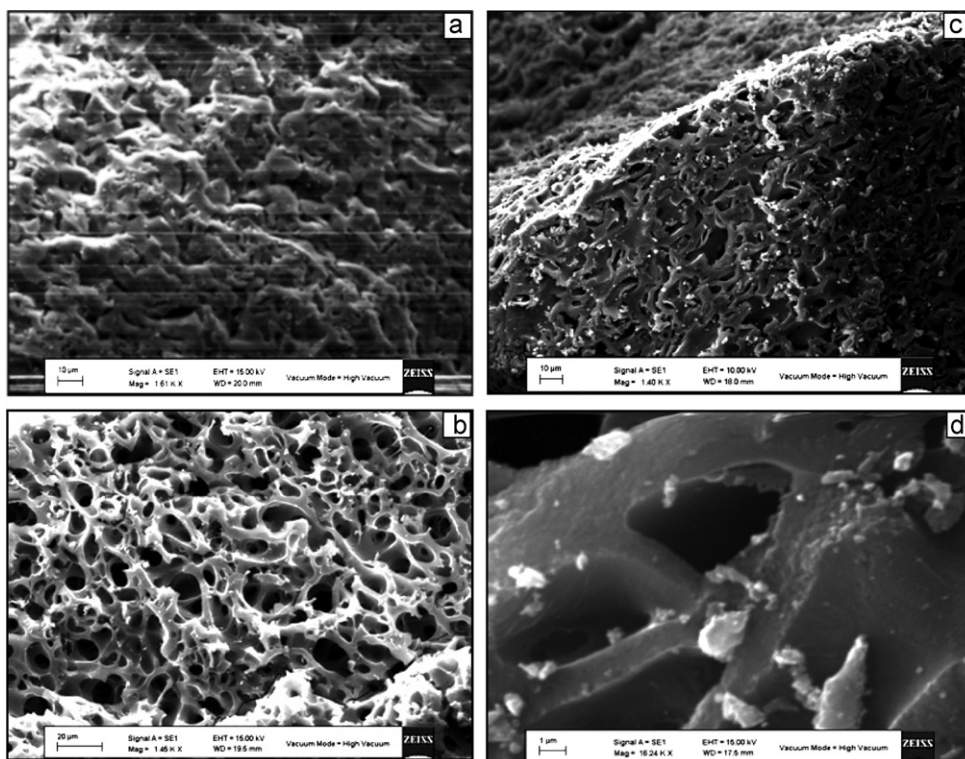


Fig. 8. The SEM figures obtained ACs from (a) 10% ZnCl_2 solution and (b) 40% ZnCl_2 -40% HCl solutions (c) at 1 μm of (b) and (d) 40% salt solution at 1 μm of (d).

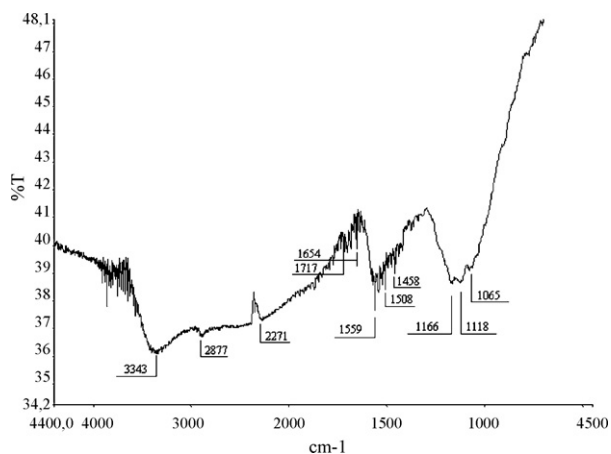


Fig. 9. Fourier transform infrared spectrum of AC impregnated with ZnCl_2 and then with HCl.

As seen in the figure, the AC has a band at 3343 cm^{-1} due to ν (O–H) vibrations in hydroxyl groups. Also the AC has carbonyl and alkyne groups and aromatic rings. These functional groups were formed, possibly due to the extraction of H element and OH groups from the structure of raw pistachio shell during the impregnation ZnCl_2 and HCl, respectively, heat treatment stages as a result of the dehydration effect of ZnCl_2 . On this AC structure carboxylate ion groups disappeared because of HCl–carboxylate ions reaction during impregnation of HCl.

4. Conclusions

In this paper, the effect of preparation conditions on the surface area and pore volume of AC from pistachio shells were investigated. Investigated conditions were ZnCl_2 solution as activating solution, activation temperature, waiting time into the solution, and acid,

base and salt as second activating reagent. It was found that the best preparation conditions for the AC obtained from pistachio shell are impregnation of 40% ZnCl_2 and 40% HCl or only 40% salt solution by waiting in solution for 1 day, activated at 900°C for 90 min along, and used as inert atmosphere CO_2 passed into water, which means the condition 5 or 8.

Briefly, the ACs having $3895\text{ m}^2/\text{g}$ of BET surface area and 4.59 cc/g of mesopore volume by BJH method and 1.86 cc/g of micropore volume by DR method can be produced by treating raw pistachio shells having $631\text{ m}^2/\text{g}$ of BET surface area and 0.03 cc/g of mesopore volume and 0.43 cc/g of micropore volume.

As a result, when raw material was impregnated HCl solution after ZnCl_2 impregnation micro- and mesoporous structures were developed considerably. Pistachio shells were convenient starting material for the preparation AC having high surface area due to its chemical structure. It can be said that ACs prepared from pistachio shells may be used for both gaseous and liquid adsorption applications depending on the different activation conditions.

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