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Preparation and characterization of activated carbon produced from pomegranate seeds by $ZnCl_2$ activation

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ABSTRACT

In this study, pomegranate seeds, a by-product of fruit juice industry, were used as precursor for the preparation of activated carbon by chemical activation with $ZnCl_2$. The influence of process variables such as the carbonization temperature and the impregnation ratio on textural and chemical-surface properties of the activated carbons was studied. When using the 2.0 impregnation ratio at the carbonization temperature of 600 °C, the specific surface area of the resultant carbon is as high as 978.8 $m^2\,g^{-1}$. The results showed that the surface area and total pore volume of the activated carbons at the lowest impregnation ratio and the carbonization temperature were achieved as high as 709.4 $m^2\,g^{-1}$ and 0.329 $cm^3\,g^{-1}$. The surface area was strongly influenced by the impregnation ratio of activation reagent and the subsequent carbonization temperature.

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1. Introduction

Almost any lignocellulosic material can be used as precursors for the production of activated carbons. The use of a suitable precursor is mainly conditioned by its availability and cost, although it also depends on the main applications of the manufactured carbon and the type of installation available. Activated carbon production from coal is an old process. Due to the high cost in the production of activated carbon from coal, the use of lignocellulosic materials as precursor for the production of activated carbons has attracted much attention. The idea is to find cheap and efficient precursor for the production of activated carbon. Activated carbon can be produced from all carbon-containing lignocellulosic materials i.e. lignin [1], macro-algal [2], defective coffee press cake [3], palm shell [4], rice husk [5] wastes of vegetable origin (e.g. nutshells, fruit stones) [6].

Processing of fruits produces a solid waste of peel/skin, stones or seeds which can be used as precursor for the production of activated carbon. There are lots of fruits contain considerable amounts of seeds. One of these fruits is pomegranate (*Punica granatum* L.). The total pomegranate production in the world is about 1 million tonnes [7]. Turkey is one of the world's largest producers of the pomegranate. The annual production of pomegranate in Turkey is about 60,000 tons [8].

Activated carbons are produced through physical or chemical activation. Chemical activation can be accomplished in a single step by carrying out thermal decomposition of raw material with chemical reagents. The most widely used chemicals include zinc chloride ($ZnCl_2$), phosphoric acid (H_3PO_4), and potassium hydroxide/carbonate (KOH/K_2CO_3). An example of the use of $ZnCl_2$ for activation is the work of Olivares-Marin et al. who prepared activated carbon from cherry stones by chemical activation with $ZnCl_2$ [9]. The influence of the carbonization temperature and the impregnation ratio on textural and chemical-surface properties of the products was studied. In another study, a series of micro- and mesoporous activated carbons were produced from paper mill sludge by chemical activation with $ZnCl_2$ [10]. It was reported that $ZnCl_2$ to sludge ratios less than 1.0 and greater than 1.5 resulted in the production of micro- and mesoporous carbons, respectively [10]. Gonzalez-Serrano et al. reported the production of activated carbons with high surface areas from chemical activation of Kraft lignin with $ZnCl_2$ [11]. The produced activated carbons were essentially microporous. Activated carbons were prepared from chemical activation of pistachio-nut shell with $ZnCl_2$ under nitrogen atmosphere and vacuum condition [11]. It was reported that the activated carbons prepared under vacuum condition slightly better results in terms of BET surface area and pore volume than those produced under nitrogen atmosphere [11].

Lignocellulosic materials such as olive and peach stones have been also used as precursors for granular activated carbon, using phosphoric acid, zinc chloride and potassium hydroxide as activating agents [12]. It was reported that whereas KOH only

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Table 1
Proximate, ultimate and component analyses of pomegranate seeds.

Proximate analysis (as received, wt%)	
Moisture	5.38
Volatile matter	78.71
Fixed carbon	14.08
ash	1.83
Ultimate analysis (dry basis, wt%)	
C	49.65
H	7.54
N	4.03
S	0.65
O[a]	38.13
GCV[b] ($MJ\ kg^{-1}$)	20.98
Component analysis (dry basis, wt%)	
Extractives[c]	6.11
Hemicellulose	25.52
Lignin	39.67
Cellulose	26.98

[a] By difference.
[b] Gross calorific value.
[c] Toluene/alcohol (2/1, v/v).

produces a widening of micropore width, $ZnCl_2$ additionally develops small mesoporosity and H_3PO_4 leads to a more heterogeneous pore size distribution [12]. In another study, several studies of activated carbons have been prepared by chemical activation of peach stones with $ZnCl_2$ [13]. It was reported that the main factor affecting the surface area and the micropore size distribution is the amount of Zn introduced in the precursor during impregnation. In addition, partial gasification in CO_2 of the carbons produced a considerable developing of surface area and porosity [13]. In our previous papers, we produced the activated carbons from oil cakes by chemical activation with sulfuric acid [14] and alkaline hydroxides/carbonates [15].

In the present study, activated carbons were prepared from pomegranate seeds by chemical activation with $ZnCl_2$. The yields, specific surface areas, pore volumes, and pore sizes of the activated carbons were determined. The effects of impregnation ratio and carbonization temperature on these properties were also studied.

Table 2
Conditions used for the production of activated carbon from the pyrolysis of pomegranate seeds with $ZnCl_2$ activation.

Carbonization temperature (°C)	Type of activated carbon	Theoretical impregnation ratio	Experimental impregnation ratio
600	BC1	–	–
800	BC2	–	–
600	PAC1	0.50	0.44
800	PAC2	0.50	0.44
600	PAC3	1.00	0.80
800	PAC4	1.00	0.80
600	PAC5	1.50	1.31
800	PAC6	1.50	1.31
600	PAC7	2.00	1.80
800	PAC8	2.00	1.80

Table 3
Yields of activated carbons and chemical recoveries in the $ZnCl_2$ activation process.

Carbonization temperature (°C)	Type of activated carbon	Theoretical impregnation ratio	Yields of activated carbon[a] (wt%)	Chemical recovery (wt%)
600	BC1	–	29.28	–
800	BC2	–	27.87	–
600	PAC1	0.5	37.75	74.43
800	PAC2	0.5	36.59	71.73
600	PAC3	1.0	38.50	80.16
800	PAC4	1.0	36.75	77.90
600	PAC5	1.5	39.16	78.68
800	PAC6	1.5	38.26	77.91
600	PAC7	2.0	39.21	81.24
800	PAC8	2.0	37.58	77.50

[a] All yields calculated on a dry basis.

2. Experimental

2.1. Materials

Pomegranate seeds were obtained from Dimes Company, Izmir, Turkey and used without further treatments. The proximate, ultimate and component analyses of pomegranate seeds are shown in Table 1. Determination of each component of pomegranate

Table 4
Main characteristics of biochars and activated carbons from the pyrolysis of pomegranate seeds with $ZnCl_2$ activation.

Type of activated carbons	BC1	BC2	PAC1	PAC2	PAC3	PAC4	PAC5	PAC6	PAC7	PAC8
Carbonization temperature (°C)	600	800	600	800	600	800	600	800	600	800
Impregnation ratio	–	–	0.5	0.5	1.0	1.0	1.5	1.5	2.0	2.0
Proximate analysis										
Volatile matter	12.10	9.50	9.58	8.22	11.47	9.01	16.59	14.08	14.83	13.39
Fixed carbon	78.74	80.41	88.36	87.53	87.41	88.92	82.00	83.43	83.05	84.88
Ash	9.16	10.09	2.06	4.25	1.12	2.07	1.41	2.49	2.12	1.73
Elemental analysis (wt%)										
C	74.86	76.19	80.54	76.94	78.89	79.21	75.16	76.66	76.94	79.34
H	1.94	1.79	2.08	1.49	2.55	1.26	2.55	2.43	3.23	2.39
N	3.05	2.84	4.21	3.79	3.69	2.87	2.89	3.57	3.08	3.30
S	0.04	0.05	0.27	0.22	0.11	0.19	0.14	0.13	0.21	0.07
O[a]	20.11	19.13	12.90	17.56	14.76	16.47	19.26	17.21	16.54	14.90
Typical properties										
BET surface area, S_{BET} ($m^2\ g^{-1}$)	2.631	2.924	709.4	455.3	697.9	743.1	856.0	692.5	978.8	823.7
Micropore surface area, S_{mic} ($m^2\ g^{-1}$)	0	0	466.3	407.4	495.7	632.6	596.9	268.9	587.6	153.8
Mesopore surface area, S_{meso} ($m^2\ g^{-1}$)	2.631	2.924	243.1	47.87	202.2	110.5	259.1	423.6	391.2	669.9
Total pore volume ($cm^3\ g^{-1}$)	0.032	0.029	0.329	0.217	0.320	0.376	0.393	0.318	0.563	0.350
Micropore volume ($cm^3\ g^{-1}$)	0	0	0.240	0.211	0.254	0.304	0.135	0.139	0.283	0.071
Mesopore volume ($cm^3\ g^{-1}$)	0.032	0.029	0.089	0.006	0.066	0.072	0.258	0.179	0.280	0.279
Average pore diameter (A°)	16.2	22.2	14.0	16.6	15.6	19.0	15.8	14.8	16.6	16.2
The point of zero charge (pH_{PZC})	7.62	4.68	9.20	9.41	8.42	9.37	7.88	7.94	7.35	9.01

[a] By difference.

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