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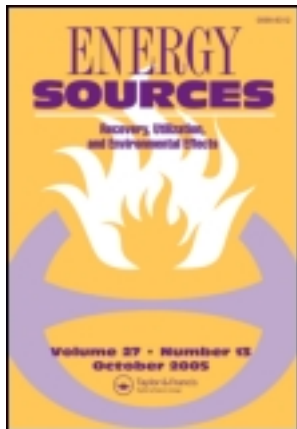
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Steam and KOH Activated Carbons from Peach Stones

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Abstract Peach-stone-based activated carbons were prepared by using steam and KOH activation at 800°C. The effects of solid/liquid phase KOH and steam activation, alkali and acidic pre-treatment, and also single-step activation on activated carbon properties were investigated comprehensively. The activated carbon produced by solid phase KOH impregnation showed the highest surface area of 835 m²/g and micropore volume of 0.412 cm³/g. Besides, single-step steam activation produced a highly microporous carbon having a relatively high surface area of 608 m²/g and micropore volume of 0.331 cm³/g. Peach-stone-based activated carbons with well-developed pore structure and functional properties were achieved, comparable to the commercial carbon, CPG-LF (Chemviron Carbon). The produced carbons were tested for Chromium (VI) removal from aqueous solution. Some of the activated carbons showed higher Chromium (VI) adsorption capacity than commercial activated carbon, CPG-LF (Chemviron Carbon).

Keywords activated carbon, chromium, KOH, peach stone, steam

1. Introduction

Sorption of organic and inorganic compounds on activated carbon depends on several factors, such as chemical and physical properties of carbon surface, speciation of adsorbate, and water chemistry. Different activation processes can be used to increase the performance of the activated carbon, as they can alter the structural and functional properties of the carbon surfaces (Mendez and Streat, 2002). In addition, the nature of the precursor affects activated carbon properties as well as carbonization and the activation process. Commercial activated carbons are mainly produced from wood, coal, and coconut shell. However, it is possible to produce activated carbon by using various agricultural by-products that have very low ash and sulphur contents. Recently, various fruit stones and shells have been used to develop lower cost activated carbons that have high adsorption capacity (Youssef et al., 2004). The important things are the availability and abundance of the precursor. Peach stone is one of the most abundant agricultural waste products from manufacturing peach juice in Turkey. The world production of peaches was 18,000,853 tons in 2008. Turkey is the sixth largest peach manufacturer in the world with the production of 551,906 tons (www.fao.org). Peach stones are generally used as

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an energy source, burned in the field, or spilled into the environment in a non-controlled way. It is possible to convert this waste product to a valuable material, activated carbon, using different activation methods.

The main objective of the current study is to prepare peach-stone-based activated carbons that are suitable for Cr(VI) removal from aqueous solutions. For that reason, steam and KOH activation were applied at different experimental conditions.

2. Experimental

All chemicals used in this work were analytical grade. Bituminous coal-based granular commercial activated carbon (Chemviron Carbon, CPG-LF) was used for the comparison.

2.1. Preparation of Peach Stone

Peach stones were obtained from a fruit juice factory in Bursa, Turkey. Peach stones were crushed and sieved to a particle size of 2–4 mm and then they were separated into two parts: the first part was used as received, the second part was pretreated in order to remove inorganic and humic substances from the main structure of the peach stone. The pretreatment process was applied as follows: (a) boiling with 1 M NaOH for 4 h; (b) washing with distilled water; (c) boiling with 0.1 M HCl solution for 1 h; (d) washing with distilled water to remove excess acid; and (e) drying in an oven at 105°C for 24 h.

2.2. Production Procedure

The carbonization and activation experiments were conducted in a horizontal quartz tube reactor with a diameter of 6 cm and a length of 55 cm. The reactor was heated by using a Protherm ASF tube furnace (Alserteknik, Turkey). The furnace had a 25-cm length of heating area. The reactor was continuously purged with nitrogen during the carbonization and activation periods. The nitrogen flow rate was kept constant at 500 mL/min by using a Dwyer MMA flow meter. Produced carbon samples were cooled to the ambient temperature in nitrogen atmosphere.

In order to carbonize, peach stones were placed into the quartz tube reactor and heated to 800°C at the rate of 5°C/min. Carbonization experiments were performed at 800°C for 2 h in nitrogen atmosphere. Carbonized materials were rinsed with hot distilled water until neutral pH was obtained. These samples were abbreviated as YKN and KN.

Steam activation was performed at 800°C with the heating rate of 5°C/min in nitrogen atmosphere. Steam/nitrogen flow was passed through the reactor for 2 h at 800°C. Approximately 50 mL/h of steam was consumed during each activation period. Steam activated carbons were rinsed with hot distilled water until neutral pH was obtained. These samples were abbreviated as YKSN, KSN, and KKSNN.

Potassium hydroxide activation was applied to the samples by using two different methods as liquid phase and solid phase impregnation. Liquid phase impregnation was performed as follows: (a) stirring with saturated KOH solution for 3 h; (b) filtration; and (c) dehydration at 120°C for 24 h. Raw material:KOH ratio was 1:2 and 1:3 by the weight. For the case of solid phase impregnation, carbonized peach stones were mixed with grounded KOH pellets in solid form with the ratio of 1:3 by the weight. Impregnated samples were placed into the quartz tube reactor and heated to 800°C at the rate of 5°C/min. Activation was performed at 800°C for 2 h in nitrogen at-

Table 1
Production conditions of carbon samples

Sample	Pre-treatment	One-step	Carbonization			Activation			
			Flow	T , °C	Time, h	Method	Flow	T , °C	Time, h
YKN	—	—	N ₂	800	2	—	—	—	—
YKSN	—	✓	—	—	—	Steam	Steam/N ₂	800	2
KN	✓	—	N ₂	800	2	—	—	—	—
KSN	✓	✓	—	—	—	Steam	Steam/N ₂	800	2
KKSN	✓	—	N ₂	800	2	Steam	Steam/N ₂	800	2
KOH2	—	✓	—	—	—	Liquid phase KOH impreg. Ratio: 1:2	N ₂	800	2
KOH3	—	✓	—	—	—	Liquid phase KOH impreg. Ratio: 1:3	N ₂	800	2
KOHS2	✓	—	Steam/N ₂	800	2	Liquid phase KOH impreg. Ratio: 1:2	N ₂	800	2
KOHS3	✓	—	Steam/N ₂	800	2	KOH impreg. Ratio: 1:3	N ₂	800	2
KOHKN3	✓	—	N ₂	800	2	Solid phase KOH impreg. Ratio: 1:3	N ₂	800	2
KOHKS3	—	—	N ₂	800	2	Liquid phase KOH impreg. Ratio: 1:3	Steam/N ₂	800	2

mosphere. These samples were abbreviated as KOH2, KOH3, KOHS2, KOHS3, and KOHKN3.

Steam and KOH activations were also combined. For that reason, carbonized peach stones were impregnated with KOH in the liquid phase as described above. The impregnated sample was placed into the quartz tube reactor and heated at the rate of 5°C/min in nitrogen atmosphere. After 200°C, the steam/nitrogen flow was passed through the reactor. Finally, temperature was kept at 800°C for 2 h in the steam/nitrogen flow. This sample was abbreviated as KOHKS3.

Activated carbon samples produced by KOH activation were rinsed with hot distilled water, and then treated with diluted HCl solution to remove excess alkali and again rinsed with hot distilled water until neutral pH. All the produced carbons were stored in a desiccator after drying at 105°C for 24 h. The abbreviations of the produced carbons and summarized production procedures are shown in Table 1.

2.3. Characterization

2.3.1. N₂ Adsorption-desorption Measurements. N₂ adsorption-desorption measurements were performed using a Quantachrome Autosorb-1-C surface analyzer. The specific surface area, total pore volume, and micropore volume were obtained from the nitrogen adsorption data at −196°C. Prior to analysis, carbon samples were degassed at 200°C for 24 h.

2.3.2. *Scanning Electron Microscope (SEM)*. SEM images of the samples were obtained by using a JEOL JSM-633SF model field emission scanning electron microscope.

2.3.3. *Surface Chemistry*. The relative concentrations of acidic surface functional groups in carbons were determined by Boehm's titration as described in previous work (Beker et al., 2010). The results of Boehm's titration were supported by Fourier transform infrared (FTIR) spectra. Analyses were performed with a Unicam Mattson 1100 model spectrometer using carbon samples dispersed in KBr pellets.

2.4. Chromium (VI) Adsorption

Chromium (VI) adsorption experiments were carried out by agitating 20 mg of activated carbon samples with 50 mL of Cr(VI) solution of 30 mg/L at pH 3 in an orbital shaker. Synthetic chromium solution was prepared by dissolving potassium dichromate in distilled water. Equilibrium Cr(VI) concentrations were determined by using Analytik Jena Specord 40 and JASCO V-530 UV (APHA, 2005). Cr(VI) adsorption capacities were calculated by using the following equation:

$$q_e = \frac{(C_0 - C_e) \cdot V}{m}, \quad (1)$$

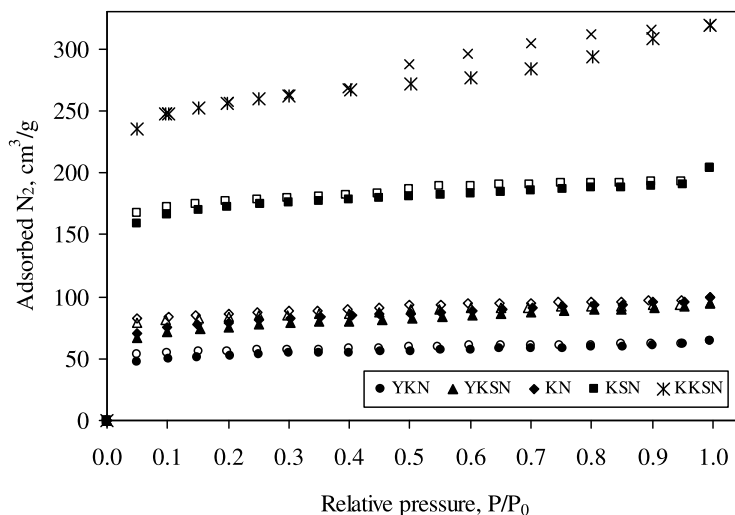
where q_e is adsorption capacity ($\text{mg}_{\text{Cr(VI)}}/\text{g}_{\text{activated carbon}}$), C_0 and C_e are the Cr(VI) initial and equilibrium concentrations (mg/L), respectively, V is the volume of the Cr(VI) solutions, and m is the weight of the activated carbon (g).

3. Results and Discussion

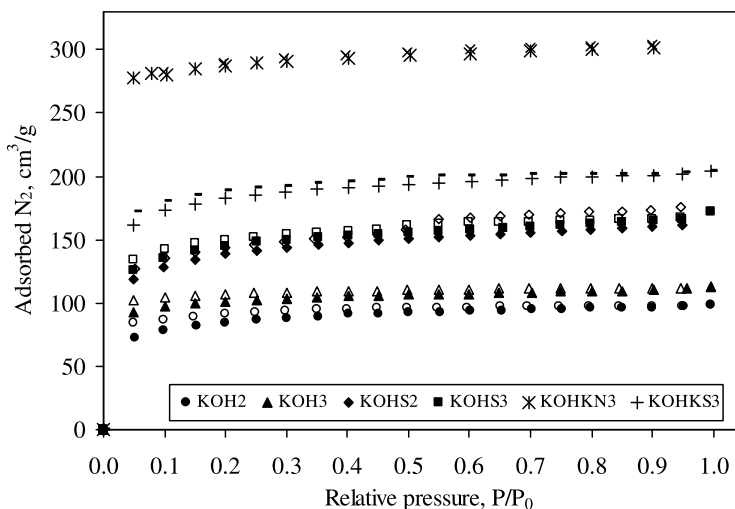
3.1. N_2 Adsorption-desorption Isotherms and Pore Structure

Nitrogen adsorption-desorption isotherms of the produced carbons are shown in Figure 1. According to the IUPAC classification (Sing et al., 1985), all the produced carbons showed Type I nitrogen adsorption isotherms. This demonstrates highly microporous structure of the carbons. Similar nitrogen adsorption isotherms were obtained for KN and YKSN samples. This indicates that both pretreatment and steam application increased the nitrogen adsorption in equal amounts. As can be seen from Figure 1a, adsorbed nitrogen volume was significantly increased since both pretreatment and steam was applied (KSN and KKSNN). However, the hysteresis of the adsorption/desorption isotherms of KKSNN sample indicates the existence of mesopores. Most isotherms, especially KOH-activated samples, showed low pressure hysteresis. This can be associated with narrow slit-like pores (Sing et al., 1985). It can be seen from Figure 1b that KOH activation was also effective to increase nitrogen adsorption. Maximum nitrogen adsorption capacity was obtained with the KOHKN3 sample, which was produced by solid phase KOH activation.

Table 2 shows the effects of activation methods on surface area and pore volumes of the produced carbons. Similar surface area and pore volume properties were observed for KN and YKSN. The surface area of carbonized carbon sample was increased from 163 to 247 m^2/g with pretreatment; this value was increased to 237 m^2/g in the case of steam application. The pretreatment method was effective as much as the steam activation. This can be interpreted that NaOH and HCl react with lignin and inorganic matters in



(a)



(b)

Figure 1. N₂ adsorption-desorption isotherms: (a) carbonized samples and steam activated carbons and (b) KOH activated carbons (full items: adsorption data; empty items: desorption data).

the main structure of peach stone causing pore development during the pretreatment period. As can be seen from Table 2, applying pretreatment and steam together (KSN and KKSNN samples) resulted in a significant rise in the surface area and pore volume, as expected. Similar surface area and pore volume values were declared in the articles related with peach stone-based activated carbons that were produced by steam activation in two steps (Arriagada and García, 1997; Galiatsatou et al., 2002; Yalcin and Arol, 2002).

Table 2
Surface area and pore volume of the
produced carbon samples

Sample	Surface area, ^a m ² /g	Total pore volume, ^b cm ³ /g	Micropore volume, ^b cm ³ /g
YKN	163	0.087	0.079
KN	247	0.136	0.120
YKSN	237	0.131	0.115
KSN	608	0.341	0.331
KKSN	755	0.445	0.348
KOH2	266	0.135	0.124
KOH3	312	0.155	0.151
KOHS2	437	0.295	0.189
KOHS3	456	0.235	0.206
KOHKN3	835	0.458	0.412
KOHKS3	569	0.281	0.258
CPG-LF	693	0.351	0.318

^aBET method.

^bDFT method.

The single-step steam activated KSN sample has more microporous structure, while the KKSN sample, which was produced in two steps, has higher mesoporosity. Further steam activation of carbonized peach stone can be caused by pore widening by reacting with pore walls, consequently, a serious increase in mesopore volume. Arriagada et al. (1997) developed an activated carbon with a high surface area depending on activation period; they observed an increase in mesoporosity with an activation period. Roman et al. (2008) also indicated that development of microporosity reaches a maximum at about 50% burn-off level, then micropores become mesopores.

As can be seen from Table 2, although steam and KOH activation was effective in increasing the surface area and pore volume individually, their combination did not improve the pore volume properties. The possible reason of this result could be that the blockage of the carbon surface by KOH particles can prevent influencing of steam molecules to the carbon surface during the activation. Moreover, some side reactions may occur that cannot be detected when KOH and steam activation applied at the same time. These reactions may influence the effect of KOH and steam activation in reverse direction, consequently, preventing pore development. It can also be said that the pore volume of the carbons are slightly increased with the KOH impregnation ratio from Table 2. This increase was not significant. The possible reason of this could be losing an amount of KOH by filtration at the impregnation step.

The highest surface area and micropore volume obtained among the samples prepared by liquid phase impregnation is 569 m²/g and 0.258 cm³/g, respectively. The KOHKN3 sample has 835 m²/g of surface area and 0.412 cm³/g of micropore volume. This result shows that the solid phase KOH impregnation is more effective than the liquid phase KOH impregnation. Bagheri and Abedi (2009) indicated similar results with corn cob-based activated carbons. It should be kept in mind that a serious amount of KOH was

lost by the filtration step in liquid phase impregnation; hence, the amounts of KOH in the furnace were not equal for the two different impregnation procedures. Consequently, activation degree of the carbons produced by liquid phase impregnation was low. Some authors informed that the liquid phase KOH impregnation of some biomass material without any filtration step was superior to the mixing with solid KOH (Ahmadpour and Do, 1997; Bagheri and Abedi, 2009). However, solid mixing is a very simple method and does not require any evaporation process, which consequently saves time.

The values belonging to the commercial activated carbon, CPG-LF, was also given in Table 2. The surface area and the pore volume of the samples, KOHKN3 and KKSN, which were prepared by different activation methods, are higher than the commercial carbon. Among all of the produced carbons, KOHKN3 has the highest surface area and pore volume. Following this, KKSN sample has the second highest surface area ($755 \text{ m}^2/\text{g}$), total pore volume ($0.445 \text{ cm}^3/\text{g}$), and micropore volume ($0.348 \text{ cm}^3/\text{g}$). On the other hand, the surface area and pore volume of the KSN sample are also relatively high, even its microporosity (97%) is higher than the others.

3.2. SEM Images

According to the SEM images, all the produced carbons have highly porous structure (Figure 2). Pretreatment and steam activation developed the pore structure of the carbons. Figure 2 also shows that the pore formation of KOH2 and KOH3 was not completed. The white spheres in the images of both samples may be attributed to the presence of potassium salt residues in the matrix. A successive physical activation can be applied to improve the pore structure of these samples. Irregular and rather wide pore structure were observed with KOHS2 and KOHS3 samples. This may be explained as the growing and breaking of micropores by further chemical activation. Also, expected pore development was not observed when steam and KOH were applied together (KOHKS3).

3.3. Surface Chemistry

The changes in surface functionalities, which were detected by Boehm's titration measurement, are summarized in Table 3. According to the obtained results, all carbon samples possess acidic oxygen functionalities in the form of phenolic groups and a small amount of lactonic groups in some. This result may be attributed to the gas phase oxidation. Shen et al. (2008) informed that the gas phase oxidation causes an increase in hydroxyl and carbonyl surface functional groups. While pretreatment and steam activation increased total acidic groups, they were not as effective in the case of basic groups (Table 3). Compared with the others, higher amounts of basic groups were observed in KOH2 and KOH3 samples (0.550 and 0.569 meq/g, respectively). This may be interpreted as the presence of residual alkali, which was not neutralized at the washing step. In summary, almost all carbon samples have a slightly acidic character except KSN, which has equal amounts of acidic and basic functional groups (0.316 and 0.319 meq/g, respectively).

The FTIR spectra of the produced carbon samples are shown in Figure 3. In general, almost all carbon samples have very similar FTIR spectra. The band in the region of $3,400 \text{ cm}^{-1}$ is assigned to the O-H stretching vibrations of the functional groups, such as phenols and alcohols (Gomez-Serrano et al., 1999; Chunlan et al., 2005; Burg and Cagniant, 2008; Shen et al., 2008). The broad peak displayed shoulders at about

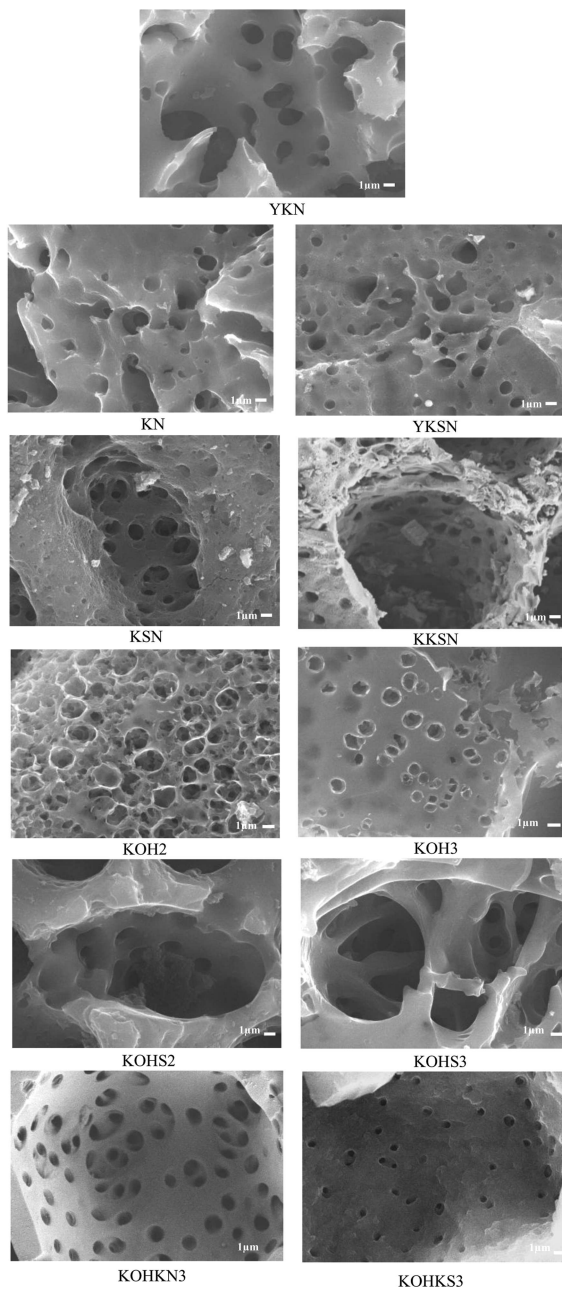


Figure 2. SEM images of carbon samples.

Table 3
Boehm's titration results

Sample	Carboxylic groups, meq/g	Lactonic groups, meq/g	Phenolic groups, meq/g	Total acidic groups, meq/g	Total basic groups, meq/g
YKN	—	—	0.218	0.218	0.327
KN	—	—	0.463	0.463	0.121
YKSN	—	—	0.539	0.539	0.296
KSN	—	0.062	0.254	0.316	0.319
KKSN	0.044	0.103	0.631	0.777	0.360
KOH2	—	0.006	0.249	0.255	0.550
KOH3	—	0.008	0.218	0.226	0.569
KOHS2	—	—	0.397	0.397	0.339
KOHS3	—	—	0.526	0.526	0.302
KOHKN3	—	0.098	0.971	1.069	0.442
KOHKS3	—	—	0.658	0.658	0.163
CPG-LF	0.157	—	0.519	0.677	0.185

—: Cannot be detected.

2,900 cm^{-1} represents C–H stretching of methyl and methylene groups (Gomez-Serrano et al., 1999) or alkyl groups, such as $-\text{CH}_3$, $=\text{CH}_2$, and $-\text{CH}_2\text{CH}_3$ (Guo and Lua, 2000). According to Arriagada et al., the band between 1,610–1,630 cm^{-1} represents aromatic structure and C=O in quinonic configuration (Arriagada et al., 1997). The C=O stretching vibration at 1,700 cm^{-1} can be discriminated in a few samples. In some cases, a very broad band was observed between 1,600–1,700 cm^{-1} that can be explained as overlapping of C=C and C=O stretching peaks. In addition, many functional groups can show an absorption peak in this region like quinonic (1,550–1,680 cm^{-1}), carboxylic (1,665–1,760 cm^{-1}), and lactones (1,675–1,790 cm^{-1}) (Figueiredo et al., 1999; Gomez-Serrano et al., 1999; Burg and Cagniant, 2008). As known from Boehm titration results, the produced carbons do not include carboxyl groups, quinonic groups and lactones can be responsible for the band at this region. While the bands in the region of 1,350–1,400 cm^{-1} can be assigned to carbonates, carboxyl carbonates, and lactones, the small peaks between 1,100–1,200 cm^{-1} can represent alcohols, C–H in ether, C–OH and O–H stretching in phenolic groups (Figueiredo et al., 1999; Gomez-Serrano et al., 1999; Burg and Cagniant, 2008).

3.4. Chromium (VI) Adsorption Capacities

Considerably high Cr(VI) adsorption capacities (18–78 mg/g) were obtained with the produced carbon samples (Figure 4). Cr(VI) sorption capacities of KKSN, KOHKS3, and KSN samples are higher than commercial carbon, CPG-LF. It is expected to obtain higher capacity with KOHKN3, which has the highest surface area and micropore volume, but its capacity is lower. This could be explained with the acidic character of KOHKN3's surface. An acidic surface is not attractive for negatively charged hydrogen chromate molecules.

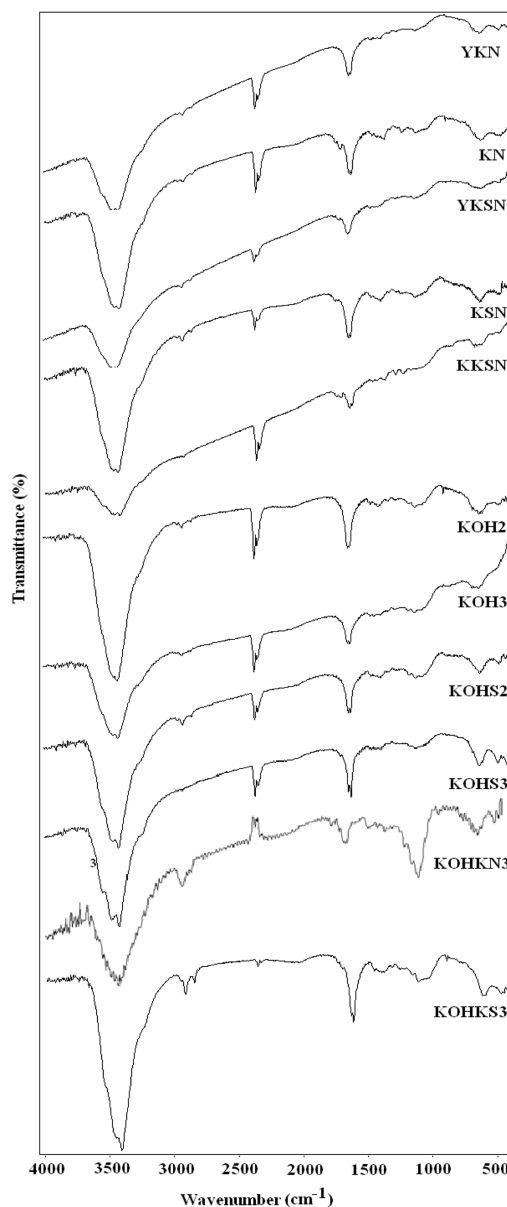


Figure 3. FTIR spectra of the produced carbon samples.

4. Conclusions

Peach stone has been converted to highly microporous activated carbons by using steam and KOH activation. Activated carbon produced from the peach stone promises economical benefits since it is cheap and an adequate candidate for water treatment; and by its preparation, a waste material has been utilized. The produced carbons can be evaluated as a potential adsorbent for removing Cr(VI) from aqueous solutions with high adsorption capacity.

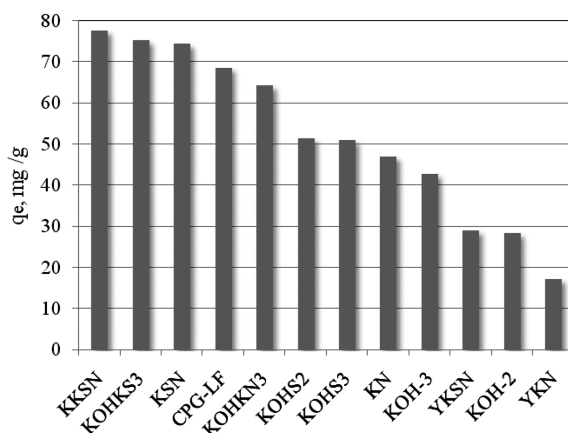


Figure 4. Cr(VI) adsorption capacities of carbon samples.

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