



The effect of process parameters on the carbon dioxide based production of activated carbon from lignite in a rotary reactor



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ABSTRACT

Activated carbons were prepared from raw lignite applying a physical activation method with CO₂ in a rotary tube furnace. The effects of process parameters were determined namely; temperature, activation time, preoxidation time, CO₂ flow rate, heating rate and rotation rate. In general micropore rich activated carbons were produced and the operating parameters had a significant influence on the product. BET surface areas were found to range from 331.5 m²/g to 696.4 m²/g, the total pore volume from 0.1768 cm³/g to 0.4446 cm³/g and yield from 61.06% to 11.06%. Raising CO₂ flow rate significantly increased the BET surface area of the product. The operation mode of the reactor was important. The BET surface area of the sample produced with rotation of the reactor is approximately 16% higher than the sample produced without rotation of the reactor, although it appears insensitive to the absolute speed of rotation. It was concluded that rotation of the reactor is an effective way to enhance the production of activated carbon, in terms of increasing surface area without a significant reduction in yield.

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1. Introduction

Coals are increasingly being used as raw materials for the preparation of active carbons because of their low cost and availability [1–4]. Biomasses [5,6] and polymers [7] have also been considered for similar reasons. Lignites [8–20] are also potential raw materials for the production of activated carbon, because there is growing interest in the preparation of low cost adsorbents.

The raw material can be activated using both chemical and physical activation methods. Various chemicals such as K₂CO₃ [5], H₃PO₄ [6,21], KOH [22], ZnCl₂ [23], have been used as activating agents in the chemical activation method. The physical activation method is a heat treatment process and consists of two basic processes; carbonisation and activation. Generally carbon dioxide, steam, oxygen and air are used as activating gases [1–4,8–12,24]. However, carbon dioxide and steam are mostly preferred due to the endothermic reaction of these fluids with the surface of the carbonaceous material. Thus the oxidation process is more controllable in the presence of CO₂ and H₂O when compared with O₂ and air [24]. Coals mostly show plastic behaviour during the pyrolysis process that adversely influences the pore development. Preoxidation helps to reduce this negative effect [25].

The following reaction mechanism between the carbonaceous surface and CO₂ has been suggested [26–28], where C(O) represents the oxygen adsorbed on the surface.



The use of lignites as precursors to prepare porous materials has been reported in the existing literature. Chemical activation methods were applied in some studies to obtain activated solid particles; the activating agents used in these investigations were phosphoric acid [10], potassium carbonate [19], potassium hydroxide [18] and zinc chloride [20]. Previous researchers have commonly characterised the product in terms of surface area, pore size distribution, morphology and surface composition. The adsorption capacity of the activated carbons was also determined using different chemical agents (notably dyes).

Steam [8,9,15] and CO₂ were also used as activating agents for the physical activation of some lignite samples. Starck et al. [15] determined the influence of demineralisation and ammoxidation on the adsorption properties of the activated carbon as well as the BET and pore size characteristics. In another steam activation study [9], lignite was pyrolysed and the quantities and properties of the gas, liquid and solid products were determined. The characteristics of the solid fraction including BET surface area, pore volume, iodine number and methylene blue adsorption were also evaluated.

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Table 1
Proximate and ultimate analysis of the lignite sample.

Proximate analysis (wt %)	
Moisture (Furnace method)	13.50
Moisture (Xylene method)	14.20
Ash (dry basis)	4.76
Fixed carbon (dry ash free basis)	51.68
Volatile matter (dry ash free basis)	48.37
Ultimate analysis (wt %, dry ash free basis)	
Carbon	67.87
Hydrogen	4.58
Nitrogen	1.45
Sulphur	0.36
Oxygen (by difference)	25.74

Different lignite samples were activated in fixed bed reactors using CO₂ and also mixtures of N₂ and CO₂ as the activating agent [11,13,16]. One research group studied the activation of lignite and they reported their results in two different articles [16,17]. The process in these studies was completed in two different stages; the sample was pyrolysed in the first stage to obtain char and the char samples were then activated by CO₂ [16] and by a mixture of N₂ and CO₂ [17] in the second stage. Skodras et al. [11] demineralised the lignite and then pyrolysed the demineralised lignite. Following this process the final product was activated in the presence of CO₂ at 800 °C and 900 °C. Karaca et al. [12,14] and Karaca [13] used the same lignite for CO₂ activation and the adsorption properties of the activated lignite were examined using methylene blue. However, there are no data reported on the BET surface area and porosity of the activated carbons.

Table 2
Experimental conditions.

Experiment ID	Temperature (°C)	Activation time (h)	Preoxidation time (h)	CO ₂ flow rate (L/min)	Heating rate (°C/min)	Rotation rate (rpm)
<i>Effect of temperature</i>						
1	750	1	–	0.3	5	13.7
2	800	1	–	0.3	5	13.7
3	850	1	–	0.3	5	13.7
4	900	1	–	0.3	5	13.7
<i>Effect of activation time</i>						
5	850	0	24	0.3	5	13.7
6	850	1	24	0.3	5	13.7
7	850	3	24	0.3	5	13.7
<i>Effect of preoxidation time</i>						
8	850	1	6	0.3	5	13.7
9	850	1	12	0.3	5	13.7
10 ^a	850	1	24	0.3	5	13.7
11	850	1	48	0.3	5	13.7
12	850	1	72	0.3	5	13.7
13	850	1	96	0.3	5	13.7
<i>Effect of CO₂ flow rate</i>						
14	850	1	24	0.15	5	13.7
15 ^a	850	1	24	0.3	5	13.7
16	850	1	24	0.45	5	13.7
<i>Effect of heating rate</i>						
17 ^a	850	1	24	0.3	5	13.7
18	850	1	24	0.3	10	13.7
19	850	1	24	0.3	15	13.7
20	850	1	24	0.3	20	13.7
<i>Effect of rotation rate</i>						
21	850	1	24	0.3	5	0
22	850	1	24	0.3	5	1.25
23	850	1	24	0.3	5	5.0
24 ^a	850	1	24	0.3	5	13.7

^a Experiment 6.

There are many studies in the literature relating to the preparation of porous materials using bituminous coals as precursors. However, literature on the preparation of porous material using lignite using CO₂ as an activating agent is limited. Indeed the lignite based studies reported were mainly conducted in consecutive chemical/physical treatment processes rather than the direct interaction with CO₂. Experiments were mainly carried out in fixed bed reactors or vertical tubes. No report of the physical activation of lignite with CO₂ in a rotary tube reactor has appeared in the available literature to date, although Zondlo and Velez [4] used a rotary reactor to study anthracite.

The objective of this study was to determine the effect of process parameters on the preparation of porous carbons, with high surface area from Turkish lignite. A rotary tube reactor was employed for the heat treatment based activation process. The effect of major process parameters including temperature, preoxidation time, activation period, CO₂ flow rate and reactor rotation, on the characteristics of the final product has been investigated.

2. Experimental

2.1. Lignite characteristics

Soma lignite (collected from the Kistrakdere region of Soma, Turkey), was used as the starting material. The proximate and ultimate analyses of the raw lignite are shown in Table 1. The ash and moisture contents were performed according to ASTM (ASTM E1755-01) and TS (Turkish Standards, TS 1561). The content of C, H, N, O and S elements were determined using an elemental analyser (LECO CHNS 932). The as-received lignite was crushed and sieved to a particle size of 150–425 μm prior to treatment. The BET surface area of the raw lignite sample is 9.3 m²/g.

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