

Potentials of Cassava Starch for the Production of Sorbitol.

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Abstract

Cassava starch is widely used as an industrial raw material for food, pharmaceutical, textile and chemical industries. Sorbitol also known as glucitol, is a sugar alcohol (polyol) with a chemical formula $C_6H_{14}O_6$; produced industrially by hydrogenation of carbohydrates using catalysts at high temperatures. It is found in nature and exists in fruits and vegetables, mushrooms, and also in human beings. Sorbitol is used in both food and non-food applications. The potentials of cassava starch for the production of sorbitol was investigated. Cassava starch was initially hydrolyzed to glucose by adding 1M sulfuric acid at 100°C for 2 h -4 h respectively. The glucose samples were subsequently reduced to sorbitol by using 0.5M sodium borohydride. Physicochemical analyses of the glucose syrup produced were done according to standard methods. Dextrose equivalent (DE) which is a measure of the reducing sugar in glucose samples was determined using dinitrosalicylic acid (DNSA) method. The sample of glucose syrup was reacted with a mixture of alkaline solution of DNSA and aqueous solution of sodium potassium tartrate. Color extinction was measured with a UV visible spectrometer. DE was calculated from the standard curve obtained from plotting absorbance versus concentration of known glucose samples. The conversion of cassava starch to glucose and sorbitol respectively was verified using Fourier-transform infrared spectroscopy (FTIR). The results show that the specific gravity and pH of glucose syrup are between 1.211 g/cm³ and 1.375 g/cm³, 6-7 respectively. The calculated apparent degrees Brix ranges between 49% and 87%. The color of glucose syrup produced was golden yellow; the dextrose equivalent of the samples is between 19 and 33. The FTIR spectra showed the presence of functional groups C=O aldehyde stretching and sp³ C-H aldehyde; C-O-alcohol stretching for glucose and sorbitol respectively. These indicate that the samples produced are glucose and sorbitol respectively. The result of the preliminary investigation indicates that cassava starch has good potential for the production of sorbitol.

Keywords: Cassava starch, Glucose syrup, Hydrogenation, Sorbitol, Sodium borohydride.

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I. Introduction

Cassava (*Manihot esculenta* Crantz) is a major food security root crop in Sub-Saharan Africa cultivated mostly for its starchy tubers. It is the second most important source of starch globally after maize [1]. Nigeria is the largest producer of cassava with about 59.5 million tons produced in 2018; representing 21% of the world total. However, about 90% of cassava produced in Nigeria are consumed locally without any value addition. Other major producers of cassava roots include Thailand (31.7%) and the Democratic Republic of Congo (30.0%) [2].

Cassava is a good energy source providing about 160 kcal/100g. Globally, about 800 million people depend on it as their primary energy food staple [3]. In addition to its food uses, evidence suggests that cassava has medicinal values for the management of diabetes and dyslipidemia [4]. Cassava is also a source of raw material for industrial applications. Some of the cassava products include high-quality cassava flour (HQCF), cassava chips, garri, ethanol biofuel feedstock, starch etc. Cassava starch finds utilization as an industrial raw material for food, pharmaceutical, textile, and chemical industries [5].

Sorbitol ($C_6H_{14}O_6$) is a sugar alcohol (polyol) with six-carbon atoms and six hydroxyl groups

(fig. 1), found in the berries of a tree called 'the mountain ash', by a French Chemist in 1872. Also, sorbitol occurs naturally in fruits such as apples, pears, peaches, apricots, and nectarines. Other natural sources include dried fruits like prunes, dates and raisins, and in some vegetables [6].

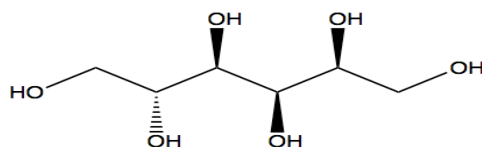


Fig. 1. Chemical structure of sorbitol.

Sugar alcohol (polyol) is a polymerised sugar with an alcohol functional group. There are seven sugar alcohols approved globally for use in food products; they are sorbitol (E420), mannitol (E421), isomalt (E953), maltitol (E965), lactitol (E966), xylitol (E967), and erythritol (E968). These sugar alcohols are certified as safe by Food and Drug Administration (FDA). They are Generally Recognized As Safe (GRAS). However, excess consumption of them may have laxative effect and other gastrointestinal symptoms like flatulence, bloating and abdominal discomfort [6]. Sorbitol (E420) is approximately 60% as sweet as sucrose (table sugar) with an average caloric value of 2.7 kcal/g compared to 4 kcal for sucrose [6- 8]. It is highly hygroscopic, chemically inert, and stable at high temperatures. Unlike sugar, sorbitol does not undergo Maillard reactions [6, 9]. Currently, sorbitol represents the world's most consumed polyol in nutrition, cosmetics, medical and industrial applications [10, 11]. The global value in 2018 stands at more than 2.5 million metric tons, with the market demand currently growing at 2-3% annually. The Sorbitol market is expected to register a compound annual growth rate of 5.5% for the period 2018-2023. [12]. This trend is due to extensive applications of sorbitol in various industries (fig. 2).

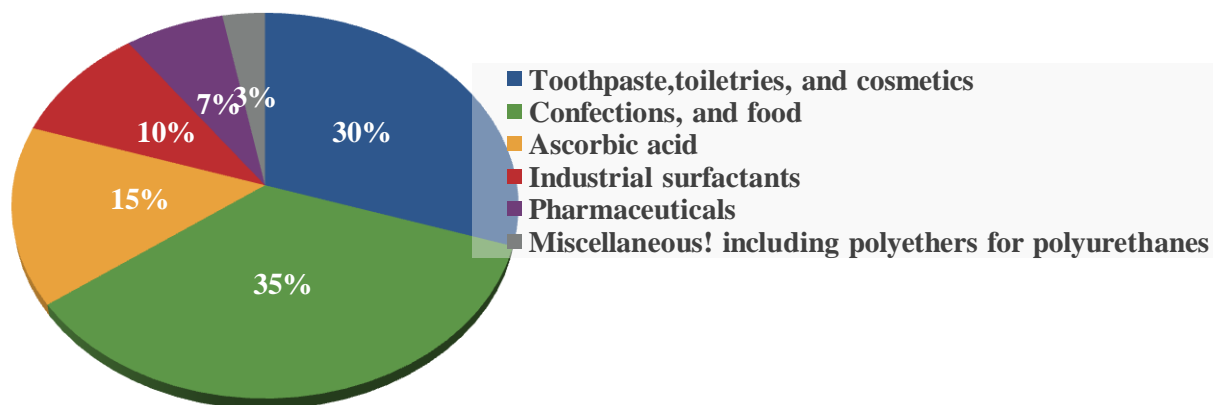


Fig. 2. End Use Pattern for sorbitol.

Adapted from Kirschner, M., 2007. ICIS Chemical Business [13].

Sorbitol is used in food and beverages (as a sweetener, texturizer, and softener), in pharmaceutical as a carrier due to its solubility properties, sweet taste, and cooling sensation, cosmetics as a humectant and an emulsion stabilizer, resins as a polyol, toothpaste as a sweetener, humectant, etc.[13]. Sorbitol is non-cariogenic due to its resistance to digestion by oral bacteria. It does not break down to sugars and starches that promote tooth decay. It serves as a major raw material in the manufacture of Vitamin C [11, 14]. Other uses include parenteral preparations used in osmotic diuretics in the prevention and treatment of renal failure, shock, and the treatment of various intoxications. It is also used as a food additive in the management of liver failure, for the treatment of water and mineral imbalance associated with some oedemas [15]. Sorbitol is a low-calorie nutritive sweetener widely used in food to replace sugar in food products like diet drinks, diet ice cream, mints, cough syrups, sugar-free chewing gums, flavor concentrate, dried roasted nuts, icings, and for garnishing food products [16, 17]. It has a low glycemic index of 9 as compared to sugar with a glycemic index of 69 [18, 19, 20]; and this makes it suitable as part of a diabetic diet. It has been reported that no significant increase in blood sugar was observed after intake of ice cream with 35g sorbitol [21]. The low glycemic index explains increasing consumer preference towards low-calorie foods as a result of the global increase of patients with diabetes.

According to WHO, diabetes (Hyperglycemia) is a chronic metabolic disease characterized by elevated levels of blood glucose (or blood sugar), which can cause serious health complications like damage to the heart, blood vessels, eyes, kidneys, and nerves. Type 2 diabetes is the most common type of diabetes among adults. It is caused by resistance to insulin or insufficient production of insulin thereby resulting to too much sugar in the body. Diabetes is a major cause of death globally with an estimated 463 million people affected globally; having

4.2 million deaths in 2019. The prevalence of diabetes is estimated to rise by 25% (578 million) by 2030 and 51% (700 million) by 2045 [22, 23]. In Sub-Saharan Africa, Nigeria has the highest number (approx. 3.9m) representing approximately 4.99% of adult population (20-69 years). The increase in diabetes affects the quality of life with the global diabetes-related healthcare expenditure estimated at 760 billion USD, representing a 14.5% increase from 2017 [22]. Due to the rise in diabetes-related mortality and the association between diet and diabetes, there is a renewed interest for sugar substitutes or nutritive sweeteners with low-calories. Sorbitol is one of the recommended sugar substitutes with low-calorie value for a healthy lifestyle and management of diabetes.

Sorbitol is a hydrogenated form of a monosaccharide; industrially produced by reduction of d-glucose from biomass in a high-pressure reactor using hydrogen gas in the presence of a catalyst [24 - 29]. However, epimerization of glucose in an alkaline medium into mannose can easily occur and subsequently hydrogenated into mannitol. Therefore a suitable pH 7.5 is recommended for glucose conversion to avoid epimerization [30]. Enzymatic production of sorbitol from *Z. mobilis* and *Candida boidini* has also been reported [6, 13, 31]. Glucose is reported to be the best source of sorbitol production due to its availability and low cost [32].

Glucose syrup is mainly derived from corn starch. Meanwhile, there is limited information on the use of glucose syrup from cassava starch for the production of sorbitol using a metallic hydride. The objective of the present study is to investigate the potentials of glucose syrup from cassava starch for the production of sorbitol using sodium borohydride and also the effect of reaction time on conversion of cassava starch to glucose syrup. This is the first report on the production of sorbitol by hydrogenation of glucose from cassava starch using a metallic hydride.

II. Method

2.1 Materials and chemicals

All chemicals were of analytical grade. They include Cassava starch, Sulfuric acid, Hydrochloric acid, Sodium Hydroxide, Fehling's solution A, Fehling's solution B, Filter paper, Cotton wool, Muslin cloth, Ferrous sulfate, Sodium Hydroxide, Sodium Borohydride, Dinitrosalicylic acid (DNSA), Sodium potassium tartrate.

2.2 Sample preparation

Cassava starch was purchased from a local market in Lagos, Nigeria. The starch was washed with water to remove extraneous materials. The mixture was filtered using a fine pore muslin cloth. The filtrate was kept overnight to separate out. The supernatant was decanted and the starch washed again to obtain a white odourless starch. The starch was allowed to dry under the sun until constant weight. The moisture content of the dried starch was determined in triplicate using a moisture analyser.

2.3 Production of sorbitol

Sorbitol production followed a 2-step conversion of glucose into sorbitol. Starch, a linear polymer of glucose with α -1, 4-glycosidic bonds, was hydrolyzed into glucose and subsequently hydrogenated into sorbitol.

1. Acid hydrolysis of the cassava starch

The starch slurry was prepared with 1M sulfuric acid (1:3), starch to sulfuric acid ratio. The slurry was refluxed at 100°C for a period of 2 to 4 h. respectively. Test for starch using iodine was achieved after 1 hr of refluxing. The slurry was further heated until the test for presence of reducing sugar, as evidence of hydrolysis using Fehling solution was achieved. The reaction was cooled and neutralized with 1M sodium hydroxide solution. The neutralized solution was concentrated under low heat to avoid caramelization of the sugar.

2.3.1.1 Test for reducing sugar

Dinitrosalicylic acid (DNSA) method was used for estimating the concentration of reducing sugar in the sample. An alkaline solution of DNSA (1 g DNSA dissolved in 20 mL 2M NaOH) mixed with an aqueous solution of sodium potassium tartrate (30 g in 50mL distilled water) was reacted with the glucose syrup. The mixture was placed in a boiling water bath for 5 min, then it was cooled by immersing in cold water bath. The intensity of colour as observed is an index of reducing sugar. Water was added and the extinction of the coloured solutions (orange) was read at 540 nm using a blank (control) prepared with 1 ml distilled water and 1 mL DNSA. The standard curve of absorbance versus reducing sugar concentration was plotted. The amount of reducing sugar in the sample was calculated from the standard curve.

The apparent Brix was calculated by using a pycnometer.

Calculations:

$$\text{Degrees Brix} = 231.61 \times (S - 0.9977).$$

S = apparent specific gravity.

Dilution factor (DF) = final volume / aliquot volume

The amount of carbohydrate in the sample =mg/dl x DF

2. *Hydrogenation of glucose syrup*

Sorbitol was produced by hydrogenating the aldehyde group in glucose into a hydroxyl group using sodium borohydride. Hydrogenation of glucose syrup samples was done using 0.5M of sodium borohydride solution. The metallic hydride solution was added continuously through the dropping funnel into the reaction flask containing the glucose syrup for a period of 2 h. at room temperature. At the end of the addition of the hydrogenating agent, the reaction mixture was heated between 60°C to 70°C. After 1 h, the mixture was tested for the presence of glucose. At the end of the reaction, water was added to the reaction mixture and stirred for about 30 min. The sample was evaporated and Fehling’s solution was used to test for the presence of sugar. Then sorbitol crystals were precipitated with absolute ethanol and analysed by FTIR.

4. *FTIR*

Spectral information of glucose syrup and sorbitol crystals samples was used to identify the functional groups present. FTIR spectra were recorded in duplicate at 100°C using FTIR (Agilent technologies) spectrometer in the range of wavelength from 4000-650 cm⁻¹, with background scan of 32 at 8 cm⁻¹ resolution.

III. Results and Discussion

The physicochemical properties of the glucose syrup produced are as shown in Table 1. The moisture content value (15%) was used to adjust the sample weight for the slurry. The degrees Brix was calculated based on apparent specific gravity. It is “apparent” because it is a relative value. The effect of resident time on the conversion of starch to glucose is as shown on table 1. From the result obtained, the percentage Brix and dextrose equivalent (DE) increase with higher resident time. The DE was calculated from the line equation obtained from the standard curve (Fig. 3).

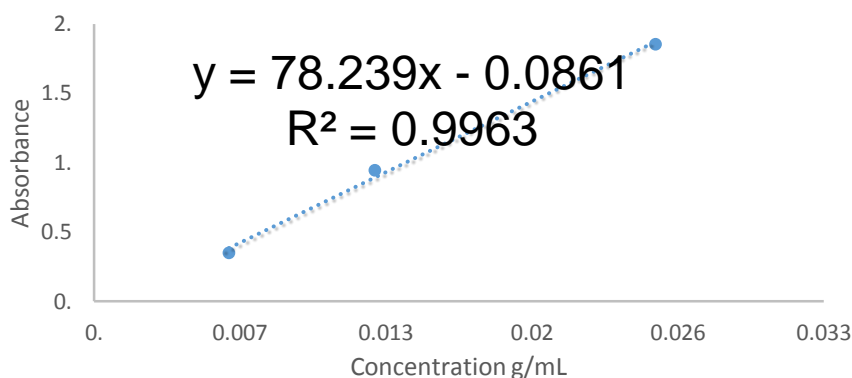


Fig. 3: Standard curve for glucose samples

The correlation equation was established using regression line:

Y = 78.239X - 0.0861 with correlation coefficient R² = 0.996

Table 1. Physicochemical properties of glucose syrup.

| Time (h.) | 4 | 3 | 2 |
|---|-------|---------------|---------------|
| Specific gravity g/cm ³ (20°C) | 1.375 | 1.249 | 1.211 |
| pH | 6-7 | 6-7 | 6-7 |
| Color | Dark | Golden yellow | Golden yellow |
| DE | 33 | 23 | 19 |
| % Apparent Brix | 87 | 58 | 49 |
| Temperature °C | 100 | 100 | 100 |

DE dextrose equivalent

Table 2 shows the relationship between resident time and DE of glucose syrup produced.

Table 2. Dextrose equivalent of samples

| Reaction time h. | Absorbance cm^{-1} | SD | DE |
|------------------|-----------------------------|-------------|----|
| 4. | 2.4868 | ± 0.042 | 33 |
| 3. | 1.7315 | ± 0.023 | 23 |
| 2. | 1.3799 | ± 0.005 | 19 |

The result obtained shows DE values between 19 and 33. The DE increased with resident time and also shows a direct relationship with the apparent Brix value. These values compare with commercially available liquid glucose syrup having DE more than 20.



Sample ID:G2 FIRO
 Sample Scans:32
 Background Scans:32
 Resolution:8
 System Status:Good
 File Location:C:\Users\Public\Documents\Agilent\MicroLab\Results\G2 FIRO_2019-07-29T23-11-18.a2r

Method
 Name:C:\Users\Public\Documents\Agilent\MicroLab\Methods\Agilent Default.a2m
 User:Admin
 Date/Time:07/29/2019 11:11:18 PM
 Range:4000 - 650
 Apodization:Happ-Genzel

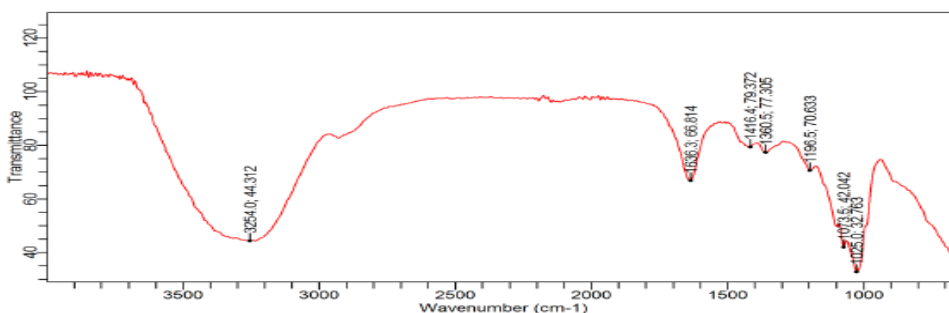


Fig. 4: FTIR spectra of Glucose sample



Sample ID: S1 FIRO
 Sample Scans: 32
 Background Scans: 32
 Resolution: 8
 System Status: Good
 File Location: C:\Users\Public\Documents\Agilent\MicroLab\Results\S1 FIRO_2019-07-29T23-17-09.a2r

Method
 Name: C:\Users\Public\Documents\Agilent\MicroLab\Methods\Agilent Default.a2m
 User: Admin
 Date/Time: 07/29/2019 11:17:09 PM
 Range: 4000 - 650
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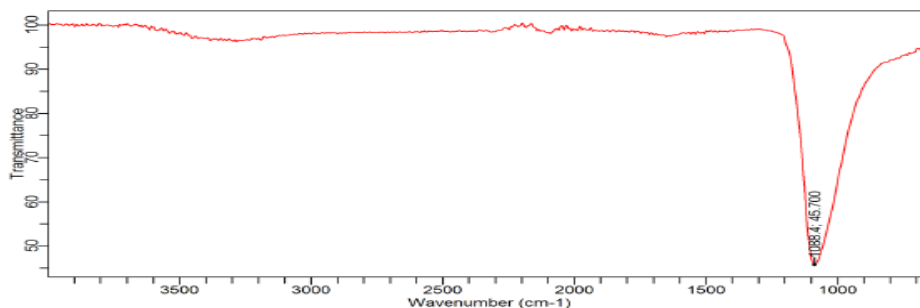


Fig. 5: FTIR spectra of Sorbitol sample

Table 3. Comparison of FTIR frequencies of glucose and sorbitol

| Wavenumber cm^{-1} | Assignments | |
|-----------------------------|--|----------------|
| | Glucose | Sorbitol |
| 3254.0 | O-H stretching | O-H stretching |
| 1636.3 | C=O aldehyde stretching | |
| 1025.0 | C-O, C-C stretching | |
| 1360.5-1416.4 | Bending of O-C-H, C-C-H, C-OH | |
| 2900-3000 | sp^3 C-H aldehyde | |
| 1088.4 | C-O-alcohol stretching | |

The obtained FTIR spectra of glucose and sorbitol samples agree with literature values. Comparison of the FTIR spectra of the samples showed a characteristic broad-band observed at 3200-3500 cm^{-1} which was ascribed to the stretching of hydroxyl groups (O-H) in the glucose sample. However, this was not very intense in sorbitol spectra. This could be due to the use of high values of nominal resolution which lead to the deformation of the absorption band and or decreased hydrogen bonding of water. Similarly, the peak area at 1636 cm^{-1} indicates the presence of free carbonyl group COO associated with aldehydes. The peak of glucose

(1025 cm^{-1}) is characteristic of a C-O stretch vibration, peak area at 1360.5-1416.4 cm^{-1} (bending of O-C-H, C-C-H, C-OH) was not visible in sorbitol spectra. Similarly, the vibrational band observed between 2900-3000 cm^{-1} ascribed to C-H stretch of aldehyde is not found in the sorbitol spectra. The most important band for the sorbitol sample presented a maximum absorption at

1088 cm^{-1} . This is ascribed to a C-OH stretching vibrations of sorbitol. According to Castro and Cassella (2016), [33] the stretching vibrations of sorbitol C-OH were centred at approximately 1046 cm^{-1} and 1084 cm^{-1} .

IV. Conclusion

The potential of cassava starch for the production of sorbitol was investigated. Glucose syrup produced from cassava starch was converted to sorbitol by using sodium borohydride as the reducing agent. Crude sorbitol crystals were obtained from the liquid sorbitol. The results obtained show that cassava starch has a potential to be used for the production of sorbitol. However, further studies are required for the separation and

purification of sorbitol after production. This preliminary study shows the potential to increase the value chain of cassava, minimize loss of cassava tubers due to poor storage, create wealth, and also maximize its overall health and economic benefits. This study provides for the first time the potentials of cassava starch for the production of sorbitol using a metallic hydride.

Conflict of interest

The authors declare no conflict of interest.

The material as contained in this manuscript has not been previously published and is not being concurrently submitted elsewhere. The authors declare there are no similar manuscript published or in press.

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