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Analysis and Process Optimization for Carbon Black Production from Corn Cob

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ABSTRACT: Carbon black which is one of the massively consumed industrial chemical, has been produced from corncob using acid hydrolysis. The research has shown the possibility of raw material substitution for carbon black production from natural gas to corn cob. This study focused on the hydrolysis of the corn cob structure into small sugar which then was pyrolysed into carbon black. The effect of three factors i.e. temperature, hydrolysis time, and dosage on acid hydrolysis of hemicellulose to produce glucose was studied. Then the amount of glucose was determined by using UV-Visible spectroscopy. The highest amount was 0.073 g/mL. However, this is not the optimal value and optimization was done on the results. Analysis was done by Design Expert Software version 7.0.0 and CCD was selected for optimization. The effect of each factor and its interaction with the other factors were vital for the hydrolysis process. High amount of glucose, by that carbon black were determined at high levels of all the three factors. The content of final product, carbon black was analysed by elemental analysis and using FTIR its functional groups was identified. Five samples were suggested from optimization and the highest amount of carbon glucose was 0.058g/mL. Using elemental analysis, the amount of carbon found in the sample was checked and it was 67.9%. In addition, FT-IR analysis was done on the sample and using origin software the graph was plotted. And finally it was confirmed that the results have close resemblance with the standard carbon black characteristics.

KEYWORDS: Carbon black, corn cob, analysis, glucose, hydrolysis

INTRODUCTION

I.

Carbon black (CB) is a manufactured product that has been in commerce for over a century. Consisting of a fine black powder of nearly pure elemental carbon (EC), it has numerous applications in a variety of commercial and consumer products [1]. The plastic and tire manufacturing sector consumes raw materials that are mainly extracted from natural gas/oil. Among the raw materials, the master batches/colorants are the additives that give the final product its colour. Carbon black is used in tires, rubber and plastic products, printing inks and coatings. These applications are related to properties of its particle size and structure, specific surface area (i.e. surface chemistry which is the composition of hydroxyl and carboxyl groups in the entire structure), conductivity and color [2].

Nearly all of the world's carbon black is produced by two carbon black manufacturing processes, furnace black and thermal black, the furnace black process being the most common. The furnace black process uses heavy aromatic oils as feedstock. The production furnace uses a closed reactor to atomize the feedstock oil under carefully controlled conditions (primarily temperature and pressure). The thermal black process uses natural gas, consisting primarily of methane or heavy aromatic oils, as feedstock material. The process uses a pair of furnaces that alternate approximately every five minutes between preheating and carbon black production [3].

Natural gas is a non-renewable resource but also largely consumed as energy source and processed to produce so many varieties of products. Almost all sectors of industries consume one or more materials that are extracted from natural gas. Among those is carbon black. Not only its competitiveness of natural gas usage but also its immense release of hazardous chemicals to the environment are not negligible [2].

There is a possibility of producing carbon black from corn-cob. Usually, the corn cob is considered as an agriculture waste. This natural and organic waste material is not used as food, as bio material releases less pollution during processing, and reduces natural gas consumption extraction of carbon black from this would be much more feasible. Corn-cob mainly consists of carbon, hydrogen, and oxygen (i.e. cellulose, hemicellulose and lignin) [4].

In this study, sulphuric acid was used for hydrolysis of corncob, which degraded bio macromolecules into small molecules, and then it also performed well in facilitating polymerization of small molecules in the next process. Carbon black were prepared via the dehydration, polymerization and carbonization of sugars derived from sulphuric acid



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hydrolysis of corncob. The reactions occurred at low temperature and atmospheric pressure due to the catalysis of sulphuric acid, which was a mild condition to produce carbon black that could be industrialization in the near future. The effects of different acid concentrations on morphology and structure of the materials have been investigated in detail. The chemical properties and structural characteristics of the hydro chars were characterized by means of different experimental techniques.

II. RELATED WORK

Carbon black (CB) is an ancient material and has been produced for over a century by several traditional processes. CB is the name of a group of manufactured fine-particle products that have a variety of different trade names and physicochemical properties, but share a chemical composition of nearly pure elemental carbon (EC). The raw materials have been mainly residual oils from refineries and from natural gas. Among several types of carbon black Furnace black is made by partial combustion of oil, Channel black is made by partial combustion of natural gas and the carbon is collected on iron channels, Thermal black is made from the thermal decomposition of natural gas, methane [5].

Carbon black has been commercially produced for over a century with the year 2008 worldwide production totalling approximately 9.8 million metric tons. Rubber applications tire-related automotive uses (tires, tubes, tread), rubber automotive products (e.g. belts, hoses, miscellaneous), and nonautomotive industrial rubber products including industrial molded and extruded products dominate worldwide CB use patterns, consuming approximately 90% of CB used in the US, Western Europe, and Japan. The remaining 10% is divided among other special CB applications that include uses as a pigment, UV absorbing, and/or conducting agent in inks, coatings, and plastics (Long et al., 2013)

III. METHODOLOGY

The experiments were conducted at Kombolcha Institute of Technology and Addis Ababa Institute of Technology, Amist Kilo campus. Some of the analysis were done at Addis Ababa University College of Natural Science, Arat kilo campus.

A. Analysis of the contents of the raw material

The first step was analysis of the corn cob its moisture content and ash content. These were done in Kombolcha Institute of Technology and Addis Ababa University Institute of Technology respectively.

1) Moisture content determination: Moisture content was determined by adopting (AOAC, 2005) method. The moisture content was determined by using digital oven. The oven was set at 105 °C for 24 hours. The sample was weighed before and after drying in an oven. First of all, weight of empty previously dried (1 hr at 100 °C) crucible with cover was taken and 0.5g of corn cob substance was placed on it. Then the crucible was placed in an air oven and dried at a temperature of 105 °C for 24 hrs. After drying, the crucible was removed from the oven and cooled in desiccator. It was then weighed with cover glass. The crucible is again placed in the oven, dried for 30 minutes, took out of the dryer, cooled in a desiccator and weighed. Drying, cooling and weighing were repeated until the two consecutive weights were the same. From these weights, the percentage of moisture in corn cob substance was calculated as follows:

$$Moisture\ Content\ (\%) = \frac{Loss\ in\ Weight}{Weight\ of\ sample} \times 100$$
(1)

2) Ash content determination: AOAC, 2005 was used to determine the total ash content. About 0.5g of corn cob substance was taken in a dry, clean porcelain dishes and weighed accurately. Hot air oven method was applied to remove the moisture. Then the sample was burnt on a gas burner. This was done to avoid the loss of sample in the muffle furnace under higher temperature. Then sample is transferred into the muffle furnace and burnt for 4 to 6 hours at a temperature of 550 °C and ignited until light gray ash resulted (or to constant weight). The sample was then cooled in a desiccator and weighed. The ash content expressed as:

$$Ash \ Content \ (\%) = \frac{Weight \ of \ residue}{Weight \ of \ sample} \times 100$$
(2)

B. Identification of the optimal value of temperature, dosage and residence time for hydrolysis

In this research carbon black was produced using furnace. To produce carbon black the first step is converting hemicellulose to simple sugar using sulphuric acid. Acid hydrolysis was needed to change hemicellulose structure. Experimental design was analysed and done by Design-Expert 7.0.0 software. The experimental design selected for this study was Central Composite Design (CCD) and the response measured was glucose amount. Furthermore, the physicochemical analysis of the glucose was carried out. The variables used in this design were dosage, temperature and hydrolysis time and the variables range were selected based on the literature and shown in Table I.



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The 20 experiments were carried out and data was statistically analysed by the Design-Expert program to find the suitable model for the glucose amount as a function of the above three variables. The ranges and the levels of the variables investigated in this research were given in Table I.

S. No.	Factors	Experimental Level			Coded Levels		
1	Temperature (°C)	70	90	110	-1	0	+1
2	Dosage (g/mL)	0.03	0.12	0.20	-1	0	+1
3	Time (minute)	30	40	50	-1	0	+1

TABLE I: Experimental and Coded Levels of Three Variables Employed for Hydrolysis

Hydrolysis conditions were investigated at sulphuric acid concentration 72 (wt. %), ratios of biomass to sulphuric acid, reaction temperatures, and reaction times as shown in the above table. The reactions were carried out in 500 ml beakers placed magnet stirrer with heater. After completion of the reactions, the mixtures were allowed to cool to room temperature and filtered through a Buchner funnel. The filtrate was used for the preparation of carbon black, the solid residue was washed to neutrality with distilled water and dried at 110 °C overnight for weight determination.

1) Determination of sugar content: The sugar content was determined through phenol-sulphuric acid method by taking anhydrous D–glucose as standard as described below.

The total sugar concentration was determined by using UV-visible spectrophotometer at 490 nm wavelength of glucose absorbance and the quantification was made from calibration curve using glucose as standard and calculation was performed by equation of the linear regression obtained from calibration curve.

Stock glucose solution was made by dissolving 0.5 g of glucose in 100mL of distilled water. Various dilutions of the stock glucose solutions were made separately by pipetting a known volume of the stock solution (1, 2, 3, 4 and 5 mL) into a 100mL volumetric flask and filling the volume with distilled water up to the mark. The concentrations made for this study were: 0.05, 0.1, 0.15, 0.2 and 0.25 mg/mL.

The basic principle of this method was that carbohydrates when dehydrated by reaction with concentrated sulphuric acid, produce furfural derivatives. Further reaction between furfural derivatives and phenol develops detectible colour. The standard procedure of this method was as follows. A 2mL aliquot of a carbohydrate solution was mixed with 1 mL of 5% aqueous solution of phenol in a test tube. Subsequently, 5 mL of concentrated sulphuric acid was added rapidly to the mixture. After allowing the test tubes to stand for 10 min, they were vortexed for 30 s and placed for 20 min in a water bath at room temperature for colour development. Then, light absorption at 490 nm was recorded on a spectrophotometer. Reference solutions were prepared in identical manner as above, except that the 2mL aliquot of carbohydrate was replaced by distilled water [7].



Fig. 1 Standard curve for glucose

C. Production of carbon black at optimal value

After hydrolysis, the next experimental procedure was preparation of carbon black. For production of carbon black, the hydrolysed sample at optimal value was required. Tubular furnace was required for production of carbon black at optimal value. Beaker for holding the sample, thermometer to measure the temperature, magnetic stirrer with heater for stirring and heating, distilled water for washing and oven were required.



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Two-hundred and fifty millilitres of hydrolysis solution were poured into a 500 ml beaker equipped with a thermometer and a mechanical stirrer and heated at 95 °C for 6 h. The solids that formed were washed to neutrality with distilled water and dried at 120 °C overnight. The solid carbon was pyrolyzed in a tubular furnace at temperatures of 400-800 °C for 1 h under nitrogen gas. The rate of temperature increase was 20 °C/min.

1) Analysis of the physical and chemical characteristics of carbon black: Elemental analysis is the qualitative detection and quantitative determination of chemical elements in the analysed samples. To detect an element, one should fix an appearance of analytic signal in the formation of precipitate or characteristic crystals, colour change, an isolation of gaseous products, an appearance of definite lines in spectrum, luminescence etc. The elemental analysis for carbon black need EA 1112 Flash CHNS/O- analyser.

Before starting the analysis, there are conditions that should be fulfilled. For oxygen determination, the system should operate in pyrolysis mode. The conditions for the analysis were carrier gas flow rate of 120 mL/min, reference flow rate 100 mL/min, oxygen flow rate 250 mL/min; furnace temperature of 900 °C and oven temperature of 75 °C. Calibration was done with 5 calibration points for every component. Sample was run in duplicate and the average values are taken.

FT-IR spectroscopy and KBr are required for analysis. Spectroscopy is the study of the interaction of energy and matter. Absorption of energy of different magnitudes causes different changes in matter. The magnitude of energy absorbed is determined by the structure of the matter under study. Certain features of molecular structure such as the presence of functional groups with IR spectroscopy, arrangement of hydrogen and carbon atoms with NMR spectroscopy, or the extent of conjugation with UV/visible spectroscopy.

Determination of the complete molecular structure of an unknown substance is made easier when more clues are there. One useful clue is the molecular formula, which is provided by mass spectrometry. However, the formula alone is usually not enough for us to propose a single structure, because even a small collection of atoms often has several constitutional isomers.

FT-IR spectroscopy of the samples as powder-pressed KBr pellets was conducted in the wave number range from 4000 to 400 cm-1 at a resolution of 4 cm-1 using a spectrophotometer at room temperature.

The structure and the composition of products were analysed by X-ray powder diffraction (XRD) (SHIMADZU XRD-6000 diffractometer employing Ni-filtered Cu K α radiation, at a scanning rate of 6°/min with 2 θ ranging from 5° to 60°).

IV. EXPERIMENTAL RESULTS

The results for the above experimental work are presented below. The analysis for data was done by using design expert software. In this section analysis result of raw material, glucose amount after hydrolysis, and product analysis are presented.

A. Analysis of the contents of the raw material

The raw material was brought from Gondar, Amhara Region, Ethiopia. After bringing the raw material it was examined for ash content and moisture content. The average ash content is 0.967 (% w/w) and the average moisture content is 5.51 (% w/w).

B. Identification of the optimal value of temperature, dosage and residence time for hydrolysis

The main components of corncob are cellulose and hemicellulose which are carbohydrates. Cellulose and hemicellulose are polysaccharide and need a method for conversion of polysaccharides into monosaccharides. Basically, there are two methods for extraction of simple sugar from cellulose and hemicellulose. Acid hydrolysis is one of the method that can be used for conversion of polysaccharide to monosaccharide. The other method for hydrolysis is enzymatic hydrolysis. The reason for hydrolysis of corn cob in this experiment is to get simple sugar. Simple sugar can then be easily converted to carbon black.

The first step of the experiment was converting cellulose and hemicellulose by using acid hydrolysis. The level and the factors are shown in the methodology. The number of runs is 20 for optimizing the factors for glucose concentration. The number of run is decided by the method of analysis chosen. For this research central composite was selected. Central composite needs only twenty runs for optimization. By using UV-visible spectroscopy the absorbance was determined after hydrolysis and then the amount of glucose concentration was determined by using standard glucose curve which was plotted. The next part is analysing the data using ANOVA analysis. The significance of the data was analysed by using design expert version 7.

1) Optimization of experimental variables on hydrolysis of corncob: The main objective of the experiment was finding the best combination of the factors that can give high amount of glucose. The production of high amount of carbon black from corncob needs high amount of glucose concentration. Which is obvious. There are different factors that affect the yield of glucose from cellulose and hemicellulose using acid hydrolysis.



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The effect of temperature on glucose concentration is shown in Fig. 2. The figure shows response surface plot of glucose concentration with respect to temperature and dosage. On the left side of the graph the response variable glucose concentration is shown. The maximum amount of glucose concentration is found when both factors temperature and dosage are at highest value. The response surface plot is curved and it indicates that dosage and temperature have interaction. At maximum value of the factors the amount of glucose concentration is estimated to be less than 0.068 g/mL. Below the response surface plot there is contour plot which strength the above statements. It is curved due to there is an interaction effect of dosage and temperature.



Fig. 2 Response surface plots the effects of temperature and dosage on glucose concentration when time is at the centre point

The response surface plot is represented on the graph by green colour. The one which is represented by yellow is contour plot on the basement of the graph. These two lines are marked by red and black colour. The line which have red colour shows when the temperature is at high value and the one with black colour shows when the temperature is at low value. Based on these two lines the one with red colour at high temperature produces high amount of glucose concentration. However, the line which is depicted by black colour at low temperature produces low amount of glucose.

From low value of the factor to higher value the amount of glucose produced is shown with different colour. The low value of the response variable is shown with blue-green colour.

Fig. 3 clearly shows response surface plot of glucose concentration with respect to temperature and time. The response variable is represented by actual and coded factors with equation in the analysis. In these representations all of the coefficients in the assumed model are significant and the quadratic model is acceptable. The response surface plot is obtained from hydrolysis temperature and time has curvature. This is an indication that there is interaction between the two factors temperature and time on glucose concentration.



Fig. 3 Response surfaces plots of the effect of temperature and time on the glucose concentration when dosage is at the centre point

The plot proposes that there were well defined optimum operating conditions. The maximum amount of glucose is found at high temperature and time. High dosage of hemicellulose high amount of glucose concentration is predictable. Using response surface plot, the significance of the coefficients in the model is checked. At the four edges of the graph the amount of response variable is shown. The maximum amount of the response variable glucose concentration is



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found when dosage and time are at maximum. On the contrary when dosage is high and time is low the amount of glucose produced is 0.054 g/mL. When hydrolysis time and dosage are at low value the amount of glucose is greater than when dosage is low and time is high.



Fig. 4 Response surface plots of the effects of dosage and time on the glucose concentration when temperature was at the centre

The maximum amount of glucose is produced when these factors are at high level. However, to make sure where to operate optimization was done.

D. Production of carbon black at optimal values of hydrolysis

The main focus of this research was on the hydrolysis of carbon black. After hydrolysis, the next step was production of carbon black. From hydrolysis experiment the optimal amount of glucose determined and based on that by selecting the maximum value carbon black was produced. After production of carbon black, the next step was analysis of the product. The product was analysed with standard.

1) Analysis of the physical and chemical characteristics of carbon black: Using EA 1112 Flash CHNS/O- analyser the elemental analysis for carbon black was investigated. Carbon, hydrogen, nitrogen, and sulphur (CHNS) are fundamental elemental components that were analysed.

No	Temp	Dos. (g/ml)	Time (min)	N (%)	C (%)	H (%)
	(°C)		. ,			
1	99.25	0.18	45.48	0.18	59.5	2.8
					6	6
2	78.81	0.12	30.32	0.21	51.5	3.2
					4	9
3	85.66	0.03	31.57	0.21	67.9	3.1
					4	4
4	105.1	0.14	37.5	0.20	56.7	2.4
	8				0	9
5	89.86	0.12	32.21	0.20	49.6	2.5
					3	8

T.	AB	LE	2:	Elemental	Anal	lysis
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(e)

Fig. 5 FT-IR analysis for (a) sample 1, (b) sample 2, (c) sample (3), (d) sample (4) and (e) sample (5)

From elemental analysis it possible to deduce how much amount of nitrogen, carbon, hydrogen and sulphur were found in the sample. From the table the sulphur part is removed because it is zero. Run number three has the highest amount of carbon content and run number five has lowest amount of carbon. This might happen due to glucose concentration after hydrolysis is low. The sulphur content for all sample is almost zero. It is found in trace amount. Sample two has highest amount of hydrogen and the lowest amount is found in sample four. To check whether the amount of elements in the samples were acceptable or not literatures written by scholars were checked. The amount of nitrogen, carbon, hydrogen and sulphur are relatively similar with what was wrote by [8].

Using FT-IR these samples were analysed and graphs were plotted. The standard IR spectrum graph of carbon black is shown in Fig. 5. When transmittance is high which indicated by peaks pointing up show weak absorption but, when the transmittance is low which is indicated by peaks pointing down show strong absorption. Using origin software version 8 the following graphs were plotted.

When carbon black analysed using FTIR the picks on the graph shows different functional groups. The band width ranging from 3800-3400 cm-1 is a proof of the presence of phenolic hydroxyl groups on the samples. The second broad band at 3300 - 2800 cm-1 was identified with O - H stretching vibrations, probably from O - H (carboxylic) group. The peak at 1724 wavelength shows C = O stretch. At band width 1574 C = C double bond is shown. The peak at wave



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number 1263 is for P = O stretch. The last one is C - O stretch and it occurs at wave number 1100. These phenomena are shown on the samples investigated.

In Fig. 5 what expected is happened, all the stretches are shown on the graphs. The graphs clearly show stretch in the band width 3800-3400 cm-1. This stretches shows phenolic (O-H) stretch. In addition, on the graphs carboxylic (O-H) stretch is shown in the band width ranging from 3300-2750 cm-1. At 1724 cm-1 wavenumber the graph point upward direction and it shows C = O. And also at 1263 P = O stretch is shown on the graph. The last thing which was checked based the standard is C - O stretch and it is shown at 1100 cm-1 on the graph. The existence of these functional groups are evidence for checking whether the samples are carbon black or not. If the sample showed these peaks in a given band width it is carbon black.

Moreover, standard graph of carbon black was checked in order to have good conclusion about carbon black. The standard graphs show the same phenomena with the samples.



For the carbon black from corn cob prepared under different temperature, dosage and time, X-ray diffraction patterns (Fig. 7) show two peaks at about 20° and 44°. The pattern shows a broad diffraction peak in a 20range of 10°–30°. Our wide-angle XRD pattern matches well with that previously reported [7] for non-graphitic carbon. While localized graphitization is manifested by the diffraction peak at 44°, which assigned to the plane (100). It was impossible to completely graphitize in such a low-temperature system, so the macroscopy of the samples shows amorphous structure.



Fig. 7 XRD of carbon black prepared under different temperature: (a) 99.25°C, (b) 78.81 °C, (c) 85.66 °C and (d) 105.18 °C.

V. CONCLUSION

Carbon black can be produced from biomass containing carbon in the form of carbohydrate. There are agricultural byproducts which should be utilized and need focus. This research aimed at extracting the glucose part of corn cob. In this research hydrolysis of corncob were done using acid hydrolysis. In this study, optimization of hydrolysis for production of glucose from corn cob was investigated. The effects of temperature, hydrolysis time and dosage on the amount of glucose were studied. UV-visible spectroscopy was used to analyze the amount of glucose. The highest amount of glucose is 0.073 g/mL. However, this amount is not optimal and need optimization. Using Design-Expert version 7.0.0, three-level-three-factor Central Composite Design (CCD) the result was analyzed. The model chosen was quadratic and the model is significant since its p-values is less than 0.05. Optimization of the factors were accomplished and the effect of factors were checked. When hydrolysis time, temperature and dosage increase the



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amount of glucose produced increases. Hydrolysis time and dosage have interaction in the production of glucose. The optimization showed that all factors have significant interaction. The optimization selected five combination of the factors and the highest optimal amount of glucose produced was 0.058 g/mL.

After optimizing the hydrolysis process the next step is pyrolysis. Five combination of the factors were selected and pyrolysis of samples were done for carbon black production. And the samples were analyzed by using elemental analysis and FT-IR analysis methods. Elemental analysis measures the percentage of carbon, hydrogen, sulfur and nitrogen. The maximum amount of glucose gave high amount of carbon black as it was expected. The maximum amount of carbon in the elemental analysis was 67.94%. This result is nice and found in acceptable region. During FT-IR analysis using KBr pellets the carbon black sample showed the same trend like the standard graphs. The graphs showed in the band width 3800-3400 cm-1, 3300-2750 cm-1, phenolic (O-H) and carboxylic (O-H) stretch respectively. At 1724 cm-1, 1263 cm-1, and 1100 cm-1 wavenumber the graph shows C = O, P = O, and C - O stretch respectively. This functional groups are evidences for the samples are carbon black.

Therefore, it is possible to conclude using elemental and FT-IR analysis what were produced are carbon black. Causal Productions has used its best efforts to ensure that the templates have the same appearance.

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