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Research Paper

The use of design of experiments for the evaluation of the production of surface rich activated carbon from sewage sludge via microwave and conventional pyrolysis

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HIGHLIGHTS

- Using of DOE for preparation of AC by conventional and microwave pyrolysis.
- The significant parameters in producing activated carbon were investigated.
- Conventional pyrolysis AC had better textural development than microwave AC.
- Temperature and holding time had significant influence on the S_{BET}.
- Reduction of production cost of activated carbon.

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ABSTRACT

Experimental design and response surface methodology were used for the preparation and comparison of activated carbon produced from sewage sludge by two types of pyrolysis: conventional furnace and microwave. The preparation method was performed following a full fractional factorial design (2^3), including pyrolysis temperature or power radiation, holding time and chemical activation agent, and specific surface area (S_{BET}) of prepared activated carbon. The influence of these factors on the S_{BET} of obtained carbon was investigated using an analysis of variance. Samples made by conventional pyrolysis showed overall higher S_{BET} values than samples synthesised by the microwave method. The optimum parameters for the preparation of activated carbon using the conventional pyrolysis have been identified as: pyrolysis temperature of 500 °C, holding time of 15 min, and a ratio of $ZnCl_2$:sludge of 0.5. Microwave pyrolysis is found to be optimal when operating at 980 W for 12 min. Under these conditions, S_{BET} values of 679 and 501 m^2g^{-1}, respectively, have been obtained. The analysis of nitrogen adsorption/desorption isotherms revealed the presence of micro and mesopores in the activated carbon. The most important significant factor, according statistical analysis, in the variance in S_{BET} for the conventional pyrolysis samples were the pyrolysis temperature and interaction between pyrolysis temperature, holding time and ratio of $ZnCl_2$:sludge were the most important factors. The highest impact parameters for the microwave method were found for the interaction between power radiation and ratio of $ZnCl_2$:sludge and the holding time.

1. Introduction

The growing urbanisation of global society, coupled with increasingly stringent sludge reuse/disposal regulations and increasing public pressure, is forcing both public and private sludge generators to evaluate their sludge management strategies [1,2]. Conventionally, waste sludge is disposed of via incineration, landfilling, and as soil conditioner in agriculture. However, in recent years, new applications for sewage sludge, such as production of ceramic materials [3,4] and activated carbon [5–7] have been explored.

Activated carbon is generally produced from natural starting materials (e.g. coconut shells) by pyrolysis under inert atmosphere at

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elevated temperatures. The temperature treatment can be conducted by conventional heating in a furnace, or by microwave-assisted pyrolysis. The main difference between conventional pyrolysis and the microwave-assisted method is the way the heat is generated. Microwaves supply energy directly to the carbon bed [8,9]. Energy transfer is not by conduction or convection as in conventional heating, but microwave energy is readily transformed into heat inside the particles by dipole rotation and ionic conduction [8,9]. Thus, it has the advantages of rapid temperature rise, uniform temperature distribution, and energy savings over conventional thermal methods [8,10]. However, current literature is not clear about which method produces adsorbents with higher specific surface areas (S_{BET}). Different work comes to different conclusions about different starting materials; for instance, with oil palm shell as the starting material, conventional pyrolysis obtains higher S_{BET} than microwave [11]. Conversely, activated carbon prepared from olive pits obtained higher S_{BET} with the microwave-assisted heating process [12,13].

The properties, especially S_{BET}, of activated carbon depend on variables like feedstock type and source, pyrolysis temperature, radiation power, and holding time [13,14]. Because of these very controllable parameters, pyrolytic methods are highly versatile processes where it is possible to optimise these variables to get activated carbon with higher S_{BET} [13,14].

The properties of activated carbon such as S_{BET} can be improved using experimental design by response surface methodology (RSM). RSM is a very valuable tool for this purpose as it presents statistical models which can be used to understand the interactions between the parameters that have been optimised [15–17]. RSM has been applied widely in various processes for the optimisation of experimental conditions, and should prove useful for the preparation of activated carbon. As reported recently, RSM was applied in the production of AC using different precursors such as Turkish lignite [17], Albizia lebbeck seed pods [18], Bamboo [19], Jatropha Hull [20], polycarbonate [21] and coconut shell [22].

To this point, it does not appear that RSM has been applied to study the comparison of the preparation of activated carbon from sludge sewage by conventional pyrolysis versus microwave-assisted pyrolysis.

This work aims to apply RSM to evaluate how microwave and conventional pyrolysis methods, and the pyrolysis conditions: pyrolysis temperature/applied microwave power, holding time, and ratios of chemical agents, can be optimised for maximum effect on the S_{BET} of activated carbon from sewage sludge.

2. Materials and methods

2.1. Experimental design

In this work, a 2^3 full factorial design with 3 central points was studied. The factors used for the production of activated carbon from sewage sludge were studied with standard RSM in order to identify and optimise the effective process parameters. In addition, this method helps to analyse the interaction between these parameters [15,16]. With this method, a core factorial is created that forms a cube with sides that are two coded units in length (from −1 to +1); noted in the Table 1. Table 1 shows the ranges and the levels of the variables examined and their combinations in this study. The studied variables were: activation temperature or microwave power ($X1$), chemical impregnation ratio ($X2$), and holding time ($X3$). In order to minimise the effects of the uncontrolled factors, the experimental sequence was randomised, as can be seen in Table 1. It shows the number of runs and their respective parameter combinations to activated carbon samples made by conventional pyrolysis and microwave. It was assumed that the relationship between the three independent variables and the experimental S_{BET} data follow a linear equation, as expressed in Eq. (1):

$$R = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_1 X_2 + \beta_5 X_1 X_3 + \beta_6 X_2 X_3 + \beta_7 X_1 X_2 X_3 + \varepsilon \quad (1)$$

Where R is the predicted response; X_1 to X_3, coded variables; β_o, the constant coefficient; β_1 to β_3, the linear term coefficients; β_4 to β_6, the interaction coefficients between two variables and β_7, the interaction coefficients between three variables.

2.2. Preparation of sludge-derived activated carbon

The raw material used for preparing the activated carbon was sewage sludge obtained from a municipal wastewater treatment plant in Porto Alegre, Brazil. First, the sludge was dried at 105 °C for 24 h until no further weight loss could be detected. Subsequently, it was crushed with a grinder, and sieved to a size range

Table 1
Design of experiments for preparation of activated carbon by conventional and microwave pyrolysis.

Factor	Name	Conventional			Microwave		
		Variable level					
		−1	0	+1	−1	0	+1
		−1	0	+1	−1	0	+1
X1	Temperature and power	500	650	800	700	840	980
X2	Hold time	15	37.5	60	8	10	12
X3	Ratio: $ZnCl_2$/sludge	0.5	1.0	1.5	0.5	1.0	1.5

Experiments samples	Coded variables			Conventional			Microwave		
	T_f	t_r	m	T_f	t_r	m	Pow	t_r	m
500-15-0.5 and 700-8-0.5	−1	−1	−1	500	15	0.5	700	8	0.5
800-15-0.5 and 980-8-0.5	+1	−1	−1	800	15	0.5	980	8	0.5
500-60-0.5 and 700-12-0.5	−1	+1	−1	500	60	0.5	700	12	0.5
800-60-0.5 and 980-12-0.5	+1	+1	−1	800	60	0.5	980	12	0.5
500-15-1.5 and 700-8-1.5	−1	−1	+1	500	15	1.5	700	8	1.5
800-15-1.5 and 980-8-1.5	+1	−1	+1	800	15	1.5	980	8	1.5
500-60-1.5 and 700-12-1.5	−1	+1	+1	500	60	1.5	700	12	1.5
800-60-1.5 and 980-12-1.5	+1	+1	+1	800	60	1.5	980	12	1.5
650-37-1.0 and 840-10-1.0	0	0	0	650	37.5	1.0	840	10	1.0
650-37-1.0 and 840-10-1.0	0	0	0	650	37.5	1.0	840	10	1.0
650-37-1.0 and 840-10-1.0	0	0	0	650	37.5	1.0	840	10	1.0