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Research Article

STUDIES ON SOME PHYSICOCHEMICAL PROPERTIES OF NATIVE AND MODIFIED STARCHES FROM *DIGITARIA IBURUA* AND *ZEA MAYS*

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ABSTRACT

This study aims at evaluating a tablet excipient from local source *Digitaria iburua* starch which is used locally as food because of its high carbohydrate content, it was thought that the starch from *Digitaria iburua* may serve as a tablet excipient. The Physico-chemical properties of starches from *Digitaria iburua* starch and *Zea mays* and potentials as pharmaceutical excipients were evaluated and compared..*Digitaria iburua* starch was extracted for this study by steeping the grains in water and wet-milled after 24hours.0.1N sodium hydroxide solution was added to the sieved starch. The starch was dried in the oven at 48^{o}C. *Zea mays* (.Maize stach B.P) was used as a reference standard for comparism. The percentage yield for starch from *Digitaria iburua* crop was found to be 62.21% while the yield for the modified starch was 73.18% .

The results showed that *Digitaria iburua* starch and maize starch BP have almost similar , Carrs index, moisture content, true density, tapped density, bulk density, Hausner ratio, swelling power and mean particle size, However, *Zea mays* starch showed superiority in some properties such as moisture sorption capacity and Angle of repose. The photomicroscopy shows that *Digitaria iburua* starch has predominantly polygonal with occasional spherical or oblong granules with occasional striation especially with the spherical granules. The study revealed smilarity and some differences in physico-chemical properties between the starches. However, the starch met the basic requirements for pharmaceutical use and application.

Keywords: Evaluation, Starch, Excipient, *Digitaria iburua*, Physico-chemical, *Zea mays.*

INTRODUCTION

Starch is one of the earliest excipients to be used for pharmaceutical dosage forms. Depending on the application, starch acts as a diluent, disintegrating agent or binder. Starches are the major polysaccharide food reserve of seeds,stems and roots of plants,with definite chemical structures and composition.The starch polymer consists of D-g lucose units linked together through –(1 4) bonds to form a linear component called amylase and in addition –(1 6) branch points.The branched form of amylase is called amylo-pectin.Amylose and amylopectin are the major chemical components of starch granules.Generally,amyloses have good structural properties because they pack closely ro form strong,rigid and insoluble material unlike amylopectins which are readily soluble in aqueous systems,thus giving good thickening properties [1]The proportion of amylase in starch depends on its botanical source and this may vary from 10% to 30% [2].Starches with low amylose content was found have better pharmaceutical uses.[3]

Starch in its pure form is a white,amorphous,relatively tasteless solid,odourless and insoluble in cold water and in organic solvents such as ethanol,ether and acetone.Starch is hygroscopic and absorbs water when equilibrated under normal atmospheric condition until the amount present is 10-17%[4,5] Official starches available recommended by British Pharmacopocia 2002 [6] for pharmaceutical industries are:-

- Maize starch obtained from caryopsis of zea mays L.
- Rice starch obtained from caryopsis of oryza sativa L.
- Wheat starch obtained from caryopsis of triticum aestivum, L(T.vulgare)
- Potato starch obtained from tuber of solanum tuberosum L.
- Tapioca starch obtained from mainhot utilissima.

To proffer solution to the importation of starch for tabletting, researchers have evaluated local starches for use as pharmaceutical excipients.

Nasipuri ,(1975) evaluated cassava starch (*Manihot utilissima*) as a tablet binder and disintegrant and found out that to be as efficient as potato starch.

Yam starch (*Diascorea rotundata*) was also evaluated by Nasipuri ,(1979)[8] as pharmaceutical binder and disintegrant and he found out that the starch so obtained was suitable as a binder and disintegrant.

Nasipuri ,(1979) [7] also evaluated cocoyam starch (*Colocasia esculenta*) as binder and disintegrant and found that the product is as suitable as potato starch.

Opakunle et al (1978) [9] investigated yam (*Diascorea rotundata*) and cassava (*Manihot utilissima*) starches as diluents and glidants, they found out these suitable for those purposes.

Esezebo and Ambujam (1986)[10] evaluated plantain (*Musa paradisiaca*) starch as tablet binder and disintegrant with maize starch as standard. They concluded that plantain starch has twice the binding properties of maize starch and half the disintegrant property.

Iwuagwu et al (1986)[11] studied African bitter yam (*Diascorea dumetarum*) and White yam (*Diascorea rotundata*) starches for their binding and disintegrating properties vis-à-vis potato starch and found that white yam tuber starch have stronger binding property than potato and African bitter yam. However the disintegrant properties were similar.

Deshpande et al (1987)[12] studied the binding and disintegrating properties of starch extracted from sorghum (*S. bicolor*) and found that it was as good as maize starch.

Muazu (2010)[13] evaluated the glidant properties of fonio (*Digitaria exilis*) starch and concluded that it can be used as glidant in tabletting.

Akande (1988) [4] investigated pearl millet starch as tablet binder and disintegrant using maize starch as standard. He concluded that millet starch can be use as both binder and disintegrant.

Garr (1988) [14] found sorghum starch as useful as maize starch, in respect of binding and disintegrating characteristics.

Kunle (1988) [5] studied the effect of some local starches (fresh yam tuber, cassava and cocoyam) on the properties of granules and tablet formulation and concluded that these are adequate as binders and disintegrants.

Many local starches have been studied extensively but no work seems to have been reported on *Digitaria iburua*despite its abundance in Nigeria especially Northern Nigeria.

Digitairia iburua is also called Black Fonio. And it is grown in Northern Nigeria States-Zaria ,the Jos-Bauchi-Plateau regions of Nigeria as well As the Northern regions of Togo and Benin [15, 16]

The objective of this study is to conduct a physico-chemical studies on the Native and Modified starches obtained from *Digitaria iburua* grains so as to investigate on their suitability for use as a pharmaceutical excipient compared to standard pharmaceutical excipient maize*(Zea mays)* starch B.P

MATERIALS AND METHODS

Digitaria iburua grains was obtained from Sabon Gari market in Zaria, Kaduna State, Nigeria. Paracetamol (May and Baker (Nigeria), Maize starch and Talc (B.D.H. Laboratories, U.K) and magnesium stearate Hopkin and Williams, U.K.). They were all utilized as obtained.

METHODS

Extraction of starch from*Digitaria iburua* grains

Digitaria iburua grain was thoroughly washed and all foreign material removed. The washed seeds were allowed to steep in water for about 24 hours; the steeped grains were crushed using a blender (Philips Cucina HR 1757, Japan). Enough quantity of water was added to the pulp which was then passed through a 180µm sieve. The starch as a residue was collected allowed settling and 0.1N sodium hydroxide was added to separate the starch and protein materials as well as to neutralize the slight acidity. Excess sodium hydroxide was removed by washing several times with distilled water.

The clear supernatant fluid was poured away while sedimented starch was collected on a tray and air-dried on a table. Using pestle and mortar the dried starch lumps were ground and the fine powder passed through a sieve (180µm).

Preparation of modified/ Pregelatinised *Digitaria iburua* starch(pgs)

A 400g of *Digitaria iburua* starch powder was weighted and put in a beaker.A small quantity of cold water was added to make it into a paste.Boiled water was then added to make up to 5liters and the beaker was then put on a hot plate with continuous stirring until a translucent paste was formed .The paste was poured on a tray and dried in an oven at 40^0c to obtain a dried pregelatinised starch.

Determination of percentage yield

The percentage yield of *Digitaria iburua* was determined from the weight of grains used which was noted as W_0 and the final starch obtained from the procedure noted as W_1. For the pregelitinised starch, quantities of *D.iburua* powder before and after pregelitinisation were noted as W0 and W1 respectively. Percentage yield X was then calculated as

$Y = W_0 - W_1/W_0 \text{ X } 100$ ..... (1)

Solubility test: A 1g of *Digitaria iburua* starch was weighed and poured into a beaker containing 1ml, 2ml, 10ml, 1L and 10L distilled water at 25°C and was stirred, and the solubility was observed. Same procedure was repeated using 65% alcohol as a solvent. The procedures were repeated for pregelitinised starch and maize starch.

Iodine test: Using BP (2002) starch identification test, 1g of starch was boiled with 15ml of water and allowed to cool. A few drops of 0.1N Iodine solution were added to 1ml of the mucilage and the colour changes recorded.

Acidity test: A 10g of *Digitaria iburua* starch was added to 96%v/v alcohol which was previously neutralized using 2 drops phenolphthalein solution as indicator. The mixture was shaken for an hour using an automated beaker shaker, filtered and 50ml of the filtrate titrated with 0.1N NaOH solution. And the quantity recorded. Same was done for Maize starch and PGS.

Determination of pH: 10g of *Digitaria iburua* starch was weighed into 15ml distilled water and was properly mixed. The mixture was poured into boiling distilled water to make up 100ml of slurry. The slurry was allowed to cool. Using a pH meter (kent EIL 7055), the pH of the slurry was measured. Same was done for Maize starch and PGS.

Determination of starch hygroscopicity

A 2g of starch poured in an evaporating dish was exposed to atmospheric condition by placing in open space and left for 24 hours observed every 6 hours. The final weight of starch was recorded and percentage lost was recorded.

Determination of moisture content of starch

A 3g of starch was weighed into an evaporating dish and placed in an oven set at 105ºC. The starch was weighed periodically until constant weight was attained. The test was repeated and the mean of the three recorded using the formula

$MC = W_0 - W_1 / W_0$..... (2)

Where MC is the moisture content and w_0, w_1 are initial and final weights of the starch.The same procedure was done for PGS and Maize starch powders.

Microscopic examination of starch

Small quantity of starch in glycerol was mounted on a microscope. The size of starch particles was measured; their shape was also observed.

Determination of flow properties of starch

Angle of repose: A funnel was mounted on a laboratory stand at a height of 10cm from the table-top. 50g of *Digitaria iburua* starch was poured into the funnel with the tip closed. The tip-plug was removed and the starch was allowed to flow, the height and diameter of the starch heap were measured. Same was done for maize starch and PGS. The angle of repose, θ, is given by the following equation:

$\theta = \tan^{-1}(h/r)$.....(3)

Where h is height of conical powder heap and r is the radius of the circular base

Flow rate: using Erweka Flow tester, 50g each, of the individual starches respectively were allowed to pass through its orifice and the time taken was recorded. Mean of three readings was taken as the flow rate of the starches.

Determination of Starch density

***Bulk density*:** 20g each, of individual starches or granules respectively were poured through a short-stemmed glass funnel into a 200ml graduated glass cylinder and the volume occupied by the starch/granules was read and the bulk density calculated.

$$\text{Bulk density} = \frac{\text{mass of the starch/granule}}{\text{volume of the starch/granule}} \quad (4)$$

Tapped density: Graduated cylinder containing 50g *Digitaria iburua* starch powder was dropped on a bench 50 times from a height of about 20mm and the respective volumes recorded. Same was done for maize starch and PGS powder and the tapped densities was then calculated in g/ml

Carr's Index: The difference between the tapped and bulk density divided by the tapped density was calculated and ratio expressed as a percentage.

Hausner ratio: (i.e. the ratio of tapped density to bulk density) was calculated for all the starches.

Determination of Starch true density: The specific gravity bottled method was adopted, and xylene was used as displacement fluid. The bottle was cleaned and filled with xylene, all spilled over liquid (xylene) was wiped off with an absorbent cloth. The weight of the bottle filled with xylene was noted as (a), the bottle was emptied and cleaned, 2g of starch was weighed into the specific gravity bottle, the weight of the starch powder was noted as (w). The specific gravity bottle containing the starch was almost filled with xylene, stirred with glass rod and allowed to stand for 10 minutes for air bubbles to be released. The bottle was then carefully filled with xylene and the final weight of the bottle was noted as (b). starch true density was the calculated as:

$$\ell = w/[(a+w)-b]S \quad \text{.....(5)}$$

Where ℓ is the particle density of starch and S is the specific gravity of xylene = 0.86

Determination of swelling power

Granules were prepared from extracted *Digitaria iburua* starch, pregelitinised starch and maize starch using water as solvent. The wet mass was passed through 1.7mm wire mesh, which was then dried in an oven at 40ºC. With a 12.5mm punch and die set, the granules were compressed into tablets. Weight and dimension of tablet were recorded. Two tablets each were placed in a desiccator at 98% relative humidity at room temperature for five days. The weight and volume of the two tablets were recorded after five days. The difference between the initial and final volume was calculated and expressed as a percentage swelling power of the starches. The procedure was repeated and the mean of the two was taken as the swelling power. The swelling power, S, is

$$S = V_0 - V_{1.../} V_0 \quad \text{.....} \quad (6)$$

Where v_0, v_1 are respective initial and final volumes of the tablet

Determination of moisture sorption capacity

10g of individual starches was spread evenly in Petri dishes; the Petri dish was placed in a desiccator with 98% relative humidity at room temperature. The samples were periodically weighed until a constant weight was attained. The percentage increase in weight was calculated and taken as the moisture sorption capacity. The above experiments were repeated thrice and the average/mean of the readings was recorded.

Sieve analysis of starch powders

A 50g of the powders was weighted and put in the uppermost sieve of a set of sieves arranged in decreasing sizes. The sieves were then mounted on a sieve shaker and shaken for 10mins.The powder retained on each sieve after 10mins was measured and recorded.

RESULTS AND DISCUSSION

Table 1: Shows the percentage yield of starches obtained from *Digitaria iburua* grains and *Digitaria iburua starch*

Material	Percentage yield
Digitaria iburua (Native)starch	62.20%w/w
Pregelitinised (Modified)starch	73.18%w/w

The percentage yield of starch obtained from the grains of *Digitaria iburua* not as much as the yield obtained when *Digitaria iburua* starch was pregelatinised/Modified..

Table 2: Comparism of result of identification tests of *D.iburua*, PGS and maize starch BP

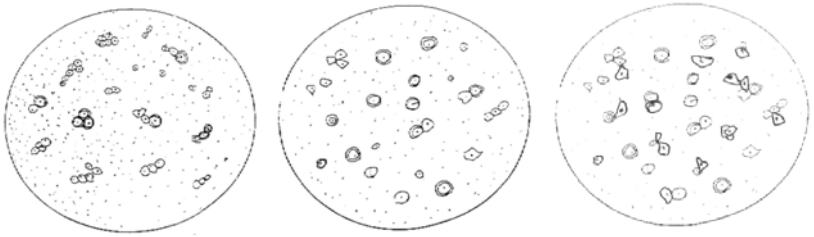
Identification test	*D. iburua*	PGS	MS BP
Solubility test	Insoluble	Insoluble	Insoluble
Iodine test	Positive	Positive	Positive
Acid test	Positive	Positive	Positive
pH	7.2	6.9	6.5

The three starch powders were insoluble in water and 65% ethanol,with blue-black colour on addition of iodine and positive to acid test.

Table 3: physicochemical parameters of Native and Modified *Digitaria iburua* starch powders compared to Maize starch B.P. powder

Physicochem.ical parameters	*Digitaria iburua* /Native starch	Pregelitinised /Modified starch	Maize(*Zea mays)* Starch B.P
Flow rate (g/sec)	5.93	5.07	5.40
Angle of Repose(°)	25.60	21.30	35.96
Carr's Index (%)	24.4	17.8	31.60
Moisture content (%w/w)	11.33	11.75	12.10
True Density (g/ml)	2.08	1.50	1.48
Tapped Density (g/ml)	0.59	0.86	0.76
Bulk Density (g/ml)	0.44	0.71	0.52
Moisture Sorption (%)	10.5	8.00	14.84
Swelling Power (%)	22.50	21.03	21.07
Hausner ratio	1.34	1.22	1.46

The flow rate, true density and swelling power of *Digitaria iburua* starch powder analysed was higher than PGS and Maize starch B.P



Digitaria Iburua Maize starch PGS

Fig. 1: Morphological structure of particles of *Digitaria iburua*, Maize starch

The percentage yield of starch powder extracted from the grains of *Digitaria iburua* was 62.20%w/w(Table 1)while the yield obtained from pregelatinising the starch was 73.18%w/w. Both yields have proved to be good .Any loss from the product could have been as a result of envisaged processing loss.Pregelatinisation of starch involves the rupture of the organized structure of the starch particles to form a granular and gelatinization involves the disorganization of the crystalline like lattice arrangement into a disorganized amorphous form[17]

Result of identification test was shown on Table 2 British Pharmacopeia (2002[6] identification tests for starch carried out showed that DI, PGS, and Maize starch (MS) as standard were positive to iodine and acid test and practically insoluble in water.

The pH of the starches were in the ranking order of MS >DI >PGS in terms of acidity. The near neutral pH of PGS might be as a result of disorganization of H^+ due to the heat applied during drying of mucilage which could reduce tendencies of interaction of excipient.

Table 3 illustrated the result of physicochemical properties of all starch powders. Both the bulk and tapped densities follow the same pattern of DI < MS < PGS. From these it could be deduced that the Bulk and tapped densities increases with PGS having the highest.

These gives an idea of how well the starch powders will compress to make a tablet since the smaller the particle size, the more the resistance to flowing powders; this is because of adhesion between the powders [18].

Also both the Carr's index and Hausner ratio tells the percentage compressibility of a starch powder. Both the Carr's index and Hausner ratio have a similar pattern of PGS<D.I <MS.

The moisture content of the starch powders ranks in the order MS>PGS>DI. This ranking could imply that the particle of DI starch may have smaller pore sizes which traps lesser amounts of water resulting to the least moisture content. Swelling Power is a parameter that is analysed in theory of disintegration which must be preceded by water penetration. The swelling power ranks in the order DI>MS>PGS. The high swelling power of DI will give good disintegrating properties. The moisture sorption is a parameter for indicating how sensitive a starch powder is to atmospheric moisture which also indicate its physical stability when formulated into tablet. The moisture sorption ranks in order MS>DI>PGS. The least value for PGS could also be due to the rupture of the organized structure of the starch particles into a disorganized amorphous form [17]

CONCLUSION

From the studies conducted it was observed that both Native and Pregelatinised form of *Digitaria iburua* Starch compared well to Maize starch B.P in terms of Physico-chemical properties and could be good excipients for Pharmaceutical industries especially in the manufactured of solid dosage forms.

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