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Extraction and Characterization of White Guinea Corn (*Sorghum arundinaceum*) Starch: A Potential Source of Pharmaceutical Raw Excipient Material

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ABSTRACT

The current research article is focused on extraction and characterization of White Guinea Corn Starch as pharmaceutical excipient. The present study is aimed at extracting and characterizing a tablet excipient from a local source, white guinea corn which is used locally as a source of food because of its high carbohydrate content. Isolated starch has been evaluated for all critical parameters like flow rate, angle of repose, Carr's index, moisture content, bulk density, tapped density, swelling capacity, hydration capacity, Hausner's ratio, iodine test, solubility, hygroscopicity, efflorescence, pH and moisture sorption capacity and compared with maize starch BP. The following physicochemical parameters were investigated: flow rate, angle of repose, Carr's index, moisture content, bulk density, tapped density, swelling capacity, hydration capacity, Hausner's ratio, iodine test, solubility, hygroscopicity, efflorescence, pH and moisture sorption capacity. Maize starch BP was used as a reference standard; starch identification tests, characteristics were evaluated. The result showed that white guinea corn starch has a good yield and has similar characteristics with maize starch BP in almost all the parameters evaluated as specified in British Pharmacopoeia by BP and Handbook of Pharmaceutical Excipients. Therefore, the starch can be used as an alternative to maize starch BP in pharmaceutical formulations especially solid dosage forms.

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INTRODUCTION

Starch is one of the earliest excipients to be used for pharmaceutical dosage forms.[1] Starches are the major polysaccharide food reserve of seeds, stems and roots of plants with definite chemical structure and composition such as amylose and amylopectin.[1,2] Starches are widely used as excipients in pharmaceutical drug manufacture. In tableting, they function as diluent, disintegrant, binder and glidant.[3,4] Guinea corn commonly called sorghum, is a grass species cultivated for its grain, normally used as food by both animal as well as human also its has rich significance in ethanol production. which is used as food, both for animals and humans and for ethanol production. Sorghum originated in northern Africa and is now cultivated widely in tropical and sub-tropical regions and it is world fifth most important cereals crop after rice, wheat, maize and barley.[5] Sorghum is the world fifth most important cereals crop after rice, wheat, maize and barley.[5] It is most at times annual, it grows in clumps that may reach over 4m high. Sorghum is annual plant and its grain are small, ranging from 3-4 mm in diameter.[2] Sorghum grows in a wide range of temperature, high altitudes, toxic soils and can recover growth after some drought. It can be said to survive harsh environmental conditions. A good quality and mature guinea corn contain starch (74.63%), dietary fibre (6.30%), fat (3.30%), protein (11.30%) and 4.47% other micro- nutrients indicating that it can be a potential source of starch.[5] The study, therefore, is aimed at extraction and characterization of starch from white guinea corn.

MATERIALS AND METHODS

Sorghum arundinaceum (white guinea corn) obtained from Samaru market in Zaria was taken to department of Biological Sciences, Ahmadu Bello University Zaria, Nigeria, for authentication and certification at the Herbarium. Other materials are Maize starch BP (BDH, UK), Sodium hydroxide pellets (Merck, Germany). They were all utilized as obtained.

Extraction of starch

A 2.5 kg of white guinea corn was washed several times with water and soaked in 4.5 litres of water containing well dissolved 45 g of 0.25 M sodium hydroxide and left for 24 hrs. The water and sodium hydroxide was decanted and then washed thoroughly with scrubbing using enough water until the bark was removed and a clear supernatant was formed. It was then grinded and filtered with a sieve following with addition of enough water to ensure complete removal of the starch during the filtration process. The filtrate was allowed to stand overnight

at room temperature (25-28^{o}C), for the starch to settle and the water on top (precipitant) was decanted remaining the starch at the bottom. Re-suspension and sedimentation operations were repeated until white product was obtained. It was centrifuged on thermo-electron machine (at 1000 rev/min for every 5 minute interval). The product was then dried in air at room temperature for 5 days and monitored every day to avoid smearing and caking using hand. It was placed in an oven at 48^{o}C for 24 hrs to avoid denaturalization. The native starch obtained was then size-reduced and passed through 180 µm sieve. The percentage yield was calculated and recorded.

Preparation of modified/pregelatinization *Sorghum arundinaceum* (PGS)

The 150 g of the native starch was dissolved in 1000 ml of distilled water in a stainless steel container and placed in a water-bath whose temperature rises with time. Thermometer was placed inside the container from time to time to determine the temperature and it was continuously stirred to avoid caking. This process continues until a gelatin was formed and the temperature was recorded. Then it was poured into a cleaned, dried, open and stainless steel tray and kept inside an oven to dry at temperature of 60^{o}C. The percentage yield after gelatinization was calculated and recorded. The weight of the PGS (W_2) was expressed as the percentage weight of the dry starch (W_1) used for producing the mucilage that gave the yield (Y), where $\mathbf{Y = (W_2/W_1).}$

Determination of physicochemical properties

The following physicochemical tests were conducted on the native starch and PGS with maize starch as the standard for comparison.

Solubility test: A 1 g of each starch was weighed and transferred into beakers containing 1ml, 2 ml, 10 ml, 1 L and 10 L distilled water at 25^{o}C. The mixture was stirred and the solubility noted. The procedure was repeated using hot water and 95% alcohol as a solvent. The tests were repeated for PGS and maize starch BP.

Iodine test: Using BP[6] starch identification test protocol, 1 g of each starch was boiled with 15 ml of water and allowed to cool. A few drops of 0.1 N iodine solutions were added to 1ml of the mucilage and the colour changes recorded. The same test was performed on PGS and maize starch.

pH test: A 1.0 g of each sample was dispersed in 100 ml of distilled water, shaken vigorously for 5 minutes and allowed to stand. The pH of the supernatant liquid was determined using a pH meter (Oaklon pH meter: pH1100 series, Singapore).

Moisture contents: A 1.0 g of each sample was weighed using an electronic balance and dried in the Gallenkamp hot air oven at 105^0C for 80 min. At every 30 minutes interval until a constant weight was obtained. The percentage loss on drying was determined as moisture content. Moisture Content = W2/W1 x100%; where W1 is the initial weight before drying and W2 is final weight after drying.

Moisture absorption capacity: A 2.0 g of each starch powder was weighed and uniformly distributed over the surface of a 70 mm tarred Petri- dish. The sample was placed in a desiccator containing distilled water in its reservoir (RH =100%) at room temperature and the weight gained by the exposed sample at the end of the five day period was noted and the amount absorbed was calculated from the weight difference.

Swelling power: The swelling capacity of each powder was estimated by a method described by Iwuagwu and Onyekweli[7]. The tapped volume occupied by 5 g of the powder, Vx, was noted. The powder was then dispersed in 85 ml of water and the volume was made up to 100 ml with more water. After 24 hr of standing, the volume of the sediment, Vv, was estimated. The swelling capacity, (S) was computed as follows: S = Vx / Vv.

Sieve analysis: A 50 g of each starch was weighed individually and placed on the set of sieves arranged in the order: 500 µm, 250 µm, 150 µm, 90 µm, 75 µm and the pan sizes. The sieves were placed on the Endecott sieve shaker and shaken for 10 minutes. The particles retained on each sieve were weighed and recorded. The cumulative frequency weights were calculated and a graph of cumulative frequency oversize (%) was plotted against sieve size (µm) (Figure 1).

Determination of flow properties

Angle of repose: A 50 g of each sample was placed in a plugged dry glass funnel held to a retort stand at 10 cm distance from the top of the table, then the funnel was unplugged and the granules allowed running freely and falling onto a clean white paper. The height (H) of the cone formed and its diameter (D) were recorded. Angle of repose was then determined using the base radius (R) of the powder heap. $A° = \tan^{-1} H/R$; where H is the height and R is the radius (D/2**).**

Flow rate: A 50 g of each sample was placed into the Erweka flowability apparatus funnel and then allowed to flow freely through an orifice. The time of flow was then noted and recorded. This experiment was repeated three times and the average reading recorded in g/sec. Flow Rate = w/t; where w is the weight (g) of the sample and t is the time taken (seconds).

Determination of starch density

Bulk density: A 50 g of each sample was weighed and poured gently through a short stemmed glass funnel into a 100 ml measuring cylinder. The volume, Vo was noted as the bulk volume and the bulk density was calculated using the formula below: Bulk Density = mass (g) / Vo (cm^3).

Tapped density: A 50 g of each sample was weighed and transferred into a 100 ml measuring cylinder. The initial volume, Vo was noted. The cylinder was then tapped 50 times on a hard table, noting the volume, V_{50} of the samples. The tapped density was then calculated using the formula: Tapped Density = Mass (g) / V_{50} (cm^3).

Carr's index: This is the percentage difference between the tapped density and the bulk density. It is also referred to as compass billing index or compressibility; Carr's index = Tapped density- Bulk density / Tapped density x 100.

Hausner's ratio: This is the ratio of tapped density to bulk density. Hausner's ratio = Tapped density / Bulk density.

Hydration capacity: A 1.0 g each sample was placed in each of four 15 ml plastic centrifuge tube to which 10 ml distilled water was added and then stoppered. The content was mixed on a vortex mixer for 2 minutes. The mixture was allowed to stand for 10 minutes and then centrifuged at 1000 rpm for 10 minutes in a bench centrifuge. The supernatant was carefully decanted and the sediment weighed. The hydration capacity was calculated as the ratio of sediment weight to the dry weight.

Hygroscopicity and Efflorescence: A 2.0 g W1 of each starch in an evaporating dish was exposed to atmospheric condition by placing in an open space and left for 24 hrs but observed at 8 hourly intervals. The final weight W2 of the sample was recorded after exposure and percentage difference calculated. H = 100(1-{W2/W1}). If the value of H, hygroscopicity value, is minus or negative it means it absorbed some moisture and if it has a positive value it

means it lost some moisture. The average relative humidity of the room was also taken into account.

RESULTS AND DISCUSSION

The flow properties of powder are essential in determining its ability as direct compression excipient. The flow rate of powder is said to be good if it below 5g/s.[8] The flow rate test carried out for the batches are 2.14g/s, 4.28g/s and 4.80g/s for native, modified and maize starch (standard) respectively, indicating that the native and modified starch flows better than standard (Table 1). The angle of repose for a powder with good flow property should be within $30^0 30^o$.[9] However, the angle of repose between 35-45^0 is a sufficient criterion for predicting the flow properties of powder.[10] From the results obtained, native starch and the modified starch have angle of repose within 28-35^0 comparable to the maize starch (standard) as shown in Table 1. This is an indication that the powders have good flowability.

The Carr's index is a measure of the flowability and compression of a powder. The maximum acceptable value for Carr's index is 15% (Remington, 2005). The lower the Carr's index of the material, the better the flowability and compressibility.[11] The native and the modified starch have Carr's index of 15.82 and 15.92 similar to the standard of 14.50 (Table 1). Hausner's ratio is an indicative of interparticulate friction. It is an indirect measurement of powder flow. Hausner's ratio greater than 1.25 indicates poor flow.[12] The results obtained are 1.20, 1.26 and 1.46 for native, modified and standard respectively; Meaning that the native and modified are better than the standard (Table 1).

The values of bulk and tapped densities reveal granule size, flowability and the rate that the powder packs down and their values are similar to the standard as shown in Table 1. The maximum moisture content prescribed for safe storage by most starch producing countries is 13%w/w. higher levels of moisture have been known to affect the flow and mechanical properties of starches and can lead to microbial spoilage and consequent deterioration in starch quality.[13] The results obtained shows that both the native and modified starches have moisture content values within the acceptable range (13.5%w/w, 11.50%w/w and 12.10%w/w for native, modified and standard respectively) (Table 1). The low percentage yield obtained for native starch (37%) indicates that it contains protein substances, roughages, fibers and other minerals, while the high percentage yield of modified guinea corn starch (95%) indicating that is more pure because it has undergone modification (Table 1). Swelling power is the ability of the material to absorb water and swell up. The swelling capacity for

native, modified and standard are 14.03, 21.67 and 21.07 respectively, is indicating that modified starch swells faster comparable to the standard than the native starch. The high swelling power of modified starch will give good disintegrating properties[14] (Table 1). The hydration capacities for native, modified and the standard are 1.485, 3.658 and 14.84 respectively indicating Meaning that maize starch easily get hydrated than the other two starches (Table 1). The moisture absorption values for the native, modified and standard starch are 7.22, 19 and 12.50 respectively. Indicating that modified starch has the highest value due to its high porosity and capillary action. The equilibrium moisture content of starch is a measure of its sorption characteristics and this may introduce instability into moisture sensitive drug products (e.g. aspirin) and might be indicative of a good disintegrant property of the starch [2,3,5,14] (Table 1)

CONCLUSION

The results of the research show that white guinea corn (*Sorghum arundinaceum*) can be compared to maize starch BP which indicates that they can be used as pharmaceutical excipients.

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Table 1: Physicochemical parameters of *Sorghum arundinaceum*, PGS and Maize Starch BP

Physicochemical parameters	*Native Starch*	*PGS*	*Maize Starch BP*
Flow rate (g/sec)	2.14	4.28	4.80
Angle of repose(^{0})	31.49	28.10	35.90
Moisture content (%w/w)	13.50	11.50	12.10
Moisture sorption capacity	7.22	19.00	12.50
Swelling power (%)	14.03	21.67	21.07
Bulk density (g/ml)	0.526	0.609	0.52
Tapped density (g/ml)	0.625	0.769	0.76
Carr's index (%)	15.82	15.92	14.50
Hausner's ratio	1.20	1.26	1.46
Solubility	insoluble	insoluble	insoluble
Iodine test	positive	positive	positive
pH	7.60	7.65	6.5
Percentage yield (%)	37	95	-
Hydration capacity	1.485	3.658	14.84
Hygroscopicity	no	yes	yes
Efflorescence	yes	no	no
Odour	Odourless	Odourless	Odourless

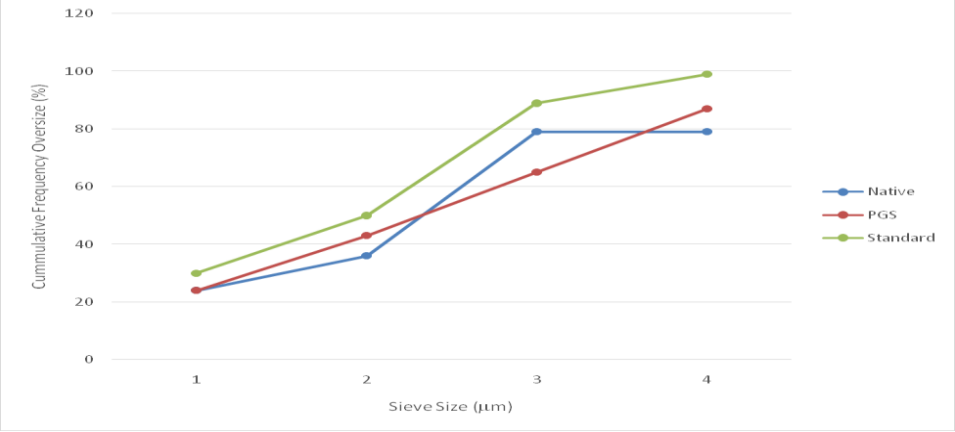

Figure 1: Shows the graph of sieve analysis of the three starches.