Research Article

COMPRESSIONAL CHARACTERISTICS OF LIMA BEAN STARCH AND THE MECHANICAL PROPERTIES OF ITS TABLETS

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ABSTRACT

Compressional and mechanical properties of lima bean starch were carried out using Heckel plot for compressional test while tensile strength was used for the mechanical properties. From kawakita plot, the value of P_k explains the plastic deformation during compression. From the Heckel plot, the higher the total relative compression density (Da), the denser is the powder in the compression phase especially during die filling.

Keywords: Compressional characteristics, Mechanical, Lima Bean, Starch.

INTRODUCTION

Lima bean (*Phaseolus lunatus* Linn) is economically and nutritionally important pulse crop but is widely cultivated like other bean species. The crop is commonly consumed among farmers in Nigeria and some other developing countries[1]. Lima bean is richer in dry matter, protein and ash than other cowpeas[2,3]. Lima bean is one of the known leguminous plants, which plays an important role in the acceptability of monotonous diets in many parts of the world[4]. The consumption of legumes range from insignificant amounts in Europe to intakes of 400 to 500g daily in some parts of Asia and Africa[5], as the major choice of their protein.

Starch is the principal dietary carbohydrate of majority of legume and cereal-based foods[6]. The modern food processing industries are increasingly dependent on the use of both native and modified starches for the manufacture of various formulated products[7,8].

The present study was undertaken to determine the physico-chemical parameters of lima bean starch and tablet formulated from the starch. The data generated were compared with the results obtained from corn starch (BP) and some other published starch samples.

MATERIALS AND METHODS

Lima bean (*Phaseolus lunatus Linn*) was brought from Oba market in Imesi-Ile, Osun state. The sample was screened and the bad seeds were eliminated and the remaining good ones were grounded into flour.

Isolation of Starch from the Sample

Five kilogram of the lima bean was soaked in distilled water at pH of 8.0 using solution of sodium hydroxide at 4°C for 12 hours. The seeds were dehulled and blended for 30 minutes. The slurry obtained after blending was later suspended in 25 litres of distilled water at pH of 8.0 using 0.5M sodium hydroxide solution. The mixture was stirred for 30 minutes and the pH maintained between 8.0 and 8.5. The suspension obtained was sieved using 75µm sieve and centrifuged for 30 minutes. The starch obtained was washed with water and air dried for 48 hours at room temperature.

Determination of the Physico chemical Properties of the Starch Sample:

Starch swelling capacity

Five grammes of lima starch was poured into a 100ml measuring cylinder and 90ml of deionized water was added and shaken vigorously for 5 minutes. Deionized water was later added to make up to the mark and the mixture was allowed to stand for 24 hours. The swelling capacity was calculated from the equation:

$$\text{Swelling capacity} = \frac{V_2 \text{ X } 100}{V_1} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

V_1 = Initial volume of the starch

V_2 = final volume of the starch

Water retention capacity

The method[9] was used with slight modification. Five grams of lima starch (w_1) was added to a 100ml measuring cylinder containing 90ml of distilled water and the mixture was shaken for 5 minutes and later made up to the mark using distilled water. A 15ml of the mixture was centrifuged for 25 minutes at 5000rpm. The supernatant was discarded and the residue was later oven dried at 70°C to a constant weight (w_2).

The water retention capacity was calculated from the equation

$$\frac{W_1}{W_2} \text{ x } 100\% \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Starch Compatibility

500g of starch powder was weighed and compressed directly into a tablet with thickness of 3.04 ± 0.01mm at zero porosity. Each tablet was compacted for 1 minute with 10 predetermined loads ranging from 0.8 – 2.4 metric tones.

The compression characteristics of the tablet (mega pascal) was carried out on a carrier hydraulic hand press using a 12.5mm die and flat-faced punches lubricated with a 2% w/v dispersion of magnesium stearate in alcohol before each compression process. While the mechanical characteristics of the tablet was determined using the method described[10].

Preparation of tablet from the starch

Tablet with a hole (1.54mm diameter) at the center was made using an upper punch with a hole through the center and a lower punch fitted with a pin. The tablet later ejected through the machine and stored over silica gel for 24 hours prior to determination of its physico-chemical properties[10].

Determination of the tensile strength of the tablet

The mechanical strength of tablet compressed was assessed by the tensile strength. The tensile strength of the normal tablet (T) and tensile strength of a tablet containing a hole (t) were determined at room temperature by diameter compression using an Erweka hardness tester and then applying the equation.[11]

$$T = \frac{2F}{\pi dt} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

d = tablet diameter (m); F = Load (mN) needed to cause fracture; tablet thickness.

Compression characteristics were analysed using the[12,13] techniques, the mechanical properties of the tablets were determined using their tensile strength and brittle fracture index (BFT)[11,14]. The Heckel equation was used to relate the relative density Da, of the tablet powder bed during compression to the applied pressure, P, using the equation.

$$\ln \{1/(1-Da)\} = KP + A \quad \ldots\ldots\ldots\ldots\ldots\ldots(4)$$

The slope of the straight line portion, K, is the reciprocal of the mean yield pressure, Py, of the material. The relative density, Da, can be calculated from the intercept A using the equation

$$Da = 1 - e^{-A} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

The relative density Da of the powder at the point when the applied pressure equals zero, Do, is used to describe the initial rearrangement phase of densification as a result of die filling. The relative density, Db, describes the phase of rearrangement at low pressures and is the difference between Da and Do:

$$DB = Da - Do \quad \text{--------------------------} \ (6)$$

The Kawakita equation was used to study the powdered tablet compression using the degree of volume reduction (c) and is written as:

$$C = (V_0 - V_p)/V_0 = abP/(1 + bp) \quad \ldots\ldots\ldots\ldots (7)$$

In practice, the above equation can be further rearranged to give

$$P/C = p/a + 1/ab \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots(8)$$

Where V_0 = initial bulk volume of the powder and V_p is the bulk after compression. The constant a is equal to the minimum porosity of the material before compression While constant b is related to the plasticity of the material. The reciprocal of b gives a pressure term Pk which provides an inverse measure of the deformability of the particles during compression[15,16].

This has been shown to be the pressure required to reduce the powder bed by 50%[17,18]. The index tensile strength and brittle fracture index are related by the equation below:

$$BFI = 0.5\,[(T/T_0) - 1] \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(9)$$

Where T = tensile strength of the tablet without a hole and T_0 = apparent tensile strength of the tablet when a hole is centrally created on the tablet at the same relative density. The hole acts as a built-in stress concentrator defect.

Determination of Starch Density

The particle density of each starch was determined using the pycnometer method with benzene as the displacement fluid[19]. The bulk density of each starch powder at zero pressure (loose density) was determined by pouring the powder at an angle of 45^{0}C through a funnel into a measuring glass cylinder with a diameter of 21mm and a volume of 50mL[20,11]. The relative density, D, of each starch powder was obtained from the ratio of its loose density to its particle density. The Hausners ratio was determined by the method[21]. Hausners ratio was determined as the ratio of the initial bulk volume to the tapped volume. It was obtained by applying 100 taps to 30g of each starch sample in a graduated cylinder at a standard rate of 38 taps per minute (British Standard, 1970).

RESULTS AND DISCUSSION

The results obtained showed that lima starch has relatively high moisture content (12.7%). This value is higher than those of sorghum (7.1%), Plantain (7.8%), and corn starch (6.8%) reported[22] and yellow melon (10.0%) reported[6] (Table 1). This result indicates that lima starch has fairly good storage potential. The result presented in Table 1 showed the value of water retention capacity of lima starch to be 2.60% while that of swelling capacity was 1.80%. These values for swelling and water retention capacities were lower than the native and pre-gelatinized starches of sorghum, plantain and corn starches reported[22]. Each starch swells differently showing variableness in arrangement of the granules within the starches. Lima starch undergoes a very slow and almost restricted swelling at relatively low temperature. This indicates strong bonding forces of approximately uniform strength[23]. The swelling rate pattern of lima starch is compared with those of modified and unmodified bulma and gourd starches reported[8] which proceeded at a much slower rate. Hence, it is therefore presumed that the associative forces with the lima starch granules represent a much wider range of bond strength. This also showed that the granules of lima starch are highly associated and relatively resistant to swelling (Table 1). The particle density of lima starch was found to be $1362 kg/m^3$ while that of bulk density was $550 kg/m^3$. The particle density of lima starch is lower than those reported for varieties of hard red spring wheat starches ($1473-1491 kg/m^3$)[24] but compared favourably with that of white melon ($1350 kg/m^3$) reported[25]. The particle or granular density influences the rate and extent of packing experienced by a material during various units of operations[11]. The results of the particle and bulk densities showed that lima starch is useful for tablet in the pharmaceutical industry since lima starch particles would occupy with space. This implies that the higher the particle size the higher the space it occupied and the lesser the bulk density.

Table 1: Physical Characteristics of Native Starch

Parameters	**Results**
Shape	Spherical/Angular
True density (g/cm^3)	1.362
Tapped density (g/cm^3)	0.71
Swelling capacity (%)	1.775
Water retention capacity (%)	2.634
Bulk density (g/cm^3)	0.55
Flow rate (g/sec)	3.03
Average particle size	7.0µm – 21.0µm
Moisture content (%)	12.7

The result for tensile strength is presented in Table 2. It is observed that lima starch has high tensile strength. It has been shown that smaller particles allow greater packing density after a particle rearrangement. It is worth nothing that tensile strength of the compact of starch increases with increasing compression pressure. The high tensile strength obtained in this work means that the starch would prevent capping, lamination and chipping of the compact even during transportation[14].

Table 2: Tensile strength of Native Starch

Pressure (Mpa)	**Relative density (gcm^{-3})**	**Tensile strength (MNm^{-2})**
79.96	0.819	0.139
111.94	0.869	0.169
143.93	0.876	0.204
159.92	0.967	0.246
175.91	0.986	0.279

The result of the brittle fracture index of lima starch is presented in Table 3. Brittle fracture index (BFI) is an index used to measure the ability of the material (tablet) to relief localized stresses. A low value of brittle fracture index is desirable for the minimization or avoidance of the problem of lamination and capping and other tablet problems during the production. The BFI value of lima bean is higher than those of corn, plantain and sorghum starches reported[22]. Since lima starch has very high BFI, it implies lesser ability to increase the lamination tendency in tablets. This is also in agreement with[14] which states that as the tensile strength value increases, lamination and capping tendency in tablets also increases.

Table 3: Brittle Fracture Index of Native Starch

Pressure (M pa)	**BFI = (T/To – 1) 0.5**
79.96	2.694
111.94	2.080
143.93	1.514
159.92	1.027
175.91	0.715

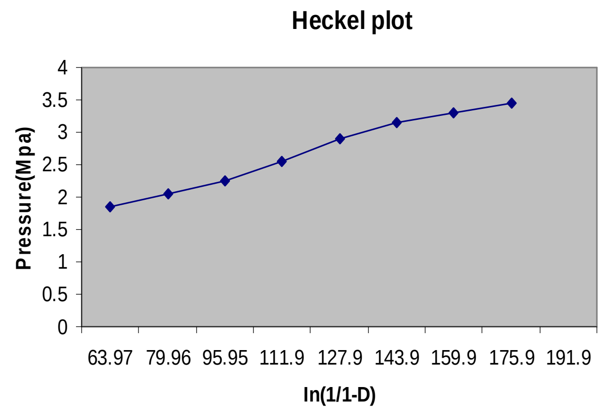

Figure 1: The Heckel Plot

Figure 1 shows Heckel plots for the lima starch. Heckel plot is the plot of In (1/1 – D) against applied pressure (equation 4). The values of mean yield pressure, Py, were calculated from the region of the plots showing the highest correlation coefficient for the linearity of >0.994 for the sample is generally between 62 - 195 Nm^{-2} The intercept, A, was determined from the extrapolation of the region used for the calculation of Py. The values of Da and Db were calculated from equations (5) and (6), respectively. The values of Py, Do, Da and Db for the sample are presented in Table 4. The different values and their interpretation are discussed in situ.

(a) Total relative compression density, Da represents the total degree of packing achieved for the starch formulated into tablet.

(b) During the initial die filing and at low pressure as a result of rearrangement process before in appreciable amount of inter-particulate binding takes place. Da value was obtained from the constant A, which is the intercept of the extrapolated linear portion of the Heckel plot[20]. The higher the Da value, the more dense is the powder in the compression phase especially during die filling and also in the early stages of downward movement of the upper punch. It can be shown from the result that lima starch has a higher Da value which implies that lima starch undergoes more rearrangement as the pressure was gradually increased.

(c) Relative density at zero pressure (Do) represents the initial packing in the die as a result of die filling. Lima starch has high Do value which is influenced by particle shape and size.

(d) Relative density at low pressure (Db) represents the phase of rearrangement of the particles in the early stage of compression, the extent depends on the theoretical point of densification at which deformation of particles begins. Db in obtained from the difference between Da and DO. The higher value of Db indicates more resistance to the movement of the particles while lower value of Da will show more fragmentation.

The mean yield, Py, is inversely related to the ability of a material to deform plastically under pressure. It is obtained as the reciprocal of the slope of the initial linear portion, b, of Heckel plot. A low value of Py indicates an intense plastic flow material while a high value of Py, indicates a material that undergoes plastic flow with difficulty. It can be observed that the Py value of lima starch is low and compared favorably with value obtained[22] for sorghum and corn starches. The low Py value obtained for lima starch, indicates that it would undergo deformation more readily. Plastic deformation of material during compaction increases the area available for inter-particulate bonding[26].

Figure 2 shows Kawakita plot for the lima starch. A linear relationship was obtained at all compression pressures employed. Kawakita plot is a plot of P/C against applied pressure N/m^2. (equation 7). The value of 1-a gives the initial relative density of the starch (D_i) while P_k value was obtained from the reciprocal of values of b. The values of a and b were obtained from the slope and intercept of the plot respectively. Di provides a measure of the packed initial relative density with the applied pressure. Lima starch has high Di value and thus implies high packed initial relative density. P_k is a pressure term and low value obtained for lima starch indicates softness and readiness for plastic deformation of lima starch under applied pressure. The plot is generally linear between 62 and 195 Nm^{-2}. Odeku and Itiola[27] showed that Py relates essentially to the onset of plastic deformation during compression and to the amount of plastic deformation occurring during the compression process.

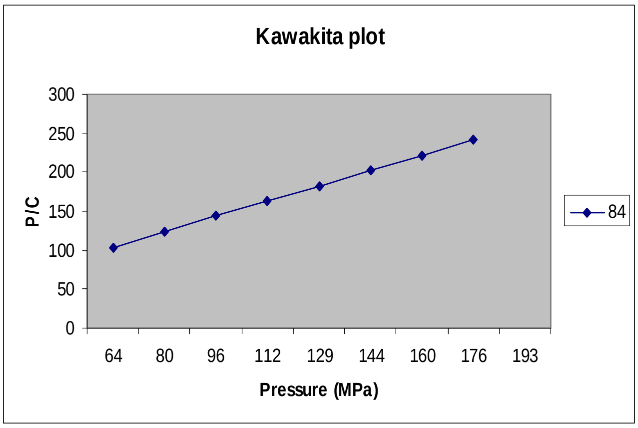

Figure 2: The Kawakita Plot

From the present results, it appears that lima starch facilitates enhanced onset of plastic deformation but reduces the total amount of plastic deformation which occurred during compression process. This may be due to higher value of tensile strength of the lima starch.

Table 4: Parameters Obtained from Density Measurements and from Heckel and Kawakita plots for the Native Starch

	Heckel Plot					**Kawakita Plot**	
Nature of Native Starch	**Py(Mpa)**	**A**	**Da**	**Do**	**Db**	**Di=(1-a)**	**Pk = 1/b**
	144.369	1.056	0.652	0.404	0.248	0.684	190.599

CONCLUSION

From the data obtained from the present study, it can be concluded that lima starch shows a favorable compression and mechanical properties. Also it has a relatively high tensile strength. This will prevent lamination and chipping of the compacts. Lima starch also has a better flow ability, packing fraction, BFI, low Py and low P_k yields which make it a good compressibility product. These properties of lima starch are compared favorably with other well-known starches. Hence, lima starch is recommended to serve as good binder in pharmaceutical and food packaging industries.

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