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Isolation and physicochemical characterization of tigernut (*Cyperus esculentus*) starch as a potential industrial biomaterial

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Abstract: Tigernut (*Cyperus esculentus*) seeds were examined for its starch composition and applicability as biomaterial in hybrid composite materials development. The physicochemical properties of the starch extracted were then compared with standard industrial maize starch. The starch was isolated using 1% w/v sodium metabisulphite solution and the obtained starch was found to be a brilliant white, crystalline, non- hygroscopic powder with yield of about 21%. The starch percentage solubility at 90°C was 2.36 with a swelling power of 13.7 and gelatinization temperature of 66°C. It had a browning temperature of 257.0 – 268.2°C, charring temperature of 281.4 – 291.6°C, water absorption capacity of 71%, pH of 5.6, foam and emulsion capacities of 2.8% and 8.17% respectively. The proximate analysis (%) was found to be: fat – 2.3, ash – 0.24, protein – 0.18, moisture – 8.67 and carbohydrates – 88.61. XRD scan (a gonio (2Θ) scan) of the starch sample at 10°C – 100°C 2Θ angle established the organic nature of the starch. Analysis of the starch sample by XRD gave amylose to be 28% and amylopectin to be 72%. The α-amylose of the starch had an orthorhombic crystal system with a high purity rate. The spectral revealed peak positions at 2Θ positions of 11.3189°, 14.9662°, 17.0105°, 17.8645°, 22.8843°, 26.4595° and 30.2574° corresponding to a Full Width at Half Maximum (FWHM) (2Θ) of 0.6593°, 0.5274°, 0.5274°, 0.3296°, 0.7252°, 0.6593° and 0.7911° respectively. The XRD analysis confirmed the starch to be of high purity and quality with a score of 83% on the ICDD database. Applicability in composite materials studies showed a high level of compatibility as binder/filler materials within the matrix and fiber materials employed. Generally, the values obtained from the characterization of tigernut starch showed that it has high potential for industrial applications especially but not limited to use as biomaterials in composites, food, textile and pharmaceutical industries.

Keywords: Tigernut, Starch, Physicochemical, XRD, Hybrid Composite, Binder/Filler, Biomaterial

1. Introduction

Starch exists as a major carbohydrate storage product in all plants containing the green pigment called chlorophyll. It is cheaply available and commonly used in many facets of life for the manufacture of industrial products. The unique properties of starch which enhances its use basically includes biocompatibility, biodegradability, gelation and modification ability to suit potential usage [1, 2].

Tigernut, botanically called cyperus esculentus is a perennial herb of both the tropics and temperate regions of the world. It can be found growing naturally as weed but can also be commercially cultivated producing rhizomes and small spherical tubers or nuts [3, 4]. So many varieties of the tigernut tubers existed though in Nigeria, two major varieties predominates namely; yellow and brown varieties. These are readily available in the market especially in the northern part of Nigeria.

Tigernut starch based on the variety or methods of extraction gives an odourless, brilliant white or off-white non-hygroscopic powder with yields varying from as low as 14% to as high as 37% depending on the size and flesh of the tubers or nut. Oil yields of as high as 27% has also being reported [5, 6, 7, 8].

Physicochemical properties of most starches are a function of the composition and proportion of amylose and amylopectin in the starch. Properties such as solubility,

swelling power, gelatinization temperature, water absorption capacity, pH, emulsion capacities and viscosity are highly dependent on the amylose and amylopectin content [2, 8, 9, 10]. Starches from different botanical sources are identified using the properties inherent in their amylose and amylopectin contents [8, 9, 10].

Thus, the aim of the research was to isolate and characterize physicochemically, starch extracted from tigernut tubers as potential biomaterial for industrial application and comparing same with standard industrial maize starch which served as a reference while at the same time applying it in the development of hybrid composite materials as a binder/filler material for composite manufacture.

2. Materials and Methods

2.1. Materials

Dried tigernut tubers were obtained from Gwagwalada market in Abuja, Nigeria. Corn starch (BP) and other reagents and solvents used were of analytical grade.

2.2. Methods

2.2.1. Starch Isolation

The dried tigernut tubers were separated from dirts and washed. The washed tubers (0.997 kg) were soaked in sodium metabisulphite solution (2 L 1 % w/v) at room temperature (27 OC). Thereafter, the tubers were removed and wet milled into slurry using a grater. The paste was dispersed in a large volume of 1 % sodium metabisulphite solution and filtered through muslin cloth. The suspension was centrifuged at 3500 rpm for 10 mins to facilitate the removal of dirts. The supernatant was carefully decanted and the mucilage scraped off. The process was repeated for three times with the mucilage on the starch scraped continuously until a pure starch was obtained. The resulting starch was dried in the sun and further dried at 60°C in a hot air oven, pulverized, weighed and stored in sample bottles for analysis.

2.3. Determination of Certain Physicochemical Properties:

2.3.1. Swelling Power

The method described by Afolayan et al (2012) was used to determine the solubility index. The starch sample (0.1 g) was weighed into a test tube and 10 ml of distilled water was added. The mixture was heated in a water bath at a temperature of 50 ^{O}C for 30 min with continuous shaking. In the end, the test tube was centrifuged at 1500 rpm for 20 min in order to facilitate the removal of the supernatant which was carefully decanted and weight of the starch paste taken. The swelling power was calculated as follows:

$$\text{Swelling power} = \frac{\text{Weight of starch paste}}{\text{Weight of dry starch sample}}$$

This was carried out over a temperature range of 50 ^{O}C – 100 ^{O}C.

2.3.2. Solubility Index

The method described by Afolayan et al (2012) was also used to determine the solubility index. Starch sample (0.5 g) was added to 10 ml distilled water in a test tube. This was subjected to heating in a water bath with a starting temperature of 50 OC for 30 min. It was then centrifuged at 1500 rpm for 30 min. 5 ml of the supernatant was decanted and dried to constant weight. The solubility was expressed as the percentage (%) by weight of dissolved starch from heated solution. This was carried out over a temperature range of 50 °C – 100 °C.

2.3.3. pH

A 20 % w/v dispersion of the sample was shaken in water for 5 minutes and the pH was determined using a pH meter.

2.3.4. Browning and Charring Temperature

The method of Builders et al (2001) was used. Some of the starch sample was put into a capillary tube, the browning and charring temperatures were determined using a melting point apparatus with model Electrothermal 9100.

2.3.5. Foam Capacity

The method of Omojola et al (2010) was used with slight modifications. Starch sample (1 g) was homogenized in 50 ml distilled water using a vortex mixer (vortex 2 Genie set at shake 8) for 5 minutes. The homogenate was poured into a 100 ml measuring cylinder and the volume recorded after 30 s. The foam capacity was expressed as the percent increase in volume.

2.3.6. Emulsion Capacity

Sample (1 g) was dispersed in 5 ml distilled water using a vortex mixer for 30 seconds. After complete dispersion, 5 ml vegetable oil (groundnut oil) was added gradually and the mixing continued for another 30 s. The suspension was centrifuged at 1600 rpm for 5 min. The volume of oil separated from the sample was read directly from the tube. Emulsion capacity is the amount of oil emulsified and held per gram of sample.

2.3.7. Gelatinization Temperature

This was evaluated using the method of Attama et al (2003). The starch sample (1 g) was put in a 20 ml beaker and 10 ml of distilled water was added. The dispersion was heated on a hot plate. The gelatinization temperature was then read with a thermometer suspended in the starch slurry.

2.3.8. Water Absorption Capacity

The method described by Omojola et al (2010) was

used to determine the water absorption capacity. The starch sample (5 % w/v) was dispersed in a pre-weighed centrifuge tube. The tube was agitated in a vortex mixer for 2 min. The supernatant was then discarded and the weight of the tube and hydrated sample taken. The weight was calculated and expressed as the weight of water bound by 100 g dry starch.

2.3.9. Structural Analysis of Starch Samples Using XRD

The structural studies of the starch samples was carried out using a PANalytical XPERT-Pro MPD Diffractometer system with a Reflection-Transmission Spinner configuration, a 2Θ PW 3050/60 goniometer system and a Reflection-Transmission spinner PW3064/60 sample stage. The scan range (2Θ) was from 10° to 100° with a step size (2Θ) of 0.0670°, scan step time (s) of 59.6900 and using CuKα monochromatic radiations Kα1 and K α2 of 1.54060A and 1.54443A respectively and K-beta of 1.39225A. The wavelength, accelerating voltage and current were 1.54060A, 40Kv and 30mA respectively. Spinning of the sample stage was enabled.

The diffraction spectra, peak list; incorporating the peak positions (2Θ), height/counts/intensities (cts), the full width at half maximum (FWHM) (2Θ), interplanar distance (d-spacing) (A) and relative intensities (%) as well as the pattern list showing the visible reference code and chemical formula, score, component name, displacement (2Θ) and scale factor was evaluated from these spectra.

The prepared powdered starch sample was loaded onto a cylindrical sample holder which was then mounted onto the XRD machine sample stage. A monochromatic beam of light was allowed to strike the mounted sample after spinning was enabled on the sample stage for the optimized time and 2Θ angle. The intensity of the rays was measured at Bragg's 2Θ angle from start angle of 10° to end angle of 100° and this was displayed as the scan spectral on a real time computer connected to the Diffractometer system. The crystallinity and other important parameters were determined from the diffraction spectral analysis using the equipment's softwares

3. Results and Discussion

3.1. Chemical Composition

Table 1 shows some physicochemical properties of the starch.

The starch obtained was a brilliant white, crystalline, non- hygroscopic powder with yield of about 21%. The yield is considered to be appreciable especially when compared with starches from other sources such as cassava and corn.

The pH is very close to that of corn starch although slightly lower [13], this could be due to the nature of the material from which the starch originated. It is comparable with the previous pH values reported for tuber starches [15] and within the pH range of 3 – 9 obtained for most starches used in the pharmaceutical, cosmetics and food industries.

Table 1. *Physicochemical Properties of Ginger and Maize Starch.*

PARAMETERS	TIGERNUT	MAIZE
Ph	5.6 ± 0.1	5.92 ± 0.2
Gelatinization temperature (^{O}C)	66 ± 0.0	73 ± 0.1
Foam capacity (%)	2.8 ± 0.1	5.0 ± 0.1
Emulsion capacity (%)	8.2 ± 0.2	5.2 ± 0.2
Water absorption capacity (%)	71 ± 0.1	93 ± 0.2
Browning temperature (^{O}C)	257.0 – 268.2	268.9 – 281.5
Charring temperature (^{O}C)	281.4 – 291.6	289.4 – 299.8

Maize starch has a higher water absorption capacity of 93 ml in 100 g of sample as compared with 71 ml in 100 g of sample for tigernut starch. The variation in water absorption could be due to different proportion of crystalline and amorphous regions within the granule.

The browning and charring temperatures is observed to be quite higher than the reported value for some other starches [12], although lower than that reported for maize starch, this shows that the starch can even be heated to a higher temperature without changing colour or charring. This quality will make it a preferable starch in industries that use starch at higher temperatures.

The starch sample was observed to have a gelatinization temperature of 66 °C which falls within the range of gelatinization temperatures commonly observed for starches.

The foam capacity is lower while the emulsion capacity is higher than that reported for maize starch which is a reflection of the high fat content and an indication that it can be used as an emulsifier.

3.2. Swelling and Solubility

The swelling and solubility profiles of tigernut and maize starches over a temperature range of 50 – 100 °C are shown in Figures 1 and 2.

The profiles show a general trend of increase with increase in temperature for the starch. This is an indication of the water absorption characteristic of the granules during heating just like other sources - anchomanes and icacina starches which shows temperature relaxation between 50 – 60 °C and 90 °C [12].

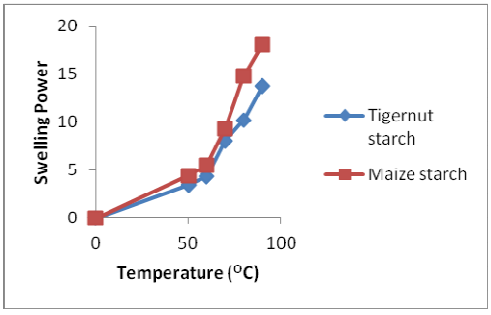

Figure 1. *Swelling profile for maize and tigernut starch.*

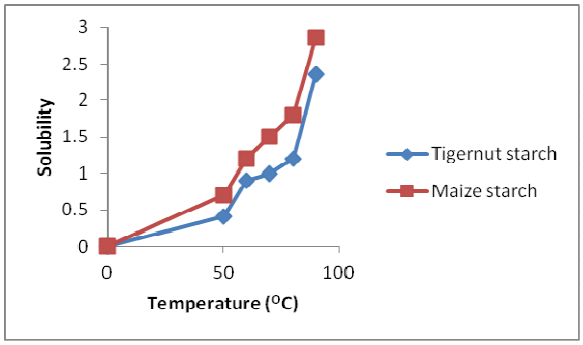

***Figure 2**. Solubility profile for maize and tigernut starch.*

The swelling power is lower than that of maize starch. Increase in swelling power is indicative of suitability of a starch being used as a disintegrant in the pharmaceutical industry [16], hence tigernut starch can be used as a disintegrant in the formulation of tablets. Also high swelling power results into high digestibility and ability to use starch in solution suggesting improved dietary properties and the use of tigernut starch in a range of dietary applications [17].

The solubility profile for both starches also shows an even increase in solubility with temperature rise.

3.3. Discussion of XRD Analysis Results

The XRD profile of the tigernut starch exhibits well resolved and intense peaks. The most intense peak height for the starch sample was recorded at 2Θ position of 17.8645° with a d-spacing of 4.96526A, FWHM of 0.3296°, height of 2641.08cts and relative intensity of 100%. This is shown in figure 3. A sharp peak indicates highly crystalline starch material as indicated in figure 3. Comparison of the XRD spectral of both the tigernut starch and the industrial maize starch indicated prominent peaks for the two starch samples appearing at almost the same 2Θ angles (positions) but at different intensities, indicating slightly different degrees of crystallinity of the starches from different sources. Figure 4 shows the XRD spectrum for the industrial maize starch while figure 5 gives the superimposed spectral for both tigernut and industrial maize starch samples. Analysis of the tigernut starch samples gave amylose content to be 28% and amylopectin to be 78%. The α-amylose of the starch had an orthorhombic crystal structure with a high purity rate. The XRD analysis confirmed the starch to be of high purity and quality with a score of 83% on the International Centre for Diffraction Data (ICDD) database. Application of the starches in the fabrication of composites revealed a high level of compatibility as a binder/filler material within the matrix and fiber material used.

4. Conclusion

Some physicochemical properties of tigernut starch were determined and compared with standard industrial maize starch. Based on the results of the yield and physicochemical characterization of tigernut starch as well as the structural characterization of the starches using the XRD, it has been discovered that it compares favourably with maize starch and is therefore a good source of starch and a potential biomaterial for industrial uses.

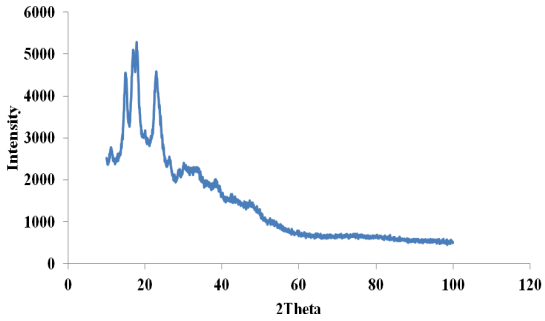

***Figure 3**. X-ray diffraction spectra of tigernut starch.*

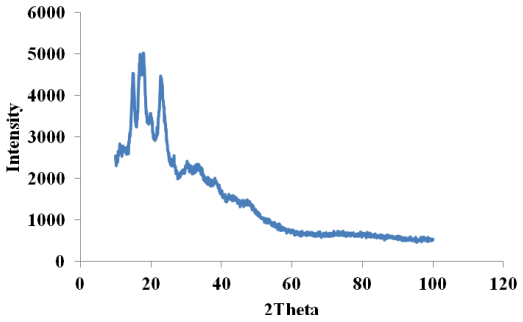

***Figure 4**. X-ray diffraction pattern of Industrial maize starch.*

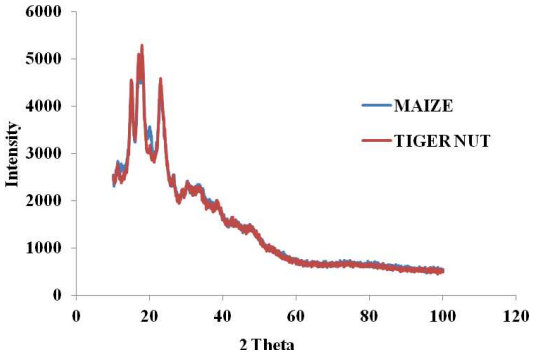

***Figure 5.** Superimposed X-ray spectra of tigernut and industrial maize starches.*

References

[1] Le Corre, D., Bras, J., Dufresne, A., Starch nanoparticles: A Review. "Biomacromolecules" 2010, 11, 1139-1153.

[2] Morthy, S.N., Starch and starch derivatives in food. "Trends Carbohydr. Chem.". 1996, 425, 133-139.

[3] Burkill, H.M., "The useful Plants of West Tropical Africa" 2nd Edition, Kew Publishing Company, Great Britian, 1985.

[4] Belewu, M.A., Abodunin, O.A., "Preparation of kunnu from unexploited rich food source: Tigernut (cyperus esculentus). Pakistan J. Nutr. 2008,7, 109-111.

[5] Defelice, M.S., Yellow nutsedge cyperus esculentus L.- Snacks food of the gods. Weed Technol. 2002, 16, 901-907.

[6] Adejujitan, J.A., Otunola, E.T., Akande, E.A., Bolarinwa, I.F., Oladokun, F.M.,: Some physicochemical properties of flour obtained from fermentation of tigernut (cyperus esculentus) sourced from Ogbomosho, Nigeria, Afr.J.Food Sci., 2009,3, 51-55.

[7] Turesson,H., Marttila, S., Gustavsson, K., Hofvander, P., et. al., Characterization of oil and starch accumulation in tubers of cyperus esculentus var, sativus (cyperus): A novel model system to study oil reserves in nonseed tissues. Am.J.Bot, 2010, 97, 1884-1893.

[8] Linssen, J.P.H., Cozijnsen, J.I., Pilnik, W., Chufa: Cyperus esculentus: A new source of dietary fibre. J. Agric. Food Sci. 1989, 49, 291-296.

[9] Manek, R.V., Builders, P.F., Kolling, W.M., Emeje, M., Kunle, O.O.: Physicochemical and binder properties of starch obtained from cyperus esculentus AAPS. Pharmscitech. 2012, 13, 379-388.

[10] Philip F. Builders. Chukwuemeka C. Mbah., Kenneth K. Adama., and Momoh M. Audu. “Effects of ph on the physicochemical and binder properties of tigernut starch”. Starch/starke 2013, 65, 1-13.

[11] AOAC (1990). Official methods of Analysis.15th Ed, Association of official analytical chemists. Washington D.C, pp: 808,831-835.

[12] Afolayan, Michael O., Omojola, Moses O., Orijajogun, Joyce O., Thomas, Sunday A. (2012): Further physicochemical characterization of Anchomanes difformis starch. Agriculture and Biology Journal of North America 3(1): 31 –38.

[13] Omojola M. O., Akinkunmi Y. O., Olufunsho K. O., Egharevba H. O. And Martins E. O. (2010): Isolation and physico-chemical characterization of cola starch; African Journal of food, agriculture, nutrition and development. 10(7):2884 – 2900.

[14] Attama A. A., Nnamani P. O., Mbonu I. K. and Adiku M. U. (2003): Effect of hypochlorite oxidation on the physicochemical properties of gladiolus starch; Journal of Pharm and allied Science 1 (1) 28-35.

[15] Coursey D. G. And Rasper V. (1967): Properties of starches of some west African yams; J. Sci. Food Agric., 18: 240 – 248.

[16] Chowdary K. P. R. & Enturi V. (2011): Preparation, characterization and evaluation of starch citrate- a new modified starch as a disintegrant in tablet formulations; International Journal of Pharm. Research and Development 12 (2) 9 – 17.

[17] Nuwamanya E., Baguma Y., Emmambux N., Taylor J. and Rubaihayo P. (2010): Physicochemical and functional characteristics of cassava starch in Ugandan varieties and their progenies; Journal of Plant Breeding and Crop Science 2 (1) 001 – 011.