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The Effect of Process Variables on Surface Areas of Ultrasound-Assisted Preparation of Activated Carbon From Alkaline Impregnated Hazelnut Shell

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Abstract

This study visualizes the effects of independent parameters on the preparation of activated carbon from hazelnut shell via ultrasound assisted KOH impregnation. The effect of operational parameters including particle size (0.78-1.85 mm), ultrasonic power density (2-190 W/L), impregnation ratio (0.015-0.06 g/mL), impregnation time (37-143 min), activation temperature (661-839 ºC) and activation time (18-72 min) on the surface areas of prepared activated carbon were investigated. Based on the results, the activation temperature and activation time have a positive effect, while particle size and impregnation ratio have a negative effect on the surface areas of prepared activated carbon. The surface area was increased by increasing activation temperature and activation time, but decreasing particle size and impregnation ratio.

Keywords: Hazelnut shell, activated carbon, ultrasound, alkaline impregnation, optimization

INTRODUCTION

Industrial waste waters contain of organic, inorganic, metallic and biological pollutant, and they cause serious disposal problems for environment. Pollution by heavy metals is one of the most serious environmental problems facing the world's life. For this reason, the concentration of heavy metals in waste water must be reduced to the maximum permissible concentration [1]. One of the oldest methods for removal of heavy metals from wastewater is using porous solid adsorbents. The properties of porous solids that render them useful for water treatment include high porosity and surface activity as well as the physical and chemical nature of the adsorptive surfaces. Compared with the other purification and separation methods, adsorption has demonstrated efficiency and economic feasibility as a wastewater treatment operation. Adsorption still remains one of the more novel chemical engineering processes in water purification, separation, mineral beneficiation, soil conversation, and many other process areas even though the concept of applying the adsorption process to treat contamined liquids and gases has been realized for many years.

Generally, granular activated carbon (GACs) were produced by activating either chemical activating or physical activating from various carbonaceous raw materials such as wood, peat, coal, lignite, and wastes of vegetable origin (e.g. grape seeds, palm-tree cobs, nutshells and fruit stones) as the two major resources [2, 3, 4]. Today, one promising approach for the production of cheap and highly efficient activated carbon is the use of cheaper and readily available non-classical materials such as hazelnut shell, coconut shell, olive-waste cakes and corn cob [3,5-13]. The combination of the chemical and physical activation processes leads to the production of activated carbon with specific surface properties. The first step involves a chemical activation step where raw agricultural materials are impregnated with a solution of dehydrating agent (for example $ZnCl_2$, H_2SO_4) to retard the formation of tars during the carbonization process. Furthermore in physical activation, they are washed, dried and carbonized in an inert atmosphere to produce the final activated carbon [2, 3, 7, 11, 14].

It is now widely accepted that ultrasound power has great potential for uses, in addition to conventional applications in cleaning and plastic welding, in a wide variety of industrial fields such as electrochemistry, food technology, nanotechnology, chemical synthesis, dissolution and extraction, dispersion of solids, phase separation, water and sewage treatment [15]. Ultrasound produces its mechanical and chemical effects through the formation and collapse of "cavitations" bubbles [16]. A significant amount of research has been published concerning with this "sonochemical effect", and collected in various recent books [17, 18]. Ultrasound exhibits also several beneficial mechanical effects in solid-liquid systems by means of the cavitations phenomenon by causing the formation of many microcracks on the solid surface, it increases the surface area between the reactants and cleans solid reactant or catalyst particle surfaces; thus, it enhances mass transfer rates [19].

The aim of this paper is to investigate the effect of process variables on surface areas of ultrasound-assisted preparation of activated carbon from alkaline impregnated hazelnut shell. The experiments were designed statistically and the particle size, ultrasonic power density, impregnation ratio and time, activation temperature and time were chosen as independent variables. The regression model obtained by means of variance analysis were used in a constrained optimization to find optimum process conditions for maximum surface areas of the activated carbon prepared by ultrasound-assisted KOH-impregnated hazelnut shell.

MATERIALS and METHODS

Activated carbon used in the adsorption experiments were supplied from project number 2003/38 supported by Atatürk University Research Foundation, Erzurum/Turkey [20].

The experimental set-up consisted of an ultrasonic power generator (Meinhardt ultraschalltechnic, K 80-5, 140W, 850 kHz), a jacketed glass reactor equipped with a titan probe (E/805/T/solo ultrasonic transducer) which is connected to the bottom of the reactor and fitted with a reflux condenser. A typical impregnation experiment (chemical activation) was carried out as follows: specified particle size and amounts of hazelnut shells and 10% KOH were loaded into the glass reactor and chemical activation process maintained the desired impregnation time. Ultrasound power (continuous mode) was adjusted using the relationship between the intensity setting of the generator, and ultrasound power absorbed by the reaction medium measured by the calorimetric method [21]. Ten percent of KOH solution was used in all the experiments [22, 23]. A constant impregnation temperature of 50 ^oC was applied by means of a constant temperature circulator. At the end of the impregnation experiment, the sample was immediately filtered, washed with hot distilled water for removal of its alkalinity, the basic and water-soluble components and dried. The impregnated sample was carbonized in a furnace (Carbolıte, CWF 1300) under N_2 atmosphere (1 kg/cm^2) at desired carbonization temperature and time for final activated carbon.

The orthogonal central composite design was applied for fitting a second-order model in this study as described in detailed in our previous experimental studies [24-26].

RESULTS

Contours of fitted second-order Eq. (1) and data from second-order design have been used to visualize the effects of independent parameters on the surface areas of the activated carbons prepared by ultrasound-assisted KOH-impregnated hazelnut shell under experimental conditions in response surface contour plots of Figs. 1–6.

$$Ys= - 9.1681 -10.5471 X1 + 0.7000 X2 -12.5003 X3 +1.7162 X4 +8.2995 X5+24.7996 X6 +12.6799 X1X1 +9.9499 X2X2+9.0234 X3X3+8.9166 X4X4+7.7605 X5X5 +11.3060 X6X6-0.6400 X1X2+3.2513 X1X3+4.3713 X1X4 - 1.2950 X1X5-6.1550 X1X6 -1.2950 X2X3 -14.8250 X2X4 +3.2513 X2X5 - 0.0837 X2X6 -0.0837 X3X4 -0.6400 X3X5 -14.8250 X3X6 -6.1550 X4X5 - 1.2950 X4X6 +4.3713 X5X6 \quad (1)$$

In Fig. 1, it is seen that the decrease of impregnation ratio and particle size in the alkaline impregnation step to prepare activated carbon from hazelnut shell increased the surface area of the activated carbon. In Fig. 2, the surface area of the activated carbon increased with the increase in activation temperature and increased with the decrease in particle size. The surface area of the activated carbon increased with the increase in activation time and increased with the decrease in particle size as shown in Fig. 3. In Fig. 4, the surface area of the activated carbon increased with the increase in activation temperature and increased with the decrease in impregnation ratio. The surface area of the activated carbon increased with the increase in activation time and increased with the decrease in impregnation ratio as shown in Fig. 5. The surface area of the activated carbon increased with the increase in activation time and activation temperature as shown in Fig. 6.

In conclusion, the surface area of activated carbon increased with the decrease in impregnation ratio. This behavior could be attributed to the ratio of alkaline impregnation to hazelnut shell which is important in sonication. The diffusion of KOH solution into the pores entails an opening and enlargement pores, which enhance the surface area of activated carbon. The enhancement of surface area of the activated carbon prepared from ultrasound-assisted KOH-impregnated hazelnut shell was attributed to higher diffusion of KOH solution into the pores of the hazelnut shells, higher surface area with the formation of many micro-cracks on the hazelnut shells surface and cleaner solid particle surfaces produced by cavitation process under ultrasound irradiation [19].

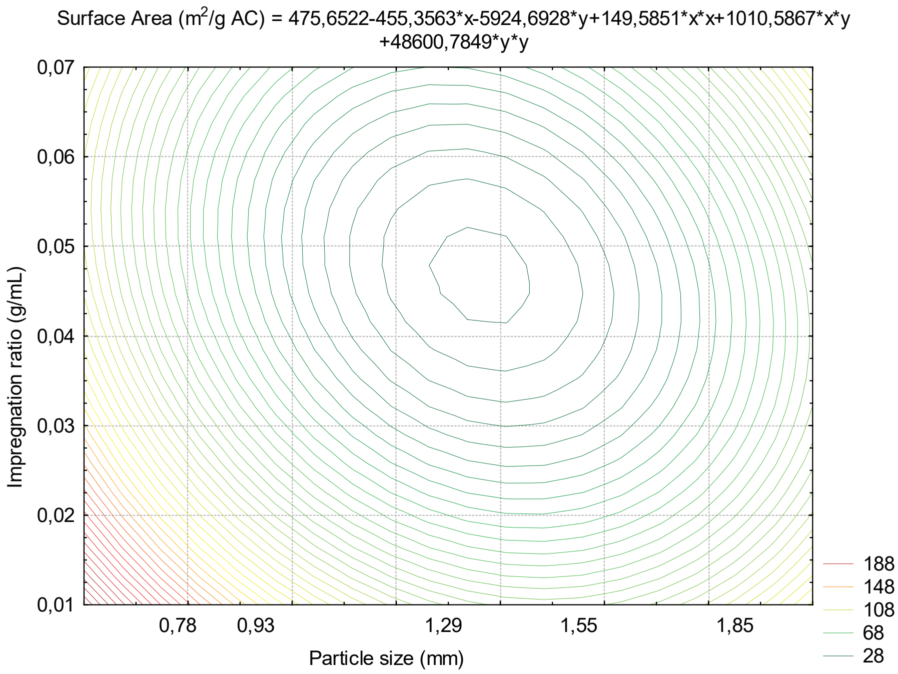

Figure 1. Response surface contour plots for the effects of impregnation ratio and particle size

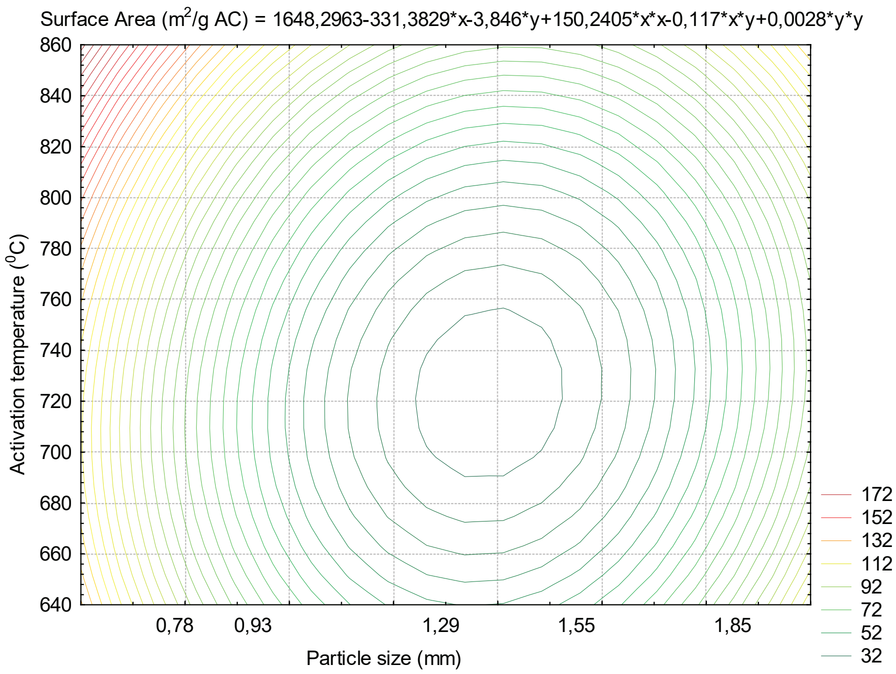

Figure 2. Response surface contour plots for the effects of activation temperature and particle size

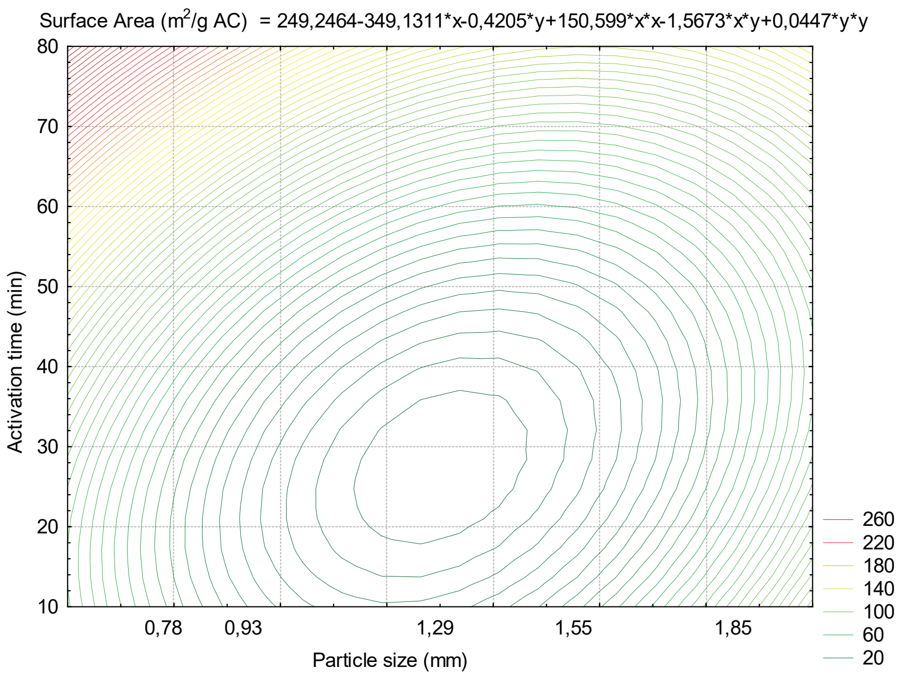

Figure 3. Response surface contour plots for the effects of activation time and particle size

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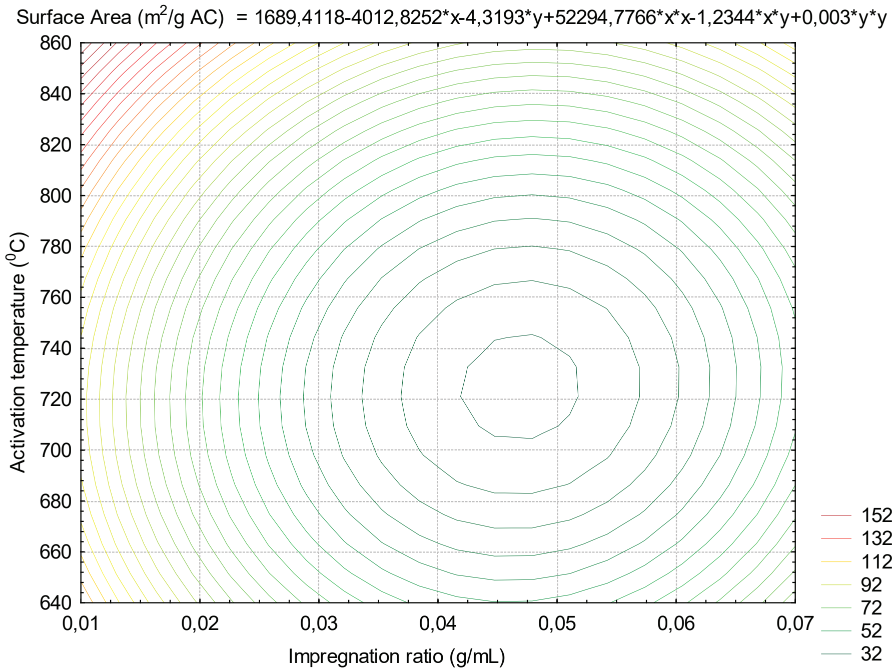

Figure 4. Response surface contour plots for the effects of activation temperature and impregnation ratio

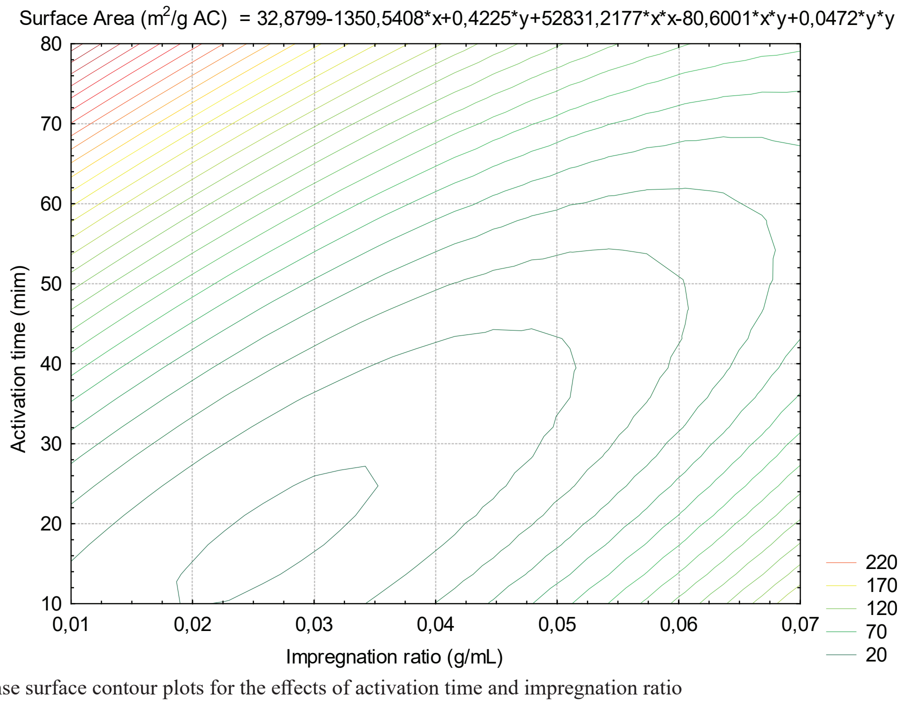

Figure 5. Response surface contour plots for the effects of activation time and impregnation ratio

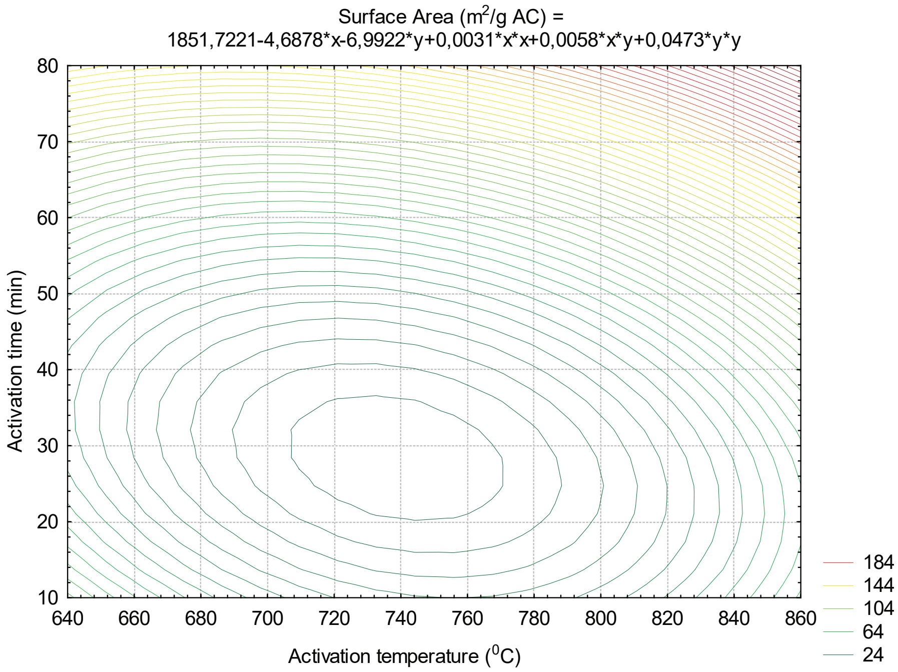

Figure 6. Response surface contour plots for the effects of activation time and activation temperature

DISCUSSION

Activated carbons with high surface areas can be prepared from agricultural waste (hazelnut shell) using ultrasonic irradiation assisted chemical activation by KOH. Based on the results, the activation temperature and activation time have a positive effect, while particle size and impregnation ratio have a negative effect on the surface areas of prepared activated carbon. The surface area was increased by increasing activation temperature and activation time, but decreasing particle size and impregnation ratio. In conclusion, the alkaline impregnation into hazelnut shells under ultrasonic irradiation was found to be beneficial for preparation of activated carbon for use as adsorbents.

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