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Original Article

Evaluation of Compressional and Mechanical properties of Cocoyam (*Colocasia esculenta*) starch flour and its Tablets

Oluwagbenle Henry Niyi, Akinwumi Olubunmi Adenike, Ayodele Olajide and Kolade KJ

Department of Chemistry, Faculty of Science, Ekiti State University, Ado-Ekiti, Nigeria

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ABSTRACT

The compressional and physicochemical parameters of cocoyam starch and the mechanical properties of its tablets were evaluated. The compressional characteristics were analyzed using density measurement, Heckel and Kawakita plots. The cocoyam starch had a high value of Hausner's ratio, indicated high densification and poor flowability of the starch. The viscosity profile of the cocoyam starch showed that the viscosity increased as the temperature was increased. The tablets without hole at the centre had higher tensile strength (T) than those tablets with hole at the centre. The brittle fracture index (BFI) of the tablets at various applied pressures had values very close to unity, particularly tablets with hole at $0.25Nm^{-2}$ applied pressure. The friability test indicated that tablets without hole had better resistance to stress, friction and abrasion than tablets with hole at the centre. The results suggested that cocoyam starch could be useful as a binder or disintegrant in tablets formulation due to good compressional and mechanical properties of its tablets.

Keywords: Evaluation, Compressional, Mechanical, Cocoyam, Starch and Tablets

INTRODUCTION

Starch is a digestible polysaccharide that has more nutritional and industrial uses than monosaccharide and disaccharide moieties [1]. The modern food processing industries are increasingly dependent on the use of both native and modified starches (and gum as well) for the manufacture of various fabricated foods [2]. As a result, there is an increase demand and pressure to search for new resources of starch from the available underutilized farm produces, for use in food, drug and allied industries. Modified and native starches have limited usefulness in food processing, since they are sensitive to heat, pH and shear stress.

Cocoyam is a member of the Araceae family and widely cultivated in the tropical and subtropical parts of the world. It produces edible starch tubers and vegetables, which are highly appreciated and grown mostly by the local farmers [3]. The common species of cocoyam are the *Colocasia esculenta* and *Xanthosoma sagittifolium* species both belonging to the Araceae family and constitute one of the six most important roots and tuber crops in the world [4].

Address for Correspondence: Prof. Oluwagbenle H. N., Department of Chemistry, Faculty of Science, Ekiti State University, Ado-Ekiti, Nigeria; E-mail: httphenryo@yahoo.com

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Cocoyam is monocotyledonous plant of one meter height or more. The above-ground stem consists of heart-shaped leaves supported by solid and erect petioles. The underground stem or corm is a compact structure embedded with nutrients and largely starch [5]. Cocoyam is propagated in a vegetative manner using corms, cormels or suckers. The size of materials is critical to obtain adequate yields. When planting material of more than 150g is used for planting, it may result in increased growth and yields [3]. Therefore, this study was undertaken with the following specific objectives: to evaluate the compressional characteristics and the mechanical properties of starch and tablet forms of cocoyam for pharmaceutical industry utilization.

MATERIALS AND METHODS

Cocoyam tubers (*Colocasia esculenta*) were obtained from a farm at Iworoko – Ekiti in Ekiti State, South - west of Nigeria.

Isolation of the starch: Cocoyam starch was isolated by the method of Moorthy and Nair [6] with slight modifications. Freshly harvested cocoyam tubers (200g) were peeled and blended using a Kenwood blender. The blended mixture was filtered through a triple layer cheese cloth and starch washed thoroughly using distilled water. The washed starch mixture was subjected to further filtration using polypropylene in which pure starch granules and water were washed as the filtrate. The filtrate was allowed to settle for 24hr, decanted, followed by centrifugation at 5000rpm for 15min in a centrifuge machine. The supernatant was discarded and protein layer scrapped off using a spatula. Potassium hydroxide (0.5M) was added to the residue in a test tube and further centrifuged for 5min to remove impurities. The supernatant was discarded and the residue was left to dry at room temperature in a crucible [7, 8]. The dried starch granules were milled, sieved, weighed and stored in a refrigerator until analyses.

Determination of moisture content: A 9g of cocoyam starch powder was weighed on a digital weighing balance and then mixed with 1cm^3 of water on a porcelain slab. The wet starch powder was placed on the pre-weighed porcelain slab and then transferred into the oven set at 60^{0}C. The starch sample was removed from the oven and weighed at 2, 5, 8, 15, 24, 30, 35, 40 and 60 min time intervals until a constant weight was obtained. Determinations were done in triplicate.

Determination of particle size of the starch: Particle size of the cocoyam starch was determined under an optical microscope. Ten grams of the starch was dispersed in deionized water. The particles were then viewed under a microscope at a magnification of 10 and then the main projected particle diameter was deduced [9]. Determinations were done in triplicate.

Determination of particle density: The particle density of the starch powder was determined by the pycnometer method using benzene as the displacement fluid. An empty 50cm^3 pycnometer bottle was weighed (W) and filled with the benzene and the excess wiped off. The weight of the pycnometer bottle together with the benzene was determined (W_1). The difference between the two weights (W_1-W) was calculated as W_2. A 2g quantity of cocoyam starch powder was weighed (W_3) and further transferred into the pycnometer bottle. The excess benzene was wiped off and then the pycnometer bottle was re-weighed again (W_4) [10].

All determinations were done in triplicate.

Determination of bulk density: The bulk density of the cocoyam starch at zero pressure (loose density) was determined by adding 20g of the starch particles at an angle of 45^0 through a funnel into 50cm^3 glass cylinder of 20mm diameter [11]. 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 graduated cylinder at a standardized rate of 30 taps per minute [12].

Flowability of starch: The flowability of the cocoyam starch was determined using the Hauser's ratio and the Carr index. The Hauser's ratio was determined as the ratio of initial bulk volume to the tapped volume [13].

Determination of starch swelling capacity: The procedure described by Bowen and Vadino [14] was used. Starch powder (10g) was poured into a 100ml measuring cylinder and the bulk volume measured (V_1). Deionized water (90cm^3) was then added and the mixture was well shaken for 5 min and then made up to 100cm^3. The dispersion mixture was allowed to stand for 24hr before the sedimentation volume was read (V_2). Determinations were done in triplicate.

Determination of starch water retention capacity: This was determined using the method of Ring [15]. To 10g of cocoyam starch in a 100cm^3 measuring cylinder was added 90cm^3 of deionized water and the dispersion was well shaken for 5 min and then made up to 100cm^3. 15cm^3 of the dispersion mixture was centrifuged for 25min at 5000rpm and the supernatant was discarded and the residue weighed (W_1). The residue was then dried at 70^{0}C to a constant weight (W_2). Determinations were done in triplicate.

Determination of starch viscosity: Four grams of cocoyam starch was weighed into a beaker containing 100cm^3 of distilled water to form a mixture. The mixture was heated in a water bath and a thermometer was used to monitor the mixture temperature. The viscosity of the heated starch mixture was read on a viscometer (Brookfield Viscometer) with two spindles at temperatures of 50^{0}C, 60^{0}C, 70^{0}C and 90^{0}C at 50 and 100rpm. Then the viscosity was read at 90^{0}C. Thereafter, the starch mixture was cooled to 50^{0}C in a water bath and the viscosity was taken at 50 and 100rpm respectively.

Preparation of the tablets

A 500mg of the cocoyam starch powder was compressed on a Carver hydraulic press (Fred Carver tableting machine, model C, USA) at the applied pressures of 0.25, 0.50, 0.75 and 1.0Nm^{-2} for 30 sec. Then 10.55mm die and faced punches were lubricated with a 1% (w/v) dispersion of magnesium stearate in acetone.

Tablets with holes were made using a die of 10.55mm with a pin at the middle [11, 16].

Friability tests on tablets: Five tablets were taken from tablets made (with and without holes) at different pressures of 0.25, 0.50, 0.75 and 1.0Nm^{-2}, and weighed. The weights of five tablets were recorded. The tablets were put in the friabilator (DBN friability Tester, England) and then calibrated at 100rpm; the friabilator was switched off and the weights of the tablets were taken again. The weight differences were then deduced [17].

Determination of tablet hardness: The tablet hardness was determined on a Pfizer hardness tester, England. Each of the tablets with holes and without holes 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 and the compression led to splitting of the tablets into two equal halves. The crushing strength of the tablet was read on the hardness tester and recorded.

Determination of relative density of 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.

DISCUSSIONS

Table 1 shows some of the microscopic and physicochemical properties of the cocoyam starch. The Hausner's ratio provides an indication of densification. The cocoyam starch had high value of the Hausner's ratio, indicated high densification and poor flowability of the starch.

The shape of the starch particles as viewed under the microscope was oval / spherical. Table 2 shows the results for the viscosity of the starch at both 50 and 100rpm, at various elevated temperatures. It was observed generally, that viscosity increased as the temperature was increased.

The ability to imbibe and absorb water is desirable during production of baked foods and tableting [21]. The adjustment of water ratio in starch may be necessary to obtain the required viscosity. Water retention may depend on the nature and type of starch granules.

The temperature at which starch granules begin to swell rapidly is called gelatinization temperature [22]. The gelatinization temperature range is distinctive for various types of starches. At these temperatures, some of the smaller molecules of amylose become disentangled and are leached into the surrounding water. When heated above 100ºC, amylose molecules will diffuse out of the granules and pass into solution and the viscosity is increased i.e the starch solution thickens forming a sol.

The value of the particle density of cocoyam starch was higher than those of crim wheat (1.475g/cm^3) reported by Medcalf and Gilles [23] and gourd starch (1.450 - 0.300g/cm^3), white melon (1.350 - 0.200g/cm^3) reported by Ogungbenle [1]. This implied that cocoyam starch was denser than crim wheat starch, gourd starch and white melon starch. The ranking is therefore; cocoyam starch > gourd starch > white melon starch. This explains the usefulness of cocoyam starch in tableting in the pharmaceutical industry.

Figure 1 shows the Heckel plot for the starch, the value of the mean yield pressure P_y (inverse of the slope K) was calculated from the region of the plot showing the highest correlation coefficient of 0.9657. The intercept "A" was determined from the extrapolation of the region used for the calculation of P_y. The values of D_a and D_b were calculated from equations 11 and 12 respectively. The value of D_0 was calculated as the ratio of the bulk density to the particle density. The values of D_a, D_b, D_o and P_y are presented in Table 3.

D_b is the densification of the starch at low pressure, while D_a is 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 pressure.

Table 1 Physicochemical properties of the cocoyam starch.

Particle density (ρ_s) g/cm^3	Bulk density (ρ_0) g/cm^3	Tap density (ρ_t) g/cm^3	Hausner's ratio	Carr index (%)	Swelling capacity (%)	Moisture content (%)	Water retention capacity (%)	Mean particle diameter (đ) (mm)
2.176	0.39	0.57	1.47	31.58	1.06	6.59	1.99	0.14

Table 2**:** Viscosity studies of the cocoyam starch

Temperature (^{0}C)	rpm	Viscosity (BU)	Torque
50	50	2.00	0.10
	100	6.00	0.60
60	50	2.00	0.20
	100	6.00	0.60
70	50	6.00	0.60
	100	17.00	1.70
80	50	98.00	4.90
	100	95.00	9.50
90	50	238.00	11.90
	100	207.00	20.70

Fig. 1: Heckel plot

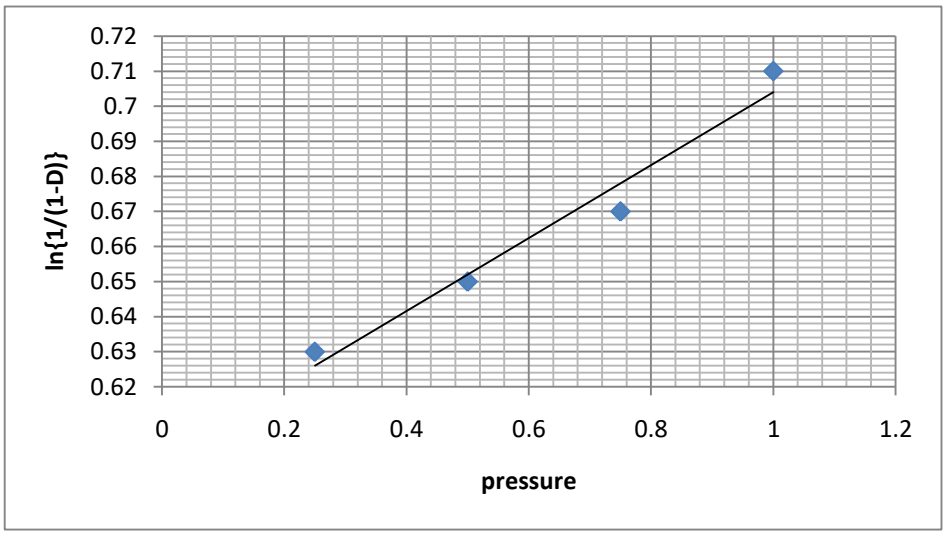

Table 3: Parameters obtained from the Heckel and Kawakita plots of cocoyam starch

	Heckel plot				Kawakita plot			
Cocoyam Starch	D_o	P_y	D_a	D_b	D_i = (1-a)	P_k	b	a
	0.179	9.615	0.451	0.272	0.678	0.00161	621.12	0.322

Fig. 2: Kawakita plot

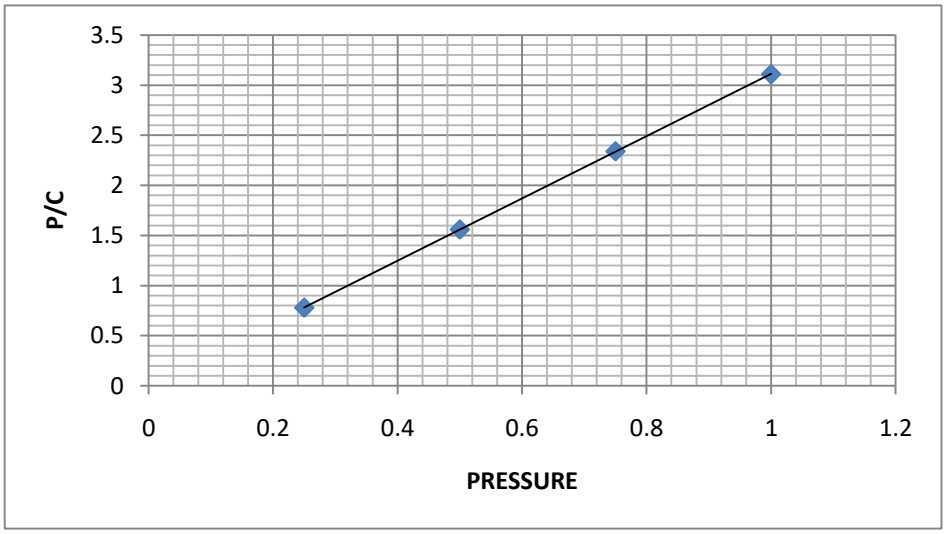

Figure 2 shows the kawakita plot of the cocoyam starch. A linear relationship was obtained from the compression applied.

The values of "a" and "b" were obtained from the slope and intercept of the plot respectively. Values of (1-a), gave the initial relative density of the starch "D_i" while P_k value was obtained from the reciprocal of the value of "b". The values of Di and P_k are shown in Table 3. D_i provides a measure of the packed initial relative density of the material. The Di value of cocoyam was higher than Di of corn starch (native and pre-gelatinized) (0.360 and 0.414) respectively while that of P_k for cocoyam starch was lower than those of native and pre-gelatinized corn starches (2.234 and 2.980) reported by Alebiowu and Itiola [8].

Tensile strength and brittle fracture index (BFI) are two important parameters which have been used as a measure of the bond strength and lamination tendency of the tablets respectively. Table 4 shows the tensile strength of the tablets without hole at the centre, apparent tensile strength of the tablets with hole at the centre and also the brittle fracture index of the tablets at different applied pressures. The tensile strength and BFI of the tablets were calculated using equations 9 and 10 respectively.

It was deduced that as the pressure was decreased, the tensile strength of the tablets with and without hole also decreased. The tensile strength of tablets without hole was greater than those that had hole at the centre. This indicated that the particles of the tablets without hole were rigid, compactible and highly compressed and hence, may not easily chip off under increased stress.

The brittle fracture index (BFI) is defined as the measure of localized stress relief within the tablet by plastic deformation, a low value of the BFI indicates the ability of the material to relieve localized stress, while a value approaching unity indicates the tendency of the material to laminate or cap. The BFI of the tablets at different pressures had values that were approaching unity particularly with tablets at 0.25 Nm^{-2} applied pressures.

Table 4: Tensile strength of the tablets and BFI values

Applied Pressure (N/m^2)	T (N/m^2)	T_0 (N/m^2)	BFI
1.00	7.73×10^5	3.58×10^5	0.579
0.75	5.32×10^5	3.03×10^5	0.378
0.50	4.98×10^5	2.03×10^5	0.727
0.25	4.4×10^5	1.57×10^5	0.914

Table 5: Friability test on tablets without hole

Pressure (Nm^{-2})	Initial weight(g)	Final weight(g)	Weight difference(g)
0.25	2.46	2.38	0.08
0.50	2.45	2.37	0.08
0.75	2.45	2.36	0.09
1.00	2.50	2.36	0.14

Table 6: Friability test on tablets with hole

Pressure (NM^{-2})	Initial weight(g)	Final weight(g)	Weight difference(g)
0.25	2.31	2.27	0.04
0.50	2.35	2.23	0.12
0.75	2.32	2.24	0.05
1.00	2.30	2.17	0.13

The relative density of the tablets was observed to have values that decreased with decreased in compression pressures. Tablets compressed at $1.0Nm^{-2}$ had the highest values of the relative density while tablets compressed at $0.25Nm^{-2}$ pressure had the least relative density. It was also observed that tablets compressed at $0.25Nm^{-2}$ had the highest values of tablets thickness while tablets compressed at $1.0Nm^{-2}$ pressure also had the least thickness values.

In terms of tablets' weights, tablets compressed at $1.0Nm^{-2}$ had the highest weights, while tablets compressed at $0.25Nm^{-2}$ had the least weights.

Tables 5 and 6 show the results of the friability test on tablets with hole at the centre and those without hole at the centre at different pressures. The differences in weights of tablets with hole at the centre were greater than those without hole. This indicated that the tablets with hole at the centre were partially friable, while those without hole at the centre were not friable and fragile. Friability is the measure of the tablet resistance to shock, friction and abrasion. The weight differences were less than 0.5 for both tablets with and without holes.

The results obtained from the hardness test indicated that tablets compressed at $1.0Nm^{-2}$ pressure had the highest crushing strength while tablets compressed at $0.25Nm^{-2}$ pressure had the least crushing strength. The crushing strength is measured in Newton (N). It is worth noting that tablets without hole at the centre had crushing strength that was higher than tablets with hole at the centre. This implied that tablets without hole were harder and tougher than tablets with hole.

The results obtained from the weight difference during the friability test on tablets are shown in Tables 5 and 6. The values were less than 0.5 for both tablets with and without hole. This indicated that both forms of tablets had good resistance to external stress, friction, abrasion and pressing.

CONCLUSIONS

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It can be concluded that the cocoyam starch can be used as an alternative source of binder and excipient in tablet formulations in pharmaceutical industry.

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