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Production and characterization of activated carbons using sugarcane bagasse from Chemelil Sugar Factory compared with commercial activated carbons

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Abstract

Activated carbons (AC) synthesized from sun dried bagasse of Chemelil Sugar Factory in Kenya were used in this study. Using molasses as a binder, ratio 1:1, the bagasse and molasses mixture was compressed into pellets in an extruder, heated to produce a char, and then activated using carbon dioxide, before finally being ground into powdered activated carbon. Carbonization was done in a retort, while the heating was done by two gas burners. A thermocouple was used to record and control the temperature, which ranged from $300 - 700^{\circ}$ C. The carbonized material (char) was cooled and ground to powder. Sample activation was conducted in an atmosphere of carbon dioxide passing through a chrome-nickel pipe with a gas distributor mounted in a furnace of porcelain cylinder with a metal sheet having one end closed. Air and nitrogen were used to control burning and the temperature of the furnace was controlled to between $300 - 600^{\circ}$ C. The activated carbons produced were characterized for their physical properties (bulk density, yield, surface area, and micro pore volume and pore size), chemical properties (ash, pH, conductivity, Iodine and molasses decolourisation) and compared with selected two commercial activated carbons. The results showed that carbon materials activated from bagasse for 5 h at a temperature of 600° C showed similar characteristics with the commercial activated carbons and therefore an inexpensive substitute for AC.

Keywords: Activated Carbon, Adsorption, BET surface area, sugarcane bagasse

1. Introduction

Biomass such as bagasse, sugar cane fiber or solid waste from sugar cane (after juice extraction) can be used in the production of activated carbon (AC) for adsorption applications (Tawan et al., 2017). Activated carbon from bagasse is a low-cost, natural and available adsorbent, and, it has been applied for the removal of many pollutants (Salihi et al., 2017; Van Tran et al., 2017). In Kenya activated carbon is mainly used in wastewater treatment. Bagasse in the sugar factories is mainly used as fuel in boilers to produce electricity and process steam, while the excess estimated at 46% is disposed off from the factory some to be used as manure in sugarcane farms. Excess bagasse is used in the making of briquettes and match sticks. Bagasse production from sugar companies in Kenya averages about 2,000,000 tons per year (Agriculture Food Authority - Sugar Directorate, Kenya, 2018). Due to high cost of commercial AC, various agricultural waste materials have been widely examined for the preparation of low cost activated carbon. Bagasse is composed largely of cellulose, pentosan, and lignin. Various studies indicate that sugarcane bagasse is a new alternative that can replace the existing products of activated carbon

precursors like coal. (Ahmedna et al., 2000; Foo et al., 2013). The adsorption capacity of an activated carbon is determined by: (i) its surface area, (ii) internal porous structure, and (iii) the functional groups at the carbon pore surface. Activated carbon materials are known to contain oxygen, hydrogen, chlorine and sulphur atoms (Chen et al., 2008). The physical, chemical and surface characteristics of activated carbon determine its efficiency in removing the targeted colorants. The properties of activated carbon include: Surface area (Zou et al., 2001), density and particle size (Abbas, 2020) and particle distribution. Other properties are: porosity, wettability, dustiness, operation temperature, electrical conductivity, moisture, and pH (Ahmedna et al., 1999), total ash (Ahmedna et al., 2000), and water extractable inorganics. The production of activated carbon from bagasse is seen as having potential to add value to the sugar industry as an extra income stream, besides the activated carbon produced being used in

*Corresponding author e-mail: lumadedeh@yahoo.com Received: 15 May 2020 Revised: 19 June 2020 Accepted: 24 June 2020 the sugar refining process as a decolorizing agent. Processing excess bagasse to produce activated carbon can provide better solutions in the utilization and management of the waste in the sugar industry. This will take care of the potential hazard of fire outbreaks in addition to reducing the cost of its disposal. The objective of this work was to (i) produce activated carbon from sugarcane bagasse of Chemelil Sugar Company at selected temperatures and times on laboratory scale, (ii) evaluate and characterize the properties of the produced activated carbon, and (iii) compare the properties and performance of the produced activated carbons with commercial activated carbons.

2. Methodology

2.1 Materials used

Sugarcane bagasse (Chemilil Sugar Company,Kenya), Cane molasses (Chemilil Sugar Company), Nitrogen (Kenya Industrial Research & Development Institute), Carbon dioxide (Kenya Industrial Research & Development Institute) and Commercial activated carbons (Sutcliff Carbons England).

2.2 Experimental method

2.2.1 Preparation of bagasse activated carbon

The bagasse was sampled from the factory and dried under the sun for three days. The bagasse sample was mixed with molasses in a mass ratio 1:1 compressed into pellets using an extruder, and then heated to produce char. The char was put through the activation stage and finally ground into powdered activated carbon. Carbonization was done in a retort, of capacity of 1.0 kg between temperature ranges of 300 - 700 °C by heating using two gas burners, which were controlled by adjustment of the gas pressure until gas evolution ceased. A Chromel-Alumel thermocouple was placed through one end of the closed tube extended to the middle of the retort to control the temperature. The gas evolved was passed through an externally cooled condenser for the removal of tar and water. After cooling, the carbonized material (char), was removed from the retort, washed with hot water, then with dilute HCl to remove salts, and then finally washed with cold water until pH was neutral. (1L/ kg 60° C) (Ozer *et al.*, 2012). The washed carbonized materials were dried in an oven at 120°C for 2h, then ground to powder ready for activation. Experiments were performed to final temperatures of 300 - 700 °C with heating rates ranging from 5 to 30 °C/min with nitrogen sweep gas flow-rate of 350 ml/min. During activation, a furnace of porcelain cylinder with a metal sheet was used and one end closed with a distributor through which a Chrome-Nickel pipe passes for gas inlet. A Chrome-Alumel thermocouple was placed in the furnace. The temperature of the furnace was controlled by the rheostat during activation, which lasted between 2 - 5h. The top of the furnace was closed to prevent any air leakage. Carbon dioxide gas was used as the activating agent. The furnace was first pre-heated to 300°C before 200g of the carbonized char was charged into it. Air and nitrogen were used to control burning. The temperature varied from $300 - 600^{\circ}$ C. The final product was again washed with hot water, then with dilute HCl to remove salts; then finally washed with cold water until neutral pH was attained, after which the sample was dried in an oven at 120°C for 2h.

2.2.2 Methods of characterization of the activated carbons

The so-produced activated carbons were characterized for their physical properties (bulk density, surface area, micro pore volume and pore size), and chemical properties (ash pH, conductivity, iIodine and molasses content. declourisation) determined. The Braunauer-Emmet-Teller (BET) specific surface areas of the samples were determined using the N₂ adsorption-desorption isotherms at 77 K in the relative pressure range of 0.05 to 0.25 as follows. The activated carbon samples were dried under vacuum at 100 °C for 3 h to remove moisture from the carbon pores. Samples were placed in an automatic Surface Area Analyzer (Micrometrics ASAP 2000), which measures the adsorption of nitrogen gas to the samples at 77 K with the help of the Micrometrics software. In the calculations, the cross-sectional area of the nitrogen molecule was taken as 0.162 nm². The total volume pore volume of the adsorbed amount of N2 was taken at the relative pressure greater than 0.98, assuming a liquid molar volume of N₂ to be $34.65 \text{ cm}^3/\text{g}$ at 77 K.

3. Results and Discussion

3.1 Production and characterization of activated carbons

A summary of the results obtained are provided in Table 1, which indicates that the yields of carbonized bagasse ranged from 15 - 26%, with slight deviations from each other, due to the different activation temperatures applied. The bagasse carbon made at temperatures of 600°C gave a yield of 19.4% and this sample was the one used for subsequent activation with carbon dioxide.

Table	1:	Carbonization	of	bagasse-based	carbons	with		
their yields at different synthesis temperatures.								

Temperature of carbonization (° C)	Yield (%) ±1
300	25.4
400	21.6
500	21.9
600	19.4
700	15.2

The results in Table 2 show that as the carbonization temperature increased, the yield of char decreased (Girgis *et al.*, 2002). It was observed that the temperature of 700°C was not conducive as some salts start to melt at temperatures above 700°C as the porosity and the pore structure is affected, this therefore results in poor adsorption. (Jun *et al.*, 2010). Table 2 provides a summary of the results obtained indicating the physical and chemical properties of the synthesized activated carbons, which were benchmarked against the commercial ones. Increasing activation temperature causes the evaporation of the volatile matter and it can be expected that there will be a high chance of macropore development (Juang *et al.*, 2001).

Carbon	BET surface	Pore	Pore size	Density	Ash	Conducti-	pН	Moistu	Decolourisation (%)	
type	area (m²/g)	volume	(Å)	(g/cm^3)	(%)	vity (µs)		re (%)	Iodine	Molasses
		(m ² /g)				at 23°C				
WP320*	1327.4	1.30	36.00	0.28	3.6	285.0	4.5	7.7	87.0	93.6
CS200*	858.5	0.54	25.60	0.35	16.8	17.1	6.7	5.3	93.5	93.8
600/C5	800.6	0.68	20.44	0.30	18.8	266.0	6.2	18.6	95.6	92.2
600/C4	771.5	0.51	23.26	0.33	19.0	245.7	6.1	20.1	82.6	85.6
600/C3	579.4	0.42	26.11	0.35	20.9	223.0	6.4	19.4	78.2	78.9
500/C5	654.0	0.50	33.22	0.32	21.7	235.8	6.2	19.8	63.7	53.3
500/C4	560.1	0.49	26.42	0.37	20.4	226.7	6.6	20.2	47.8	45.7
500/C3	437.5	0.39	26.50	0.36	22.1	237.2	6.7	21.4	34.8	31.3

Table 2: Characteristics of the activated bagasse-based carbons compared to commercial carbon samples

* Commercial activated carbon samples

At lower temperatures, the carbon dioxide does penetrate easily into the resulting macropores. The site near the entrance of the pores is eroded more easily by the reaction than the inner portions (Ko et al., 2002). The erosion of the exterior portion of the char upon activation results in disappearance of exterior pores and this explains why both the surface area and the pore volume decrease with the degree of activation at higher burn off levels. The maximum values of surface area and pore volume for bagasse carbons were obtained within the temperature range of about 600oC and activation time of 5h. Increasing both the activation time and temperature has negative effect as it causes the widening of the existing pores leading to larger pore sizes occasioned by the complete burnout of the walls between adjacent micro pores. This widening of pores does not produce the desired adsorption efficiency that is required In order to produce an activated carbon with optimum properties, a high degree of uniformity in carbonization must occur, originating from the surface to the centre of the particle (Hesas et al., 2013). In addition to the change in the pore sizes, continued activation has impact on the crystalline structure. At lower activation temperatures, a fraction of each inner particles fail to undergo carbonization, and because of this unreached and un-reacted region. So a carbonized char of fairly low quality was obtained due to the non-homogenous effect by low heat treatment of 300 or 400° C. Activation using CO₂ involves the C–H₂O and C–CO₂ reactions respectively, resulting in the removal of carbon atoms and causing the main weight loss of the resulting char (Gonçalves et al., 2016; Abdelouahab-Reddam, et al., 2014). At 700°C according to (Ahmedna et al., 2000) some salts start to melt at temperatures above 600 °C leading to roasting the bagasse and therefore the structure gets negatively affected. This leads to the formation of poor adsorption properties in the material. Porosity plot of the BET specific surface area is provided in figure 1. Decolourisation was found to be directly impacted by both the surface area and pore volume, with the

highest pore volume being 1.30 m^3/g for WP320 followed by 600C/5 at 0.68 m^3/g as shown in Table 2.



Fig 1: Plots showing the influence of synthesis time and temperature on the BET surface area of the activated carbons.

Both commercial carbons, WP320 and CS200 had high BET surface area values of 1327.4 m^2/g and 858.5 m^2/g , respectively. WP320 had low pH value of 4.5 due to activation by phosphoric acid, while the steam activated CS200 with pH of 6.7 had high ash content of 16.8% with a relatively higher density of 0.35 g/cm³. The bagasse carbons had lower BET surface area compared to the commercial carbons, with the highest being the 600/C5 sample with 800.6 m^2/g followed by 600C4 (771.5 m^2/g). This can be attributed to high carbon content and as a result they were volatized to a less extent leading to a relatively lower surface area (Pendyal et al., 1999). The lower volatilization capacity means that the carbons undergo less weight loss and are therefore relatively denser. The ash content in CS200 (16.8%) which was slightly less than the ash content in the bagasse carbons, which ranged between 18% and 22%. In WS320 ash content was very low (3.6%). Since all the bagasse-based carbons were activated in carbon dioxide, it was observed that their pH values were less acidic, ranging from 6.1 to 6.7. However, carbon dioxide activation produces an extensive surface area and a well developed microporosity (Girgis et al., 2002). H₃PO₄ is usually preferred as a chemical activation agent because it bubbles through the carbon matrix and gives a well-developed microporosity leading to a high surface area after opening up the cellulose structure. This structure once developed cannot shrink. The phosphoric acid controls the pyrolysis in such a way that formation of tar, which is an unwanted impurity together with volatization, are kept to the minimum. The H₃PO₄ has an advantage, for example, in the case of WP320, which is a wood based carbon, and it leads to higher conversion of wood into carbon (Pollard et al., 1997). In the bagasse-activated carbons, 600/C5 (pH 6.2), and 600/C4 (pH 6.1), fit well into the pH range of good colour removers. The inference here is that since carboxyls and lactones normally decompose between 600 - 800 °C, it gives them an advantage in sugar decolourisation because with no carboxyls on the surface, the negative surface charge is decreased. In the case of carboxylic acids, the fewer the number of these groups, the less the negative charge, which leads to increased adsorption.

3.2 Iodine and molasses decolourisation

The last two columns of Table 2 shows the decolourisation efficiencies for the commercial and bagasse derived activated carbons on treatment with cane molasses. The iodine and to an extend molasses decolourisation test of an active carbon is an indicator of the ability to adsorb low molecular weight compounds (Liakos and Lazaridis, 2016; Rahimi *et al.*, 2018). In figure 2, it is shown that bagasse-carbon samples (600/C5 and 600/C4) comparatively had better iodine solution decolourisation efficiency (95.6 and 93.4% respectively) due to their large surface area and pore volume. For example, 600/C5 sample (with pore volume 0.68 cm³/g) compared well



Fig 2: Comparison of percent decolourisation in molasses and iodine samples.

with the commercial carbons of decolourisation efficiency 87.0 and 93.5% in WS320 and CS200 respectively. The bagasse carbon with the least decolourisation efficiency of 34.8% was the 500/C3 sample, which can be attributed to the low surface area (437.5 cm²/g) and the relatively small pore volume. The disadvantage of all these type of carbons is that they have a high ash content, between 18 and 22%. The amount of colour removed from molasses is regarded as the activated carbon's capacity to adsorb coloured bodies especially sugar colorants since most of the impurities in the syrup during industrial sugar processing end up in the molasses.

Carbons with high capacity for colour removal from molasses are potentially good sugar syrup decolourizers. The commercial carbons CS200 and WP320 show high decolourisation values (93.8 and 93.6% respectively). The bagasse derived carbon, which achieved the best molasses decolourisation efficiency, were 600/C5 (92.2%) and 600/C4 (85.6%), with pore volumes of 0.68 cm³/g and 0.51 cm³/g respectively. The bagasse carbons with a decolourisation percent above 90% gave a good indication that the carbons have a good capacity to remove high molecular weight sugar colorants (Aljohani et al., 2018). Among the commercial carbons WP320 (93.6 %) came second after CS200 (93.8 %). This is because the sample has a larger pore size diameter 36.0 Å to trap larger molecular colorants, which had been newly formed. A look at other properties of 500/C5 with surface area 800.6 m^2/g has a pore size of (20.44 Å), which is a good size for syrup decolourisation, but its low density (0.30 g/cm^3) and of course the high ash content (18.8%) are its drawback (Kaushik et al., 2017). High ash content leads to higher electrical conductivity. More electrical conductivity of the activated carbon means more ash content available for leaching into the sugar syrup. The leachable ash is undesirable and can be regarded as a colourant which then cancels the decolourisation effect of the activated carbon (Mudoga et al., 2008).

4. Conclusion

The laboratory prepared activated carbon obtained from sugarcane bagasse by direct carbonization for 5 h at 600 $^{\circ}$ C (600/5) under a stream of carbon dioxide has a decolourisation performance that can compete with commercially used activated carbons. Among the studied physical characteristics of the activated carbons, iodine decolourisation performance appear to be the best indicator followed by the molasses decolourisation This work has demonstrated that bagasse derived activated carbon 600/5 and commercial activated carbons WP320 and CS 200 possess similar characteristics. This is due to their physical, chemical, and adsorption properties, particularly their high surface area and pore volume accompanied by low ash content. The 600/5 is a potential low cost alternative to the existing commercial carbons. Bagasse therefore can be a used as an inexpensive substitute for activated carbon precursor, and will require further development while observing the processing parameters.

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