

PRODUCTION OF ACTIVATED CARBON FROM SOUTH AFRICAN SUGARCANE BAGASSE

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Abstract

Activated carbons were produced from compressed South African sugarcane bagasse in a pyrolysis furnace by adopting the physical method of processing viz. carbonization followed by partial gasification with steam. Feasible processing conditions were determined by varying the temperature, hold times and partial pressure of steam in the reactor at a heating rate of 10°C/min. Consequently, the char and activated carbons were characterized with respect to iodine number, methylene blue number, molasses number, surface area, pore volume and pore size distribution. Increasing temperature and the partial pressure of steam increased the degree of activation within a smaller residence time thereby producing activated carbons with, for example, a BET surface area of 995 m²/g, iodine number of 994 mg/g, molasses number of 700 and methylene blue number of 256 mg/g. Activated carbons exhibiting the best properties were produced by pyrolysis at 680°C and a hold-time of 1 hour followed by activation with steam at 900°C for 2 hours reaching a 50% burn-off. The resultant activated carbon's ability to adsorb colour from raw sugar was also investigated and compared to a commercial reference carbon. Results reveal that activated carbon produced from sugarcane bagasse has a significant potential in the sugar industry.

Keywords: activated carbon, sugarcane, bagasse, pyrolysis, activation, decolourisation

Introduction

During the 1999/2000 season the South African sugar industry crushed just over 21 million tons of sugarcane and produced 5 million tons of bagasse at 51% moisture. Bagasse is the fibrous waste left after the extraction of sugar juice from crushed cane. This by-product has been used in many processes viz. being burnt as fuel to provide energy for the plant, processed into pulp for papermaking, used as a reactant in the chemical industry, and used as an additive in animal feed. The South African sugar industry also produces refined sugar whereby the process involves the removal of colour from raw sugar. Since bagasse is a highly carbonaceous agricultural byproduct, a natural outlet would be to use bagasse as a feedstock in the manufacture of activated carbons, which can then be used in the decolourisation process. In South Africa, the diffusion process is used to extract sugar from the cane, which gives the bagasse potentially different physical and chemical properties to bagasse produced in other countries.

Activated carbon is an invaluable adsorbent used extensively in industries such as food processing, pharmaceuticals, chemical, petroleum, mining, nuclear, automobile and vacuum manufacturing to purify, decolorize, deodorize, dechlorinate, detoxicate, filter, recover salts and used as catalysts and catalysts supports (Bansal *et al.*, 1988). Granular and powder activated carbons are produced commercially from precursor materials such as anthracite and bituminous coal, lignite, peat, wood, coconut shells, and nutshells (Pollard *et al.*, 1992). Activated carbons are manufactured chemically or physically or by using a combination of both these methods. The physical method is a two-step

process involving pyrolysis of the raw material in an inert atmosphere followed by activation at higher temperatures with an oxidizing gas such as steam or carbon dioxide. The chemical process occurs in one step with the activating agents (ZnCl_2 , H_3PO_4 , H_2SO_4) mixed into the raw material and the end product is washed to remove the excess chemicals.

Many lignocellulosic agricultural by-products have been converted successfully into activated carbons such as rice hulls by Ahmenda *et al.* (1997), Pendyl *et al.* (1999), Yousef and Mostafa (1992), Tekker *et al.* (1997), rice straw by Pollard *et al.* (1992) and bean curd refuse by Muroyama *et al.* (1995, 1996). Jiaan Xia *et al.* (1998) and Higa *et al.* (1989) produced granular activated carbons from sugarcane bagasse by the physical method using steam as the activating agent. Lavarack (1997) and Bernado *et al.* (1997) produced powder activated carbons from sugar-bagasse and illustrated their ability to decolourise raw sugar. Lutz *et al.* (1997) carried out low temperature experiments on sugarcane byproducts and produced high surface area, activated carbons. Pendyl *et al.* (1999) and Ahmenda *et al.* (1997) combined sugarcane bagasse with different binders such as coal tar, corn syrup and molasses and produced granular activated carbons. Their studies revealed that bagasse bound with corn syrup produced activated carbons with the greatest potential to adsorb colour bodies from raw sugar.

The adsorption capacity of an activated carbon depends mainly on its surface area, pore volume and pore size distribution characteristics. The pore sizes are defined as micropores (less than 2 nm), mesopores (2-50 nm) and macropores (greater than 50 nm). The micropores contribute largely to the surface area, whereas the macropores act as channels to the micropore surfaces.

The quality of the activated carbon produced depends much on the type of raw materials used, the processing conditions and unit operations (Gergova *et al.*, 1994; Mackay and Roberts, 1982a and b; Rodriguez-Reinoso and Solano, 1989). According to Paturau (1989), the composition of bagasse varies with respect to the variety of cane, its maturity, the method of harvesting and finally the efficiency of the milling plant. Hence, activated carbons produced from sugarcane bagasse differ from country to country.

The objective of this study was to produce activated carbons from South African sugarcane bagasse, characterize them, determine their sugar decolorizing ability and compare them to a commercial carbon.

Materials

The sugarcane bagasse and molasses were obtained from Illovo Sugar Mill located in Sezela in South Africa. Sugar used for binding was commercial Illovo refined sugar. The commercial carbons that were used as reference carbons were Norit PN2 (peat parent material, steam activated, powder) and Chemviron CaneCal (coal parent material, patented activation, granular) carbons.

Methods

After viewing the ultimate use of the resultant products and the availability of chemicals and utilities at local sugar mills and refineries, the physical method of manufacture was chosen, with steam as the activating agent. Both pyrolysis and partial gasification take place in the same reactor on a semi-batch scale.

Raw material preparation

As-received sugarcane bagasse was dried at 110°C to remove moisture and crushed for 15 minutes in a food processor giving rise to bagasse particles with the following average size distribution (Table 1) having an average diameter of 420 μm .

Table 1. Size Distribution of crushed bagasse.

| Size range / μm | Cumulative mass % |
|----------------------------|-------------------|
| + 1000 | 18 |
| + 710 / - 1000 | 26 |
| + 500 / - 710 | 38 |
| + 355 / - 500 | 54 |
| +250 / - 355 | 69 |
| +180 / -250 | 79 |
| - 180 | 100 |

The crushed bagasse samples were compressed into cylindrical pellets using a die and piston placed in a hydraulic press and applying a 110 kN force. The resultant pellets were compact having a density between 700-900 kg/m³. In order to produce granular activated carbons, the crushed bagasse was initially mixed with refined sugar prior to being compressed, following work by Evans *et al.* (1999).

Experimental procedure

A schematic set-up showing the reactor and downstream processes is illustrated in Figure 1. The reactor is a 55 mm (i.d.) stainless steel cylindrical pipe surrounded by a ceramic insulation layer. The energy required for the process is provided by a 4 kW heating coil which is embedded in the ceramic insulation. A wire mesh basket connected to a mass balance via a ceramic rod sits in the hot zone of the reactor. All inlet and exit pipes to the reactor are heated to prevent condensation of the gases. The temperature is controlled via a thermocouple linked to a PID temperature controller.

The bagasse pellets were inserted into the wire mesh basket. During pyrolysis nitrogen gas entered the bottom of the reactor through the balance housing and into the hot zone of the furnace to provide an inert atmosphere. The sample was pyrolysed at a heating rate of 10⁰C/min until the desired temperature was reached and held at constant temperature for a specific hold time. The exit gases leaving the reactor were cooled to room temperature using ice as the cooling medium. The gas/liquid medium was separated and the gas further cleaned to remove tarry matter before entering a gas analyzer or being vented. Char with a rudimentary pore structure is left behind in the wire mesh basket.

The resultant chars were then subjected to partial gasification with steam. The steam enters the reactor by bubbling nitrogen gas through a set of dreschel bottles kept at constant temperature of 92°C. Controlling the nitrogen gas flowrate controls the partial pressure of the steam in the reactor. Gases exiting the reactor during this stage followed the same route as in the pyrolysis phase. The activated carbon left behind in the reactor was cooled with nitrogen gas to room temperature before being removed. Variation of the reaction time under constant temperature and partial pressure of steam determined the best activation parameters with respect to surface area, iodine and methylene blue numbers.

The mass and temperature were continuously logged with time in one-second intervals for the entire process i.e. from the time the raw material (bagasse pellets) enters the reactor until the end product is produced.

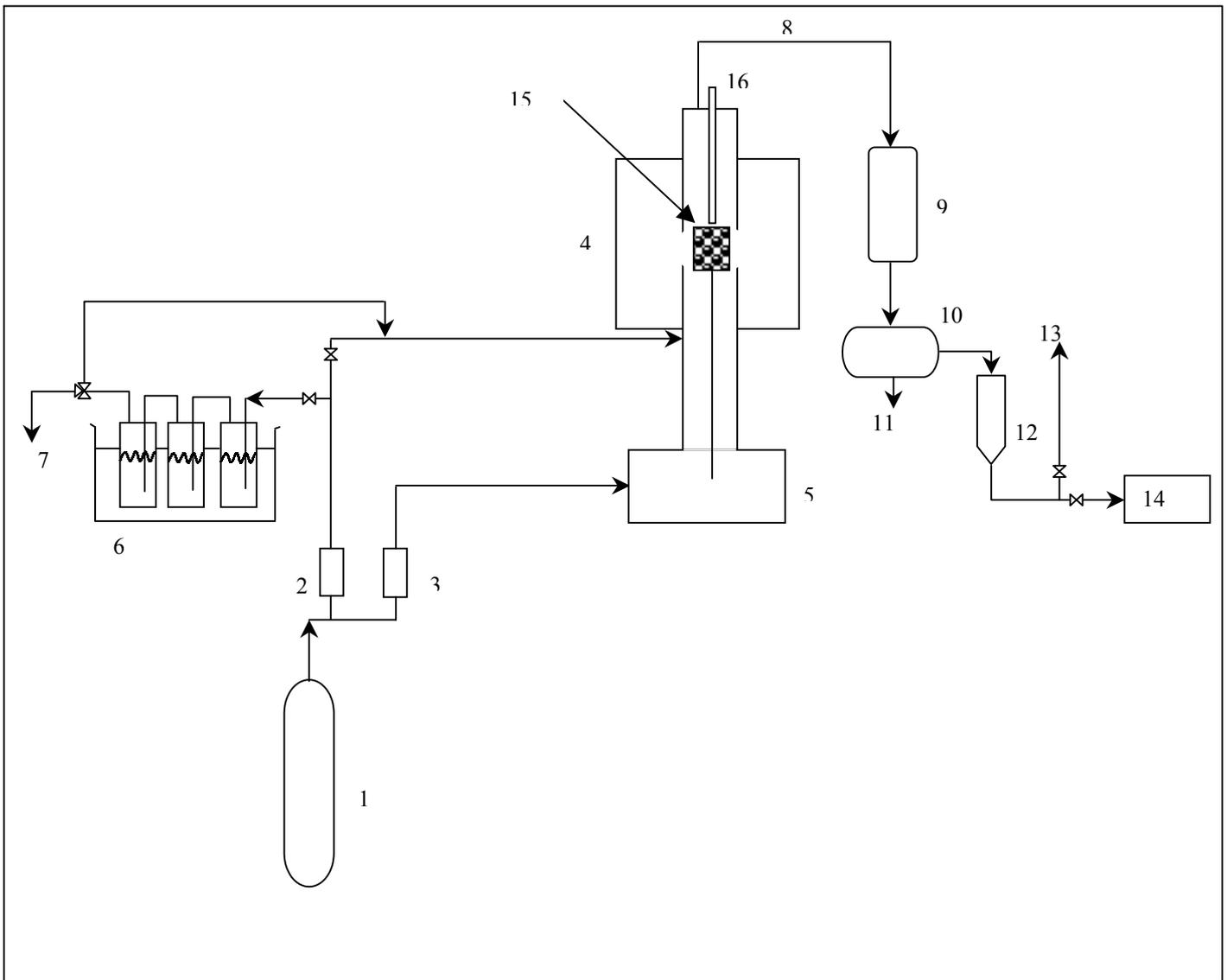


Figure 1. Schematic diagram of the activated carbon process.

1 Nitrogen gas, 2 & 3 rotameters, 4 reactor, 5 mass balance, 6 steam set-up, 7 steam sampling valve, 8 exit gases, 9 condenser, 10 separator, 11 liquid, 12 gas cleaning filter system, 13 gases to vent, 14 gases to gas analyzer, 15 wire mesh basket, 16 thermocouple.

Characterization

Yield and % burn-off

Burn-off is the percentage weight loss of pyrolysed char on a dry basis, which occurs during activation, and yield is the mass of carbon left after pyrolysis and activation (Pendyal *et al.*, 1999). These parameters were evaluated using equations 1 and 2.

$$\text{Burn-off} = [(w_{ba} - w_{aa}) / (w_{ba})] \times 100 \quad (1)$$

and

$$\text{Yield} = [w_{aa} / w_{rm}] \times 100 \quad (2)$$

w_{ba} = dry weight before activation

w_{aa} = dry weight after activation

w_{rm} = dry weight of raw material before carbonization.

Iodine number

Iodine number is the mass (mg) of iodine adsorbed from a standard 0.1 N (0.05 M) iodine solution, when the equilibrium iodine concentration is exactly 0.02 N (0.01 M). The iodine number is a relative indicator of porosity in activated carbons. For high surface area carbons (greater than 900 m²/g), iodine number is numerically similar to BET surface area measurements, whereas for low surface area active carbons, this one-to-one correlation of surface area with iodine number falls away. The iodine number for chars, activated and reference carbons were measured according to the standard test method for activated carbons found in ASTM D4607-86.

Methylene blue number

The methylene blue number is the mass (mg) of methylene blue adsorbed per gram of activated carbon and is determined on pulverized carbon that has been allowed to reach an equilibrium loading with the methylene blue solution. Methylene blue is rapidly adsorbed by the meso- and macropores of an activated carbon, giving an indication of the surface area of the carbon in this pore size range. The procedure illustrated by Aktasorb Systems was used to determine the methylene blue number for chars, activated carbons produced from bagasse and reference carbons.

Surface area, pore volume, pore size distribution and average pore size

Surface area, pore volume, pore size distribution and average pore size were obtained from nitrogen adsorption isotherms at 77K using an ASAP 2010 Surface Area Analyzer. Langmuir, t-plots, BET and BJH models were used to evaluate the above-mentioned characteristics. The BET (Brunauer *et al.*, 1938) equilibrium adsorption equation based on the assumption of multi-layer adsorption shown below (Equation 3) was used in determining the surface area.

$$\frac{1}{W [(P_0/P)-1]} = \frac{1}{W_m C} + \frac{(C-1)}{W_m C} \frac{P}{P_0} \quad (3)$$

Where: W is the weight of nitrogen adsorbed at a given P/P₀
W_m is the weight of gas giving monolayer coverage
C is a constant related to the heat of adsorption.

In terms of volume adsorbed equation 3 is represented by equation 4:

$$\frac{V}{V_m} = \frac{C(P/P_0)}{[1-P/P_0][1+(C-1)(P/P_0)]} \quad (4)$$

By plotting P/P₀ vs. 1/V[(P₀/P)-1] over the range of 0.05 < P/P₀ < 0.35 the quantity of nitrogen gas adsorbed in the monolayer was determined. This was then used to determine surface area (S):

$$S = \frac{V_m \sigma N_A \rho}{MW} \quad (5)$$

Where : σ is the area of an adsorbate molecule

ρ is the adsorbate liquid density

N_A is Avogadro's number (6.022 x 10²³ number/mole)

MW is the molecular weight of the adsorbate molecule (nitrogen).

The Barrett-Joyner-Halenda (BJH) method (Barrett *et al.*, 1951), based on a model viewing the adsorbent as a collection of cylindrical pores, was used to evaluate the pore size distribution. This model takes into consideration capillary condensation in the pores using the classical Kelvin equation, which is based on assumptions of a hemispherical liquid-vapor meniscus and a well-defined surface tension. The BJH theory also includes the thinning of the adsorbed layer through the

use of a reference isotherm, while the Kelvin equation is only applied to the "core" fluid. In the original BJH formulation, the ratio of core radius to pore radius is assumed constant as a simplifying approximation.

Molasses number

The molasses number was used to determine the decolorizing capacity of the activated carbons. A solution of final molasses was treated with the prepared carbons and the reference carbon of known molasses number. The filtrate absorbances were measured and molasses number of the sample was calculated from the ratio of the absorbance values of the sample and the reference carbon.

Raw sugar decolourisation

Varying amounts of activated carbons were added to 66 Brix sugar liquor. The slurry mixture was stirred for 24 hours at 80EC. The samples were filtered to remove the suspended carbon particles, adjusted to pH of 7.00 and colour measured at 420 nm using a UV-VIS spectrophotometer. A blank containing no activated carbon was subjected to the same experimental conditions.

The percent colour removed was determined by:

$$\% \text{ colour removed} = [(\text{colour of blank} - \text{colour of carbon treated sample}) / \text{colour of blank}] \times 100 \quad (6)$$

Results and Discussion

Raw bagasse

Ash and moisture tests were also performed in a muffle furnace and drying oven on the as-received bagasse. Bagasse contained 48% moisture with an ash content of 1.8%. A later sample used had an ash content of 5,4%. An approximated elemental analysis (Table 2) on the dry bagasse was determined using a scanning electron microscope. Scanning electron micrographs (Figure 2) revealed that the surfaces of the bagasse fibres contained an abundance of large pores, which further enhanced its potential as a raw material in producing activated carbons.

Table 2. Elemental Analysis of raw sugarcane bagasse.

| Element | %(dry basis) |
|----------------|---------------------|
| Carbon | 17.89 |
| Oxygen | 75.60 |
| Aluminium | 1.47 |
| Silica | 2.67 |
| Phosphorus | 0.75 |
| Sulfur | 0.07 |
| Potassium | 0.04 |
| Calcium | 0.58 |
| Iron | 0.40 |

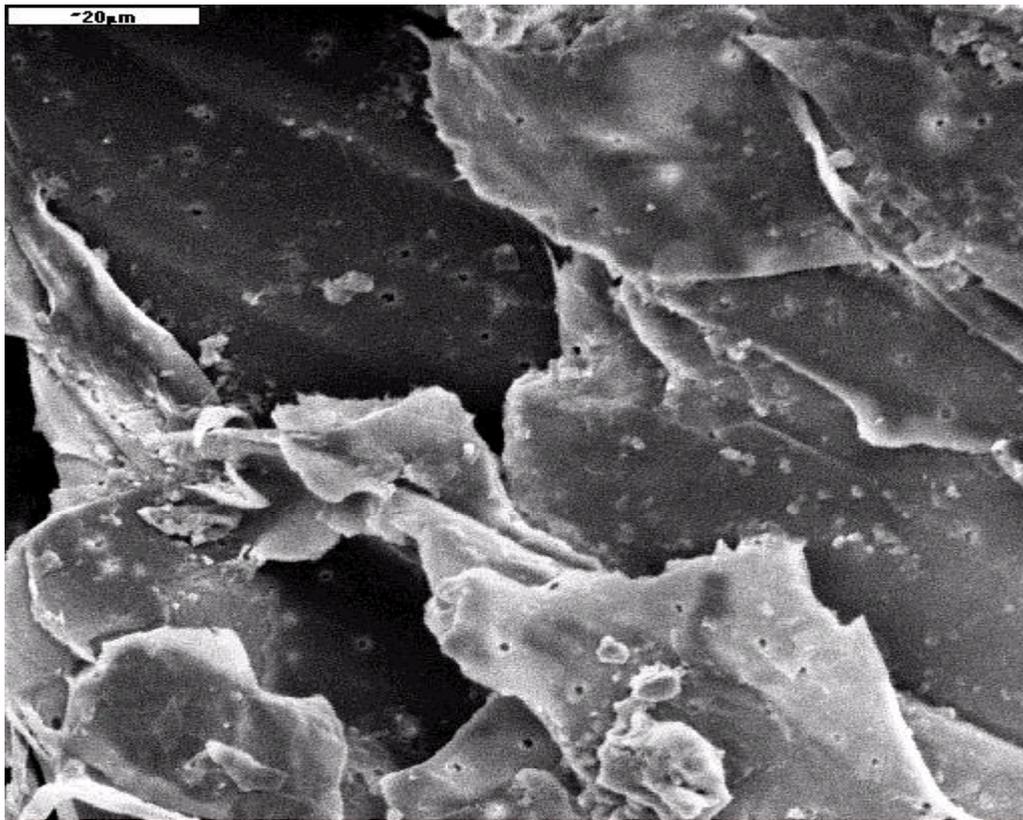


Figure 2. Scanning electron micrograph of raw bagasse showing pores on surface.

Pyrolysis

Bansal *et al.* (1988) reported that the heating rate during carbonisation, the final carbonization temperature and hold time are important factors that affect the pore volume, pore size distribution, surface area and mean pore diameter of the final activated carbon product. The best pyrolysis conditions for South African bagasse were determined with respect to iodine and methylene blue numbers (see Figure 3).

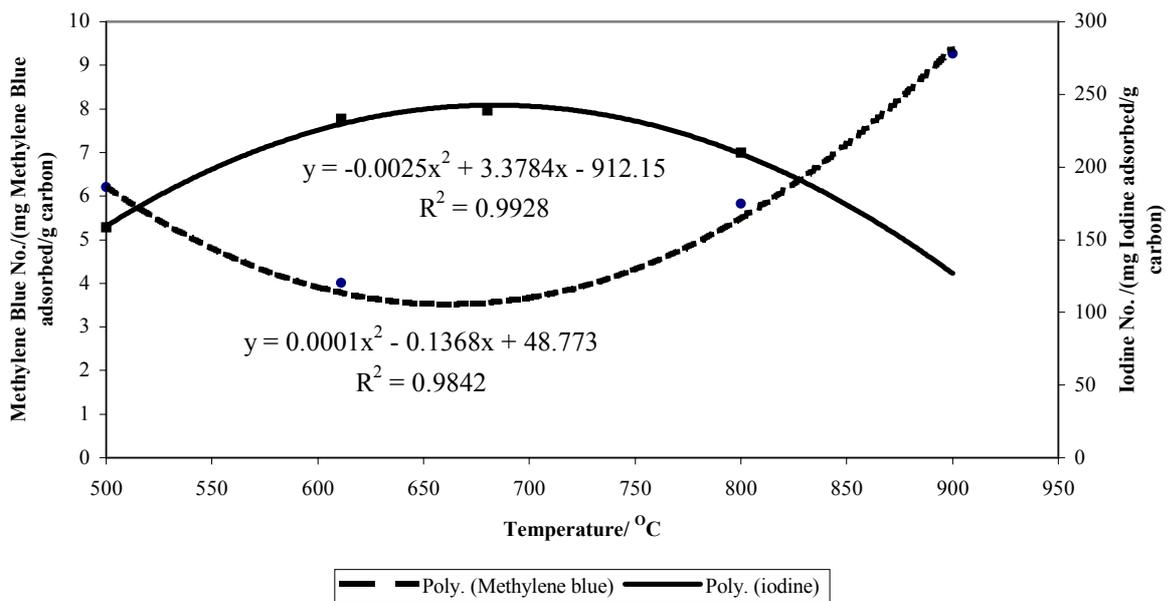


Figure 3. Graph of methylene blue and iodine numbers for carbons produced at a heating rate of 10°C/min at different pyrolysis temperatures held at a hold-time of 1 hour.

Carbon produced at 680°C at a heating rate of 10°C/min and hold-time of one hour had the highest iodine number and lowest methylene blue number indicating the conditions for pyrolysis. Iodine number also gives an indication of the microporosity of a carbon. Thus the carbons with a higher amount of micropores allow for the surface area to be increased during activation as opposed to meso-macroporous carbons (high methylene blue numbers).

The characteristics of the chars produced at the determined pyrolysis conditions can be seen in Table 3.

Table 3. Properties of bagasse char pyrolysed at heating rate of 10°C/min, final temperature of 680°C and hold-time of 1 hr.

| Property | Units | Value |
|---|--------------------|-------|
| Methylene blue No. | mg/g carbon | 3.1 |
| Iodine No. | mg/g carbon | 238 |
| Single point surface area | m ² /g | 331 |
| BET surface area | m ² /g | 320 |
| Langmuir surface area | m ² /g | 423 |
| BJH adsorption cumulative surface area of pores * | m ² /g | 28 |
| BJH desorption cumulative surface area of pores * | m ² /g | 27 |
| Single point total pore volume | cm ³ /g | 0.17 |
| BJH adsorption cumulative pore volume of pores * | cm ³ /g | 0.050 |
| BJH desorption cumulative pore volume of pores * | cm ³ /g | 0.045 |
| Average pore diameter | A | 21.0 |
| BJH adsorption average pore diameter (4V/A) | A | 67.7 |
| BJH desorption average pore diameter (4V/A) | A | 67.8 |

*between 17–3000 A diameter

A= Angstroms

Rather high surface areas were created during the pyrolysis stage.

The profiles of mass and temperature with time (one-second intervals) can be seen in Figure 4. Between 200 and 400°C there is a rapid decrease in mass. During this stage all volatile matter was removed, leaving behind a char with an elementary pore structure. At the end of this stage a 21% yield of char was obtained having the elemental composition given in Table 4. Large amounts of light gases, liquid hydrocarbons and tar were also produced.

Table 4. Elemental Analysis of pyrolysed sugarcane bagasse.

| Element | % (dry basis) |
|----------|---------------|
| Carbon | 22.36 |
| Nitrogen | 30.69 |
| Oxygen | 30.52 |
| Ash | 2.91 |

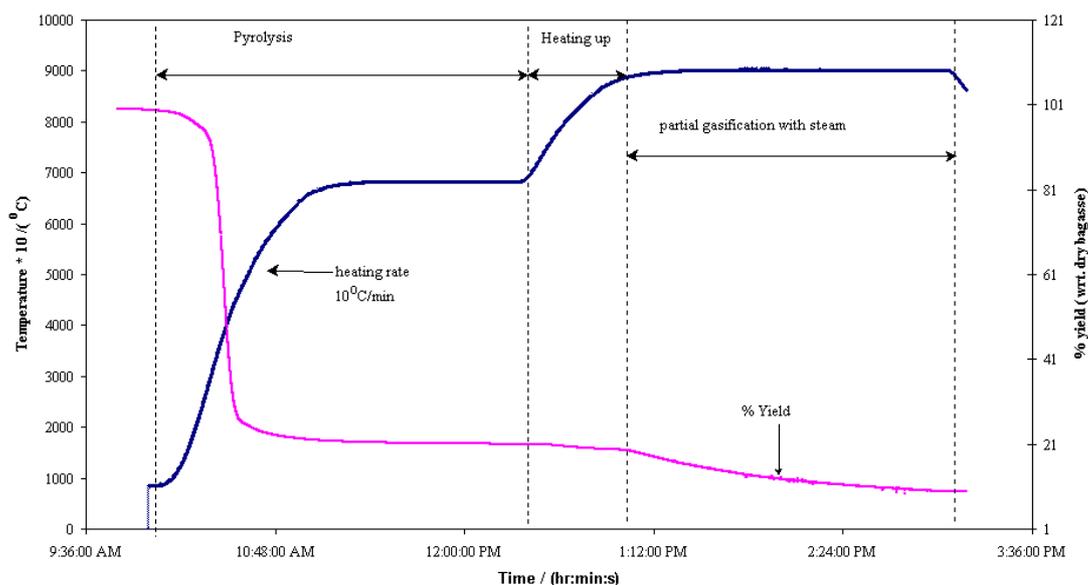


Figure 4. Graph showing temperature and mass profile for pyrolysis and activation processes in the pyrolysis reactor of bagasse.

Activation

The chars produced after pyrolysis were subjected to partial gasification at different constant temperatures, steam partial pressures and hold times. Figure 5 illustrates the effect of the partial pressure of steam in the reactor by varying the nitrogen gas flowrate in the system. This activation experiments were carried out for 4 hours at constant temperature of 800°C. The greater the steam/nitrogen ratio, the higher the methylene blue and iodine numbers and surface area of the activated carbon produced.

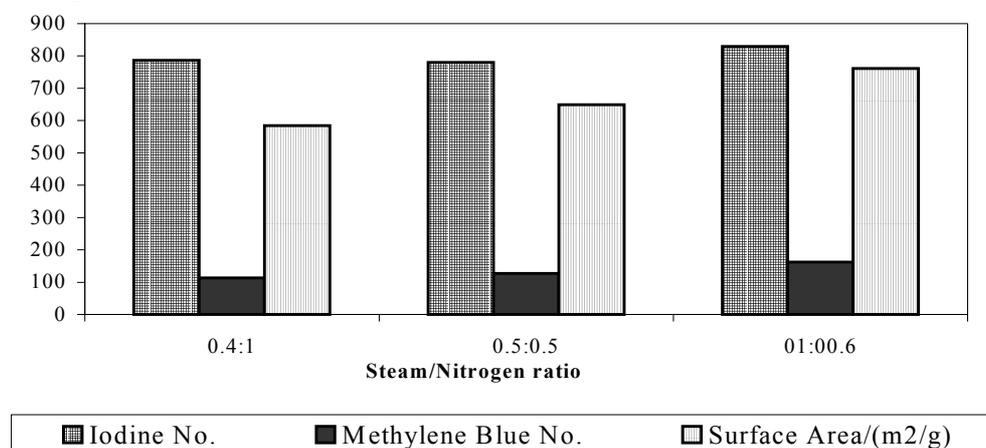


Figure 5. The effect of varying partial pressure of steam on the activated carbon properties carried out at 800°C for 4 hrs.

Activation temperature also plays a fundamental role in determining the properties of the activated carbon produced. Figure 6 shows the effect of varying activation temperature on activated carbon characteristics. The experiments carried out at 700 and 800°C were exposed to steam for 4 hours whereas the experiment at 900°C was only for 2 hours. The gasification reaction proceeded at a faster rate at 900°C reaching the desired burn off percent more quickly than those at lower temperatures. Activated carbons with the best properties were produced at 900°C in 2 hrs. The mass loss and temperature profile can be seen in Figure 4. A 50% burn-off was reached at the end of

2 hours with a 10% yield. The processing conditions depend much on the ultimate use of the carbon. In this case the activated carbon was produced for the purpose of decolourising raw sugar. In order to remove the colour bodies from sugar, the carbon has to have a large meso-macroporous pore size distribution. Table 5 shows the pore volume and pore sizes for activated carbons produced at 900°C.

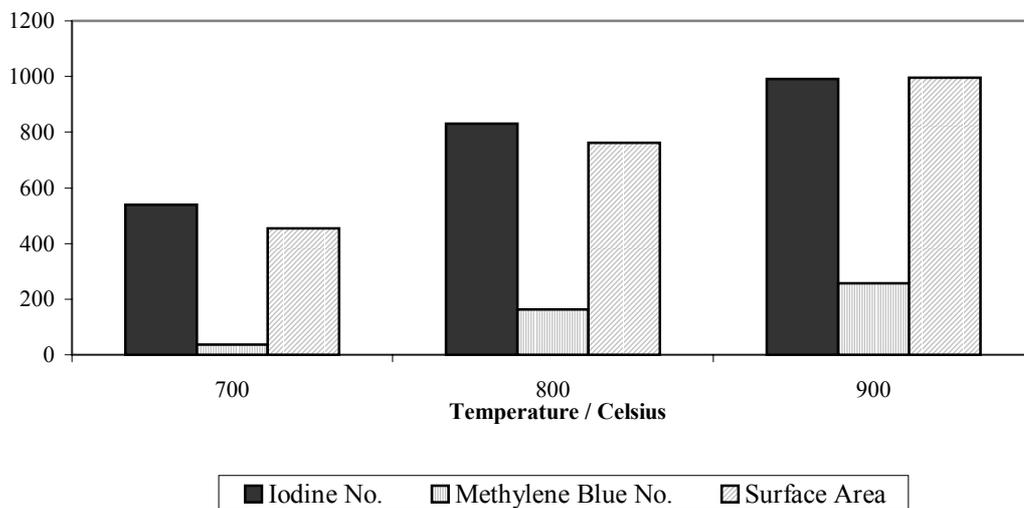


Figure 6: The effect of temperature on activated carbon properties.
NB. Experiments at 700 and 800°C were carried out for 4 hrs,
whereas experiments at 900°C were carried out for 2 hrs.

The above-mentioned carbons produced were powder activated carbons. The quality of raw material also affects the characteristics of the activated carbon produced. The cane brought into the mills varies in terms of composition as mentioned, particularly in terms of ash content. Hence different grades of bagasse are produced, and thus different grades of activated carbon were produced as seen in Table 5. As the ash content appears to have a significant effect on the carbon properties, it may be necessary to reduce the ash in bagasse by a cleaning step if the levels are too high, before pyrolysis. Granular activated carbons were also produced using refined sugar as the binder. The processing conditions were the same as the powder activated carbons but activation was carried out for a period of 2.5 hours. The activated carbon was extremely hard and its properties are also shown in Table 5. The characterization experiments were also carried out on a reference carbon and the comparison is tabulated in Table 5.

The activated carbons produced compare well with the standard carbon. The molasses number is much higher for the prepared carbons than for the reference as they contain more meso- and macropores and are thereby able to adsorb large colour bodies contained in molasses solution. These increased pore volumes and surface areas are reflected in the greater values for the BJH surface areas and pore volumes. The carbon with higher ash content gave values closer to the reference carbon, and a similar molasses number.

The activated carbons produced have a high sugar decolourising potential as can be seen by the isotherms displayed in Figure 7. Commercial carbons (Chemviron CaneCal and Norit PN2) were tested in parallel with the experimental carbons, but problems were experienced during the CaneCal tests and the results are not presented. Both the powder and granular carbons are excellent decolourisers and compare well with the commercial carbon. Approximately 80% colour removal was achieved using 0.5 g carbon/100 g Brix of brown liquor. This shows that activated carbons prepared from sugarcane bagasse have the ability to replace existing commercial sugar carbons.

Table 5. Comparison of prepared and reference activated carbons.

Sample 1 - Powder activated carbon with low ash content (28%)

Sample 2 - Powder activated carbon with higher ash content (56%)

Sample 3 - Granular activated carbon (ash content 14%)

Sample 4 - Cane Cal reference carbon (ash content 13%)

| Area | Sample | | | | |
|--|--------------------|------|------|------|------|
| | Units | 1 | 2 | 3 | 4 |
| Single point surface area | m ² /g | 1000 | 608 | 954 | 1077 |
| BET surface area | m ² /g | 995 | 606 | 942 | 1063 |
| Langmuir surface area | m ² /g | 1345 | 823 | 1273 | 1429 |
| BJH adsorption cumulative surface area of pores between 17 and 3000 A diameter | m ² /g | 463 | 274 | 358 | 204 |
| BJH desorption cumulative surface area of pores between 17 and 3000 A diameter | m ² /g | 517 | 308 | 404 | 224 |
| Volume | | | | | |
| Single point total pore volume | cm ³ /g | 0.82 | 0.47 | 0.69 | 0.62 |
| BJH adsorption cumulative pore volume of pores between 17 and 3000 A diameter | cm ³ /g | 0.60 | 0.36 | 0.46 | 0.27 |
| BJH desorption cumulative pore volume of pores between 17 and 3000 A diameter | cm ³ /g | 0.64 | 0.37 | 0.48 | 0.28 |
| Pore Size | | | | | |
| Average Pore diameter | A | 33 | 31 | 29 | 23 |
| BJH Adsorption average pore Diameter (4V/A) | A | 52 | 52 | 51 | 52 |
| BJH Desorption average pore Diameter (4V/A) | A | 50 | 48 | 47 | 49 |
| Iodine No. | mg/g | 992 | 671 | 1096 | 1000 |
| Methylene Blue No. | mg/g | 256 | 174 | 250 | 242 |
| Molasses No. | | 701 | 327 | 607 | 230 |

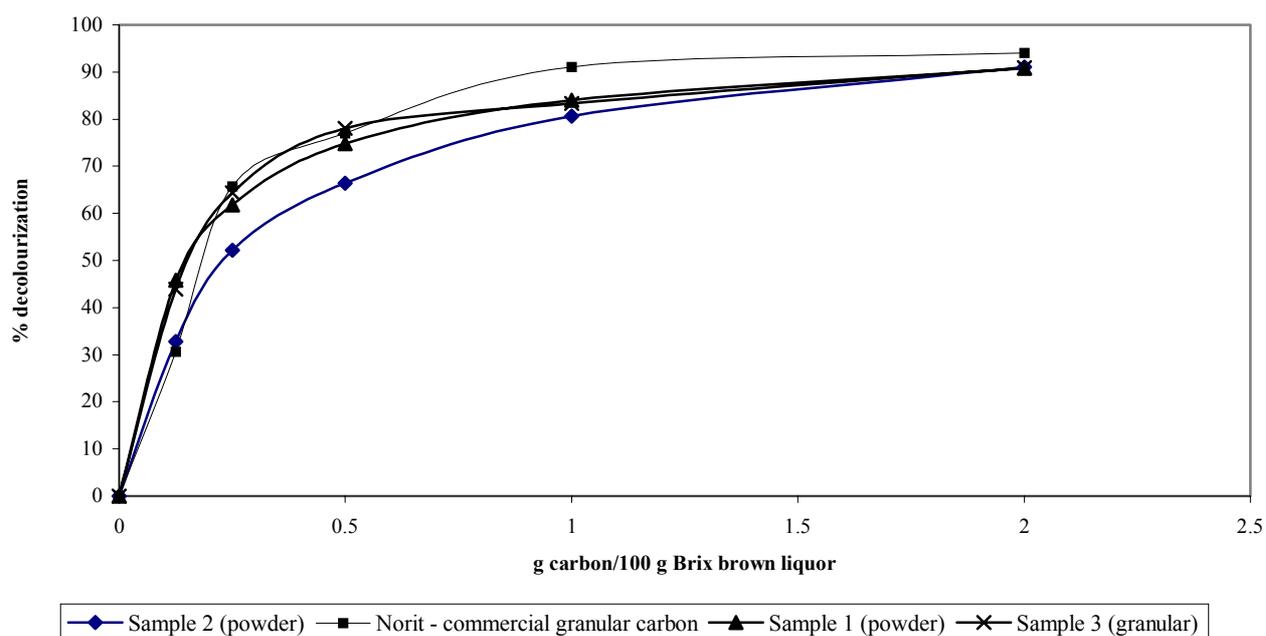


Figure 7. Adsorption isotherms of activated carbon for sugar decolourisation.

Conclusion

Using sugarcane bagasse as a parent material in the manufacture of activated carbons produces unique activated carbons having excellent sugar decolorizing capacity. The quality of the activated carbon produced depends on the method and processing conditions such as heating rate, temperature, hold-time and activating medium. Increasing the activation temperature and partial pressure of activating gas (steam) increases the degree of activation in a shorter time. Both powder and granular activated carbons can be produced, whereby the powder can be used for batch adsorption and granular for continuous systems. Their commercial production would, however, require scale-up to pilot plant using the established process coupled with its determined processing conditions.

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