



## Comparison of the effectiveness of decolourisation of of sugar syrup by activated carbons from sugarcane bagasse from Chemelil sugar factory with commercial activated carbons

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### Abstract

Six bagasse based carbons and two commercial activated carbons (AC) were evaluated for efficiency in the level of decolourisation of sugar syrup. The effect of bagasse activated carbon dosage in the range 0.2 –1.0 g per 100 ml syrup was determined. The Braunauer-Emmet-Teller (BET) specific surface area, pore size and pore volume of the samples were determined using the N<sub>2</sub> adsorption-desorption isotherms. The bagasse activated samples were analysed using standard methods including UV-Visible spectroscopy to evaluate the effectiveness for percent colour removal from sugar syrup and compared with the two commercial activated carbons. The colour removal capabilities of all carbons were measured at 1/100 (w/w) dosage. The results showed activated carbon from bagasse activated for 5h at a temperature of 600<sup>0</sup>C (600/5) was comparable to commercial activated carbon (WP 320) by their high surface areas and syrup decolourisation efficiency. Their pH values were also in the range 6-8 that makes them suitable for sugar refining.

Keywords: Activated carbon, decolourisation, sugar syrup

### 1. Introduction

Sugar producers have to find economic ways of making sugar of low colour in order to attain the quality standards. The decolourisation of syrup, apart from giving the desired white colour of the sugar crystals in the factory also results in the improvement on the crystallization process of the sugar crystals in the factory. This is because the adsorbable surface-active substances, potentially harmful, which would slow down the crystallization of sugar are removed. There are four systems of decolourisation which have been tried in sugar refining process namely: Bone char, activated carbon (GAC) (coal base, ion exchange resins and flocculants (Ahmedna *et al.*, 2000). Decolourising carbons have capacity for adsorption of surface active and colloidal impurities. The decolourisation process actually removes more than colour because colourants that interact with colour precursors, colloidal materials, organic nonsugars, are also taken out. Carbons having equal surface area but having been prepared by different methods or different activation treatments have different functional groups and hence different adsorption characteristics. This is due to the formation of different types and concentrations of surface oxides on the carbon surfaces, (Dubinin, 1987). The carbon – oxygen surface structures are important in influencing the surface characteristics which include the interaction with polar

and non-polar adsorbates, (Bansal *et al.*, 1988; Boehm, 1994). The elemental composition of activated carbon typically comprises 85-90 % C, 0.5 % H, 0.5 % N, 5 % O, and 1 % S, the balance of 5-6 % representing inorganic (ash) constituents. In addition to hydrogen and oxygen, calcined sulphur, nitrogen, chlorine and other elements can also be present in active carbon (Balci, *et al.*, 1994). Most of the available surface area of activated carbon is nonpolar in nature. As a result, carbon adsorbents tend to be hydrophobic and organophilic. During the cane juice processing, the sugars and non-sugars present in the juice are subjected to heat, varying pH, air (oxygen), lime and form coloured compounds under favorable reaction conditions. Examples of non-coloured compounds are amino acids, hydroxy acids, aldehydes, iron and reducing sugars. (Nick, 1992; Clarke *et al.*, 1984; Carpenter, 1985). There are also the reactions with bacteriocides. The bacteriocides are meant to prevent microbiological activity causing inversion of the sucrose molecule. Some colours may be developed from chemical

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Received: 19 May 2020

Revised: 12 June 2020

Accepted: 18 June 2020

action on the non-sugars present in the cane and some may occur as a result of decomposition products formed. The reducing sugars formed decompose due to heat and alkaline solutions leading to reactions with other non-sugar materials such as polyphenols and amino compounds. When sucrose is inverted, by means of acid or by the bacteria known as *leuconostoc mesenteroid*, the two reducing sugars develop colour when subjected to heat and alkaline conditions. Fructose decomposes more rapidly than glucose, followed by sucrose. The chemical reactions, which occur between the alkalis and the reducing sugars result in yellow solutions that will turn dark brown on heating forming lactic acid.

The most significant coloured substances that develop during the sugar process can be classified in three general groups. They are Melanins, Melanoidins and caramels (Mudoga *et al.*, 2008). This study investigated the effectiveness of decolourisation of sugar syrup by activated carbons made from sugarcane bagasse from Chemelil sugar factory and gave a comparison with the commercial activated carbons. The colouring materials in sugar have their origins in the colourants or colour precursors present in the juices and syrup from which the sugar is extracted, therefore the need for decolourisation of syrup. Recent research on decolourisation of syrup using activated carbon from bagasse was done by (Arunee *et al.*, 2010) who investigated the decolourisation of melanoidin by activated carbon obtained from bagasse bottom ash (BBA). Other syrup decolourisation studies have been done by (Arslanoglu *et al.*, 2012) where cations and colourants removal from sugar juice were conducted by using modified sugar beet pulp, (Khadija Qureshi *et al.*, 2007; Girgis *et al.*, 1994); Ahmedna *et al.*, 2000 and Devnarain *et al.*, 2002), In the experiments of (Niraj *et al.*, 2011), activated carbons were produced from sugarcane bagasse in a muffle furnace by adopting the physical method of processing and the activated carbons were characterized for adsorption capacity in terms of iodine and decolourizing power. Acid carbons may be better decolourizers (Paton and Smith, 1983), but in sugar refining a highly acidic carbon will not be used because the acid would cause inversion of sucrose to noncrystallizable sugars with subsequent lower yield of the crystal sugar. Distinctly alkaline carbons will also cause colour development through alkaline degradation of organic impurities. (Bernardin, 1985)

## 2. Methodology

### 2.1 Materials

Sugarcane syrup was obtained from Chemilil Sugar Company and commercial activated carbons WP320, CS 200 were obtained from Sutcliff Carbons England.

### 2.2 Preparation of activated carbon from bagasse and analysis

Six activated carbons were made from bagasse at various temperatures and times and activated with carbon dioxide. Carbonization of bagasse was done in a retort, of capacity of 1.0 kg at a temperature range of 300 – 700 °C. Heating using

two gas burners, which was controlled by adjustment of the gas pressure until gas evolution ceased.

Activation experiments were performed to final temperatures of 500 – 600°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. The temperature of the furnace was controlled by the rheostat during activation, which lasted between 3 – 5 h. Carbon dioxide gas was used as the activating agent. The furnace was first pre-heated to 300 °C before 200 g of the carbonized char was charged into it. Air and nitrogen were used to control burning. The temperature varied from 500 to 600° C. The final product was 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 2 h. The Braunauer-Emmet-Teller (BET) specific surface area, pore size and pore volume of the samples were determined using the N<sub>2</sub> adsorption-desorption isotherms at 77 K in the relative pressure range of 0.05 to 0.25.

## 2.3 Experimental method

### 2.3.1 Syrup

The syrup was obtained from the Chemelil Sugar Company. A series of grab samples of the syrup were collected at the point just before the sugar boiling and crystallization step in the factory. The sampling was done several times for a period of 48 hours. The samples were stored in the refrigerator in a plastic container. The syrup decolourisation procedure was done using UV-Visible spectroscopy where the effect of activated carbon dosage in the range 0.2 –1.0 g per 100 ml syrup was determined to evaluate the effectiveness for percent colour removal from sugar syrup (Mudoga *et al.*, 2008).

## 3. Results and Discussion

In Table 1(a) the low temperature active carbons give lower percent values for syrup decolourisation and so they represent less efficiency in adsorption and therefore not suitable for use in syrup decolourization. These are 500/C3, 500/C4, and 500/C5. The high temperature active carbons 600/C5, 600/C4 gave higher values for syrup decolourisation. This is because low temperature char contains a larger quantity of volatile matter. Increasing temperature will cause the evaporation of the volatile matter and can be expected that there will be a high development of macropores. The place near the entrance of the pores is more excessively eroded by the reaction than the inner portions (Pendya *et al.*, (1999a). Table 1 (b) shows the bagasse activated carbons, 600/C5 (pH 6.2), 600/C4 (pH 6.1), fit well into the pH range of good a colour remover of 6-8. The inference here is that since at 600 °C – 800 °C carboxyls and lactones normally decompose, it gives the carbons an advantage in sugar decolourisation because with no carboxyls on the surface, the negative surface charge is decreased hence increase in adsorption and therefore high decolourisation. The

most effective carbon was WP 320 with a syrup decolourisation efficiency of 73.1 % with a surface area of 1327.4 m<sup>2</sup>/g. It was followed by bagasse activated carbon 600/C5 (65 %) surface area 800.6 m<sup>2</sup>/g of and also pore volume (0.68 m<sup>3</sup>/g), CS 200 (60.9 %), 600/C4 (45 %) then 500/C5 (43.9%), 600/C3 (40.1%), 500/C4 (39.5%), 500/3 (28.3% in that order. The large surface area, low ash content and large pore size diameter make WP 320 and 600/C5 good syrup decolourizing agents.

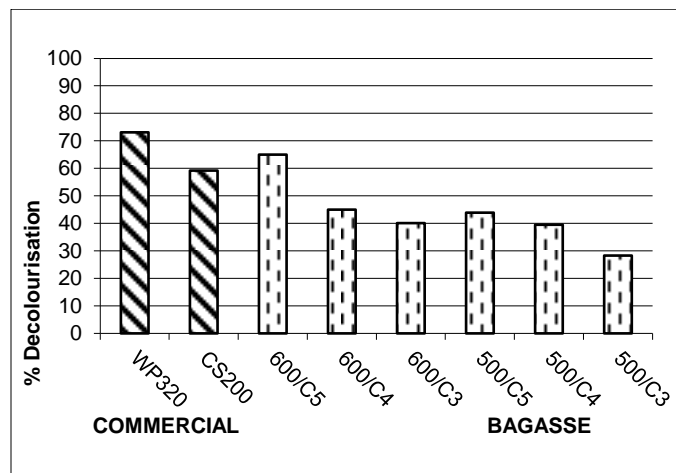
**Table 1 (a). Physical and chemical properties of the activated carbons and their corresponding syrup decolourisation percentages**

Carbon type	BET surface area (m <sup>2</sup> /g)	Pore volume (m <sup>3</sup> /g)	Pore size (Å)	Density (g/cm <sup>3</sup> )	Ash (%)
WP320*	1327.4	1.30	36.00	0.28	3.6
CS200*	858.5	0.54	25.60	0.35	16.8
600/C5	800.6	0.68	20.44	0.30	18.8
600/C4	771.5	0.51	23.26	0.33	19.0
600/C3	579.4	0.42	26.11	0.35	20.9
500/C5	654.0	0.50	33.22	0.32	21.7
500/C4	560.1	0.49	26.42	0.37	20.4
500/C3	437.5	0.39	26.50	0.36	22.1

**Table 1(b). Physical and chemical properties of the activated carbons and their corresponding syrup decolourisation percentages**

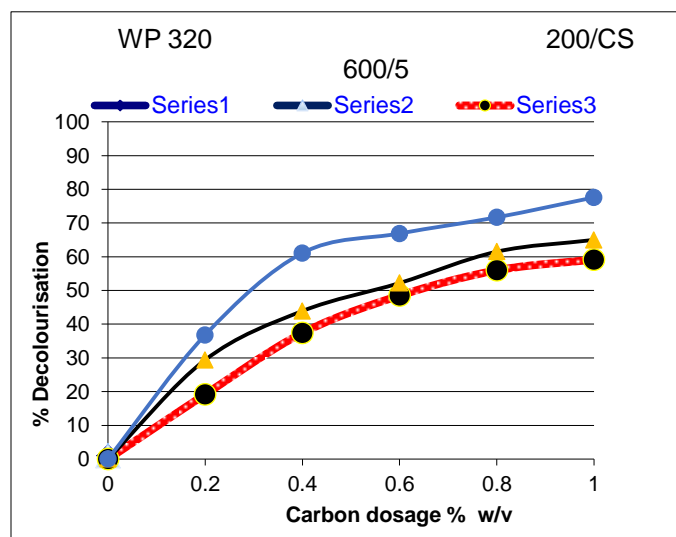
Carbon type	Conductivity (µs) at 23°C	pH	Moisture (%)	Sugar Syrup Decolourisation (%)
WP320*	285.0	4.5	7.7	73.1
CS200*	17.1	6.7	5.3	59.2
600/C5	266.0	6.2	18.6	65.0
600/C4	245.7	6.1	20.1	45.0
600/C3	223.0	6.4	19.4	40.1
500/C5	235.8	6.2	19.8	39.5
500/C4	226.7	6.6	20.2	39.5
500/C3	237.2	6.7	21.4	28.3

The syrup decolourisation percentage of commercial and bagasse activated carbons are shown in figure 1. Activated carbon pH may influence colour by changing the pH of the sugar solution. Such a change affects the pH sensitive fraction of solution colourants causing unreliable colour measurements (Ahmedna *et al.*, (1997b).



**Fig 1. Percent cane syrup decolourisation by commercial and bagasse activated carbons (1% w/v)**

The high molecular weight colourants, mostly polysaccharides are removed during clarification of juice in the factory through flocculation and coagulation followed by settling at the bottom of the clarifier. A plot of carbon dosage, M for all the commercial carbons and one bagasse 600/C5, versus dosage 1% w/v on a rectangular coordinate yields a curvilinear graph as shown in figure 2.



**Fig 2. Decolourisation isotherms of syrup as a function of dosage for the carbons for WP320, CS 200 and 600/5**

The carbon dosage is important because it determines the extent of decolourisation and may be used predict the cost of carbon per ton of sugar syrup to be treated. An increase in the quantity of activated carbon at first quickly raises the adsorption effect and then gradually tapers off. Therefore, excessive additions of carbon are not economical. Even with an infinite increased quantity of carbon the tendency is not toward 100% adsorption. This may be due to the presence of a

mixture of various colouring substances in the syrup, some which are practically unadsorbable by activated carbon. Freundlich isotherm is the most commonly used in environmental engineering as are empirical relations for adsorption process (Geessner and Hassan, 1987; Abe *et al.*, 1990). Each individual type of AC has an own isotherm curve and breakpoint characteristics, which help to predict the adsorptive capacity of particular activated carbons. From this, a design estimate for adsorptive life is obtained and once the breakpoint has been reached a reactivation process becomes necessary. The Freundlich isotherms are also used to predict the surface heterogeneity the adsorbent as well as multi-layer of coverage of the surface. The isotherms are straight lines which means that these carbon-carbon-solution system can be defined in terms of Freundlich isotherms. The Slope of these lines are rather steep which means that for countercurrent applications they are useful. The relationship between the carbon dosage and the adsorption of colourant is described by the empirical Freundlich adsorption equation:

$$X/M = KC^{1/n}$$

The Freundlich equation in logarithmic form appears as:

$$\log X/M = \log K + 1/n \log C$$

In this equation, X is the units of colourant held by M units of carbon with which such adsorption was affected. X equals  $C_0 - C$ ; that is the difference between the original and final concentration colourant.  $C_0$  is the concentration of colourant remaining unadsorbed at equilibrium. The two constants K and 1/n are particular to the system, and depend on the carbon used and the nature of the colourant. C, the concentration of colourant may be expressed in colour units. Plotting X/M verses C, the residual colour concentration, on a logarithmic scale yields a straight line graphs as shown in figure 3.

The amount of adsorbate held per g of activated carbon was expressed as the difference between initial and residual percent of colour divided by the carbon dosage in g/100 g syrup. Similar approaches were used by (Agudo *et al.*, 2002; Celebi and Kincal, 2007) in their isotherm expressions. The dependable linearity K, of the straight line – plot is useful checks on detecting adsorption abnormalities while the constant 1/n, is important to when considering the kind of dosage. The magnitude of n is gives an idea about the favorability of adsorption process. When  $n > 1$ , it indicates that the adsorption is favourable which according to the results obtained (WP 320, 7.353 600/CS, 12.44 and CS 200 13.44) was in agreement (Mudoga *et al.*, 2008). The values of K and 1/n were obtained from the intercept and slope of the linearized isotherm are given the Table 2. From the Freundlich isotherms, two bagasse-based activated carbon 600/CS and CS 200) were a suitable adsorbent for sugar colour bodies as their because  $n > 1$ .

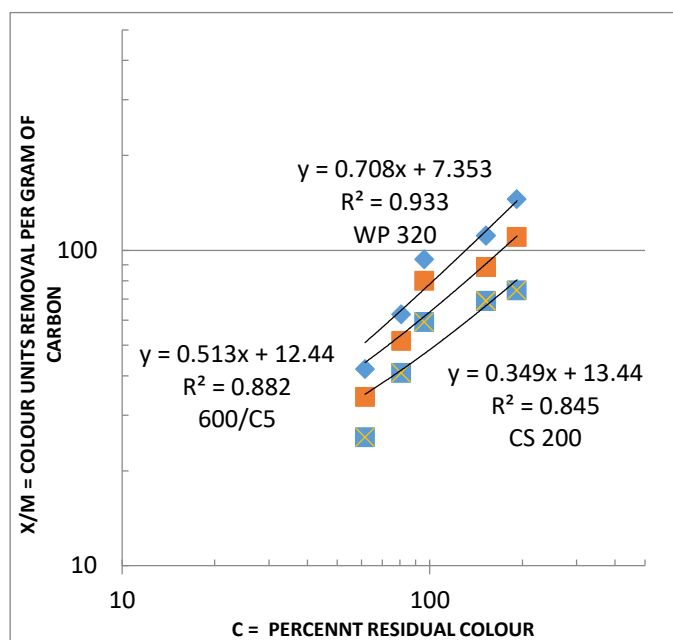


Fig. 3. Freundlich plot isotherms for the carbons

Table 2. The K and 1/n values for the activated carbon isotherms in Fig. 3

Carbon	K	1/n
WP 320	0.708	7.353
600/C5	0.513	12.4
CS 200	0.349	13.44

The adsorption mechanism depends on the physical and chemical properties of the syrup and also the mass transfer. The influence of the amount of adsorbent on the adsorption capacity has been studied (Nadeem *et al.*, 2008) and found that as the adsorbent mass is increased at fixed pH and initial adsorbate concentration, the adsorption capacity of the adsorbent was decreased. Similar results have been reported by (Ozmihchi and Kargi, 2006). They ascribed this phenomenon to the fact that at low adsorbent dosage, the adsorbate molecules were in excess of the binding sites on the surface of the adsorbent, whereas the opposite holds for high amounts of the adsorbent, that is the binding sites were in excess. The latter case will result in the incomplete occupation of the binding sites and thus lack of adsorbent surface saturation. Nevertheless, some literature indicate an increase in the removal efficiency of the adsorbent by increasing the adsorbent amount (Hakan Demiral *et al.*, 2010; Erika Suescún-Mathieu, 2014). Hence, an increase in the specific adsorbent loading will decrease the concentration of the adsorbate more efficiently and thus increase the removal efficiency. However, to determine the adsorption capacity, the mass of the adsorbent should be taken into consideration. In figure 3, it can be seen that at high residual colour concentration in the liquor, the colour loading on the carbon (X/M) is greater at lower levels. For example, in the

case of 600/C5, the first 0.2 g of carbon effected 147 colour units per gram with a decolourisation of 29.4 % however the second 0.2 g increased the decolourisation to only 44.4 % an increase of only 15 % and equivalent to 109 colour units per gram. The decolourisation percentages of 600/5 are comparable to WP 320, the first 0.2 g of carbon effected 36.7 % decolourisation (184 units per gram) and the second 0.2 g increased the decolourisation to only 58.5 % (152.8 units per gram) an increase of 21.8 %.

#### 4. Conclusion

This work has demonstrated that commercial activated carbon WP 320 with 73.1%, CS 200 with 59.2% and bagasse 600/C5 with 65.0% decolourisation efficiencies respectively are suitable for sugar syrup decolourisation and may be used raw sugar refining. The syrup decolourisation efficiency by bagasse based activated carbon, 600/C5 was close to WP 320 and therefore a potential low cost alternative for use in raw sugar refining.

#### Acknowledgement

The authors acknowledge Chemilil Sugar Company for providing sugar syrup, Kenya Industrial Research & Development Institute for providing the laboratory facilities and Sutcliff Carbons England for providing the commercial activated carbons.

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