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Extraction, Characterization and Dextrinization of Starch from Six (6) Varieties of Tubers from Iwo Osun State Nigeria for Application in the Production of Adhesives

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Abstract: Tuber crops are widely consumed; they come in various varieties with great nutritional and non- nutritional values. They are very rich in starch. The starch content of each of the six varieties of Nigerian tubers was determined, each of the tubers was wet fractionated in the laboratory and the starch was recovered. The composition of the tubers (dry matter, protein, ash and mineral content) and starch isolation (moisture, starch, protein, phosphorus, fat and carbohydrate) were determined. Substantial differences in the starch content of the tubers were observed. The dry starch recovery ranged from 10.20% to 18.30% and the wet starch recovery ranged from 15.70-32.40%. Moisture content ranged from 13-15.80%, viscosity 275.08 -382.33. The carbohydrate, ash, fat and protein content of the six varieties of the tubers vary from 45% to 53.8%, 1.0 to 2.0%, 19.56 to 21.99% and 10.96-14.29% respectively. All six starches were insoluble in water and ethanol and proved positive to iodine and Fehling's test. Extracted starch was dextrinated using 0.1M and 0.2M HCl separately at 160 and 100°C to obtain yellow dextrin and white dextrin respectively which were soluble in water. Modified starches were formulated into adhesives. Results obtained indicated satisfactory strength for the adhesives. Thus starches suitable for the production of adhesives were obtained.

Keywords: Dextrinization, Starch, Tuber, Adhesive

1. Introduction

Tuber crops are widely consumed and they come in various varieties with great nutritional and non- nutritional values. Tuber crops being a rich source of carbohydrate has encouraged its consumption, the non–food applications of tuber crops due to its starch content have promoted the preferential use of starch in industries to other polysaccharides [1]. Tropical root and tuber crops are a pertinent source of food security since they are climatic resilient and can grow on less productive and marginal lands. In addition, productivity is higher in comparison with cereals and horticultural crops, constituting a source of income for farmers [2] they are cultivated in almost all parts of the world and it is quite cheap and a readily available raw material for adhesive production [3].

Starch is the most significant form of carbon reserve in plants in terms of the amount made, the universality of its distribution among different plant species, and its commercial importance. It consists of different glucose polymers arranged into a three- dimensional, semicrystalline structure-the starch granule [4]. Since it is one of the most abundant natural carbohydrate polymers and is non-allergenic, it is generally recognized as safe (GRAS), and low-cost [5] thus the reason why it finds application widely.

Starch is synthesized transiently in organs, such as leaves, meristems and root cap cells, but its major site of accumulation is in storage organs, including seeds, fruits, tubers, and storage roots [4].

Starch, the food reserve homopolysaccharide of plants [6], is a biocompatible, biodegradable, nontoxic polymer [7], which occurs widely in nature and commonly used [8].

The application of starch includes in adhesives, agrochemicals, cosmetics and toiletries, detergents, paper making additives, pharmaceuticals, paints, textiles, water purification, biodegradable plastics and as super–adsorbent materials to mention a few, partly because of the wide range of functional properties such as gelatinization, pasting, retrogradation, water absorption capacity, swelling power, and solubility derived from it in its various natural and modified forms, and partly because of its low cost relative to alternatives [9]. The functional properties vary considerably from one botanical source to another [10], with variety and environmental conditions [11], composition and structures of the starches, which include amylose/amylopectin ratio, crystalline structure, granular size, molecular weight of the starches and chain length distribution of amylopectin [12]. Starch is principally a constituent of some kind of adhesive [3]. Modification of these starches will increase their chances of finding more applications especially in the industries for a variety of applications. The process of modification may be physical or chemical. The physical modification which does not involved any chemical reaction of starch with a modifying reagent is referred to as physical modification of starch and the products are known as physically modified starches. However, most modifications of starches are performed through chemical processes. The chemical reactions of starch (hydrolysis, esterification, etherification, oxidation and cationization) are generally exploited in the industry to produce converted or modified starches fit for different purposes in the industry [13]. It has been proven that starch, especially starches from yam show large variability in their physicochemical and functional properties hence can have diverse applications in both food and non-food industries [14] therefore dextrinization can create a variety of useful products such as starch with greatly increased water solubility [15].

2. Methodology

2.1. Collection of Materials

Freshly harvested tubers were obtained from Odo Ori market in Iwo Local Government Area Osun State Nigeria and studied namely:

i. Freshly harvested (*Manihiot esculanta*)
ii. Fermented cassava (*Manihiot esculanta*)
iii. Two varieties of cocoyam (Xanthomonas; white and red cocoyam)
iv. Sweet potatoes (*Ipomea batatoes*)
v. Bitter yam (*Discora sp*; locally called Esuru)

2.2. Preparation of Materials

The freshly harvested tubers were washed with distilled water and barks peeled off manually with a knife without losing the cortex. The peeled tubers were washed, grated and weighed the same day they were harvested. 500g of the cassava was allowed to ferment for 24h.

2.3. Extraction of Starch

A fine sieve was used to separate the starch granules from the fiber; the residue was washed with distilled water severally.

The extracted starch was allowed to settle, then the supernatant was decanted off and the starch was washed with fresh clean was to remove the proteins. The starch cake obtained was dried in the sun. The white cocoyam and bitter yam took longer to dry. The weight of the dried starch for each tuber was determined and stored in airtight containers.

2.4. Physical Measurements of Starch Extracts

i. Solubility test

One (1.0) g of each extracted starch sample was dried at 80°C in an oven for 12hrs. Then to each sample, 5ml of distilled water was added inside a 50ml beaker and 5ml ethanol separately. The mixture was stirred gently, then vigorously and allowed to stand for 12 hrs.

ii. Moisture content

In a dry and pre-weighed crucible, 5.0g each starch extract was introduced and placed in an oven for 6hrs at 100°C, then cooled in a desiccator and weighed. This process was repeated until as constant weight was obtained. The percentage moisture content was calculated using the equation given below.

$$\%\ \text{moisture} = \frac{weight\ loss}{weight\ of\ sample} \times 100$$

iii. Determination of crude protein

The micro-kjedahl method was used for the determination of the crude protein function of nitrogen

iv. Determination of pH

The pH f the extracted starch was determined by a pH meter using BDH buffer tablets (pH 4 and pH 10) to standardize the pH meter before actual measurements at room temperature.

v. Determination of crude fat

Crude fat was extracted from the starch samples using n-hexane in a soxhlet extractor. 5.0g of starch was used, extract was separated from solvent using a rotary evaporator and the fatty residue was weighed and % Fat was calculated using

$$\%\ \text{Fat} = \frac{Loss\ in\ weight\ of\ sample}{Original\ weight\ of\ sample} \times 100$$

vi. Determination of carbohydrate content

This was determined by difference using the equation given below

$$\%\ \text{Carbohydrate} = 100 - (Moisture + Fat + Ash + Protein)\%$$

vii. Determination of mineral content

The mineral content was determined using an Atomic Absorption Spectrophotometer (AAS) after digesting the starch samples. The elements determined are Phosphorus, Iron, Zinc, Sodium, Lead, Copper, Calcium, Magnesium and Manganese.

viii. Determination of acid factor measurement

The quantity of 0.1M HCl required for 25.0g suspension to get a pH of 3.0 is referred to as acid factor. Freshly prepared 0.1M HCl was titrated against 25g each of oven dried starch, stirred in 50ml of distilled water in 250ml conical flask until pH 30 was obtained.

ix. Determination of viscosity

Three 3.0g on 100% dry matter basis of starch was weighed into a canister which was inserted into the viscometer to measure the viscosity.

Calculation of dried sample weight

Since at 100% dry matter, 3g of starch is required, R Visco Analysis Weight= Sample dry matter

Therefore RVA WT- 3 =Y

Volume of water =25ml-Y

2.5. Qualitative Tests on Starch Extracts

i. Fehling's test for reducing sugars

0.1g of each sample was weighed into a test tube; 2ml of 0.5M HCl was added and warmed for 2minutes after which a few drops of Fehling's solution were added.

ii. Iodine test

0.1g of each extracted starch was weighed into a beaker and 100ml of boiled water and stirred to give a clear solution allowed to cool and 3-4 drops of iodine solution was added.

2.6. Quantitative Chemical Tests on Starch Extracts

i. Determination of total ash content

5.0g of starch sample was ashed in a muffle furnace at 525ºC for 2hrs, cooled and weighed. The percentage ash was calculated using the relationship.

$$\% \text{ ash}=\frac{Weight\ of\ reidue\ in\ gram}{Weight\ of\ sample\ in\ gram} \times 100$$

2.7. Dextrin Production

This process was carried out in four steps

i. Acidification

Starch samples were dried in oven to reduce moisture content to about 12.5%, then 25.0g of each sample was weighed into a 100ml beaker for yellow dextrin and 5ml of 0.2% HCl was added drop-wise at intervals with vigorous stirring to ensure uniformity. 2.5ml of 0.1% HCl was used instead for white dextrin

ii. Pre-drying

The acidified starch samples were air dried in the laboratory for 24hrs

iii. Conversion

The pre-dried acidified starch samples were placed in 200ml beaker and heated in an oil bath for 18hrs at 160°C (100°C for white dextrin) with continuous stirring.

iv. Cooling: the produced dextrin was introduced into an ice bath for 20minutes, then stored in a desiccator

2.8. Qualitative Analysis on Dextrin

i. Iodine test

Using the same process as was described for starch.

ii. Solubility test

Solubility in ethanol: 10ml of 95% ethanol was added to 0.1g of dextrin sample and stirred vigorously without heating.

Solubility in water: 1.0g of dextrin was added to 5ml of distilled water with continuous stirring and left for 12hrs.

2.9. Adhesive Production from Dextrin

8.70g of dextrin of each sample was weighed inside a beaker. 21.80g of water was added and the mixture heated to 70°C, then 1.50g of borax was added and the temperature increased to 90°C. 2ml of phenol was added to the mixture.

3. Results

The starch from the six (6) tubers are presented in the tables below.

Table 1. *Percentage yield of starch from the six (6) tubers, Physical measurement of starch extract, Viscosity and iodine test.*

Sample	Fresh cassava	Fermented cassava	Cocoyam (white)	Cocoyam (red)	Sweet potato	Bitter yam
Wet % starch yield	32.4	30.00	20.00	15.70	25.00	30.00
Dry % starch yield	18.30	17.60	11.00	10.20	15.00	16.00
% Moisture Content	13.00	13.80	15.60	15.80	15.60	14.40
pH	3.96	8.44	5.64	4.73	6.05	5.84
Acid factor	4.60	6.00	15.50	5.40	5.90	9.00
Peak viscosity value	303.75	382.33	306.33	229.42	306.00	275.08
Iodine test and Fehling's test	Positive	Positive	positive	Positive	positive	Positive
Solubility in water	Insoluble	Insoluble	insoluble	Insoluble	insoluble	Insoluble
Solubility in ethanol	Insoluble	Insoluble	insoluble	Insoluble	insoluble	Insoluble

Table 2. *Mineral content of starch from the six (6) tubers.*

Sample	PO^{4-} PPM	Ca PPM	Mg PPM	K PPM	Na PPM	Mn PPM	Pb PPM	Fe PPM	Cu PPM	Zn PPM
Fresh cassava	6.12	15.06	3.43	52.33	0.41	0.25	0.32	0.40	1.80	2.83
Fermented cassava	7.83	20.84	5.59	22.38	1.34	0.25	0.42	0.64	4.40	3.44
Cocoyam (white)	5.14	9.43	2.75	37.38	0.49	0.17	0.22	0.56	0.41	1.32
Cocoyam (red)	6.58	7.88	2.75	27.38	0.77	0.25	0.32	0.40.	2.68	3.76
Sweet potato	15.24	25.43	7.10	79.38	1.26	0.33	0.32	1.62	0.77	2.92
Bitter yam	0.00	12.67	4.79	3377	1.49	0.33	0.42	1.05	2.58	2.89

Table 3. *Proximate analysis of starch from the six (6) tubers.*

Sample	Fresh cassava	Fermented cassava	Cocoyam (white)	Cocoyam (red)	Sweet potato	Bitter yam
% Ash	2.00	2.00	2.00	1.00	2.00	1.00
% Protein	13.06	10.96	14.29	13.91	12.96	11.28
% Carbohydrate	50.62	53.38	47.47	44.40	51.08	50.13
% fat	21.32	19.86	20.64	25.09	19.56	21.99

4. Discussion

Starch has found numerous applications; the application of starch in adhesive production is a vital one as the choice of raw material for adhesive production rests squarely on determinants such as cost, availability, convertibility and starch yield.

Starch consists of amylose and amylopectin and from the results obtained it is noted that starch is insoluble in water due to the presence of amylopectin. The percentage of starch recovery from the six tubers ranged from 10.20% to 18.30% lowest from Cocoyam (red) highest from fresh cassava.

From table 1, the starch obtained from cocoyam (red) has the highest moisture content (15.80%) while that from fresh cassava has the lowest moisture content (13.00%).

The pH varies from 3.96-8.44. The fermented cassava has the highest and the fresh cassava having the lowest.

From table 2, the carbohydrate content of all the tubers is above 45%. However, fermented cassava has the highest percentage of carbohydrate (53.38%) and sweet potato (51.08%) having a higher percentage of carbohydrate than fresh cassava (50.62%).

The white cocoyam has the highest percentage of protein (14.29%) than the other tubers, while the red cocoyam has the highest percentage of fat (25.09%). Fresh cassava (21.32%) has slightly higher percentage of fat than fermented cassava (19.86%).

Trace metals analyzed includes Ca, Mg, K, Na, Mn, Pb, Cu, Zn, PO^{4-}. The concentration of potassium in all the starch is highest of all the minerals analyzed. Followed by Calcium, then phosphorus and magnesium

White dextrins were insoluble in water and ethanol, while the yellow dextrins were soluble in because it undergoes hydrolysis thereby converting the starch to glucose.

Hence it proves positive to Fehling's solution for reducing sugars. White cocoyam gave brown dextrin because it took a loner time to dry. Therefore this reveals that yellow dextrin is soluble in water to give a colourless solution while starch is insoluble in water.

The adhesives produced with the yellow dextrin obtained gave a fairly satisfactory strength. The strengths varied slightly which could be due to non-homogenous acidification, unequal heat distribution amongst other latent factors.

5. Conclusion

Adhesives obtained from all the tubers shows an indication that starch obtained from other tubers could be used for industrial production of adhesives. Thus the importance of starch continues to be on the upward trend due to its versatility. Its availability in these six varieties of tubers shows and proves that starch is availability in abundance; starch from tubers has transcended its traditional use as a source of carbohydrate in food to more sophisticated non-food applications. Its susceptibility to modification, which transforms the native properties into more desirable and malleable characteristics fit for different purposes has led to its growth in modern technological application.

Starches will naturally remain insolubility in cold water and loss of viscosity easily, these 'weaknesses' are lost after modification of starch by dextrinization thus increasing its utilization in industry for the production of value added products.

6. Recommendation

The dextrinization of starch from tuber should be further researched into so as to provide more applications for the starch from edible and non-edible tuber crops.

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