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Production and characterization of water-steam activated carbons made from sugarcane bagasse and molasses pellets

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ABSTRACT. Pellets made from sugarcane bagasse and molasses in weight proportions 1.0:0.5 and 1.0:1.0 (wt%) were water-steam activated during 1, 2 and 4h, at 850°C, by two processes, namely, (1) pyrolysis and activation in two different assays or (2) pyrolysis and sequenced activation in a single experiment. All carbons showed basic characteristic, with approximately pH 10.5. Microporous texture was predominant with negligible macroporosity. Longer activation time provided a larger specific surface area with maximum BET 824.30 $m^2 g^{-1}$. In fact, the stage process was more effective than the sequenced one for the same activation time. Although pore size distribution (PSD) analysis showed pore size mode of approximately 1.50 nm, a second mode about 0.8 - 0.9 nm developed in over-two-hour activation periods in the stage process.

Keywords: specific surface area, BET, DR, pore size distribution, DA, HK.

Produção e caracterização de carvões ativados com vapor de água, originados de *pellets* de bagaço e melaço de cana-de-açúcar

RESUMO. *Pellets* constituídos por bagaço e melaço de cana-de-açúcar nas proporções em peso de 1,0:0,5 e 1,0:1,0 foram ativados com vapor de água, por 1, 2 e 4h, a 850°C, através de dois processos: pirólise e ativação em dois experimentos diferentes ou um experimento único com pirólise e ativação em sequência. Os carvões produzidos apresentaram caráter básico com pH em torno de 10,5. Texturalmente, são carvões com predomínio de microporosidade e macroporosidade praticamente desprezível. Quanto maior o tempo de ativação, maior a área superficial específica, chegando ao valor máximo BET de 824,30 $m^2 g^{-1}$ sendo o processo em etapas mais efetivo do que o sequencial. Os valores modais do diâmetro dos poros obtidos no estudo da distribuição de tamanho de poros ficaram em torno de 1,50 nm, e, no processo em etapas, houve o desenvolvimento de uma segunda moda em torno de 0,8 - 0,9 nm, para tempos de ativação acima de 2h.

Palavras-chave: área superficial específica, BET, DR, distribuição de tamanho de poros, DA, HK.

Introduction

According to ROY (1995), activated or active carbon (AC) is a microcrystalline non-graphite carbon processed to develope internal porosity and thus to increase the specific surface area and the volume of pores. In fact, carbons are excellent adsorbents for purifying, filtering, bleaching, deodorizing, de-chlorination, de-intoxication, removal or modification of taste and the concentration of liquid and gas materials. The above applications are relevant to several economic sectors within different areas, such as food, pharmaceutical, chemical, oil, mineral, nuclear, car industries. Treatment of drinking and industrial water and of atmospheric air should be specially mentioned.

Several agricultural products such as sugarcane bagasse, coconut and nut shell, rice straw and husk, soybean husk and babasu palm coconuts may be transformed into activated carbon as a low cost alternative to commercial carbon adsorbents (AHMEDNA et al., 2000a). Sugarcane bagasse has been the focus of much interest. Trials with different activation techniques have been undertaken on the preparation and characterization of activated carbon from sugarcane bagasse (AHMEDNA et al., 2000a and b; BERNARDO et al., 1997; EMMERICH; LUENGO, 1994; ERLICH et al., 2005, 2006; GONÇALVES et al., 2006; PENDYALL et al., 1999a and b).

The characteristics of the final product may be different since it depends on prime matter, activating agent and conditions of activation process. Consequently, activated carbons vary in total specific surface rates, size and distribution of pores.

Activation process which may or may not include the pre-treatment of samples is determined by heating and final temperature rates in the process employed. Water steam, carbon dioxide and oxygen (air) are the most frequently employed activating agents in physical activation.

Current paper aims to the characterization of carbons produced from a mixture of sugarcane bagasse and molasses foregrounded on Gonçalves et al. (2006) but replacing activating agent (CO_2) by water steam.

Material and methods

Prime matter

Sugarcane bagasse and molasses (2006 harvest) used in carbon preparations were provided by the Usina Santa Terezinha, in Iguatemi, Maringá, Paraná State, Brazil. They were dried in an oven at 105°C for 1h to avoid fungus proliferation. Sugarcane bagasse was then sieved with a 10 mesh (1.68 mm) and conditioned in a hermetical sealed container prior to its use in the fabrication of pellets. Sugar cane molasses was stored in a fridge at –10°C to avoid degradation.

Preparation of pellets

Dry bagasse was added to the molasses to obtain mixtures with mass ratios bagasse:molasses of 1.0:0.5 and 1.0:1.0. Mixtures were then dried in an oven at 105°C for 1h and placed in a desiccator. Pellet manufacture was undertaken by compacting approximately 2 g of the mixture in a hydraulic press at a pressure of 850 kgf (83.36 MPa) during 30 seconds, by a cylindrical stainless steel pastille machine with internal diameter 2.0×10^{-2} m and 5.0×10^{-2} m long. Height of pellets was approximately 0.5×10^{-2} m.

Experimental module

Pyrolysis and pellet activation as subsequent stages were undertaken in an experimental module with discontinuous, fixed bed reactor manufactured at the Department of Physics of the State University of Maringá (DFI/UEM), Maringá, Paraná State, Brazil.

Figure 1 shows the module with the sample in a quartz tube with open extremities, which had been inserted in a horizontal electric furnace. Type K thermocouple is placed together with the sample and connected to a PID temperature controller. A system of valves locked to the reactor's entrance provides nitrogen or nitrogen with water steam to the sample. Water steam is produced in an electric boiler with a power rheostat and a rotameter controls nitrogen flow. Two Kitasato flasks are installed in series at the reactor exit. The first collects condensed volatile products released in the pyrolysis and activation processes while the second, filled with water, maintains the system sealed and at local atmospheric pressure. A flexible tube installed at the exit of the second flask releases the non-condensed volatile products.

Pyrolysis assays with pellets

Groups of about 30-32 g of pellets (16 pellets) were submitted to pyrolysis assays carried out under room temperature and pressure N_2 flow rate of 150 mL min.$^{-1}$ up to 850°C at a heating rate of 14°C min.$^{-1}$ and a soaking time of 1 hour.

Samples were cooled while in the furnace, at inert atmosphere, up to 100°C. They were then removed and placed in the desiccator till room temperature was reached. The samples were then weighed and their pyrolyzed masses obtained. Masses of condensed volatile products in the collector were determined at the end of the process.

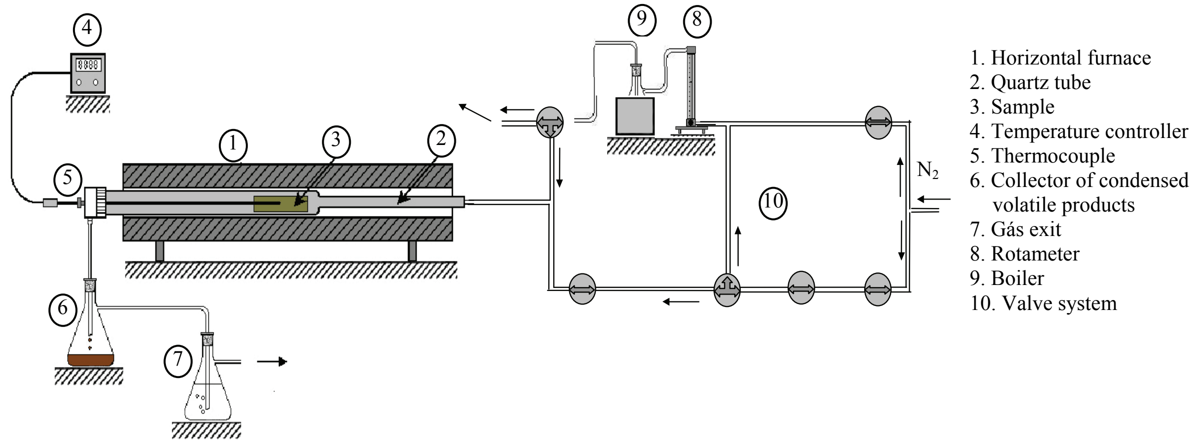

Figure 1. Scheme of the horizontal reactor utilized for the production of pyrolyzed and activated carbon.

Activation assays of pyrolyzed pellets

Carbons from the pyrolysis process were dried in an oven at 105°C during 12h, weighed and reintroduced into the reactor. The gasification of the pellets was carried out at 850°C with a room temperature and pressure N_2 flow rate of 250 mL min.$^{-1}$ bubling through water at 67°C and activation time of 1, 2 and 4h.

Mass determination of the activated carbons was done after cooling the samples in the furnace under inert atmosphere, up to 100°C and in a desiccator till room temperature was reached.

Pyrolysis and sequential activation assays

In these assays immediately after the pyrolysis soaking time (1h at 850°C) started the steam water activation, for periods of 1, 2 or 4h. Cooling process was similar to that described above but with the sole assessment of final mass after activation.

Activated carbons produced

Four types of activated carbons, totaling 24 samples, from different mass ratios and type of assay, were produced in duplicate. Figure 2 shows the sequence of assays in current experiments and explains the nomenclature used for each type of activated carbon produced.

Determination of pH of activated carbons

An Erlenmayer flask with 50 mL of neutralized distilled water (pH = 7.0) and 0.5 g of carbon (dried basis) was kept shaking during 20 minutes in a thermostatized bath (Dubnoff – TE 053 TECNAL) at 90ºC. When the solution cooled, pH was read by a pHmeter DIGIMED DM-22.

Textural characterization of activated carbons

The porosity study of activated carbons was based on the analysis of physical adsorption isotherms of N_2 (77 K). Activated carbons were crushed and sieved in order to obtain particle sizes between 7 and 16 mesh, the most common granulometry of commercial activated carbons. After vacuum dried at 110ºC, they were analyzed in a Quantachrome adsorptometer, model NOVA-1200, of the Department of Chemical Engineering of the State University of Maringá (DEQ/UEM), Maringá, Paraná State, Brazil.

Autosorb for Windows® (1.19 version) was used to determine specific surface areas and pore size distribution according to methods: BET – Brunauer–Emmett–Teller (BRUNAUER et al., 1938), DR – Dubinin–Radushkevich (DUBININ; RADUSHKEVICH, 1947), BJH – Barrett–Joyner–Halenda (BARRETT et al., 1951), t–plot (HALSEY, 1948), DA – Dubinin–Astakhov (DUBININ; ASTAKHOV, 1971) e HK – Horvath–Kawazoe (HORVATH; KAWAZOE, 1983).

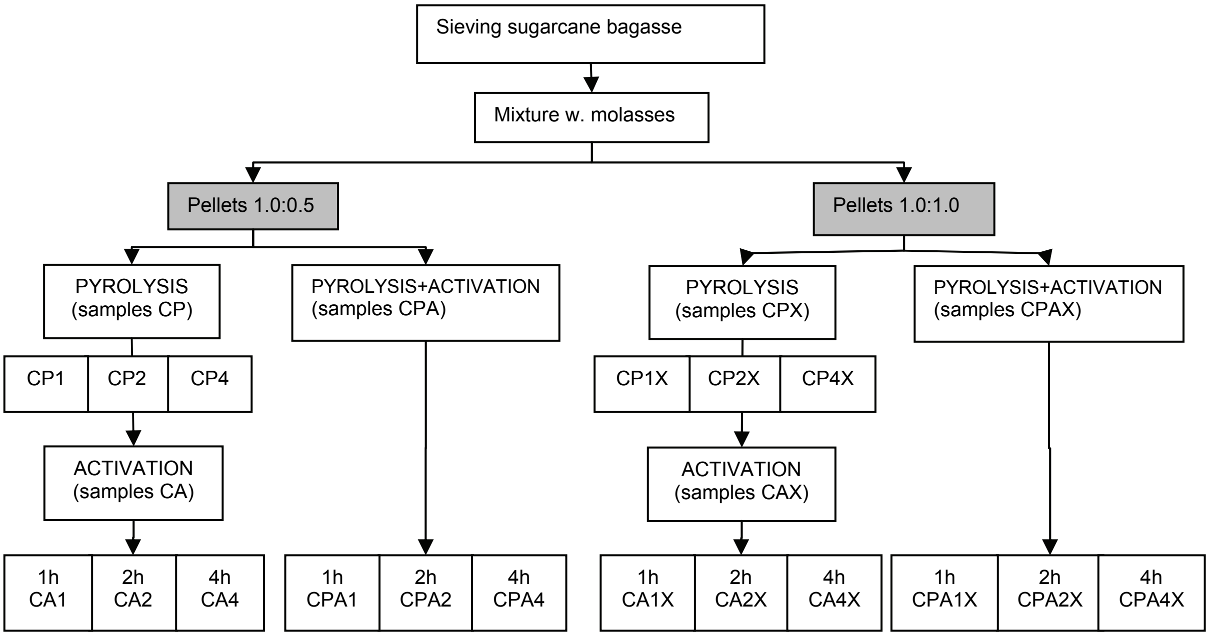

Figure 2. Diagram of pyrolysis and/or activation assays with nomenclature of samples produced.

Results and discussion

Mass yield of pyrolysis and activation processes

Pyrolysis of four sample groups with different mass ratios and/or obtained from different processes (samples CP and CPX, CA and CAX - Table 1) yielded between 18.0 and 27.6% of carbonized material being slightly higher for the samples 1.0:1.0 (samples X). Yield difference of condensed volatile products among these same groups was slightly higher and was due to a larger quantity of molasses of samples X. It has been verified that, in all cases, the higher the activation time, the lower is the mass yield.

There was no significant difference in mass yield values of activation comparing the non-sequential and sequential assays (Table 1) since the pyrolysis stage, undertaken to eliminate all volatile products, was the same for both processes.

Table 1. Yields from pyrolysis and activation processes (RP = yield of carbonized material; RC = yield of condensed volatile products; RA = yield of activated material; RT = total yield; R = yield of activated carbon; RCs = yield of condensed products).

Activation of pyrolyzed pellets						Sequential activation		
Pyrolysis			Activation					
Sample	RP (%)	RC (%)	Sample	RA (%)	RT (%)	Sample	R (%)	RCs (%)
CP1	25.54	43.36	CA1	93.64	23.92	CPA1	25.51	43.27
CP2	26.47	36.09	CA2	82.57	21.86	CPA2	23.92	40.50
CP4	26.79	38.70	CA4	67.22	18.00	CPA4	18.74	47.58
CP1X	26.94	44.14	CA1X	91.27	24.59	CPA1X	25.99	40.39
CP2X	27.58	40.69	CA2X	88.53	24.42	CPA2X	24.86	36.20
CP4X	27.27	41.49	CA4X	73.56	20.07	CPA4X	21.51	39.26

RP= 100 x (final pellets mass/initial mass). RC= 100 x (final mass of condensed products/initial mass). RA= 100 x (final mass of activated products/final mass of pellets). RT= RC x RA. R = 100 x (final mass of activated products/initial mass). RCs= 100 x (final mass of condensed products/initial mass).

In similar experiments with CO_2 as activating agent, Gonçalves et al. (2006) obtained mass yields for samples with bagase/molasses ratio 1.0:0.5 of approximately 22 and 15% respectively for 2 and 4h of activation. These results were similar to the 22 and 18% obtained in the current experiments. This fact showed that there was no significant influence of the activating agent in the production of activated carbons from mixtures of sugarcane bagasse and molasses.

The sum of yield rates of activated carbons and condensed volatile products (RC + RT and R + RCs), of approximately 65%, showed that the release of non-condensed volatile products occurred during the two types of experiment. These products could be also condensed at temperatures lower than room temperature.

The experimental procedures utilized allowed to verify that mass yields of activated carbons from sugarcane bagasse pellets with molasses as a binding factor did not depend on the activating agent and on the type of activating assay undertaken. They rather depended slightly on the quantity of molasses and were directly dependent on activation time.

pH of activated carbons

Table 2 shows that pH rates for different samples had slight variations, ranging between 10.35 and 10.57. Regardless of the activation process, in samples with ratio bagasse mass:molasses at 1.0:0.5, pH increased with an increase in activation time, similar to results obtained by Gonçalves et al. (2006). Such correlation is not so clear in the case of other samples.

The smaller values, between 9.61 and 10.29, determined by Gonçalves et al. (2006) showed that the change from CO_2 to steam water as activating agent produced carbons with more pronounced basic characteristics.

Table 2. pH values of different samples of activated carbon.

Sample	pH	Sample	pH	Sample	pH	Sample	pH
CA1	10.35	CPA1	10.35	CA1X	10.36	CPA1X	10.37
CA2	10.45	CPA2	10.45	CA2X	10.41	CPA2X	10.46
CA4	10.53	CPA4	10.57	CA4X	10.41	CPA4X	10.42

Textural characteristics of activated carbon

N_2 adsorption isotherms of activated carbons (Figure 3) have the characteristic form of Type 1 isotherms, according to BDDT classification (BRUNAUER et al., 1940), in which small micropores are filled at very low relative pressures. The variation of isotherm curves demonstrated that increase in activation time provided a progressive development of smaller micropores showed by the curve flection increase from 1h towards 4h of activation.

Specific surface area of activated carbons was analyzed by the methods t-plot, BET, DR and BJH applied to the adsorption curve (Table 3). Results for micropores by the methods t, BJH and DR vary in a similar and coherent manner with that of total specific surface area determined by BET (Figure 4). The two-stage activation process (samples CA) was more effective than the sequential one (samples CPA), particularly for lower activation periods. The CA total surface areas or micropores areas being higher than the CPA results for the same activation time. Carbons produced from pellets with mass ratio 1,0:1,0 (samples "X") were, as a rule, less activated than the corresponding ones with mass ratio 1.0:0.5.

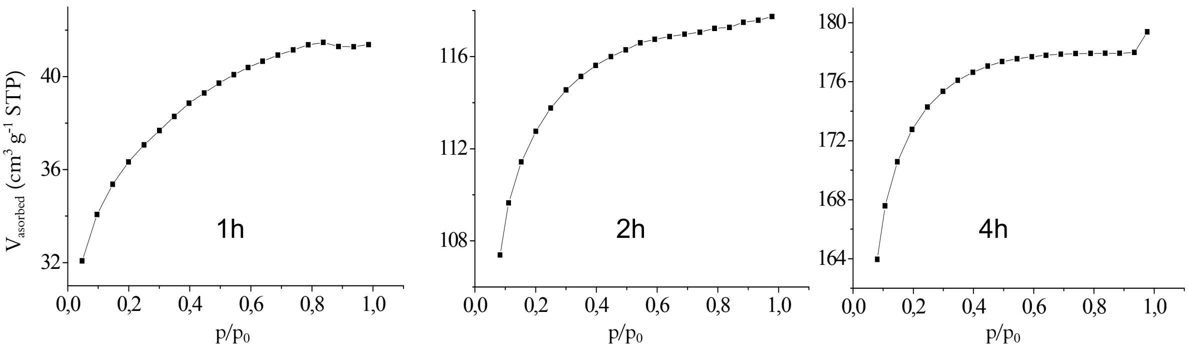

Figure 3. N_2 adsorption isotherms (77K) of sequential series activated carbons.

Table 3. Specific surface areas of pores (methods BJH, DR, BET and t).

Sample	BJH							DR	BET	t–plot Method			
	Total	Microp.		Mesop.		Macrop.		Microp.	Total	Microp.⋆		External	
	(m^2 g^{-1})	(m^2 g^{-1})	(%)	(m^2 g^{-1})	(%)	(m^2 g^{-1})	(%)	(m^2 g^{-1})	(m^2 g^{-1})	(m^2 g^{-1})	(%)	(m^2 g^{-1})	(%)
CA1	76.03	48.41	63.7	27.59	36.3	0.03	0.04	224.90	166.30	96.09	57.8	70.25	42.2
CPA1	13.12	11.46	87.4	1.66	12.7	0.00	0.00	56.31	42.87	36.48	85.1	6.39	14.9
CA2	48.85	44.30	90.7	4.55	9.3	0.00	0.00	608.70	450.90	400.00	88.7	50.85	11.3
CPA2	122.90	80.77	65.7	41.93	34.1	0.20	0.16	346.70	260.90	171.60	65.8	89.38	34.3
CA4	131.00	109.30	83.4	21.70	16.6	0.00	0.00	1101.00	824.30	754.80	91.6	69.49	8.4
CPA4	130.10	106.00	81.5	24.00	18.5	0.10	0.08	812.60	615.40	553.30	89.9	62.08	10.1
CA1X	14.96	36.26	66.8	18.01	33.2	0.00	0.00	164.90	125.50	89.56	71.4	35.92	28.6
CPA1X	12.98	7.85	60.5	5.13	39.5	0.00	0.00	41.17	31.21	19.36	62.0	11.85	38.0
CA2X	70.34	50.63	72.0	19.69	28.0	0.02	0.03	508.30	374.60	306.50	81.8	68.10	18.2
CPA2X	86.79	66.59	76.7	20.18	23.3	0.02	0.02	294.90	247.30	204.90	82.9	42.41	17.2
CA4X	113.90	87.73	77.0	25.97	22.8	0.20	0.18	780.00	578.90	504.20	87.1	74.68	12.9
CPA4X	156.90	120.40	76.7	36.50	23.3	0.00	0.00	781.40	591.10	510.40	86.3	80.75	13.7

⋆=(BET – external area).

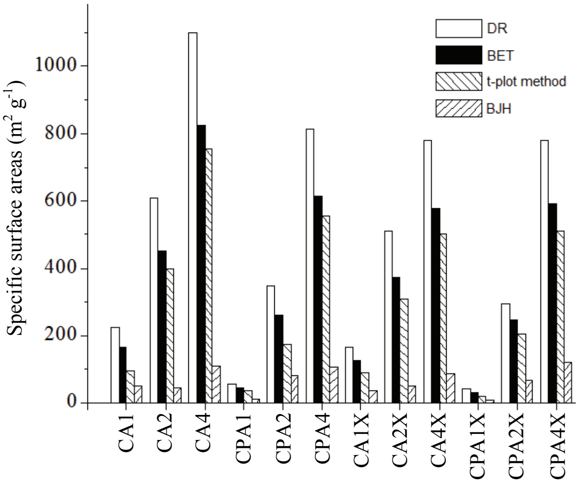

Figure 4. Specific surface areas of micropores DR, t-plot method and BJH, compared with total BET porosity.

BJH method provided smaller values when compared with those obtained by other methods, probably due to the inclusion in its formulation of r_K radius of Kelvin's equation. This radius is related to capillary condensation what is an almost impossible phenomenon in such small pores. Results may, therefore, represent only the more strongly adsorbed part of the adsorbate.

Very low specific surface areas, BET rates 30 - 40 m^2 g^{-1} (samples CPA1 and CPA1X) and 120 - 160 m^2 g^{-1} (samples CA1 and CA1X) indicated slightly activated carbon, or rather, carbons with a slightly developed micro-porous structure. These values are significantly higher for samples with activation times of 2h (BET between 300 - 600 m^2 g^{-1}) and 4h (BET between 580 - 825 m^2 g^{-1}).

The highest values of specific surface area were registered for carbon CA4, produced in two stages with a 4h activation time. Results on specific surface areas by the Dubinin-Radushkevich method always showed higher values when compared with those of other methods (Figure 4) e should be closer to their true value. In fact, it is a specific method for Type 1 isotherms, so it is more adequate to the porosity characterization of micro-porous materials, especially activated carbons (GREGG; SING, 1982).

The BJH (Barret, Joyner and Halenda) method, applied to adsorption isotherm, was also utilized to classify the pores of the activated carbons produced. According to IUPAC classification (SING et al., 1985) pores with a diameter less than 2 nm was classified as micropores; pores with diameter between 2 and 50 mm as mesopores and those with a diameter over 50 nm as macropores.

All carbons had a wide predomination of microporosity over mesoporosity and almost negligible macroporosity (less than 1%).

Microspores' specific surface area was also analyzed by the "t-plot" method which determine the external surface area corresponding to mesopores and, by difference with regard to the area determined by the BET method, calculate the specific surface area of the material's microporous portion. Results corroborate those by BJH method since micropores also predominate, especially in samples with longer activation period (Table 3).

The pore size distribution is another relevant characteristic of activated carbons. The Dubinin-Astakhov (DA) method, which provides data on total distribution of pores, and the Horvath-Kawazoe (HK) method, which details microporosity from the relatively low pressure region, were employed in the analysis of adsorption isotherms. The above methods provide the modal diameter of pores in the carbons under analysis. Results for the pore size distribution by both methods showed average modal values close to 1.50 nm revealing micropores predomination (Table 4).

Table 4. Pore size distribution mode by DA and HK methods.

Samples	DA (nm)	HK* (nm)	Samples	DA (nm)	HK* (nm)
CA1	1.740	1.488	CA1X	1.600	1.417
CPA1	1.480	1.427	CPA1X	1.640	1.452
CA2	1.260	1.527	CA2X	1.480	1.542
CPA2	1.740	1.542	CPA2X	1.480	1.388
CA4	1.400	1.477	CA4X	1.480	1.533
CPA4	1.420	1.433	CPA4X	1.480	1.433
Average	1.51	1.48	Average	1.53	1.46

*micropores distribution only.

Distribution of pores according to DA method was similar among the different types of carbon under analysis. The unimodal distribution, with mode values ranging from 1.400 nm to 1.740 nm, also showed that activated carbons were essentially microporous with small quantities of small mesopores (Figure 5). A decreasing trend in the modal size of pores existed in more activated samples (2 and 4h) when compared to samples activated during 1h. Further, 2 and 4h activated modes related to samples X were the same. Although the method was developed for heterogeneous porosity, recent studies have shown that the Duninin-Astakhov equation described homogeneous and heterogeneous systems (TERZYK et al., 2002).

HK method, which details microporosity, is based on an equation regardless of radius r_K of Kelvin's equation related to capillary condensation. Results of its application showed distribution differences among the employed activation processes (Figure 6). All slightly activated samples (1h) presented unimodal distribution. The sequential process (samples CPA) maintained the unimodal distribution in the activated samples for 2 and 4h, while the two-stage-process (samples CA) brought about a less intense second mode, corresponding to pores with diameter of 0.8 and 0.9 nm for samples, respectively, with mass ratio 1.0:0.5 and 1.0:1.0.

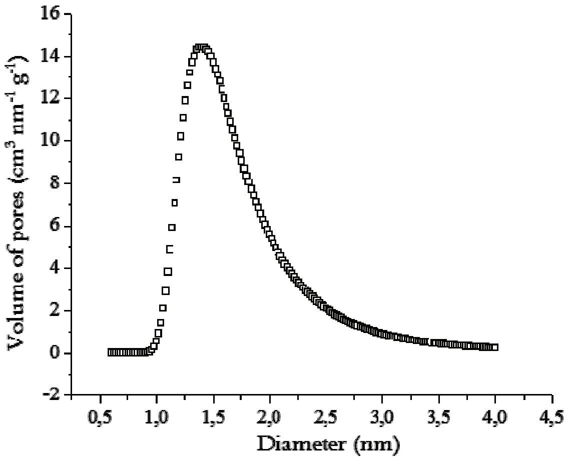

Figure 5. Pore size distribution by Dubinin-Ashtakov method (sample CA4).

Bimodal distribution is best defined in samples CAX. This suggests that greater amounts of molasses may have caused the development of smaller micropores in larger quantities. Second mode in samples CA may explain the higher rates of the specific surface area in these samples when compared to those in CPA (Table 4).

The above characteristic of the analyzed activated carbons may condition its further utilization since the smaller pores may be inaccessible and depend, for instance, on the size of the molecules that should be removed. In this case, a greater total specific surface area only will not indicate the highest reactivity of the activated carbon.

The experiments and analysis develloped during this work showed that mass yields of activated carbon from sugarcane bagasse and molasses pellets depend directly on activation time and only slightly on the quantity of molasses. On the other hand, they were not dependent on the activating agent and on the type of activation assay undertaken.

Higher pH values (between 10.35 and 10.57) than those by Gonçalves et al. (2006), between 9.61 and 10.29, indicated that the change of the activating agent CO_2 by steam water produced more basic carbons.

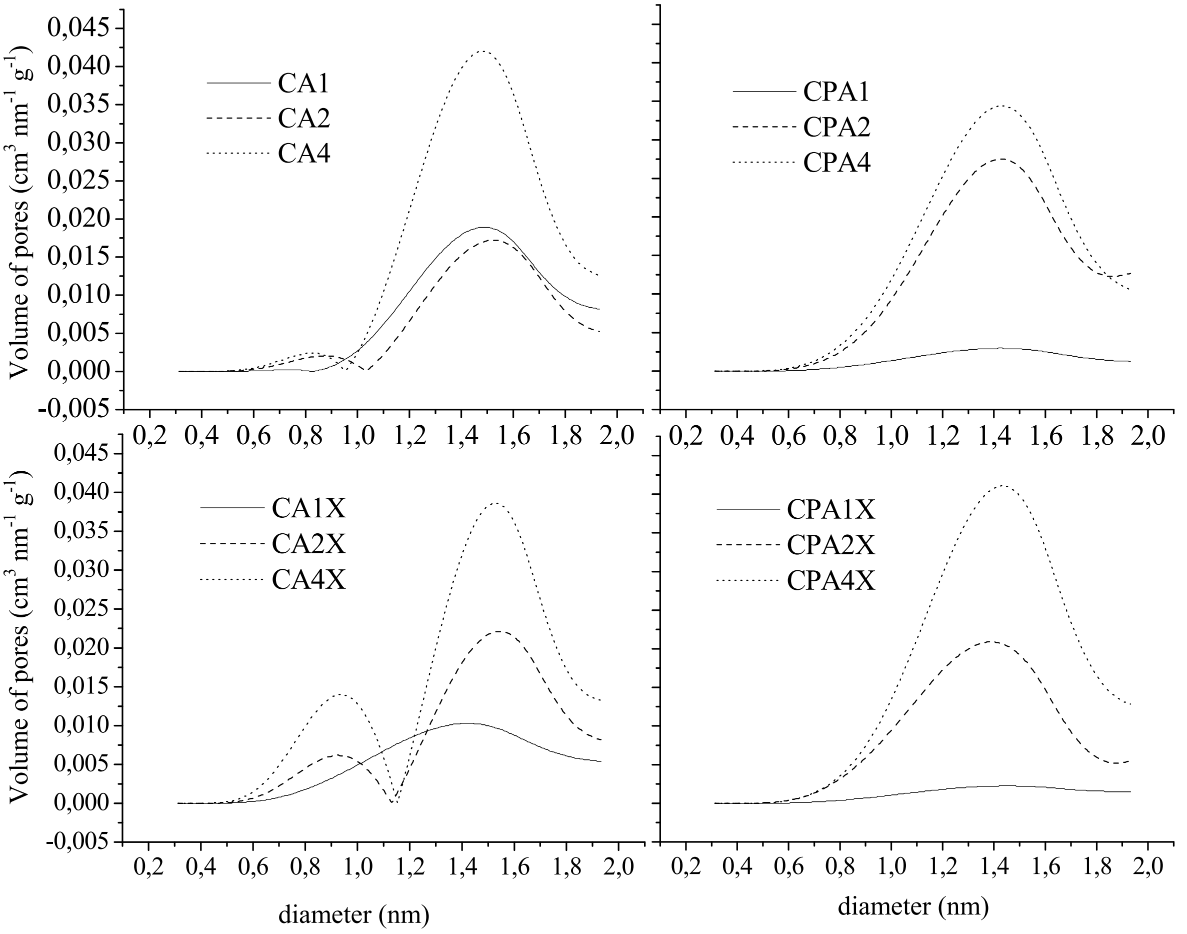

Figure 6. Pore size distribution by the Horvath-Kawazoe method.

N_2 adsorption isotherms were Type 1, with a predomination of micropores. The two stage activation process was more efficient than the sequential one since it promoted the development of greater specific surface areas for the same activation time. The highest values of specific surface area were found in carbon CA4, produced in two stages with a 4h activation.

Pore size distribution with average modal values around 1.50 nm emphasized the predomination of micropores. However, there was a sharp difference in pore distribution among the activation processes. Whereas the sequential process had a unimodal distribution, in the two-stage process the samples activated for 2h and 4h showed a bimodal distribution. The above characteristic coupled to total specific surface area may condition a higher or a lower carbon reactivity and, consequently, its utilization.

Conclusion

Experimental procedures utilized in current work and in by Gonçalves et al. (2006) showed that the use of the by-products of the sugar and alcohol industry, such as sugarcane bagasse and small quantities of molasses, are viable for the manufacture of activated microporous carbons which may later be re-utilized, for example, in the same industry for the bleaching of sugarcane broth or for the adsorption of staining molecules in other liquid media.

Results revealed which experimental parameters studied may be modified in order to obtain activated carbons for specific use. Generally, their use depends on the dimension of the molecule that must be adsorbed and on the selectivity potential of the porous structure that must be produced.

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