WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
SJIF Impact Factor 8.084
Volume 9, Issue 9, 768-780. Research Article ISSN 2277– 7105

PHYSICOCHEMICAL PROPERTIES AND TENSILE STRENGTHS OF NATIVE AND TABLET FORMS OF WATER YAM (*DIOSCOREA ALATA*) STARCH FLOUR

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Article Received on
03 July 2020,

Revised on 24 July 2020,
Accepted on 14 Aug 2020,

DOI: 10.20959/wjpr20209-16375

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ABSTRACT

The physicochemical and mechanical properties of water yam (*Dioscorea alata*) starch were determined. The mechanical properties were determined using density measurement and the Heckel and Kawakita plots. Water yam starch and tablets had low flowability and less densification during die filling at low applied pressures but there was increase in the value of plastic deformation during the compression process. The mean yield pressure P_y, which is the inverse of the starch plasticity P_k, while the brittle fracture index as well as the tensile strength of the starch and tablets at different intervals were found to be moderate in values.

KEYWORDS: Physicochemical, Tensile strength, Tablet, Starch.

INTRODUCTION

Starch is a macromolecule that possesses dietary significance as a primary role. The secondary role is from technological and industrial points of view. Starch has ability to dictate or modify the texture, physiology and consistency of finished products. Recommendations to increase the intake of carbohydrate in western diets were originally a consequence of the recommendations to decrease the fat intake and keep protein intake unchanged.[1,2] Small granular starch has been reported to be good filler for biodegradable plastic films[3] and also has been suggested to provide a better mouth feeling as a lipid substitute.[4]

Water yam (*Dioscorea alata*) is specie of the yam family (*Dioscoreaceae*). It is a monocotyledonous, tuberous, fibrous, root vegetable. It is one of the oldest food crops so far

and was introduced to West Africa by the Portuguese and Spanish.[5] Water yam is tropical crop most widely grown, distributed and extensively cultivated specie of all the yams. It is also known as ten month yam or greater yam. It is popularly called "Ewura" in Yoruba land and the main staple food in Ijebu area of Western Nigeria.[6] It is planted within the month of March and April which is the beginning of rainy season and also at the beginning of dry season (October to November). It matures in 8 – 10 months and keeps better in storage than other yam species because it can remains dormant for several months. It grows best at temperature between 25^0C and 30^0C. Water yam has an advantage for sustainable cultivation due to its comparatively good agronomic characteristics. It grows in shorter time than the white yam and possesses a fibrous root system.

Water yam tubers have variable shapes; its tubers vary in number from one to five. The flesh of the tuber ranges in color from white to purple.[7] Water yam contains a higher proportion of water than either white or yellow yam. The leaves are distinctively different from others being heart-shaped, long and broad at the petiole.[8] The present work is aimed at the determination of the physicochemical properties and tensile strengths of native and tablet forms of water yam starch flour.

MATERIALS AND METHODS

Water yam tubers (*Dioscorea alata*) were bought at a market in Ondo town, Ondo state in South west, Nigeria.

Starch Isolation and Purification

Water yam starch was isolated by the method of Moorthy and Nair[9] with modifications. The water yam tubers were peeled and 450g was blended in a Kenwood blender. The blended water yam was made into a slurry mixture with the addition of distilled water. The mixture was then filtered through a triple layer cheese cloth and starch was washed thoroughly with distilled water. The washed starch mixture was subjected to further filtration using a polypropylene screen in which pure starch in solution form are washed out with distilled water as a filtrate. The filtrate was left to settle for 3 hours and then decanted. The mixture was then centrifuged at 200rpm for 20minutes in a centrifuge machine to allow the starch to settle under gravitational force. The supernatant was discarded and the protein layer was scraped off using a spatula. The residue starch was then washed with 0.5MPotassium hydroxide solution and the mixture was further centrifuged for 15minutes at 200rpm. The supernatant was later discarded while the thick starch that settled was scrapped out into a

crucible and left to dry at room temperature.[10] The dried pure starch granules were milled into fine powder, sieved, weighed and stored prior to analyses.

Determination of moisture content

Nine grams of water yam starch powder was weighed on a digital weighing balance and then mixed with 1ml of water in a porcelain slab. An oven was pre-set to 60^0C and allowed to stabilize for 10minutes. The wet starch powder was placed on a pre-weighed porcelain slab and transferred to the pre-set oven while the oven door firmly closed. The starch was brought out of the oven and weighed again at time intervals until a constant weight was obtained. Determinations were done in triplicate.

The moisture content was calculated using the equation.

$$\text{Moisture content (\%)} = \frac{\text{Amount of water loss X 100}}{\text{Amount of dry sample}} \quad \ldots\ldots(1)$$

Amount of water loss = $W_o - W_f$

W_o= initial weight of sample

W_f = final weight of sample

Determination of particle density

The particle density of the starch powder was determined by the pycnometer method using benzene as the displacement fluid (11). An empty 50ml capacity pycnometer was weighed (W), then filled with benzene and the excess was wiped off. The weight of the pycnometer bottle and benzene was determined (W_1). The difference in the two weights (W_1- W) was calculated as W_2. A 2g quantity the water yam starch powder was weighed asW_3 and transferred into the pycnometer bottle. The excess benzene was wiped off and the pycnometer was weighed again (W_4). The particle density was calculated using equation 2.

$$\rho_s = \left[\frac{W2xW3}{50(W_3 - W_4 + W_2 + W)}\right] \quad gcm^{-3} \ldots\ldots(2)$$

All determinations were done in triplicate

Determination of bulk density

The bulk density of the water yam starch powder at zero pressure (loose density) was determined by pouring 20g of the starch at an angle of 45^0 through a funnel into a glass cylinder of 50ml capacity with a diameter of 20mm. The bulk density was calculated using equation 3 (12, 13).

$$\rho_o = \frac{w}{\pi r^2 h} \text{.....(3)}$$

Where W = Weight of sample

r = radius.

h = height of the sample in the cylinder (cm).

All determinations were done in triplicate

Determination of tap density

The tap density was determined by applying 100 taps to 20g of the starch powder in a standard flask at the rate of 30 taps per minute.[14]

Determination of particle size of the starch

Particle size of the water yam starch was determined using an optical microscope at a magnification of 10. Starch particles (10g) were dispersed on a microscope slide, a drop of glycerine was added to it and the particles were viewed under a microscope and mean projected particle diameter was obtained.[15] Determinations were done in triplicate.

Determination of the starch water retention capacity

This was determined using the method of Ring.[16] To 10 grams of water yam starch was weighed into a 100ml measuring cylinder and 90ml of deionized water was added and the dispersion was shaken vigorously for five minutes. Deionized water was added to make up the solution to 100ml. 15ml of the resultant solution was centrifuged for 25minutes at 5000rpm. The supernatant was discarded and the residue was weighed (W_1). The residue was then dried at 70^0C to a constant weight (W_2) in an oven. The water retention capacity was calculated as follows:

$$\text{Water retention capacity} = \frac{W1}{W2} \text{.....(4)}$$

Determinations were done in triplicate

Flowability of the starch

The flowability of the water yam starch was determined using Hausner's ratio and Carr Index. The Hausner's ratio was determined as the ratio of the initial bulk volume to the tapped volume.[17] The Carr Index (% Compressibility factor) was calculated as follows:

$$\text{Carr Index} = \frac{\text{Tap density} - \text{Bulk density}}{\text{Tap density}} \text{..... (5)}$$

Determination of starch viscosity

Four grams of water yam starch was added to a 250 ml beaker. 100ml of distilled water was measured and poured into the beaker to form a mixture. The mixture was heated in a water bath and a thermometer was used to check its temperature. The viscosity of the heated starch mixture was read on viscometer (Brookfield Viscometer) using spindle two (2), at temperatures of 50^0C, 60^0C, 70^0C and 90^0C at 50 and 100 rpm. After taking the viscosity reading at 90^0C, the mixture was cooled down to 50^0C in a water bath and the viscosity was taken at 50 and 100 rpm respectively.

Determination of starch swelling capacity

The method described by Bowen and Vadino[18] was used. Water yam starch (10g) was weighed into a 100ml measuring cylinder and the bulk volume measured (V_1). Deionized water (90ml) was added and the dispersion was well shaken for 5minutes. Water was added to make up 100 ml and the dispersion mixture was allowed to stand for 24hours before the sedimentation volume was read (V_2). The swelling capacity was calculated as follows:

$$\text{Swelling Capacity} = \frac{V_2}{V_1} \quad \ldots\ldots\ldots\ldots\ldots\ldots (6)$$

Determinations were done in triplicate

Preparation of starch tablets

A 500g of the water yam starch powder were compressed on a Carver hydraulic press (Fred Carver tableting machine, Model C, USA) at pressures of 0.25, 0.50, 0.75 and 1.0 N/m^2. The 10.55mm die faced punches were lubricated with 1% w/v dispersion of magnesium stearate in acetone.

Tablets were perforated at the middle using a die of 10.55mm with a pin in the middle to obtain tablets with holes.[13,19]

Friability test on tablets

Five tablets (with holes and without holes) were taken at different pressures of 0.25, 0.50, 0.75 and 1.0 N/m^2.The weights of the tablets were recorded and the tablets inserted into a friabilator (DBN Friability Tester, England) and made to rotate at 100rpm. At this velocity, the friabilator was switched off and the weights of the tablets were taken again. The weight difference was then deduced.[20]

Determination of tablets Hardness, Tensile strength and Brittle Fracture (BFI)

The hardness of each tablet was determined on a Pfizer hardness tester, England. Each tablet (with and without hole) was placed in between the anvil of the hardness tester diagonally and the knob at the base of the tester was rotated in order to compress the tablets.[21] The compression led to the splitting of the tablets into 2 halves. The crushing strength of each tablet was read on the tester and recorded. The tensile strength of the tablet was calculated using the equation 7.[22]

$$T \text{ or } T_o = \frac{2F}{\pi dt} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (7)$$

Where: T = tensile strength of tablets without hole

T_o = apparent tensile strength of tablets with holes

d = tablet diameter (mm)

t = tablet thickness (mm)

The BFI (Brittle Fraction Index) of the tablets were calculated using equation 8.

$$BFI = 0.5\ \{(\frac{T}{To})\text{-}1\} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (8)$$

Determination of the relative densities of the tablets

The weight and dimension of the tablets were accurately determined using an electronic balance and a micrometer screw gauge (for tablet thickness determination) respectively.

The relative densities of the tablets "D" were calculated using equation 9

$$D = \frac{w}{\pi r^2 h\ \rho s} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (9)$$

Where: r = radius of the tablet (cm)

h = thickness of the tablet (cm)

w = weight of the tablet (g)

ρ_s= particles density(g/cm^3)

Compressional characteristics were analyzed using Heckel and Kawakita equations.[23,24]

Heckel plot

Heckel plot of $\text{In } [\frac{1}{(1-D)}]$ against the applied pressure (P) was constructed for the different tablet formulations. The Heckel equation is as follows [23]

$$\text{In } [\frac{1}{(1-D)}] = KP + A \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots [10]$$

K is the slope, A is the intercept

The reciprocal of the slope, K is denoted by P_y, which is the mean yield pressure). The value of the D_a can be calculated from the value obtained from the intercept using equation 11:

$$D_a = 1\text{-}e^{-A} \quad \ldots\ldots (11)$$

The relative density of the powder at the point when the applied pressure is zero, D_o is the ratio of bulk density to the particle density.

D_b is the difference between D_a and D_o

$$D_b = D_a - D_o \quad \ldots\ldots [12]$$

Kawakita plot

This was determined based on the relationship between the degree of volume reduction of tablet, C, and applied pressure, P (N/m^2) as shown in the equation 13 [24]

$$\frac{P}{C} = \frac{1}{a}(P) + \frac{1}{ab} \quad \ldots\ldots (13)$$

RESULTS AND DISCUSSION

Table 1 shows the microscopic and physicochemical properties of the starch. The shape of the starch particle as viewed under the microscope was spherical in shape. The Hausner's ratio, which is determined as the ratio of the initial bulk volume to the tapped volume, provides an indication of densification. The water yam starch has high value of the Hausner's ratio, indicating high densification and poor flowability of the starch.

Table 1: Microscopic and physicochemical propertiesof water yam

Particle density ρ_s (gcm^{-3})	Bulk density, ρ_o (gcm^{-3})	Tap density ρ_t (g/cm^{-3})	Hausner's ratio	Carr index %	Swelling capacity (%)	Moisture content (%)	Water retention capacity (%)	Mean particle diameter (mm)
1.67	0.53	0.71	0.75	25.35	1.06	5.554	1.70	0.30

Water retention capacity is the measure of the extent to which the starch will retain water. The value of water retention capacity reported for cocoyam starch was lower than that of native corn starch (3.03).[25] Some starches with high retention capacities when added to formulations may imbibe a disproportionate amount of water and dehydrate other components and verse versa. The presence of polar head groups in the starch is another important measure of water retention ability of starch. The amount of bound water determined in this way was both absorbed an adsorbed on the surface by the starch

granules.[26] Denaturation and pre-gelatinization during heat treatment of starch disrupt starch granules thereby releasing amylopectin which may be responsible for swelling of the starch. The starch particle density of water yam was higher than those values reported for Durum wheat starch[27] and bulma cotton starch (1.475± 0.100 g/cm^3) reported by Ogungbenle.[26] The particle or granule density influences the rate and external packing experienced by a material during various units of operation.[13]

Table 2 shows the results of the viscosity of the starch at both 50 and 100 rpm and at different elevated temperatures. The viscosity of the water yam starch was increased as the temperature increased. The values of the viscosity on cooling at 50^{0}C were 2.0 and 7.0 BU at 50 and 100rpm respectively. Viscosity is related to the compactibility of the starch granule structure of the starch. These values were lower than that at increased temperature of 90^{0}C (18.0 and 25.0BU).

Table 2: Viscosity properties of the water yam starch

Temperature (^{0}C)	Rpm	Viscosity (BU)	Torque
50	50 100	2.0 7.0	0.3 0.7
60	50 100	6.0 7.0	0.3 0.7
70	50 100	6.0 8.0	0.6 0.8
80	50 100	10.8 15.0	1.0 1.5
90	50 100	18.0 25.0	0.9 2.5

Figure 1 shows the Heckel plot for the starch. The values of the mean yield pressure P_y (inverse of the slope k) were calculated from the region of the plot showing the highest correlation coefficient of 0.9818. The intercept A was determined from the extrapolation of the region used for P_y. The values of D_a and D_b were calculated from equations 12 and 13 respectively. D_o is the ratio of the bulk density to the particle density. The value of D_a, D_b, D_o and P_y are presented in Table 3. D_b represents the densification of the starch at low pressure, D_a represents the total degree of packing achieved at zero and low pressure.

The mean yield pressure P_y, is inversely related to the ability of a material to deform plastically under the applied pressure.

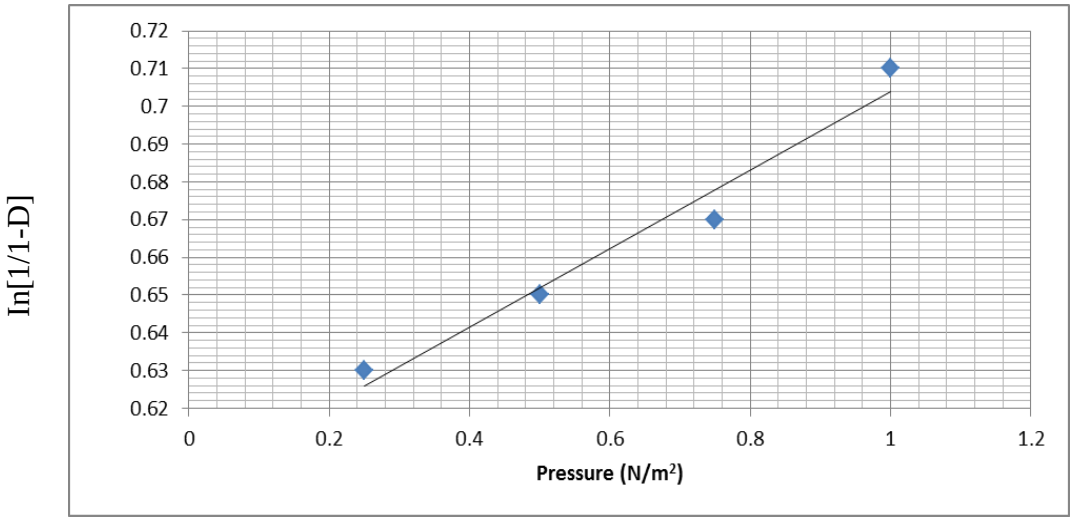

Fig. 1: Heckel plot of the water yam starch

Table 3: Parameters obtained from the Heckel plot

D_o	P_y	D_a	D_b
0.317	6.94	0.59	0.28

Figure 2 shows the Kawakita plot of the water yam starch. A linear relationship was obtained by the plot of the ratio of the applied pressure to the degree of volume reduction against the applied pressure with a coefficient correlation of one for the starch.

The values of "a" and "b" were obtained from the slope and intercept of the plot respectively. Values of 1 – a; provide the initial relative density of the starch. D_i , D_b and P_k are shown in Table 4. D_i provides a measure of the packed initial relative density of the material.

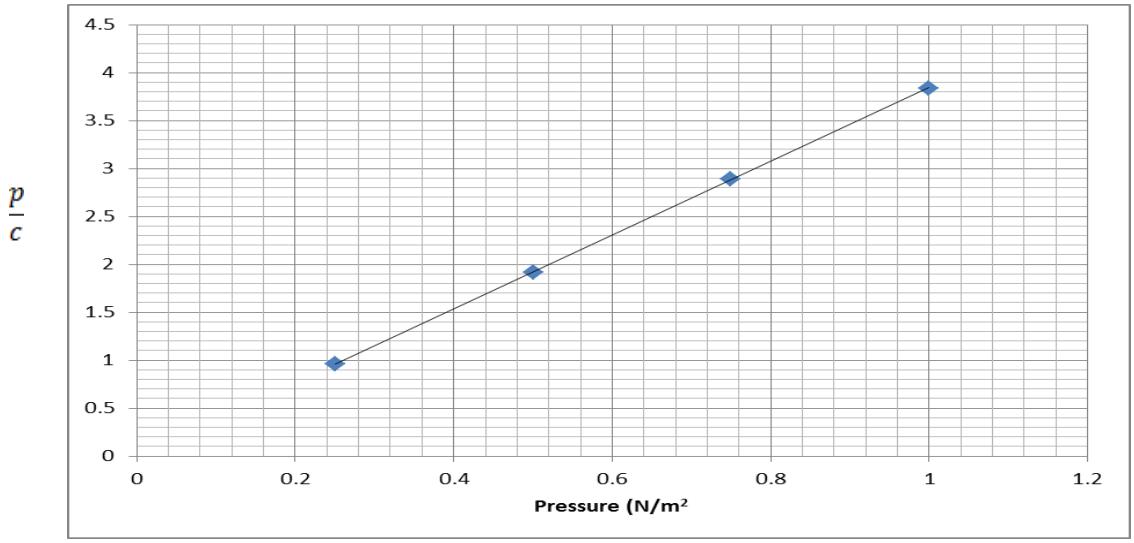

Fig. 2: Kawakita plot of the water yam starch.

Table 4: Parameters obtained from the kawakita plot

D_i [1 – a]	P_k	b	a
0.741	0.00129	772.20	0.259

The value of P_k obtained for water yam starch was lower than those of native and pre-gelatinized plantain starch (2.346 and 3.095)[25] while that of Di was higher than those of native and pre-gelatinized plantain starch (0.366 and 0.398).[25]

Tensile strength and brittle fracture index are two important parameters which have been used as the measure of the bond strength and lamination tendency of the tablets respectively. Table 5 shows the tensile strength and lamination tendency of the tablets without hole at the middle and also the brittle fracture indices of the tablets at different applied pressures. The tensile strength and brittle fracture index of the tablet were calculated using equations 7 and 8 respectively. The BFI is a measure of localized stress relief within the tablet by plastic deformation, a low value of the BFI indicates the ability of the material to laminate or cap. The BFI values of the tablets at different pressures had values that were approaching unity especially with tablets made at 0.25Nm^{-2} pressure.

It was observed that as the pressure decreased, the tensile strength of the tablets with and without holes also decreased. The tensile strengths of the tablets without holes at the centre were greater than that those with holes at the centre.

Table 5: Tensile strengths and the brittle fracture indices (BFI) of the tablets

Pressure (Nm^{-2})	T (Nm^{-2})	To (Nm^{-2})	BFI
1.0	5.48×10^5	3.44×10^5	0.297
0.75	5.27×10^5	2.68×10^5	0.483
0.50	4.87×10^5	2.35×10^5	0.536
0.25	4.50×10^5	2.07×10^5	0.587

Tables 6 and 7 showed the results of the friability test for tablets with holes at the middle and those without holes at different applied pressures. The weight difference of the tablets with holes at the middle was greater than those without holes. This indicated that the tablets with holes in the middle were partially friable while those without holes were not friable. Friability is the measurement of the tablet resistance to shock, friction and abrasion.

Table 6: Friability test on tablets with holes

Pressure (Nm^{-2})	Initial weight (g)	Final weight (g)	Weight differencse (g)
1.0	2.41	2.29	0.12
0.75	2.42	2.28	0.14
0.50	2.45	2.37	0.08
0.25	2.50	2.39	0.11

Table 7: Friability test on tablets without holes

Pressure (Nm^{-2})	Initial weight (g)	Final weight (g)	Weight difference (g)
1.0	2.47	2.37	0.10
0.75	2.48	2.39	0.09
0.50	2.46	2.39	0.07
0.25	2.48	2.36	

CONCLUSION

The results obtained from the studies would provide information on physicochemical and mechanical properties of water yam starch. The results showed that water yam starch had high tensile strength, brittle fracture index and densification values. The friability test results showed that water yam starch had higher plastic deformation while tablets without holes in the middle had better and higher external stress, shock and friction than those with holes. Therefore, water yam starch could be recommended as binder, disintegrant and filler in food and drug formulations.

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