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Production and Quality Assessment of Glucose Syrup from Selected Biomass Sources

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Authors' contributions

Author DIG designed and supervised the study as well as producing the final manuscript. Author KGT managed the analyses of the study and produced the first draft of the manuscript. Author BDI managed the literature searches and proof read the first draft of the manuscript. All authors read and approved the final manuscript.

Original Research Article

Received 11th September 2013 Accepted 28th October 2013 Published 12th December 2013

ABSTRACT

Aims: Studies were conducted on the production and quality evaluation of glucose syrup from three different types of biomass: Eucalyptus sawdust (soft wood), Mahogany sawdust (hard wood), and Rice husk.

Study Design: A 3x1x1x3 Factorial design was used.

Place and Duration of Study: Department of Food science and Technology, University of Agriculture, Makurdi, Nigeria, between October 2009 and October 2013.

Methodology: The biomass samples, after thorough cleaning, were pretreated with 1% Sodium hydroxide (NaOH) solution and then hydrolyzed with 5% Hydrogen tetraoxosulphate six (H_2SO_4) in an autoclave at 120^oC for 30 min. The extract was filtered to obtain clear glucose solutions, which were evaluated for some physico – chemical and nutritional properties using standard methods of analysis.

Results: There were significant differences (P = .05) in the yield and glucose content of the syrups, with values ranging from 59.33% to 70.40% and 21.63% to 25.20% respectively, with Eucalyptus sawdust having the least value and Rice husk the highest. There were also significant differences (P = .05) in the nutritional properties with values ranging from 1.60% to 2.01% (crude fat), 2.40% to 3.01% (ash), 4.50% to 5.08% (crude

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protein), 81.57% to 87.99% (moisture content), and 3.11% to 9.60% (carbohydrate). Rice husk had the highest moisture, protein and carbohydrate contents, while soft wood had the highest fat and ash contents. Mineral and heavy metal content also showed a similar trend. Toxicant content also differed significantly (P = .05) in the different biomass samples with soft wood and rice husk giving higher and lower values respectively. **Conclusion:** Biomass therefore has the potential of being converted to useful products such as glucose syrup. Rice husk produced the best quality glucose with higher yield, followed by soft wood and hard wood in that order.

Keywords: Glucose; syrup; soft wood; hard wood; rice husk; yield; toxicants.

1. INTRODUCTION

Agricultural and forestry products, though very useful to man, generate a lot of waste products (Biomass) from their processing, which constitute environmental hazards. Such wastes include agricultural by products such as cereal husks and hulls, corn cobs, wood dropping or residues (sawdust), polyethylene packaging materials, plastics and animal excreta. If not reprocessed into useful products such as particle board, burned in a sawdust burner or used to make heat for other milling operations, sawdust and rice husks may collect in piles and contribute harmful leachates into local water systems, creating environmental hazards [1]. For example, water-borne bacteria digest organic material in leachates, but use up much of the available oxygen, thus creating a high Biological Oxygen Demand (BOD), which could suffocate fish and other aquatic organisms as well as exert a detrimental effect on beneficial bacteria. Of greater concern are substances such as lignin and fatty acids, which protect trees from predators while they are alive, but can leach into water and poison wild-life. These slowly get broken down as the crops or their residues decay, thus getting into runoffs, causing toxicity to a broad range of organisms [2].

Chemically, wood (sawdust) and cereal husks are composed of 60 - 68% polysaccharides (cellulose, hemicelluloses and lignin) and other extractives and non - extractive extraneous materials. The extractives include fats, waxes, tannins and resins, while the non-extractives include inorganic components such as silica and oxalates [3]. The cellulose and hemicelluloses components of these materials are essentially long, molecular chains of sugars, which are protected by lignin, which serves as the glue that holds the components together. The large quantities of ligno-cellulolusic wastes, generated through forestry and agricultural products as well as industrial processes, particularly from agro-allied industries such as breweries, paper, pulp, textiles and timber industries, are therefore potential sources of sugar. Conversion of these biomass sources to useful products (such as glucose) will serve the dual purpose of waste management and wealth generation, thus ensuring food security.

As early as 1922, Kressman hydrolyzed wood products into sugars for the microbiological production of alcohol [3]. Khan [4] also reported that pure cellulose could be totally hydrolyzed by treatment with a strong mineral acid, followed by dilute acids. Weak alkalis such as 0.05N Sodium hydroxide (NaOH) have also been used. Several other researchers have produced glucose syrup from biomass for the purpose of conversion to ethanol [5]. However, comparative information on the yield and quality parameters of glucose syrup from different types of biomass is scanty.

Several technological hurdles have limited the production of food grade glucose for human use. First, the separation of lignin from cellulose and hemicelluloses to make the materials available for hydrolysis is difficult. Secondly, the hydrolysis of cellulose and hemicelluloses occurs at different rates, thus creating the possibility of over-reaction of one of them. Thirdly, the hydrolysis of the materials produces a variety of sugars (glucose, hexose and pentose) [5].

The current effort is therefore, aimed at comparatively assessing the production yield and quality of glucose syrup from sawdust (soft and hard wood) and rice husk, with a view to determining if it is economical and could be used directly as human food or as a raw material for the production of useful industrial products for human consumption.

2. MATERIALS AND METHODS

2.1 Material Procurement

The biomass samples used in this experiment were obtained as follows: Mahogany and Eucalyptus sawdust were collected from North Bank timber mill, while the Rice husk was obtained from Olam Rice Mills at North Bank, Makurdi, Nigeria. The biomass samples were collected from the sources in a clean polythene bag and taken to the laboratory for preparation.

2.2 Preparation of Raw Materials

The biomass sources were washed with tap water until when the water got clear. They were then sun dried to about 10.0 - 12.0% moisture content, after which they were digested with 5.0% caustic soda. The pulp was washed with clean water, until when it was free from the caustic soda according to the method of [6]. They were further dried milled into powder and sieved through a sieve (2mm).

2.3 Hydrolysis of the Biomass

The hydrolysis of biomass samples was carried out in an autoclave with the volume of approximately 3I, equipped with an electric heater and temperature control according to [6]. Initially, 20.0g each of the biomass was weighed into a conical flask of 500ml capacity, to which 200ml of 5.0% concentrated Hydrogen tetraoxosulphate six (H_2SO_4) was added. It was then heated to $120^{\circ}C$ for 30 minutes to hydrolyze into an extract. Thereafter, 20 ml of 5.0% Sodium hydroxide (NaOH), and 10.0g activated carbon were added to neutralize, decolourize and deodourize the extract.

2.4 Analyses

2.4.1 Physical properties

Production Yield was determined by material balancing, while glucose content, Total Soluble Solids (⁰Brix) were determined using a hand refractometer (ATAGO, Model R5000) as described by [7]. Total Dissolved Solids were also determined by the method of [7], while the viscosity of the glucose syrups was determined using a digital viscometer (A&D Vibro, Model SV-10) as described by [8]

2.4.2 Chemical properties

Moisture, crude protein, crude fat, and ash contents were determined using the method of [7], while carbohydrate was determined by difference [9]. pH was determined by the method of [10], while minerals and heavy metals were determined using Atomic Absorption Spectrophotometer (BUCK SCIENTIFIC, USA, Model 210 VGP) as described by [11].

2.4.3 Toxicants

Total phenols, phytates, oxalates, alkaloids, hydrogen cyanide and polyaromatic hydrocarbons were all determined by High Performance Liquid Chromatography using a single wavelength isocratic HPLC (Buck Scientific BLC – 10, 254nm UV absorbance detector) as described by [7]. About 2g of sample was weighed into a 25ml volumetric flask and made up with buffer/de-ionized water. The solution was filtered using HPLC grade filter paper. The standard was injected with a 100µl syringe. The chromatograph data was recorded and a calibration curve was prepared. Then the samples were injected loaded, and the sample chromatograph recorded. Using the calibration curve, the quantity of toxicant was determined.

3. RESULTS AND DISCUSSION

3.1 Physical Properties

The results in Table 1 show a significant difference (P = .05) in the percentage yield and glucose content of the syrups from the three biomass sources with values ranging from 59.33% to 70.40% and 21.63% to 25.20% respectively. Rice husk recorded the highest yield of 70.40% and glucose content of 25.20%. This could be as a result of the presence of some broken rice in the husk, which is translated to the high glucose content of 25.20%. These results agree with the findings of [12] who reported that, biomass react differently when exposed to acid hydrolysis conditions because of their relative degree of molecular order. Okeke and Obi [13] reported that there exist both economic and technical factors which favor dilute acid hydrolysis of α – cellulose to glucose, so as to maximize the glucose yield. Harris [14] predicted a maximum sugar of 65% with 0.4% acid at 120°C, the yield being based on percentage conversion of available α – cellulose to glucose. This result is also consistent with previous work by [15] as well as [16] where high yield and glucose content of 68% and 26% respectively were recorded, using dilute acid at 120°C.

Total dissolved solids (TDS) ranged from 12.01% (softwood) to 18.43% (rice husk). Total Dissolved Solids is a measure of the combined content of all inorganic and organic substances contained in a liquid in molecular, ionized or micro-granular (colloidal sol) suspended form. TDS is used as an aggregate indicator of the presence of a broad array of chemical contaminants [17].

Total soluble solids content of a solution is determined by the index of refraction. This is measured using a refractometer, and is referred to as the degrees Brix. Brix is the term used when a refractometer equipped with a scale, based on the relationship between refractive indices at 20°C and the percentage by mass of total soluble solids of a pure aqueous sucrose solution. This tests the solids concentration of a sucrose containing solution. It is widely used during fruit and vegetable processing to determine the concentration of sugar in

the products. The total soluble solids of the glucose syrups ranged from 30.40° Brix (softwood) to 36.70° Brix (rice husk) [18].

The refractive index of the syrups ranged from 1.35 (softwood) to 1.38 (rice husk). In optics the refractive index or index of refraction of a substance (optical medium) is a dimensionless number that describes how light, or any other radiation, propagates through that medium. Since refractive index is a fundamental physical property of a substance, it is often used to identify a particular substance, confirm its purity, or measure its concentration. Refractive index is used to measure solids (glasses and gemstones), liquids, and gases. Most commonly it is used to measure the concentration of a solute in an aqueous solution. For a solution of sugar, the refractive index can be used to determine the sugar content [19].

The viscosity of the glucose syrup samples ranged from 1.20mm²/s (softwood) to 1.29mm²/s (rice husk) a fluid is a measure of its resistance to gradual deformation by shear stress or tensile stress. For liquids, it corresponds to the informal notion of "thickness". For example, honey has a higher viscosity than water.

Though there were significant differences (P = .05) in the refractive index, total dissolved solids and viscosity, no significant difference (P = .05) existed in the total soluble solids (⁰Brix) value.

Parameter	Soft Wood (Eucalyptus)	Hard Wood (Mahogany)	Rice Husk	LSD
Yield (%)	59.33±2.57 ^b	60.77±1.02 ^b	70.40±0.80 ^a	3.30
Glucose Content (%)	21.63±0.96 ^b	23.57±1.06 ^a	25.20±2.75 ^a	3.56
Total Soluble Solids (⁰ Brix)	30.40±0.31 ^ª	32.57±0.30 ^a	36.70±0.9 ^ª	4.32
Total Dissolved Solids (%)	12.01±1.72 ^b	16.23±3.36 ^ª	18.43±0.92 ^a	4.49
Refractive index	1.35±0.01 ^c	1.36±001 ^b	1.38±0.04 ^ª	0.01
Viscosity (mm²/s)	1.20±0.05 ^c	1.24±0.01 ^b	1.29±0.01 ^a	0.03

Table 1. Physical properties of Glucose Syrup form Various Biomass Sources

Values are means ± standard deviation of triplicate determinations.

Means with the same superscript in the row are not significantly different (P = .05)

3.2.1 Proximate composition

Table 2 shows results of proximate composition and pH of the syrups from the three biomass sources. There was no significant difference (P = .05) in the crude fat and ash contents which ranged from 1.60% to 2.01%. The high fat content recorded by Eucalyptus sawdust is in agreement with the reports of [20] that Eucalyptus sawdust contains volatile, aromatic oil known as eucalyptus oil, whose main use is medicinal and constitutes an active ingredient in expectorants and inhalants.

The carbohydrate content ranged from 3.11% to 9.60%, with rice husk recording the highest value, which could be as a result of some escaped broken rice seeds thus contributing high amount of carbohydrate to the husk. This is in agreement with the reports of [21], that hydrolysis of ligno-cellulolusic materials of forest and Agricultural biomass follows pseudo first order homogeneous reaction.

Crude protein content ranged from 4.50% to 5.08%. The percentage of protein obtained in Rice husk was lower than Eucalyptus sawdust though they were not significantly different (*P*

= .05). Crude fibre was not detected in the three sources of biomass, probably because it was the filtrate that was used for analysis.

There was no significant difference (P = .05) in the pH values in the samples with the different treatments. pH values ranged from 6.90 in rice husk to 7.00 in hard wood and 7.20 in soft wood after neutralization.

Parameter	Soft Wood (Eucalyptus)	Hard Wood (Mahogany)	Rice Husk	LSD
Moisture (%)	87.99±0.5 ^a	83.77±0.48 ^a	81.57±0.32 ^b	3.05
Crude Protein (%)	5.08±0.80 ^a	4.50±0.36 ^b	4.73±0.15 ^a	0.46
Crude Fat (%)	2.01±0.11 ^a	1.80±0.17 ^a	1.60±0.30 ^a	0.42
Crude Fibre (%)	Not detected	Not detected	Not detected	-
Ash (%)	3.01±0.95 ^a	2.40±0.44 ^a	2.50±0.27 ^a	0.73
Carbohydrate (%)	3.11±0.34 [°]	7.53±0.38 ^b	9.60±1.3 ^ª	0.39
pH	7.20±0.92 ^a	7.00±0.37 ^a	6.90±0.53 ^a	0.56

Table 2. Proximate Composition of Glucose Syrup form Various Biomass Sources

Values are means \pm standard deviation of triplicate determinations. Means with the same superscript in the row are not significantly different (P = .05)

3.2.2 Mineral composition

Results of the mineral contents of glucose syrup from the three sources of biomass are presented in Fig. 1. There were significant differences (P = .05) in the sodium, potassium, phosphorous, magnesium and iron contents of the glucose syrup samples obtained from the three sources of biomass with values ranging from 1.00 to 8.93mg/100g, 2.30 to 9.10mg/100g, 2.40 to 3.20mg/100g, 5.80 to 13.00mg/100g and 1.10 to 3.20mg/100g respectively, while there was no significant difference (P = .05) in the calcium content. This may be probably due to the inherent composition of the minerals present in the three different sources of biomass.

Minerals are chemical elements which promote chemical reactions in the body and help to form body structures. They are recognized as major (i.e. requiring over 100milligrams per day) or trace (i.e. requiring 100milligrams or less per day) [22]. They are very essential for normal body functions at different stages of life and deficiency can lead to different disease conditions.

The level of minerals found in the glucose syrups is not adequate in meeting the daily allowances (RDAs) as recommended by [23].



Fig. 1. Mineral Composition of Glucose Syrup from Various Biomass Sources

3.2.3 Heavy metals

Although there is no clear definition of what heavy a metal is, density is in most cases taken to be the defining factor. Heavy metals are thus commonly defined as those metals having a specific density greater than $5g/cm^3$ [24]. Fig. 2 shows results of heavy metals content of glucose syrups from the three sources of biomass. The heavy metals content differed significantly (P = .05) ranging from 0.50 to 3.50mg/100g (Mercury), 0.63 to 3.50mg/100g (Cadmium), 0.63 to 2.50mg/100g (Lead) and 0.63 to 6.50mg/100g (Selenium) with soft wood having the highest values for Mercury and Selenium while hardwood had the highest values for Cadmium and Lead.

Heavy metal toxicity can result in damaged or reduced mental and central nervous function, lower energy levels, and damage to blood composition, lungs, kidneys, liver, and other vital organs. Long-term exposure may result in slowly progressing physical, muscular, and neurological degenerative processes that mimic Alzheimer's disease, Parkinson's disease, muscular dystrophy, and multiple sclerosis. Allergies are not uncommon, and repeated long-term contact with some metals (or their compounds) may cause cancer [24]. However, inorganic mercury is converted to organic compounds, such as methyl mercury, which is very stable and accumulates in the food chain, causing illness in humans and birth defects in

offspring of affected individuals. Ingesting lead can cause anaemia, kidney disease and damage to the nervous system. Lead toxicity is of particular concern in children because it is associated with IQ deficits, behavior disorders, delayed growth, impaired hearing, and possible hypertension and kidney disease in later life [24].

The values of heavy metals in these syrups are quite high compared to safety specifications by [25]. Further purification will be needed for the syrups to be safe for human consumption.





3.2.4 Toxicants

Fig. 3 presents results of toxicants content of glucose syrup from the three sources of biomass. The content of toxicants differed significantly (P = .05) in the syrup samples. Values ranged from 2.20mg/100g to 3.00mg/100g for Total phenols, 9.60mg/100g to 11.41mg/100g for phytates, 9.80mg/100g to 10.21mg/100g for oxalates, and 6.30 to 8.20mg/100g for polyaromatic hydrocarbons, while Hydrocyanic acid could not be detected.

Phenols are anti – nutritional factors which inhibit the activity of digestive and hydrolytic enzymes such as beta-amylase, trypsin and lipases, thus decreasing the availability of reducing sugars, vitamins and minerals [11]. The total phenol content in these syrups is less

than the value of 12.00mg/100g reported by [26] for the leaves of lima beans and 14. 31 to19.31mg/100g reported by [27] for the leaves of different cultivars of cassava; both of which are commonly consumed vegetables.

The phytate content of the glucose syrup obtained from the three sources of biomass is quite low, compared to the value of 12.82mg/100g reported by [28] for the fresh cassava leaves. Udensi [29] reported a phytate content 0f 45.80mg/100g for cowpea, which is widely consumed and regarded as safe. Phytate forms insoluble salts with metals such as calcium, iron, zinc and magnesium, thus rendering them unavailable for absorption in the body. It also adversely affects protein and starch digestibility. Though the smallest "toxic" dose of phytate in human is not known, it appears that high doses are required for any appreciable effect [30].



Fig. 3. Toxicant Composition of Glucose Syrup from Various Biomass Sources

The oxalate content of the glucose syrup obtained from the three sources of biomass is in the same range with results obtained by [31] for regularly consumed Nigerian leafy vegetables ranging from 4.32mg/100g for lettuce to 15.08mg/100g for spinach. Gernah and Sengev [30] reported oxalate content of 4.32mg/100g for boiled and 8.68mg/100g for fresh *Moringa oleifera* leaves respectively.

Apart from causing irritations, oxalates forms insoluble complexes with some metals thus leading, especially, to reduced calcium availability/metabolism [32]. Gordon [22], however,

reported that the risk of calcium deficiency diseases due to consumption of oxalate rich foods is very rare. This is because humans are able to efficiently utilize very low amounts of calcium in their food [31].

Hydrocyanic acid was not detected in all the three samples of glucose syrup obtained as a result of the hydrolysis of the three sources of biomass. This is consistent with the findings of [33] that cyanic levels could be reduced if not eliminated by steeping in water, fermentation or application of high temperature during processing. Consumption of food with large amount of cyanide can result in death or chronic neurological disorder such as Tropical Ataxic Neuropathy (TAN) [34,35].

Polycyclic aromatic hydrocarbons (PAHs), also known as poly-aromatic hydrocarbons or polynuclear aromatic hydrocarbons, are potent atmospheric pollutants that consist of fused aromatic rings and do not contain hetero-atoms or carry substituents [36]. Naphthalene is the simplest example of a PAH. PAHs occur in oil, coal, and tar deposits, and are produced as byproducts of fuel burning (whether fossil fuel or biomass). PAHs are one of the most widespread organic pollutants. In addition to their presence in fossil fuels they are also formed by incomplete combustion of carbon-containing fuels such as wood, coal, diesel, fat, tobacco, and incense [37].

As a pollutant, they are of concern because some compounds have been identified as carcinogenic, mutagenic, and teratogenic. PAHs known for their carcinogenic, mutagenic, and teratogenic properties are benz[a]anthracene and chrysene, benzo[b]fluoranthene, benzo[j]fluoranthene, benzo[j]fluoranthene, benzo[a]pyrene, benzo[ghi]perylene, coronene, dibenz(a,h)anthracene($C_{20}H_{14}$), indeno(1,2,3-cd)pyrene($C_{22}H_{12}$), and ovalene [38]. High prenatal exposure to PAH is associated with lower IQ and childhood asthma. The Centre for Children's Environmental Health reports studies that demonstrate that exposure to PAH pollution during pregnancy is related to adverse birth outcomes including low birth weight, premature delivery, and heart malformations. Cord blood of exposed babies shows DNA damage that has been linked to cancer. Follow-up studies show a higher level of developmental delays at age three, lower scores on IQ tests and increased behavioral problems at ages six and eight [38].

The values of polyaromatic hydrocarbons (PAHs) reported in these syrups are quite high compared to safety specifications by [25]. The syrups are therefore unsafe for consumption as they are.

4. CONCLUSION

Glucose syrup was successfully produced from the three sources of biomass selected, with a reasonable yield of 59.33% to 70.40%, and a corresponding glucose content of 21.63% to 25.20%. While the glucose syrups contain reasonable levels of macro and micro nutrients, they also contain high levels of toxicants and heavy metals, thus making them unsafe for direct human consumption. There will be need for further processing of the syrups to remove or reduce toxicants and heavy metals before they can attain food grade status.

ACKNOWLEDGEMENTS

We wish to acknowledge the laboratory staff of the Department of Food Science and Technology, Federal Polytechnic for carrying out all the analysis in this study.

COMPETING INTERESTS

There are no competing interests as far as we know.

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Peer-review history: The peer review history for this paper can be accessed here: http://www.sciencedomain.org/review-history.php?iid=366&id=5&aid=2705