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Production of Activated Carbon from Jojoba Seed Residue by Chemical Activation Using a Static Bed Reactor

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Abstract: The production of chemically activated carbon from Jojoba seed residue was experimentally investigated using a laboratory-scale static bed reactor. The effects of process variables such as activation time, activation temperature, particle size, chemical reagents (KCl, ZnCl₂ and H₃PO₄) and impregnation ratio on adsorption capacity of activated Jojoba seed residue were studied. The highest iodine number and yield were obtained by using zinc chloride as an activating reagent. The results also showed that, increasing activation time, activation temperature, impregnation ratio and/or decreasing particle size improved the adsorption capacity. An activation time of 90 min activation temperature of 600°C, impregnation ratio of 1.0 and particle size of 0.5-0.717 mm were found to be the best conditions.

Key words: Activated carbon, Jojoba seed residue, chemical activation, static bed reactor

INTRODUCTION

Activated carbons are used in many applications such as, water and wastewater treatment, chemical and petroleum industries and in mining. The source of the most commonly used activated carbon in commercial practice is peat, coal, lignite, wood and coconut shell^[1]. On the other hand agricultural by-products are lignocellulosic wastes that may offer an inexpensive additional source of activated carbons^[2,3].

One of the most important physical properties of activated carbon is the surface area. Activated carbons have an extensive internal pore structure that presents a high surface area available for adsorption^[4]. Usually, the surface properties of the produced activated carbon are affected by the starting material and activation scheme. The effect of starting material is due to the fact that carbon materials present important differences in their properties and their reactivity^[5,6]. Activated carbons are produced by three methods, physical, chemical and combined (physical and chemical) procedures^[1]. Physical activation involves carbonization followed by gasification at higher temperatures, whereas in chemical activation, the raw material is impregnated with chemical reagent such as, KCl, ZnCl₂ or H₃PO₄ then heat-treated at moderate temperatures^[3]. In general the residues from carbonization and activation have a large total pore volume and a high

internal surface area of the range between 300 and 2500 m² g⁻¹^[7,8].

As mentioned earlier, in recent years, many different agricultural residues were used to produce activated carbon, e.g. palm stone^[9], rice hulls, rice straw, sugarcane bagasse and pecan shells^[1,10-12].

Jojoba seed residue is very cheap and available in Jordan. It has been used experimentally to recover radioactive metals from nuclear wastewater as well as to remove toxic heavy metals from industrial water^[13].

The present work aims to study the effect of different parameters on the activation process of Jojoba seed residue by chemical activation in a static bed reactor, with the focus on the effect of the activation time, the particle size, the activation temperature, the impregnation reagent (KCl, ZnCl₂ and H₃PO₄) and the impregnation ratio on the adsorption capacity of the produced activated carbon.

MATERIALS AND METHODS

Materials: Jojoba seed were obtained from the Center for Agricultural Research at Jordan University of Science and Technology (JUST). Jojoba oil was extracted from the seed using Soxhlet extractor and hexane as a solvent. Details of the extraction method can be found elsewhere^[13]. The chemicals used were Zinc Chloride (98% ACS reagent), Potassium Chloride (99%) and

Table 1: Correction factor for iodine number test^[17]

Residual filter normality	0.0000	0.0001	0.0002	0.0003	0.0004	0.0005	0.0006	0.0007	0.0008	0.0009
0.008	1.1625	1.1613	1.16	1.1575	1.155	1.1533	1.1513	1.15	1.1475	1.1463
0.009	1.1438	1.1425	1.14	1.1375	1.1363	1.35	1.1325	1.13	1.1288	1.1275
0.01	1.125	1.1238	1.1225	1.1213	1.12	1.1475	1.1163	1.115	1.1138	1.1113
0.011	1.11	1.1088	1.1075	1.1063	1.1038	1.1025	1.1	1.0988	1.0975	1.0963
0.012	1.095	1.0938	1.0925	1.09	1.0888	1.0875	1.0863	1.058	1.0838	1.0825
0.013	1.08	1.0788	1.0775	1.0763	1.075	1.0738	1.0725	1.0713	1.07	1.0688
0.014	1.0675	1.0663	1.065	1.0625	1.0613	1.06	1.0583	1.0575	1.0563	1.055
0.015	1.0538	1.0525	1.0513	1.05	1.0488	1.0475	1.0463	1.045	1.0438	1.0425
0.016	1.0413	1.04	1.0388	1.0375	1.0375	1.0363	1.035	1.0333	1.0325	1.0313
0.017	1.03	1.0288	1.0275	1.0263	1.025	1.0245	1.0238	1.0225	1.0208	1.02
0.018	1.02	1.0188	1.0175	1.0163	1.015	1.0144	1.0138	1.0125	1.0125	1.0113
0.019	1.01	1.0088	1.0075	1.0075	1.0063	1.005	1.005	1.0038	1.0025	1.0025
0.020	1.0013	1	1	0.9988	0.9975	0.9975	0.9963	0.995	0.995	0.9938
0.021	0.9938	0.9925	0.9925	0.9913	0.99	0.99	0.9888	0.9875	0.9875	0.9863
0.022	0.9863	0.985	0.985	0.9838	0.9825	0.9825	0.9813	0.9813	0.98	0.9788
0.023	0.9788	0.9775	0.9775	0.9763	0.9763	0.975	0.975	0.9738	0.9738	0.9725
0.024	0.9725	0.9708	0.97	0.97	0.9688	0.9622	0.9675	0.9675	0.9663	0.9663
0.025	0.965	0.965	0.9638	0.9638	0.9625	0.9625	0.9613	0.9613	0.9606	0.96
0.026	0.96	0.9588	0.9588	0.9575	0.9575	0.9563	0.9563	0.955	0.955	0.9538
0.027	0.9538	0.9525	0.9525	0.9519	0.9513	0.9513	0.9506	0.95	0.95	0.9488
0.028	0.9488	0.9475	0.9475	0.9463	0.9463	0.9463	0.945	0.945	0.9438	0.9438
0.029	0.9425	0.9425	0.9425	0.9413	0.9413	0.94	0.94	0.9394	0.9388	0.9388
0.030	0.9375	0.9375	0.9375	0.9363	0.9363	0.9363	0.9363	0.935	0.935	0.9346
0.031	0.9333	0.9333	0.9325	0.9325	0.9325	0.9319	0.9313	0.9313	0.93	0.93
0.032	0.93	0.9294	0.9288	0.9288	0.928	0.9275	0.9275	0.9275	0.927	0.927
0.033	0.9263	0.9263	0.9257	0.925	0.925					

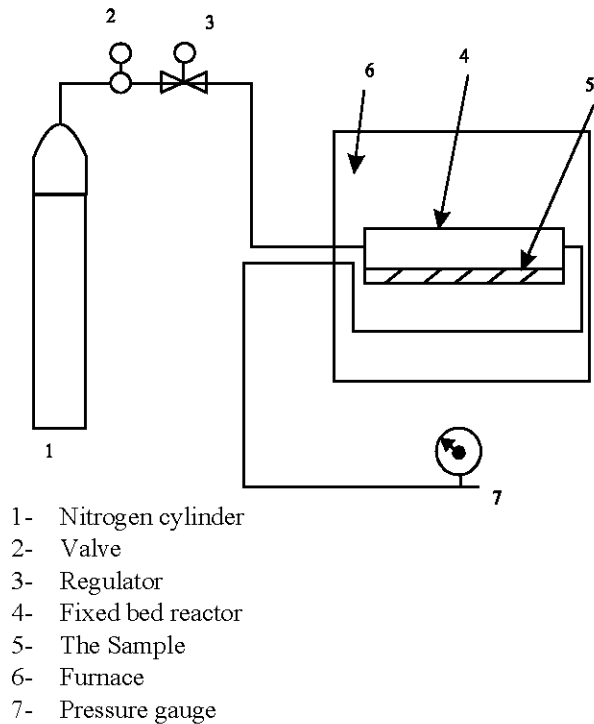


Fig. 1: Activation Apparatus

Phosphoric acid (85% wt.) all of them were purchased from Sigma - Aldrich.

Methods

Preparations of activated carbons: The solid residue was washed with water to get rid of the impurities and then dried at 105 °C to ensure that all water and hexane were removed. The dried Jojoba seed samples were crushed and sieved to obtain a particle size in the range of 0.2-2.0 mm.

In order to activate the seed, 50 g of a specified particle size were mixed with 300 mL of distilled water and predetermined amount of one of the reagents (KCl, ZnCl₂, or H₃PO₄). The mixture was shaken at 85±1°C for 7 h in a water bath using impregnation ratios from 0.2 to 1.8 gram reagent/gram sample, followed by heating until complete evaporation^[14].

The solid particles were dried at 105°C for about 24 h to ensure complete drying of the particles. After that, the solid particles were heated in a laboratory-scale static bed reactor that was designed and fabricated for the purpose of the present study. A schematic diagram of the experimental setup is shown in Fig. 1 The solid particles were heated gradually to reach the desired temperature within one hour, then kept at that temperature (i.e., activation temperature) for certain time called (activation time). Pure nitrogen was injected to the reactor in order to maintain a positive pressure of 3 psig inside the reactor to prevent any inlet air.

At the end of each experiment, the activated products in the reactor were cooled to 200-330°C by

passing a nitrogen flow at room temperature into the reactor to prevent oxidation of the sample. The activated products were removed from the reactor and then thoroughly washed and stirred with 500 mL solution of 0.10 N HCl for 4 h then mixed with distilled water for 24 h at room temperature to ensure complete salt removal. The solution was decanted and the activated particles were dried at 105°C for 24 h, weighed and stored^[15,16].

Determination of yield and iodine number: The yield is defined as the amount of activated carbon obtained for a given amount of the raw material. In this study, the standard test used in the characterization of the produced activated carbon was the iodine number (The iodine number demonstrated the adsorption capacity). The iodine number is defined as the milligrams of iodine adsorbed by one gram of carbon when the iodine concentration of the residual filtrate is 0.02 N^[17].

The iodine number of the carbon was calculated by using the following equation:

$$\text{Iodine \#} = \frac{X}{M} * A \quad (1)$$

Where, $\frac{X}{M}$ is given by the following equation:

$$\frac{X}{M} = \frac{((N_1 * 2.693) - 2.2 * N_2 * 126.3) * V_1}{M} \quad (2)$$

Where:

X: Weight of iodine adsorbed in milligram

M: Weight of activated sample in grams

N₁: Normality of iodine solution

N₂: Normality of thiosulfate solution

A: Correction factor obtained from Table 1 after the calculation of the residual filtrate normality (N_R)

$$N_R = \frac{N_2 * V_1}{50} \quad (3)$$

The capacity of a carbon for any adsorbent is dependent on the concentration of the adsorbent in the medium in contact with the carbon. Thus, the concentration of the residual filtrate must be specified or known so those appropriate factors may be applied to correct the concentration to agree with the definition. The amount of the sample to be used in the determination is governed by the activity of the carbon. If the residual filtrate normality N_R is not within the range 0.008-0.0334 N given in Table 1, the procedure should be repeated using a different weight of sample.

RESULTS AND DISCUSSION

Effect of activation time: Results shows that the iodine number increases with the activation time up to 90 min where the maximum iodine number is obtained (Fig. 2). On the other hand, the yield decreases with the increase in time. The increase in iodine number and the decrease in yield are due to the release of the volatile materials. Further increase in time more than 90 min leads to a slight increase in the iodine number; this may be due to an increase in the ash content per unit weight of the activated carbon. The highest iodine number was obtained at the activation time of 90 min with an acceptable percentage yield, thus this time period may be considered suitable for Jojoba seed residue activation. Kirubakaran *et al.*^[15] and Edwards *et al.*^[18] obtained similar results for coconut shells and for charcoal, respectively.

Effect of activation temperature: It can be seen from Fig. 3 that the iodine number increases as activation temperature increases up to 800°C. On the other hand, the yield was observed to decrease sharply due to a decrease in volatile matters. At low temperatures, activation was very low due to the inefficient heat transfer. However, at 700°C it was observed that as activation temperature increased there was no significant increase in the iodine number. This may be due to the temperature stability and catalytic activity of zinc chloride (boiling point 732°C)^[19]. After 800°C it was expected that the percentage yield decrease significantly due to the destruction of Jojoba seed residue structure.

Effect of particle size: Figure 4 shows that the increase in particle size up to 0.69 mm decreases iodine number slightly, then, the iodine number was observed to decrease sharply for further increase in particle size. Decreasing the particle size increases the specific surface area of the activated product, which helps the volatile matter to escape easily from the particle, thus enhancing the pore development. Even though smaller particles show better activation, the yield at small particles was very low. The best range of particle size diameter was 0.5-0.717 mm. Figure 5 shows that the iodine number for activated Jojoba seed residue was higher than that without activation.

Effect of impregnation ratio: It can be noted that as the impregnation ratio increases. The iodine number and the yield increase (Fig. 6). This was due to the increase in the selective oxidation as the impregnation ratio increases, however, above impregnation ratio of 1.0, the increase of the impregnation ratio was marginal, which did not

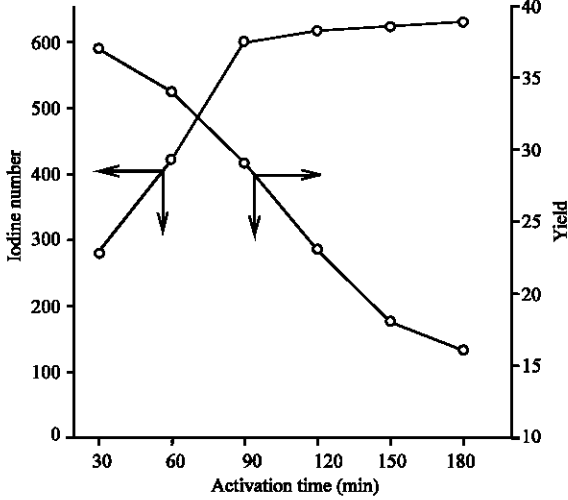


Fig. 2: Effect of activation time on iodine number and yield for Jojoba residue ($T = 600^{\circ}\text{C}$, Impregnation ratio = 1.4, impregnation reagent KCl and particle size = 0.5-0.717 mm)

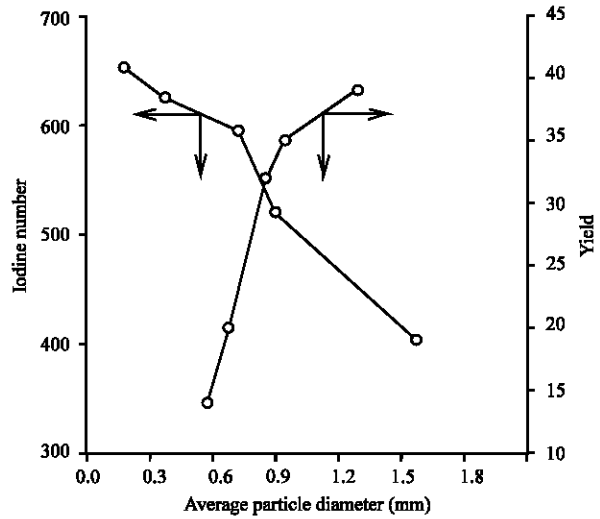


Fig. 4: Effect of particle size on iodine number and yield for Jojoba residue ($t = 90$ min, $T = 600^{\circ}\text{C}$, Impregnation ratio = 1.4 and impregnation reagent KCl)

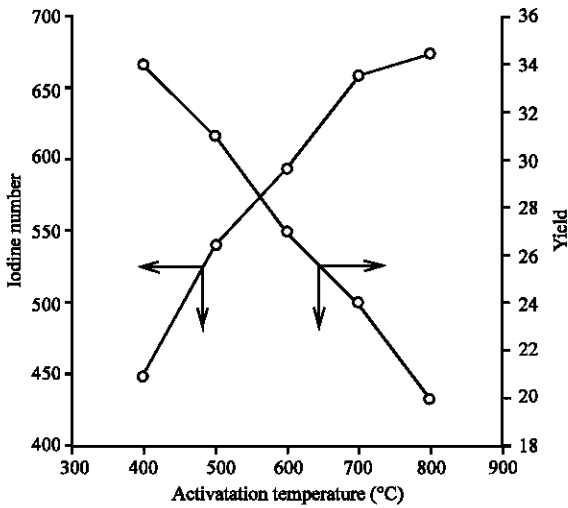


Fig. 3: Effect of activation temperature on iodine number and yield for Jojoba residue ($t = 90$ min, Impregnation ratio = 1.4, impregnation reagent KCl and particle size = 0.5-0.717 mm)

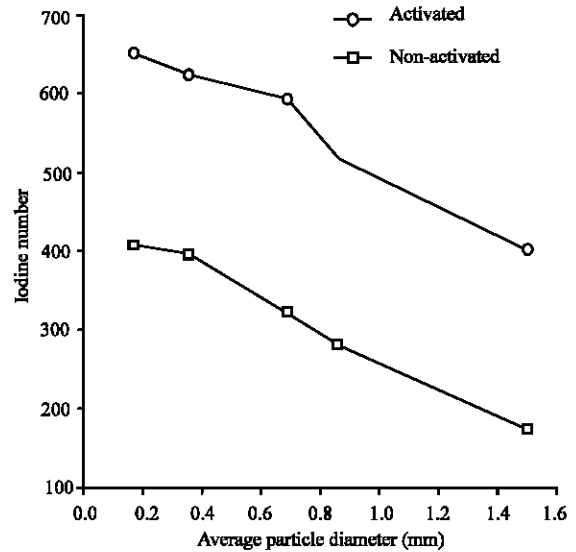


Fig. 5: Effect of particle size on iodine number for activated Jojoba residue ($t = 90$ min, $T = 600^{\circ}\text{C}$, impregnation ratio = 1.4 and impregnation reagent KCl) and Jojoba residue without activation

enhance the activation process. This means that the amount of reagent which exists when the impregnation ratio equal 1.0 was sufficient to complete the activation.

Effect of activating reagent: Figure 7 shows the effect of activating reagent on the iodine number. It can be noted that the activated product, impregnated with zinc chloride, gave an iodine number higher than that impregnated with potassium chloride and phosphoric acid. This may be due

to the vigorous carbonization reaction promoted by zinc chloride compared to potassium chloride and phosphoric acid; it might be also due to the higher capability of zinc chloride to act as a dehydrating agent than potassium chloride and phosphoric acid, which increased the concentration of carbonaceous materials. Impregnation with a solution of zinc chloride produces activated carbon with higher developed porosity^[14].

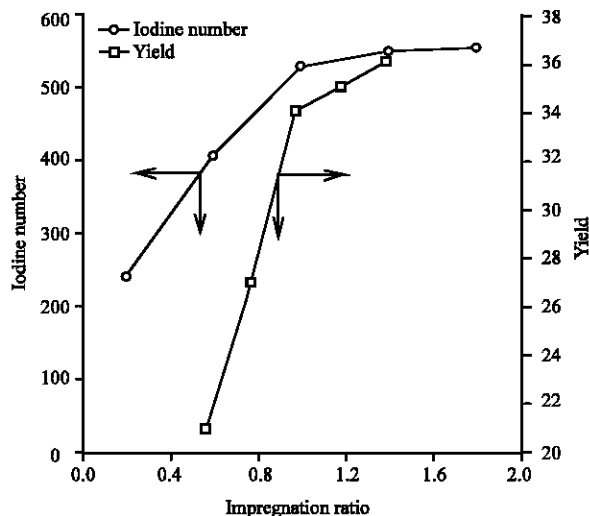


Fig. 6: Effect of impregnation ratio (gram reagent/gram sample) on iodine number and yield for Jojoba residue (t = 90 min, T = 600°C, particle size = 0.5-0.717 mm and impregnation reagent KCl)

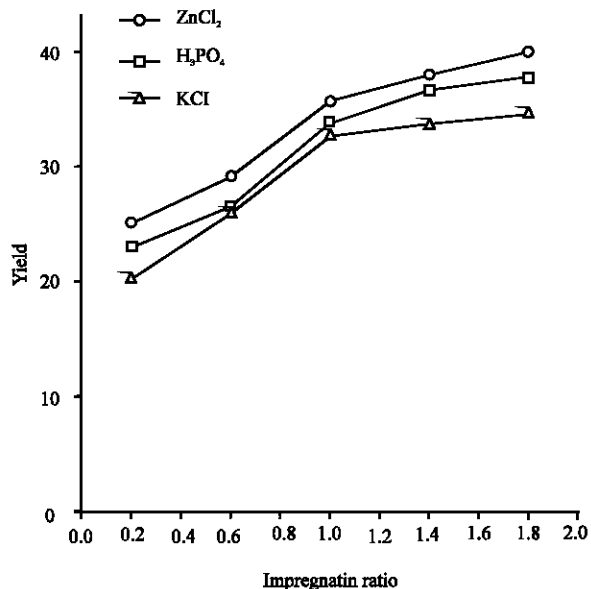


Fig. 8: Effect of impregnation ratio (gram reagent/gram sample) on the yield for Jojoba residue (t = 90 min, T = 600°C, particle size = 0.5-0.717 mm and impregnation reagent KCl, H₃PO₄ and ZnCl₂)

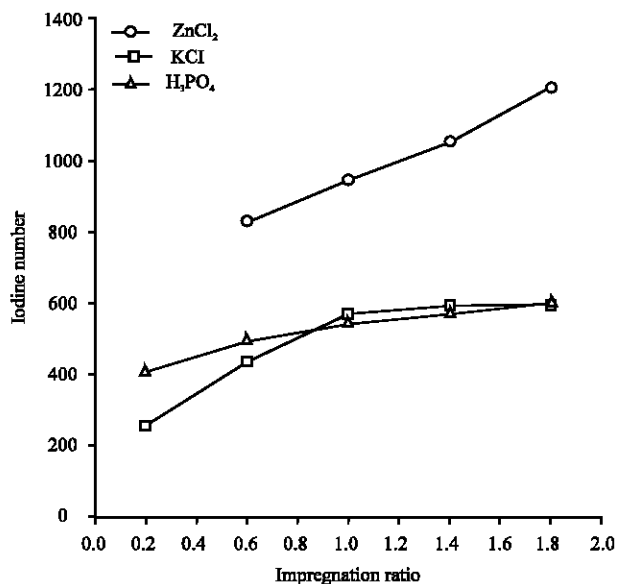


Fig. 7: Effect of impregnation ratio (gram reagent/gram sample) on iodine number for Jojoba residue (t = 90 min, T = 600°C, particle size = 0.5-0.717 mm and impregnation reagent KCl, H₃PO₄ and ZnCl₂)

The effect of activating reagent on yield is shown in Fig. 8. It can be shown that zinc chloride gave a higher percentage yield than potassium chloride and phosphoric acid for the same experimental conditions. This was because zinc chloride was a better dehydrating agent than both potassium chloride and phosphoric acid.

CONCLUSION

On the basis of the experimental study carried out in this work, it can be concluded that Jojoba seed residue can produce a suitable activated carbon in comparison with other raw materials such as, Jift, Almond shells and Coconut. The yield of the activated carbon increases as the particle size and the impregnation ratio increase and decreases with increasing the activation time and the activation temperature. The iodine number (adsorptive capacity) increases with increasing the activation time, the activation temperature and the impregnation ratio, but decreases with increasing the particle size. Activated Jojoba seed residue impregnated with zinc chloride produced activated carbon with higher adsorptive capacity than that produced using potassium chloride and phosphoric acid.

Under the experimental conditions investigated for the chemical activation of Jojoba seed residue in the static bed reactor, activation time of 90 min, activation temperature of 600°C with particle diameter of 0.5-0.717 mm and an impregnation ratio of 1.0, for the three reagents may be taken as optimal parameters.

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