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Production and characterization of activated carbon from a bituminous coal by chemical activation

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In this study, the use of a bituminous coal for the production of activated carbons with chemical activation was investigated. The effects of process variables such as chemical reagents, activation temperature, impregnation ratio and carbonization temperature were investigated to optimize these parameters. The resultant activated carbons were characterized in terms of iodine number, BET surface area, and total and micropore volumes. Acidic surface groups were established by Fourier Transmittance Infrared Spectroscopy (FTIR) technique.

Key words: Activated carbons, activation, BET surface area, Fourier Transmittance Infrared Spectroscopy (FTIR).

INTRODUCTION

Activated carbon can be defined as carbonaceous material having high porosity and internal surface area and cannot be characterized by any distinctive chemical formula. Commercial production of activated carbon is performed by pyrolysis and activation of wood, petroleum coke, anthracite and bituminous coals which have high carbon contents. In addition, agricultural wastes and by-products, hulls of almonds, hazelnuts, and coconuts, and stones of olives, apricots and peaches are also used in activated carbon production (Arol and Yalcin, 1993; Bansal et al., 1988; Qttowa et al., 1997; Stavropulos and Zabaniotu, 2005; Amaya et al., 2007; Solano et al., 2000; Robau-Sanchez et al., 2005; Evans et al., 1999; Zondlo and Velez, 2007; Dongel et al., 1998).

The problems of the ecosystem are increasing with developing technology. Heavy metal pollution is one of the main problems due to their toxicity to human life

(Badmus et al., 2007). Organic pollutants and phenol and its derivatives found in industrial waste waters and as a main components in plastics are considered to have toxic effects on human health even when present in small concentrations (Yener and Aksu, 1999; Qadeer and Akhtar, 2005; Qadeer and Rehan, 2002). Toxic metal compounds coming to the earth's surface not only reach the earth's waters (seas, ponds, lakes, and reservoirs), but can also contaminate underground water in trace amounts by leaking from the soil after rain and snow (Qadeer and Rehan, 2002; Uzun and Guzel, 2000). Therefore, the elimination of toxic metal compounds from water and waste water is important. Activated carbons, with their high porosity, are extensively used in industrial purification and chemical recovery operations (Teker and Imamoglu, 1999; Lillo-Rodenas et al., 2005; Lozano-Castello et al., 2001). Currently, natural gas storage has been attracting interest among researchers in the materials and adsorption field. Microporous activated carbons were found to be suitable materials for natural gas storage (Azevedo et al., 2007). High surface area, high surface reactivity, suitable pore distribution and mechanical strength are the desired properties for an activated carbon. Depending on the usage, activated carbons are available as powder, granulated and pressed (laminated) sticks in various sizes and shapes (Arol and Yalcın, 1993).

Basically, there are two different processes for the preparation of activated carbon, the so-called physical

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Abbreviations: BET, Brunauer, Emmett and Teller (1938) method to measure specific surface area of solids according to physical adsorption of gas molecules on a solid surface, m^2/g; **V_{TOT}**, total pore volume, cm^3/g; **V_{MIC}**, micropore volume, cm^3/g; **V_{MES}**, mesopore volume, cm^3/g; **TGA**, thermal gravimetric analysis; **TG**, thermal gravimetry; **DTG**, derivative thermal gravimetry; and **FTIR**, Fourier Transform Infrared Spectrum Analysis.

Table 1. Proximate analysis of sample (dry basis).

Ash (%)	Volatile matter (%)	Fixed carbon (%)	Total S (%)
10.10	35.60	54.30	0.71

and chemical activation. In physical activation, the first step is carbonization of the raw material and this is performed in a temperature less than 700°C in an oxygen-free environment. Carbonization means obtaining charcoal from the raw material and the produced charcoal, having a low surface area, is not an active product. The second step is to convert the carbonized product to porous activated carbon having high surface area by activating it in high temperatures (800 - 1000°C) in an oxidizing environment. At this step, pores and vessels form during the entrance of oxidizing gases into the carbonized product and removal of reaction products through particles. Substances used in physical activation are steam, CO_2 or air. Chlorine, sulphur vapors, SO_2 and ammonia are also used in this process (Lillo-Rodenas et al., 2005; Lozano-Castello et al., 2001). The chemical activation process is done by uncarbonized materials. Firstly, raw material is treated to destroy the structure by mixing with oxidizing chemicals like $ZnCl_2$, H_3PO_4, KOH, NaOH etc. in low temperatures. Then, this activated product is carbonized in an airless environment at 400 - 1000°C and thus pyrolitic disintegration is reached. In this process, substances charge to the surface to restore the carbon structure and pore structure develops as surface area increases (Lozano-Castello et al., 2001). Chemical activation is a well-known method preparing activated carbons which has been object of great study in the last years as it presents several advantages compared to physical activation. An important advantage of chemical activation is that the process allows us to obtain very high surface area activated carbons (Lillo-Rodenas et al., 2003; Lillo-Rodenas et al., 2004).

In this work, laboratory studies to produce active carbon from bituminous coal by chemical activation are presented.

MATERIALS AND METHODS

Materials

In this study, the sample was taken from Turkish Hardcoal Enterprises Amasra Colliery. The experimental sample was the classified size group -0.840 + 0.250 mm material. Result of proximate analysis of sample compatible to ASTM D 3176 is given in Table 1.

Methods

Chemical activation process was applied in this work. Active carbon production was performed in two steps, namely activation followed by carbonization. Activation studies were carried out at low temperature and high temperature conditions and named as first group and second group experiments, respectively.

First group experiments

In this group of experiments, the sample was activated by mixing and heating it with chemical reagents at low temperatures. $ZnCl_2$, Na_2CO_3, H_3PO_4, NaOH and KOH were used as chemical reagents. Impregnation ratio between chemical reagents and precursor was chosen as 4/1, 2/1, 1/1 and 0.75/1. The sample was stirred continuously in each chemical reagent of 50% solution at 80°C for 30 min. The activated sample was filtrated by vacuum and dried at 100°C for 24 h. After the activation process, the carbonization process was applied to the product obtained. During the carbonization process, N_2 was introduced to the system by a flow rate of 1000 ml/min. The carbonization process was conducted in a programmable tube furnace. The furnace was programmed in such a way that heating rate shall be 5°C/min and after reaching the final temperature of 800°C it will then remain at that temperature for 2 h. At the end of this period the furnace was left to cool itself. The carbonized product was stirred with 0.5 N HCl at 85°C on a heating magnetic stirrer for 30 min and then it was filtered. Then it was rinsed with distilled water several times until the pH of the activated carbon-distillated water mixture has exceeded 6. Finally, the obtained product was dried at 100°C for 24 h and characteristic properties of obtained activated carbon like iodine number (mg/g), BET surface area (m^2/g) and pore distribution (micropore, mesopore)(cm^3/g) were determined.

Second group experiments

In this group of experiments, the sample was activated by boiling it with chemical reagents of NaOH and KOH. The reagents, $ZnCl_2$, Na_2CO_3 and H_3PO_4, resulted in activated carbon having low iodine number in the first group experiments were eliminated. The sample was put into a volumetric flask with 50% chemical reactive solution so that impregnation ratio between chemical reagents and precursor is 1/1. A condenser was mounted to the flask to prevent evaporation. The mixture was boiled at 120°C for 10 h in an oil bath. At the end of this period, the mixture was cooled, filtrated by vacuum and then dried at 100°C for 24 h. The carbonization process was applied to the sample after the activation process. The effect of temperature on carbonization was investigated by selecting furnace final temperature as 600, 700, 800, 900 and 1000°C. The furnace was programmed in such a way that heating rate shall be 5°C/min and after reaching the final temperature it will then remain at that temperature for 2 h. At the end of this period the furnace was left to cool itself. N_2, having a flow rate of 1000 ml/min, was introduced to the system during the carbonization process. Then, the carbonized product was mixed with 250 ml 0.5 N HCl and the mixture was heated and stirred at 85°C for 30 min in a heating magnetic stirrer and it was filtrated by vacuum. Then the obtained activated carbon was rinsed with distilled water several times until the pH of the activated carbon-distillated water mixture has exceeded 6. The obtained product was dried at 100°C for 24 h. Finally, characteristic properties of obtained activated carbon like iodine number (mg/g), BET surface area (m^2/g) and pore distribution (micropore, mesopore)(cm^3/g) were determined.

Considering better properties of the products in this group of experiments, thermal analysis (TG and DTG) and Fourier Transform Infrared Spectrum (FTIR) analysis were also performed in the characterization.

The iodine number was determined as compatible to ASTM D
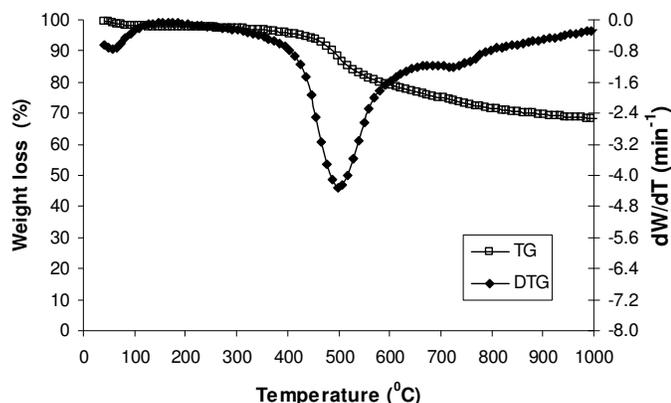
Figure 1. TG and DTG curves of coal sample used in experiments.

4607-94 standard. Thermogravimetric analysis of the sample and products was performed by using a PL 1500 TGA in an inert environment under flowing N_2 with a flow rate of 10 ml/l. For the porosity characterization of the samples, physical adsorption of N_2 at 77 °K was carried out in an automatic system (Quantachrome Autosorb 1). Micropore volumes (V_{MIC}) were calculated from adsorption isotherms, applying the Dubinin-Radushkevich (DR) equation. Total pore volume (V_{TOT}) was determined at 0.95 p/p₀ and mesopore volume (V_{MES}) was calculated as the difference between V_{TOT} and V_{MIC}. A Perkin Elmer FTIR Spectrometer was employed to determine the functional groups of the active carbons. All the spectra were taken with a 1 cm^{-1} spectral resolution, and were obtained at 298 °K. Samples were diluted with KBr. A KBr pellet was prepared by mixing 1 mg of finely dried ground coal sample with 50 mg KBr. The pellet 1 cm diameter was pressed under 8 tons of pressure for 2 min.

RESULTS AND DISCUSSION

TG and DTG curves of the coal sample taken from Amasra and having -0.840 + 0.250 mm size are given in Figure 1. Regarding TG and DTG curves in Figure 1, the sample has displayed typical coal behavior. It has lost water content up to 100 °C and exhibits no mass loss until 400 °C. After 450 °C mass loss rate has increased depending on the removal of volatiles of sample and at 900 °C total mass loss has reached 30% and reached to a steady level about 1000 °C.

Characteristic properties of the activated carbons obtained by the first group of experiments performed at 80 °C activation temperature are given in Table 2. Since iodine numbers of activated carbons obtained by using Na_2CO_3, and H_3PO_4 were very low even to consider them as activated carbon. Therefore, no further properties of these materials were determined. Although the characteristic properties of activated carbon obtained by $ZnCl_2$ activation were far better than Na_2CO_3 and H_3PO_4 considering with NaOH and KOH, it was also regarded low and no further test was implemented.

In the first group of experiments, BET surface areas of activated carbon obtained by using NaOH are higher than those obtained by using KOH at the same impregnation

rates. BET surface area and total pore volume obtained by using 2/1 impregnation ratio with NaOH, cannot be reached by use KOH ever at 4/1 impregnation ratio. Fierro et al. (2007) stated that BET surface areas of activated carbon produced from kraft lignin by chemical activation using KOH is higher than that using NaOH. However, they also claimed that among BET surface areas of activated carbons produced from anthracite by using the same chemicals under the same conditions, BET surface area of the activated carbon produced by NaOH is higher than that produced by KOH. The reason is that since Na^+ is smaller than K^+, it can both penetrate more deeply into the carbon structure and develop a higher number of smaller pores than K^+ can do (Lillo-Rodenas et al., 2001).

According to the test results given in Table 2, both BET surface areas and micropore volumes change depending on the impregnation ratio. Impregnation ratios corresponding to the maximum iodine number, BET surface area, total and micropore volumes were found as 4 for both KOH and NaOH. When the impregnation ratio was increased from 2 to 4, then BET surface area and micropore volume increase. BET surface areas of 869.9 m^2/g by use KOH has increased to 1295 m^2/g when impregnation ratio increased to from 2/1 to 4/1 by use of same reagent. These values are 1403 m^2/g and 1566 m^2/g by use NaOH at 2/1 and 4/1 impregnation, respectively.
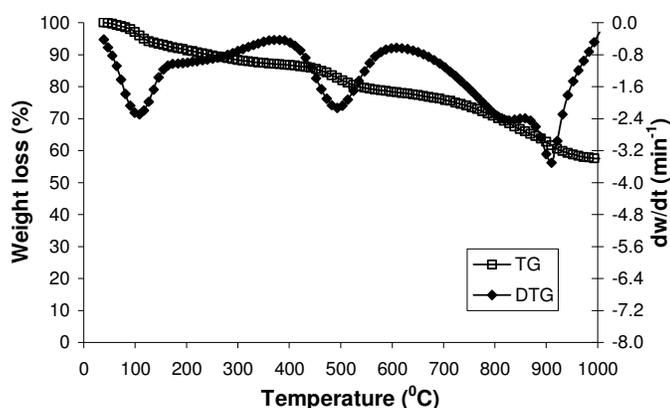
In a similar manner, Robau-Sanchez et al. (2005) stated that in the production of activated carbon by using KOH, when the impregnation ratio is increased from 2 to 4, the BET surface area also increases. According to Fierro et al. (2007), mesopore volume remains low and increases very slightly with the impregnation ratio (R), above R = 3, the micropore volume decreases; however, no additional mesopores are created. In the study performed by Otowa et al. (1997), KOH/coke weight ratio was varied by 1 to 10. The surface area and pore volume increased proportionally to the KOH/coke weight ratio up to about 4. A surface area greater than 3000 m^{2}g^{-1} was obtained at a KOH/coke ratio of about 4.

Particle sizes of the obtained activated carbons have changed depending on the impregnation ratio. Particles appear to be micronized at an impregnation ratio of 4/1. Therefore, the impregnation ratio can be used as an adjustable parameter to control the characteristic and physical parameters of activated carbons from coal.

Lillo-Rodenas et al. (2001) has activated Spanish anthracite by hydroxides. He has applied the impregnation process (changing between 1/1 and 4/1) and physical mixing in which samples are directly mixed at room temperature with a given amount of NaOH in the activation process. After both the impregnation process and physical mixing, the samples were carbonized in a furnace under flowing nitrogen. The nitrogen flow rate was varied 80 ml/min to 1000 ml/min. When the impregnation ratio was increased over 3, BET surface areas of activated carbons have changed between 1000 and 2700

Table 2. Characteristic properties of activated carbons obtained by first group of experiments.

Reagent	Temp. (°C)	Iodine Num. (mg/g)	Surf. Area (m²/g)	V_{TOT} (cm³/g)	V_{MIC} (cm³/g)	V_{MES} (cm³/g)	V_{MIC}/V_{TOT}
KOH	4/1	1107	1295	0.5616	0.5153	0.0463	91.76
KOH	2/1	850	869.9	0.3873	0.3609	0.0264	93.19
KOH	1/1	750	785	0.3596	0.2948	0.0646	81.26
NaOH	4/1	1163	1566	0.719	0.5911	0.1275	82.21
NaOH	2/1	1045	1403	0.6943	0.5816	0.1127	83.77
NaOH	1/1	657	752	0.4001	0.3119	0.0882	77.96
Na_2CO_3	4/1	96	--	--	--	--	--
Na_2CO_3	2/1	88	--	--	--	--	--
Na_2CO_3	1/1	70	--	--	--	--	--
$ZnCl_2$	4/1	560	519	0.24	0.1847	0.055	76.96
$ZnCl_2$	2/1	540	512	0.20	0.1385	0.615	69.25
$ZnCl_2$	1/1	437	446	0.1716	0.0941	0.775	54.84
H_3PO_4	4/1	95	--	--	--	--	--
H_3PO_4	2/1	70	--	--	--	--	--
H_3PO_4	1/1	52	--	--	--	--	--

Figure 2. TG and DTG curves of activated carbon obtained by KOH activation.
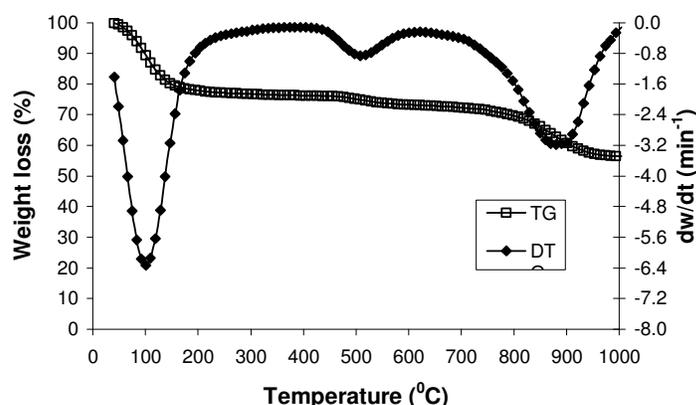
Figure 3. TG and DTG curves of activated carbon obtained by NaOH activation.

m²/g depending on the amount of nitrogen flow. BET surface areas and micropore volumes have increased by the increased amount of nitrogen. Researchers were planning to carry out further test in varying nitrogen flow rates to determine its effect on the process.

TGA analysis was performed for activated carbons obtained by activation using KOH and NaOH. TG and DTG curves obtained from these analyses are given in Figures 2 and 3 respectively.

A weight loss of about 7% occurs up to 150°C for the sample activated by KOH. It is known that this weight loss is because of water formed from dehydration of KOH. 14% of weight loss, which is rather slow, has occurred between 150 - 430°C. There was a dw/dt (rate of weight loss) peak in temperatures in between 440 and 540°C. Rate of weight loss has decreased between 540 - 600°C, increased between 600 - 800°C and then in-

creased rapidly at temperatures above 800°C. The reason for the appearance of weight loss in the KOH activated sample, though it has not been seen for the original sample to the temperature of 400°C, is again because of dehydration of KOH and loss of H. This occurs by the displacement of –H groups bounded to coal structure with –OK groups. Total weight loss at 900°C was 37%, and temperatures above 900°C the rate of mass loss gradually decreased and reached to a steady level at about 1000°C.

20% of rapid weight loss is encountered of up to 150°C for the sample activated by NaOH. The reason for this is water formed by hydrolysis of NaOH. No weight loss was recorded between 150 - 480°C; it has slowly increased between 480 - 600°C and increased rapidly at temperatures above 600°C. It can be said that weight loss, seen in the original sample between 400 -

Table 3. Characteristic properties of activated carbons obtained by second group of experiments.

Reagent	Temp. (°C)	Iodine Num. (mg/g)	Surf. Area (m^2/g)	V_{TOT} (cm^3/g)	V_{MIC} (cm^3/g)	V_{MES} (cm^3/g)	V_{MIC}/V_{TOT}
KOH	1000	920	1385	0.65	0.5914	0.0586	90.98
	900	1070	1596	0.71	0.6579	0.052	92.66
	800	730	1015	0.47	0.4006	0.0694	85.23
	700	300	-	-	-	-	-
	600	250	-	-	-	-	-
NaOH	1000	1010	1461	0,7505	0.6818	0.0687	90.85
	900	810	1210	0.68	0.6101	0.0699	89.72
	800	768	945	0.53	0.4724	0.0576	89.13
	700	640	692	0.44	0.3887	0.0513	88.34
	600	590	616	0.41	0.2916	0.1184	71.12

600°C, has not been seen in the NaOH-activated sample, which resulted from bounding of tar products to the sample structure. Total weight loss is 43% and this amount is slightly high with respect to that of KOH activated samples. It can be concluded that KOH depresses the volatile matter of coal slightly better than NaOH or it bounds coal to its structure.

The second group of tests were planned to determine the effects of activation conditions and carbonization temperature by fixing an impregnation ratio of 1/1 to produce granulated activated carbon having a high BET surface area and microporosity.

The effect of carbonization temperature was investigated in the second group of tests. Iodine numbers, BET surface areas, total and micropore volumes of produced activated carbons depending on the carbonization temperature were determined. Characteristic properties of activated carbons produced are given in Table 3.

As seen in Table 3, BET surface areas obtained at 800°C are 1015 m^2/g and 945 m^2/g by using KOH and NaOH, respectively. However, in the first group of experiments, these levels of surface area cannot be produced at the same impregnation ratio as shown at Table 2. This improvement in surface areas, considering fixed carbonization conditions, may well be because of increased activation temperatures and periods. Iodine numbers and BET surface areas produced for KOH-activated carbons have increased with increasing temperatures, had a peak at 900°C and decreased above 900°C. However, no such behaviors were observed for NaOH-activated carbons; instead, a similar relationship was observed among carbonization temperature, iodine number and BET surface area. These parameters have increased with increasing temperatures.

FTIR spectrum analysis was used to investigate variations in the functional groups of the coal sample used in experiments and obtained activated carbon samples. FTIR technique is a useful analytical method for the identification of constituents of active coal samples. The increase and decreases in the spectrum peaks shows

how the experimental conditions affect the structure of material. The spectra of coal samples could be analyzed in terms of the fixed mix of functional groups. OH and NH stretching in between 3100 - 3500 cm^{-1}, C-H aromatic in between 3000 - 3100 cm^{-1}, C-H aliphatic in between 2800 - 3000 cm^{-1}, C=O and C-O stretching in between 1640 - 1750 cm^{-1} attributed to phenolic ester, carboxylic acid and conjugated ketonic structures. The region in between 700 - 900 cm^{-1} contains various bands related to aromatic, out of plane C-H bending with different degrees of substitution (Mastalerz and Bustin, 1995). FTIR spectrum of coal sample used in the experiments are illustrated in Figure 4 and spectrums of activated carbons produced by KOH and NaOH activation at various carbonization temperatures are given in Figure 5 and 6 respectively.

The characteristically broad coal absorptions which were observed in the coal samples used in the experiments are clearly absent in activated carbon samples over 700°C, which is indicative of the complete carbonization of the coal samples. These FTIR spectra (Spectra at 1000, 900 and 800°C in Figure 5 and Spectra at 1000, 900 and 800°C in Figure 6) reflect the differences between two activation reagents. The bands at 2800 - 2980 cm^{-1} (C-H aliphatic stretching) and 1375 - 1465 cm^{-1} (C-H aliphatic bending) are more intense in KOH activated coals. The spectra of oxygen-rich KOH activated samples at 1000°C show two peaks at 1655 and 1703 cm^{-1} that were attributed to phenolic esters, carboxylic acid and conjugated ketonic structures (Supaluknari et al., 1998). When the carbonization temperatures were increased over 800°C the intensity of the 3394 cm^{-1} has three obvious periods. The intensity of this band was (O-H stretching) increased as the temperature increased. When FTIR spectrum at 1000°C in Figure 5 and 6 are compared, which were activated in KOH and in NaOH, respectively, it shows that the degree of carbonization is higher for the sample activated by NaOH as it can be appreciated by the lower intensity of the O-H band at 3422 cm^{-1}. The main differences between both types of carbonization product are observed on the characteristic
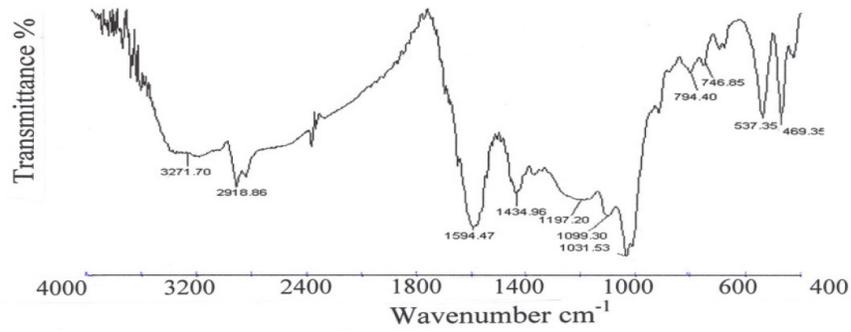
Figure 4. FTIR spectra of the coal sample used in the experiments.
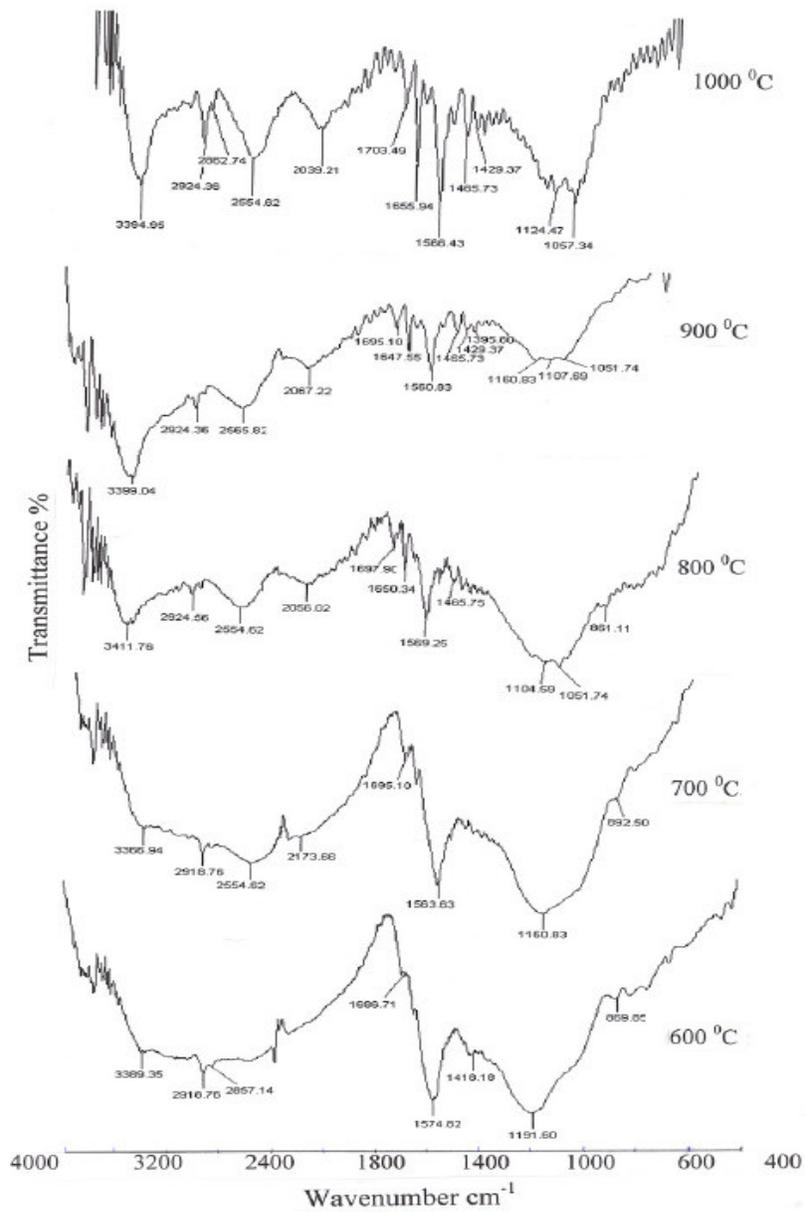
Figure 5. FTIR spectra of the activated carbon obtained by activation with KOH at different carbonization temperatures with impregnation ratio (KOH/precursor, g/g) of 1/1.
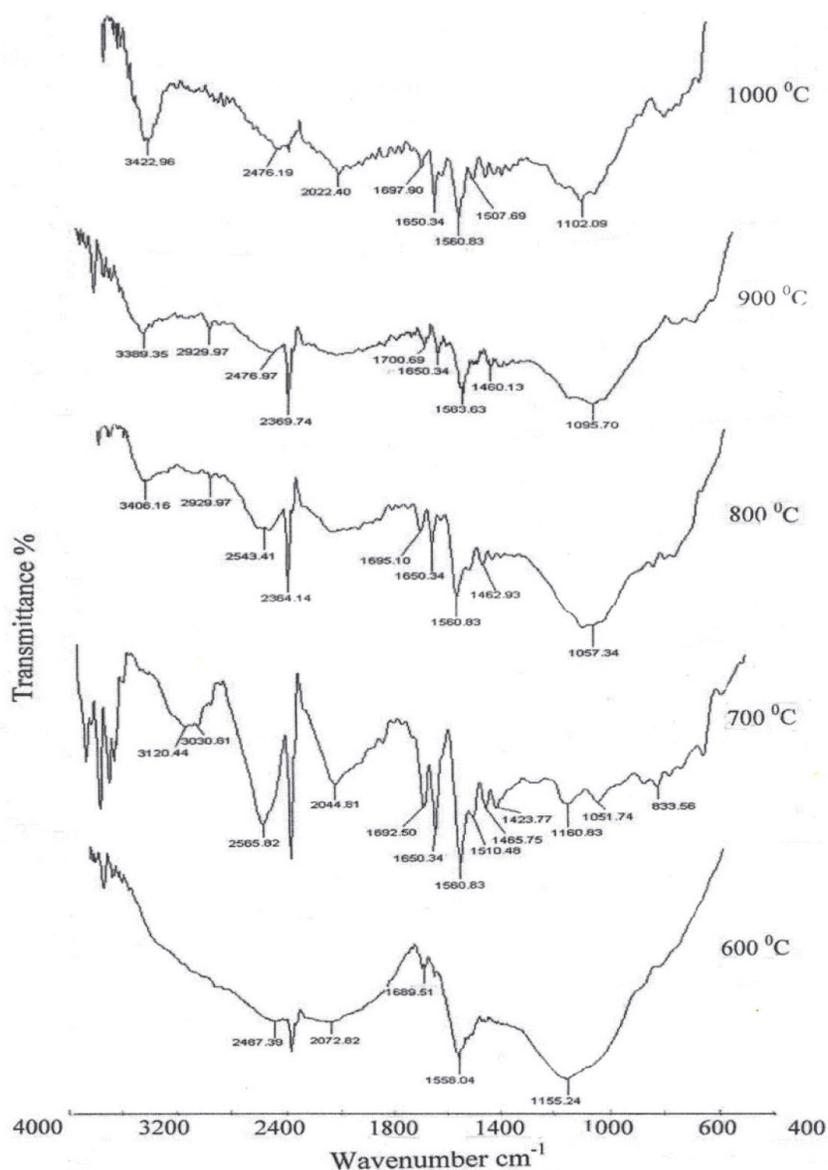
Figure 6. FTIR spectra of the activated carbon obtained by activation with NaOH at different carbonization temperatures with impregnation ratio (NaOH/precursor, g/g) of 1/1.

band of C-H aromatic stretching. It can be observed that KOH activated coal samples have much higher content to aromatic C-H than NaOH activated coal. On the other hand, the analysis of the C-H aliphatic stretching and bending region for the activated coal samples, show that aliphatic C-H mainly as $-CH_2-$ and this indicate that most of the aliphatic structures in the activated coal samples exist as cyclic structures.

Conclusions

The use of bituminous coal as a precursor for the preparation of activated carbons using KOH, NaOH and other chemical reagents was analyzed in detail. The influence of several variables such as impregnation ratio and carbonization temperature was studied.

In the first group of experiments, in which the impregnation ratio between the chemical reagent and the precursor were 4/4, 2/1, 1/1, it was seen that BET surface areas change between 1295 and 785 m^2/g for KOH activated active carbons and 1566 and 657 m^2/g for NaOH activated ones. BET surface areas and total pore volumes of KOH activated active carbons are higher than that of NaOH activated ones. Increasing the amount of activating reagent (increasing impregnation ratio) has another effect; the activation level is also increased, but the resultant pore texture is different.

According to the results of the second group of experi-

ments, in which the impregnation ratio is fixed to 1/1 to determine the effects of carbonization temperature, activation level increases with increasing temperature up to 900ºC but it decreases over 900ºC for active carbons obtained by KOH. For active carbons obtained by NaOH, increasing the carbonization temperature raises the activation level. Activated carbon with a BET surface area from 1596 - 1015 m^2/g were obtained by activation with KOH, 1461 to 945 m^2/g were obtained by activation with at NaOH at a impregnation ratio between chemical reagent and precursor: 1/1. When FTIR spectrum at 1000ºC (in Figure 5 and 6) are compared, it shows that the degree of carbonization is higher for the sample activated by NaOH as it can be appreciated by the lower intensity of the O-H band at 3422 cm^{-1}.

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