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FULL PAPER

Characterization of Activated Charcoal Obtained from Malt Bagasse

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Abstract:

Nowadays the concern about the environment has pushed the development of new technologies capable of reducing the human impact on earth. The utilization of malt bagasse to produce activated charcoal reduces the impact of this reject on the environment. The present work produced and activated the charcoal of malt bagasse using phosphoric acid (H_3PO_4), potassium hydroxide (KOH) and sodium acetate (CH_3COONa) as activating agent in a range from 1 to 3% of concentration. The activated charcoal was produced in three particle sizes limit (20, 40 and 60 ASTM) resulting in 11 experiments through full factorial design 2^3. Analysis of yield, apparent density, pH, conductivity and mesoporosity were performed. The average yield obtained was 20.2%. The charcoal with 60 mesh and chemically activated with 1% of sodium hydroxide showed the highest density (0.360 g/cm³), while the average density was 0.257 g/cm³. The mesoporosity analysis indicated that all experiments adsorbed more than 99% of methylene blue (50 mg/L) present in the solution. In this way, the activated charcoal obtained from malt bagasse present characteristics to be an excellent adsorbent.

Keywords: activation; adsorbent; brewing industry residue; dye

1. Introduction

The textile sector presents great difficulty in carrying out the treatment of its effluents due to the high costs imposed, when opting for an alternative with lower cost, it can occur that the treatment is not so effective, and consequently the dyes are discharged into water resources. The presence of dyes in the effluents is highly visible, affects aesthetics, water transparency and the solubility of gases in the receiving bodies, also reducing the capacity of regeneration of the water bodies due to the reduction of the penetration of sunlight and consequent alteration of the processes of photosynthesis [1].

The adsorption presents as an alternative method in the treatment of effluents containing dyes, as it makes use of adsorbent materials of high adsorptive capacity such as activated carbon [2]. Due to adsorption capacity, activated carbons are very useful in separation of mixtures and purification of liquids [3]. Charcoal is a carbonaceous material with a specific porous surface and provides an internal surface area ranging from 80 m²/g to 1200 m²/g [4]. Activated carbon has a well-developed pore structure and high adsorption capacity is mainly associated with the size distribution of pores, surface area and pore volume [5].

However, the use of charcoal in the process makes the process costly, one of the alternatives for adsorption is the utilization of biomass. The application of biomass fits perfectly into the concept of sustainable development, in addition to it reduce costs and do not harm the environment [6]. Bagasse is a by-product of the brewing industry; thus, it is a low-cost raw material. It has characteristics to be a good adsorbent because it has high carbon content and it presents as raw material with great potential for production of activated carbon [7].

The objective of this work was to characterize

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charcoal obtained from 20, 40 and 60 ASTM malt bagasse activated chemically with phosphoric acid, potassium hydroxide and sodium acetate at concentrations from 1 to 3% by means of a Full Factorial Design 2^3

2. Results and Discussion

Characterization of the charcoal

The results of charcoal characterization can be seen in Table 1 below.

Table 1. Characterization of the activated carbon obtained through Full Factorial Design 2^3.

	Variables			Responses			
Assay	X1	X2	X3	Density (g/cm³)	pH	Conductivity (μs/cm)	AMI (mg/g) (%)
1	-1	+1	+1	0.256	3.68	134	4.999 (99.99%)
2	-1	+1	+1	0.184	3.34	144	5.000 (100%)
3	+1	-1	+1	0.277	10.22	838	4.991 (99.99%)
4	+1	-1	+1	0.178	9.91	778	4.936 (99.93%)
5	-1	+1	-1	0.269	3.93	130	4.992 (99.99%)
6	-1	+1	-1	0.214	3.86	58	5.001 (100%)
7	+1	-1	-1	0.360	9.92	616	4.984 (99.98%)
8	+1	-1	-1	0.299	9.53	565	4.949 (99.94%)
9	0	0	0	0.280	6.70	120	5.001 (100%)
10	0	0	0	0.278	6.72	115	4.998 (99.98%)
11	0	0	0	0.277	6.71	118	5.002 (100%)

The average yield was 20.2%. The density of the coal ranged from 0.178 to 0.360 g.cm^{-3}, at a mean of 0.257 g.cm^{-3}. As expected, the H_3PO^{4-} activated samples showed acid character, as well as those activated with KOH had a basic character, the activated carbon with $C_2H_3NaO_2$ remained neutral in the range of zero biomass loading point previously performed. The highest and lowest conductivity tests were, respectively, the tests 3 (60 ASTM, KOH, 3%) and 6 (20 ASTM, H_3PO_4, 1%).

Moletta [8] characterized amylaceous biomass activated carbon with 10% H_3PO^{4-} and obtained an AMI of 78.9% for neutral pH and 70.4% for acidic pH. Thus, the activated carbon obtained from the malt bag presented better results for this parameter, since for both acid and neutral pH, it showed removal of up to 99%.

The mesoporosity analysis indicated that all assays adsorbed more than 99% methylene blue present in the solution. Analysis of Variance (ANOVA) was performed considering significant parameters with p-values less than 5% ($p < 0.05$), shown in Table 2. The ANOVA indicated an explained percentage of variation of 99.48% and a value for F_{calc} well above the tabulated value (8.81). The value for R^2 suggests that this model is adequate for evaluating the behavior of the particle diameter with regard to variations in flow.

Table 2. ANOVA of the methylene blue index.

SV	SQ	DF	MS	F_{calc}
Regression	0.005117879	9	0.000568653	82.25
Waste	2.66667E-05	3	8.88889E-06	
Total	0.005144545	12		

SV: source of variation, SQ: sum of squares, DF: degrees of freedom, MS: middle square, R^2: 99,48, $F_{9,3;\,0,05}$: 8.81

The value of $F_{calculated}$ for the regression was significant and 9.3 times greater than the $F_{tabulated}$. The Equation 1 describes the performance predicted by the model as a function of the coded variables in the reduced model that contains only the statistically significant terms, where X_1 = grain size, X_2 = activator and X_3 = concentration. It can be seen that the dye removal (Y) was affected by all the independent variables.

$$Y = 5.00 - 0.01X_1 - 0.018X_1^2 - 0.017X_2 - 1.1\times10^{-16}X_3 - 0.013X_1X_2 - 0.004X_1X_3 \quad (1)$$

Figure 1 presents the experimental results versus the results predicted by the fitted model, showing good agreement between both. A regression line is a straight line that describes how a response variable y changes as an explanatory variable x changes [9]. The percentage of variation (R^2) explained by the model was high indicating that the model conforms to the experimental data (99.48%), as shown in Figure 1. The closer to 1 the value of R is, the more results obtained experimentally are in agreement with the results of the model, indicating that this model can represent the experimental analysis.

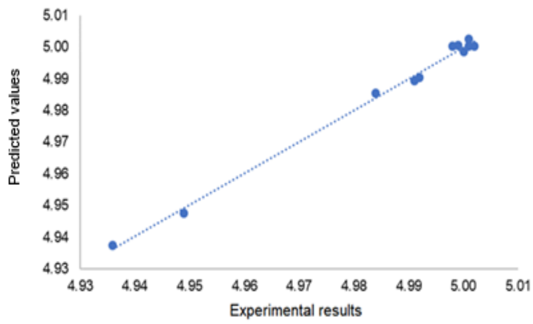

Figure 1. Experimental values versus values predicted by the model.

According to Moreno [10] the cross-section of the methylene blue molecule is approximately 0.8 nm. Therefore, it is more accessible to the mesoporos region. Thus, the AMI can be used to estimate the mesoporosity of an activated carbon.

2. Material and Methods

Adsorbent: malt bagasse was supplied by the micro brewer Schaf Bier, located in the city of Francisco Beltrão, Paraná, Brazil. For the preparation of the raw material, it was washed with tap water to remove the impurities, followed by drying in an oven. After grinding of the grains using a knife mill, different grades (20, 40 and 60 ASTM) were obtained.

Carbonization: was done using malt bagasse in the 20, 40 and 60 ASTM granulometry. Five grams of biomass were left in porcelain capsules previously dried in an oven for 24 hours at 60°C. The capsules were placed in muffle at 350°C were held for 1 h.

Activation: was done through a Full Factorial Design 2^3 according to Table 1, with three central points, totaling 11 assays to study the effect of granulometry (20, 40 and 60 ASTM), the activators (phosphoric acid, potassium hydroxide and sodium acetate), and their concentrations (1, 2 and 3%).

Physical activation: the charcoal samples were placed in previously dried crucibles and taken to the muffle at 200 °C for 1 hour.

Apparent density: determined by the "packaged" weight / volume ratio of the activated carbon in a test tube. The sample was introduced in a pre-weighed 10 mL beaker, subjected to slight lateral impacts, until no variations were observed in the compacted volume. Then the set was weighed into an analytical balance. The weighted instead mass of the coal was divided by the measured volume, to calculate the apparent density of each sample in $g.cm^{-3}$ [11]

pH: 0.1 g of activated charcoal was weighed into a 125 ml Erlenmeyer flask, then 10 mL of distilled water was added. The mixture was boiled for 5 min. After cooling to room temperature, another 10 mL of distilled water was added, to then measure the pH of the suspension using pH meter [11].

Conductivity: the method described by Ahmedna [12] was used. A 1% (w/w) solution of the charcoal was initially prepared in water under stirring for 20 min at 25 °C. The analysis were performed in a conductivity meter.

Mesoporosity - methylene blue index (AMI): the AMI determination means the mass of methylene blue removed per mass of coal used and is based on the methodologies of Mocelin [13] and Moreno [10]. The method consists of using a mass of 0.1 g of coal in 10 mL of 50 $mg.L^{-1}$ solution of methylene blue and subjecting this mixture to stirring on the stirrer table at 160 rpm until the equilibrium concentration is established. After 24 h, the supernatant is centrifuged at 3000 rpm for 20 min. The remaining concentration of the methylene blue is obtained from the spectrophotometer reading at 665 nm. From Equation 2 the calculations of the mass of methylene blue adsorbed per gram of activated carbon are carried out.

$$AMI\ (mg.g^{-1}) = 50\ (mg.L^{-1})*0.1(L) - AMr\ (mg) \quad (2)$$

On what:

AMI = milligrams of methylene blue adsorbed per one gram of charcoal;

AMr = milligrams of methylene blue remaining in solution.

4. Conclusions

From the obtained results, it is verified that malt bagasse presents the necessary characteristics to be used as a viable raw material to obtain activated carbon and to be used as an excellent adsorbent, considering that it is a residue easily found, with lower cost compared to commercially available charcoal.

In the methylene blue index analysis column, all tests showed removal above 99%, indicating that the porosity of the charcoal is able to adsorb the dye, in addition to the activated charcoal with sodium acetate presented neutral pH, not requiring the demand of reagents to neutralize the effluent which would make the process more expensive.

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