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Studies on the Physico-Chemical Properties of Starches from *Sorghum bicolor* and *Zea mays*

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ABSTRACT

The physicochemical properties of starches from *Sorghum bicolor* and *Zea mays* and their potentials as pharmaceutical excipient were evaluated. The starch was prepared by steeping in water and wet milling after 24 hours. A 0.25M sodium hydroxide pellets were added to the water to separate protein matter and neutralize acidity. The starch was then dried in the oven; this was the native starch which was then modified by pregelatinization. *Zea mays* (maize) starch BP was used as a reference standard for comparison. The following physicochemical properties were investigated: flow rate, angle of repose, Carr's index, Hausner's ratio, and moisture content, moisture sorption capacity, swelling capacity, bulk density, tapped density, particle size, solubility, iodine test, hydration capacity, gelatinization temperature and pH. The study revealed some similarities and differences in physicochemical properties between the starches. However, the three starches met the basic requirements for pharmaceutical use and application.

INTRODUCTION

Starch is cyst polymeric compound consisting of about 27% linear polymer (amylose) and 73% branched polymer (amylopectin) which are associated in the crystal lattice that is practically insoluble in cold water or alcohol but soluble in boiling water, giving a colloidal solution which forms a translucent jelly on cooling.[1] The USP[2] described starch as irregular angular white masses or a fine powder consisting chiefly of polygonal, rounded or spheroidal grains up to 35 microns in diameter and usually with a circular or several rayed central cleft. Starch is one of the most widely used excipient in the manufacture of solid dosage forms. Starches are usually modified to impart good flow and compressional characteristics. Several types of modified starches have been studied as tablets excipients[3] evaluated the incorporation of starch and a modified one within the granules of several drug formulations and found that the modified starch generally exhibited improved processing characteristics and tablet properties compared with the native starch. Starch is widely used as diluent, disintegrant, binder and glidant.[4-6] The pharmaceutical grade starch in Nigeria today is imported and this is reflected in increased production cost and erratic supply of raw materials. There is, therefore, a need to look inwards for the development of locally available starch as Pharmaceutical excipient binder that would compare with the existing imported ones.

Sorghum bicolor L. (Moench) is an indigenous crop to Africa, and though commercial needs and uses may change over time, sorghum will remain a basic staple food for many rural communities.[7] The latter is especially true in the more drought prone areas of Nigeria where this hardy crop provides better household food security than maize.[7] Sorghum is mainly cultivated in drier areas, especially on shallow and heavy clay soils. The production of sorghum in Nigeria varies from 100 000 ton (130 00 ha) to 180 000 ton (150 000 ha) per annum.[7] As a general rule, medium-late maturing sorghums are higher yielding - particularly under conditions of good moisture and nutrients. However, as moisture conditions become more limiting, the earlier-maturing hybrids will have greater yield reliability. Hybrids with medium to medium-quick maturity are likely to flower in 60-65 days when sown early, and 50-55 days when sown later under higher temperatures.[4-6] Slower-maturing hybrids will generally give higher yields than quick-maturing hybrids when moisture and nutrients are not limiting. When moisture is limiting, the quicker-maturing hybrids may offer better reliability. The choice of maturity will, therefore, depend on conditions at planting (e.g. soil type, stored water) and attitude of the grower to risk.[4,6] The objectives of this study is to carry out preliminary investigations into the physicochemical properties and suitability of the native and modified form of *Sorghum bicolor* starches as compared to *Zea mays* starch (maize starch B.P) as pharmaceutical excipient.

MATERIALS AND METHODS

Sorghum bicolor was obtained from Samaru market in Zaria, Kaduna State, Nigeria and Maize starch BP from B.D.H. Laboratories (U.K).

Collection of *Sorghum bicolor*

The *Sorghum bicolor* that was purchased from Samaru market Zaria, Nigeria was taken to the Department of Biological Sciences, Ahmadu Bello University, Zaria, for authentication and certification at the herbarium.

Extraction of starch

Yellow guinea corn (*Sorghum bicolor*) 2.6 kg was weighed and soaked in 4.5 liters of water containing dissolved 0.25 M sodium hydroxide pellets 45 g for 24 hours to separate the starch and protein material as well as to neutralize the slight acidity. Excess NaOH was removed by washing several times with distilled water. It was grinded and sieved, the residue was discarded and the filtrate was allowed to stand for 24 hours. The supernatant was then decanted, the sediment was centrifuged for five minutes and the upper portion was discarded while the lower portion (which contains starch) was oven dried and size reduced to powder which is the extracted starch. It was passed through sieve sized 0.8 micrometer and then oven dried for 1 hour. The percentage yield was calculated.

Pregelatinization of native *Sorghum bicolor* starch

One hundred and fifty (150) grams of the powder was weighed and dispersed in a silver dish and one liter of distilled water was added. It was then heated on a water bath and continuously stirred until translucent mucilage was formed. The mucilage was poured into a stainless steel tray which was placed in Gallenkamp hot air oven (Gallenkamp machines, model size 3, Birmingham, England) subjected at 50^0C until a constant weight was obtained over 48 hours period. The flakes were milled through the blender, sieved through 180 µm sieve mesh and stored as PGS. The weight of the PGS (W_2) expressed as the percentage weight of the dry starch (W_1) used for producing the mucilage to give the yield (Y), where $Y = 100(W_2/W_1)$.

Organoleptic properties of starch

The color, odour, taste and texture of the powdered starch was observed or determined physically using the sense organs (eye, nose, tongue and hand respectively) and the results obtained recorded.

Physicochemical properties of native *Sorghum bicolor* starch

The following physicochemical tests were conducted on the *Sorghum bicolor* starch and PGS with maize starch BP as the standard for comparison.

Moisture content test

A 2 g of the starch was placed in an oven at 105^0C and the weight of the starch was noted at time intervals of 30, 60 and 80 mins. The moisture content was calculated as percentage of the ratio of weight loss to that of the initial weight of the starch. The tests were repeated for PGS and maize starch BP.

Bulk and tapped densities

A 50 g of the powder was weighed on an electric balance and poured into a 100 ml glass measuring cylinder. The cylinder was dropped on a wooden platform from a height of 2.5 cm three times at an interval of 2 seconds. The volume occupied by the powder was recorded as the bulk volume. The cylinder was then tapped on the wooden platform for a further 50 times and the volume recorded as the tapped volume. The bulk and tapped densities were calculated as the ratio of weight to volume. The same tests were performed for PGS and maize starch BP. The data generated was used in computing the Carr's and Hausner's ratio. The equations are as follows:

Bulk or tapped densities = weight of powder/ volume of powder

Carr's index = [(Tapped density - Bulk density) /Tapped density] x 100

Hausner's ratio = Tapped density / Bulk density

Flow rate

Fifty (50) grams of the powder was placed in an Erweka flow apparatus (Type GD7, Erweka - Apparatebau G.M.B.H Heusentamm, West Germany) and allowed to flow through the funnel orifice. The time taken for the powder to flow through the orifice was noted and the flow rate was determined as the ratio of mass (g) to time (s). The test was repeated for PGS and maize starch BP. The means of three determinations were recorded. Flow rate = weight of powder in grams / flow time in seconds.

Particle size analysis

Fifty grams of the powder was placed on a nest sieve and allowed to vibrate for 10 minutes. The weight of material retained on each sieve was determined. The test was repeated for PGS and maize starch BP. A plot of percentage frequency distribution against particle size was drawn for each powder sample.

Angle of repose

The angle of repose (θ) was obtained using the funnel and stand method. A plugged glass funnel was mounted on laboratory stand at a height of 10 cm from the flat surface. Fifty (50) grams of the powder sample was placed in the funnel. The plug was removed and the sample allowed to flow. The height and diameter of the heap formed were noted. The same test was performed for PGS and maize starch BP. The angle of repose (θ) was calculated as: $\theta = \tan^{-1} H / R$, where θ is the angle of repose, H is the height of the conical powder heap and R is the radius of the circular base, (D/2).

Swelling power

The tapped volume occupied by 5 g of the powder, $V_{X,}$ was noted. The powder was then dispersed in 85 ml of water and the volume made up to 100 ml with more water. After 24 hr of standing, the volume of the sediment, $V_{V,}$ was estimated. The test was repeated for PGS and maize starch BP. The swelling capacity, (S) was computed as follows: $S = V_X/V_{V.}$

Moisture absorption capacity

Two grams of the powder was weighed and uniformly distributed over the surface of a 90 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 days period was noted and the amount absorbed was calculated from the weight difference. The test was repeated for PGS and maize starch BP.

Pharmacognostic test for starch

Iodine test

One gram of starch was dispersed in 5ml of water and one drop of iodine solution was added. The color obtained was observed.

Determination of pH

One gram of the 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 the pH meter (Oaklon pH meter pH 1100 series, Singapore.) The same procedure was repeated for PGS and maize starch BP.

Hydration capacity

One gram of sample was placed in each of four 10ml plastic centrifuge tube to which 10 ml distilled water was added and then stoppered. The content was mixed in the centrifuge for 2 min. The mixer was then 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 the sediment weight to the dry sample weight.

Gelatinization temperature

Two (2) and one (1) grams of the powder were placed in a beaker and 50 ml of water was added to each and was placed on a water bath, the temperature of which was allowed to rise at a rate of 5^0C/minute. The temperature at which it gelatinizes was noted as the gelatinization temperature.

Solubility

One gram of the powder was dispersed in 10 ml each of cold distilled water, hot distilled water, and ethanol and left overnight. Five (5) ml of the clear supernatant solution of each mixture was taken and heated to dryness over a water-bath. The weight of the dried residue with reference to the volume of the solution was determined as the percentage solubility of the powder in the solvent. The test was repeated for PGS and maize starch BP.

Determination of Hygroscopicity and Effervescence

Two (2) grams, (W_1) of the powder was placed in an evaporating dish and exposed to atmospheric condition by placing in open space and left for 24 hours but observed at 8 hourly intervals. The final weight (W_2) of the sample was recorded after exposure and the percentage difference calculated. Hygroscopicity (H) = 100 [1- (W_2/W_1)]. If the value of H, hygroscopic 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 noted.

RESULTS AND DISCUSSION

The results of the tests carried out are shown in tables 1- 4. Those of physicochemical tests are presented in table 4. The percentage yield of the PGS (97.6%) was found to be appreciably higher than that of the native starch (38.8%). The high yield could have been due the production of mucilage of the starch to rupture some of the crystalline microscopic granules which were neither an addition nor reduction to the final weight while the low yield of the native starch could be due to spillage during processing and presence of other constituents in the grains. All the starch powders indicated good angle of repose.[5] Both PGS and native starches indicated better flow properties which include angle of repose, lower flow rates, lower difference between bulk and tapped densities and therefore lower values of Carr's packing or consolidating indices and lower moisture content. Hausner's ratio and Carr's compressibility are used to predict the flow of granules and powders. Carr[8] predicted the values of Hausner's quotient and Carr's compressibility as 1.25 and 23 respectively and do not indicate good flow behavior. Veslasco *et al*[9] reported that angle of repose values above 50^0 is an indication of poor flow characteristics of a powder.

The result of moisture content was in the order maize starch BP > *Sorghum bicolor* native > PGS (table 4). Moisture content plays an important role in the stability of a pharmaceutical product. High moisture content tends to encourage bacterial growth and chemical degradation of excipients. High moisture content in starches affect the flow property of the granules, these could affect some parameters of tablets such as uniformity of the weight and content uniformity. Generally, 3% moisture content is considered by formulation scientists as the maximum permissible if chemical stability of hydrolysable drug content in a solid dosage form is to be assured. However, it is agreed that it is only the free mobile water fraction rather the total water content of excipients that is important in chemical stability as the bound fractions do not induce hydrolysis.[10]

The result of moisture sorption capacity was in the order PGS > *Sorghum bicolor* native starch > maize starch BP (table 4). The equilibrium moisture content of starch is a measure of its sorption characteristics and this may introduce instability into moisture sensitive drug products when such starch is used in drug formulations.[11,12] Although PGS had the lowest moisture content, it had the highest moisture sorption capacity which makes it less suitable for moisture sensitive drugs. It might be indicative of a good disintegrant property even though this result showed that the powders are sensitive to atmospheric moisture and should therefore be stored in air tight containers.

The swelling power of *Sorghum bicolor* starch was found to be 16.3%, 21.07% for maize starch BP while that of PGS was 35.6%. Swelling is generally accepted as an indication of tablet disintegrant ability.[13] It reflects the increase in volume of starch following water absorption and the PGS had the highest swelling power followed by maize starch then *Sorghum bicolor* native starch (table 4). The effectiveness of their disintegrant properties is expected to be in the same order.

The pregelatinized *Sorghum bicolor* starch has been found to be a good disintegrant in tablets because of its excellent physicochemical and pharmaceutical properties compared to native *Sorghum bicolor* and Maize starches.[14]

The particle size distribution of native starch of *Sorghum bicolor* as compared with Maize starch BP is shown in tables 2 and 3. The particle size of *Sorghum bicolor* was found to be larger than that of the Maize starch BP which translated to the flow rate of 2.056 g/sec compared to 5.4 g/sec for Maize starch BP. It has also reflected in the lower angle of repose (32.4^0) of the heap of *Sorghum bicolor* starch while that of maize starch is 35.96^0 Larger particles have smaller surface area to volume ratio. Particulate function is more of a surface phenomenon by generation of resistance to flow. This resistance is directly proportional to the surface area of the particles. The bulk property of the powder depends on the particle size and size distribution, surface area and surface to volume ratio and in the indices of physicochemical properties of a drug produced from the powders in question. Generally, as sizes of particles fall below 100 µm, they tend to be cohesive and those with lower than 100 µm hardly flow, while those with values greater than 250 µm usually possess good flow properties.[15]

CONCLUSION

Yellow guinea corn (*Sorghum bicolor*) starch has been successfully extracted from *Sorghum bicolor* cereal using standard methods of extraction which gave a moderate percentage yield. Some of the extracted starch was pregelatinized (modified) and its physicochemical properties compared well with those of unpregelatinized (native) starch and Maize starch BP. The two starches proved to be suitable for use as pharmaceutical excipients.

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Table 1: The Organoleptic properties and solubility of yellow guinea corn (*Sorghum bicolor*) Starch

Physical Character	*Native Starch*	*Modified Starch*
color	Yellowish White	Grey
Odour	Odourless	Odourless
Form	Powder	Powder
Taste	Bland	Bland
Texture	Fine	Fairly Coarse
Solubility in cold water	Insoluble	Insoluble
Solubility in hot water	Insoluble	Insoluble
Solubility in ethanol	Insoluble	Insoluble
Percentage Yield	38.8%	97.6%
PH	7.60	7.65
Gelatinization Temp.	69°C	67°C
Hygroscopicity	0%	99%

Table 2: Sieve analysis of native starch of *Sorghum bicolor*

Sieve Size (µm)	Weight Retained (g)	Weight Retained (%)	Cumulative (%)	Sieve Size × % Weight
500	5.12	10.24	10.24	5120
250	11.74	23.48	33.72	5870
180	6.38	12.76	40.10	7218
150	1.64	3.28	43.38	492
90	2.65	5.30	48.68	477
75	3.52	7.04	55.72	528
Pan	16.79	33.58	89.30	2014.8

Mean particle size = Σ (sieve size × % fraction retained/100) = 21719.8 / 100 = 217.198

Table 3: Sieve analysis of maize starch BP (standard)

Sieve Size(µm)	*Weight Retained (g)*	*Weight Retained (%)*	*Cumulative (%)*	*Sieve Size × % Fraction Retained*
500	0.13	0.26	0.26	130
250	4.89	9.78	10.04	2445
180	5.46	10.92	20.96	1965.6
150	5.08	10.16	31.12	1524
90	4.72	9.44	40.56	849.6
75	6.02	12.04	52.60	903
Pan	23.32	46.64	99.24	2798.4

Mean particle size = 10615.6 / 100 = 106.156µm

Table 4: The Physicochemical parameters of *Sorghum bicolor* Starch, Maize Starch BP and PGS

Properties	*Native Starch*	*Modified Starch (PGS)*	*Maize starch BP*
Bulk Density (g/cm^3)	0.658	0.714	0.52
Tapped Density(g/cm^3)	0.781	0.807	0.76
Carr's Index (%)	15.79	11.43	31.60
Hausner's Ratio	1.188	1.13	1.46
Angle Of Repose(^{0})	34.01	32.4	35.96
Flow Rate (g/s)	2.056	4.90	5.40
Moisture Content(%W/W)	12	11.50	12.10
Swelling Power	16.30	35.60	21.07
Hydration Capacity	1.953	2.69	2.54
Moisture Sorption Capacity (%)	23	51.5	14.84
Iodine Test	Positive	Positive	Positive
Solubility	Insoluble	Insoluble	Insoluble
pH	7.60	7.65	6.50

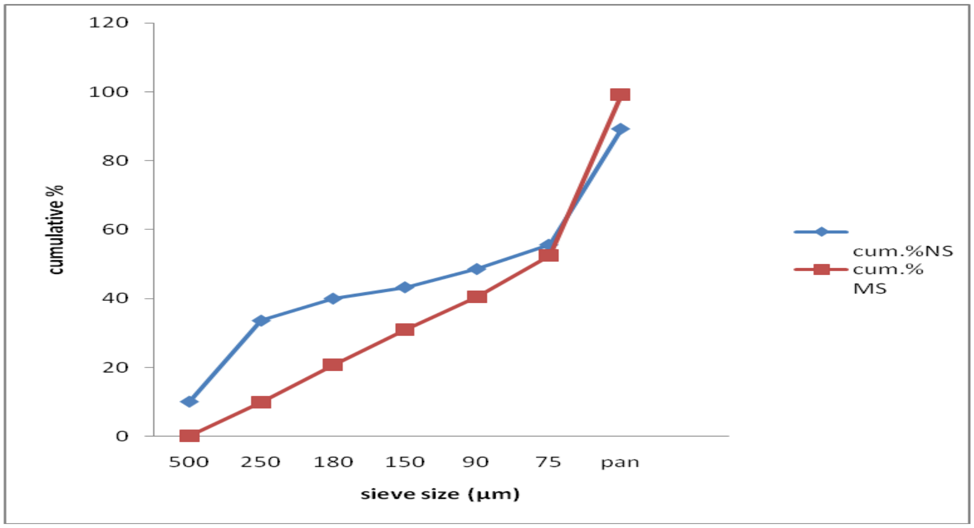

Figure.1: Graph of cumulative percent against sieve size for native *Sorghum bicolor* starch (NS) and maize starch (MS)