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Material, compressional and mechanical properties of *Borassus aethiopum* (African fan palm) starches

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Abstract
The compressional and mechanical properties of tablet formulations incorporating native and modified Borassus aethiopum starches as binder were evaluated. The native *Borassus aethiopum* starch (BAS) was modified to yield fully gelatinised starch (FGBAS) and microcrystalline starch (MBAS). The compressional properties of the starches were evaluated using geometric analysis, Heckel and Kawakita equations, while the mechanical properties were assessed through tensile strength and brittle fracture index determinations. The results show that modification had significant effect on both the compressional and mechanical properties of Borassus native starch ($p < 0.05$). The modified starches had a lower particle diameter and a corresponding increase in surface properties, and angle of repose. The ranking order for the angle of repose and surface factor is MBAS>FGBAS>BAS, while the ranking order for particle diameter is MBAS<FGBAS<BAS. The ranking order the mean values of the starches is BAS>MBAS>FGBAS, FGBAS with the least p_y values had the fastest on set of plastic deformation while MBAS with the least p_k and BFI values has highest overall plastic deformation, and least tendency to produce tablets with production defects such as capping and lamination. Modified Borassus starches show significant improved mechanical and compressional properties over the native Borassus starch.

Keywords: Borassus starches, tablets, compressional and mechanical properties

INTRODUCTION

Starch from various plants have long being used in pharmaceutical formulations most especially, tablet production, in which starches are uses as diluent/filler, disintegrant and binders, (Bos *et al.*, 1987). Researchers in the past had made significant efforts to investigate starches from various plant sources for pharmaceutical application, (Alebiowu and Osinoiki, 2010; Alebiowu & Itiola 2002; Alebiowu and Itiola 2003). The usefulness of starch in pharmaceutical formulations is attributed to its intrinsic properties such as abundance in nature, low cost, and totally biodegradable, (Alebiowu and Itiola 2003).

Even though starch is widely used in pharmaceutical industries, its usefulness is limited due to its lack of certain desired functional properties, most importantly the flow properties of starch and its low resistance to shear pressure. Modification of starches had being used to improve on its functionality as observed by various researchers, The Heckel equation relates the relative density (D) of a powder bed during compression to the applied pressure (P) and is written as, (Heckel 1964):

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

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A graph of ln{1/(1 – *D*)} plotted against P should be rectilinear with slope of *K* and intercept *A*. The reciprocal of the slope is related to the yield pressure (*Py*) of the material, and this equation has been used to distinguish materials which deformed by fragmentation from those which deform elastically on compression. From the value of *A*, the relative density (D_a) can be calculated using Humbert-Droz equation given below, (Humbert-Droz *et al.*, 1983):

$$D_a = 1 - e^{-A} \quad \ldots\ldots\ldots 2$$

The relative density of the powder at the point when the applied pressure equals zero (D_0) is used to describe the initial rearrangement phase of densification as a result of die filling. The relative density (D_b) describes the phase of rearrangement at low pressures and is given as:

$$D_b = D_a - D_0 \quad \ldots\ldots\ldots 3$$

The Kawakita equation is used to study powder compression using the degree of volume reduction(*C*), (Kawakita and Ludde 1970) and is expressed as:

$$C = (Vo - Vp) / Vo = a\,b\,P / (1 + b\,P) \quad \ldots\ldots 4$$

Where V_0 is the initial bulk volume for granular materials, *Vp* is the bulk volume after compression, *a* is a constant equal to the minimum porosity of the material before compression, *b* is also a constant that is termed the coefficient of compression and related to the plasticity of the material. The reciprocal of *b* is related to the pressure term (P_k), which is the pressure required to reduce the powder bed by 50 %, (Shivanand and Sprockel 1962; Lin and Cham 1995). In practice, the equation is rearranged to give:

$$P/C = P/a + 1/ab \quad \ldots\ldots\ldots 5$$

Heckel and the Kawakita plots have their limitations and are generally believed to exhibit linearity for many materials at high and low pressures, respectively (Celik, 1995). However, the combined use of these two plots should provide more accurate information about the compressional characteristics of powders.

EXPERIMENTAL

The materials used included corn starch B.P., xylene, hydrochloric acid (BDH Chemicals Ltd., Poole, U.K) and *Borassus aethiopum* starch (BAS) prepared in our laboratory.

Extraction of native *Borassus aethiopum* starch (BAS). The BAS was extracted from the young germinating shoots of *Borassus aethiopum* using established procedure (Buwalda, and Willemina, 1997). The extracted starch was thereafter dried at 50^0C in hot air oven (Gallenkamp, Model OV – 335, Vendon Scientific Ltd, Oldham, UK) for 72h. The dried mass was however powered using a laboratory mill (Binatone Model V26 Tokyo Japan) at a speed of 1,500 rpm. The product was collected and stored in an airtight glass container until the product is needed.

Production fully gelatinised *Borassus aethiopum* starch (FGBAS). Fully gelatinised Borassus starch was produced according to the method describe by Alebiowu and Itiola (2003).

Production of microcrystalline *Borassus aethiopum* starch (MBAS). Microcrystalline *Borassus aethiopum* starch was produced according to the method described by Buwalda and Willemina (Buwalda and Willemina 1997). A 36% w/v slurry of BAS was reacted with 28ml of 6N HCl which was added drop wise with continuous stirring of the slurry. A reaction time of 12 h was observed. The modified starch produced was recovered from the reacting medium by filtration. The product (MBAS) was re-repeatedly washed with deionized water to totally remove the acid. This was then dried in a Gallenkamp hot air oven(Philips Harris Ltd, England) at 60 ^{o}C until a constant weight was obtained. The dried MBAS was however milled and the fraction retained on 80µm sieve were collected and stored in a desiccator for further studies.

Determination of starch particle and shape. The particle size distribution of each starch

was determined by optical microscopy (Leitz, laborlux II, Germany) on 300 particles. This was used to determine the mean projected diameter (d) and the particle shape. The shape coefficient, α, for all the starches was calculated from the expression, (Odeku and Itiola 1998):

$$\alpha = \rho s\ Sw\ de + N \quad \text{……..6}$$

where N is the elongation ratio (= length / breadth). The Heywood diameter,
de, is obtained from the expression $[(4 \times 0.77 \times L \times B) / \pi]^{1/2}$μm. L and B
are the mean length and breadth of the particles respectively. The surface area of the starches, S_w (m^2/g), was calculated using the expression; (Stanley-wood, N. G. & Shubair, M. S. (1979) :

$$S_w = \alpha sv.t\ Sv / \rho s \quad \text{…….7}$$

where αsv.t is the surface-volume sieve shape factor, which was taken as 6, i.e. the shape factor for a sphere, Sv is the geometric surface area of the starches in m^{-2} obtained from sieve analysis and ρs is the particle density in gm^{-3}.

Determination of particle, bulk and tap densities. The particle density of the starch samples were determined by the pycnometer method, using Xylene as the displacement fluid. 10g of each starch were poured into a 50mL glass measuring
cylinder and its bulk volume determined. The powder was subjected to about 100 taps, (from a height of about 2.54 cm). Tapped density was determined using the ratio of the weight and the tapped volume.

Determination of Compressibility index (CI). The compressibility index for the various starch samples were calculated using the equation:

$$CI = \rho T - \rho B / \rho T \times 100\% \quad \text{…….8}$$

Where ρT and ρB are tapped density and bulk density respectively.

Angle of repose. The angle of repose which is the maximum angle that can be obtained between the self supporting cone of the powder mound and the horizontal plain was determined according to the relationship:

$$\text{Tan}\ \theta = h / r \quad \text{……..9}$$

About30g of starch powder was allowed to flow through a funnel with internal diameter of about 2.8 cm under the force of gravity to form a conical heap on a plain paper. The angle of repose was calculated as a mean of four determinations as:

Preparation of starch tablets. About 600mg of starch powder was compressed for 30 seconds into tablets with predetermined loads on a Carver hydraulic press (model C, Carver inc. Menomonee Falls, WI) using a 10.00mm die and flat faced punches lubricated with a 1% dispersion of magnesium stearate in acetone before each compression. After ejection, the tablets were stored over silica gel for 24hours to allow for elastic recovery and hardening. The weights (w) and dimensions of the tablets were determined within ± 1mg and 0.01mm, respectively. The relative density of the tablets (D) was calculated using the equation:

$$D = w / Vt.\ \rho s \quad \text{…… 10}$$

where Vt is the volume (cm^3) of the tablet.

Determination of tensile strength and brittle fracture index. Equal quantities of tablet with hole at the center and those without holes were produced by diametral compression (Fell and Newton 1970). The tensile strength of the normal tablets (T) and apparent tensile strength of the tablets containing a hole (To) were determined at room temperature using a hardness tester (AR-T M87, China) and by applying the equation:

$$T = 2F/\pi dt \quad \text{……11}$$

where T (or To) is the tensile strength of the tablet (MNm^{-2}), F is the load (MN) required to cause fracture, d is the tablet diameter (m) and t is the tablet thickness (m). All measurements were performed in triplicates. The Brittle Fracture Index (BFI) values of the tablets were calculated using the equation:

$$BFI = 0.5[(T/ To) - 1] \quad \text{……12}$$

RESULTS AND DISCUSSION

The microscopic and geometric properties of the starches are presented in table one. It was observed that modifications had significant effects on particle and surface properties of the starches ($p< 0.05$), while modification reduced the mean particle diameter but increased the surface area of all the starches. The Heywood diameter and the mean particle diameter of three starches follow the same pattern and are rank; MBAS<FGBAS<BAS. The MBAS with the least Heywood constant value and angular particle size is expected to have more particle per unit weight and this impart better homogeneity when mixing with active ingredient and other excipients in formulation, since unit operations such as milling, blending and uniform flow of powders as been showed to a large extend to depend on the particle shape and particle size distribution, (Itiola and Odeku 2005; Twitchell 2002).

Angle of repose θ of a material gives a good indication of the level of resistant of the material to flow, invariably material with $\theta < 25^o$ are consider to have excellent flow properties while material will $\theta > 40^o$ are consider as poor flowing substance. It was observed in this study that all the three starches have θ values ranging between 28.5 to 30.5^o indicating moderately flowing materials. The ranking order is MBAS>FGBAS>BAS. The angle of repose of a material is affected by the particle size distribution and it usual increase with decreased particle size, (Podczeck and Sharma 1996; Shotton and Obiorah 1973). The reduction in particle size as a result of modification may however be responsible for increased resistant to flow of the materials.

Table I: Physical and geometric properties of starches.

Starch	Geometric shape	Mean particle diameter	d_e	S_w	α
BAS	Oval/angular	9.4	1.88	0.0138	0.064
FGBAS	Oval/angular	7.4	1.89	0.0144	0.087
MBAS	Oval/angular	6.4	1.77	0.0155	0.102

Table II: Parameters obtained from densities measurements.

Starch	PD (g/cm^3)	BD (g/cm^3)	TD (g/cm^3)	Angle of repose (^{o})	HR
MBAS	1.5	0.64	0.96	30.45	1.5
FGBAS	1.41	0.6	0.85	29.41	1.42
BAS	1.39	0.58	0.79	28.48	1.36

Table III: Parameters obtained from Heckel and Kawakita plots

	HECKEL				KAWAKITA		
	D_O	D_A	D_B	P_Y	D_i	P_K	BFI
BAS	0.53	0.911	0.381	245.9	0.539	2.491	0.43
FGBAS	0.501	0.862	0.361	178.57	0.517	1.461	0.18
MBAS	0.302	0.801	0.499	208.01	0.312	1.131	0.11

Table IV: Tensile strength of starches.

Material	T	T_o	BFI
BAS	0.734	0.395	0.43
FGBAS	2.525	1.8574	0.18
MBAS	1.988	1.6295	0.11

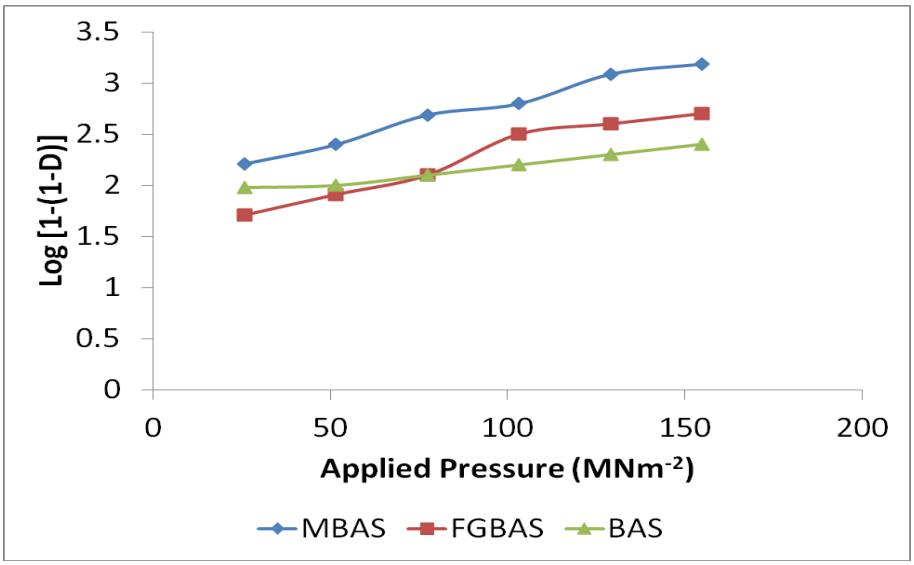

Figure I: Heckel Plot for the native and modified Borassus Starches

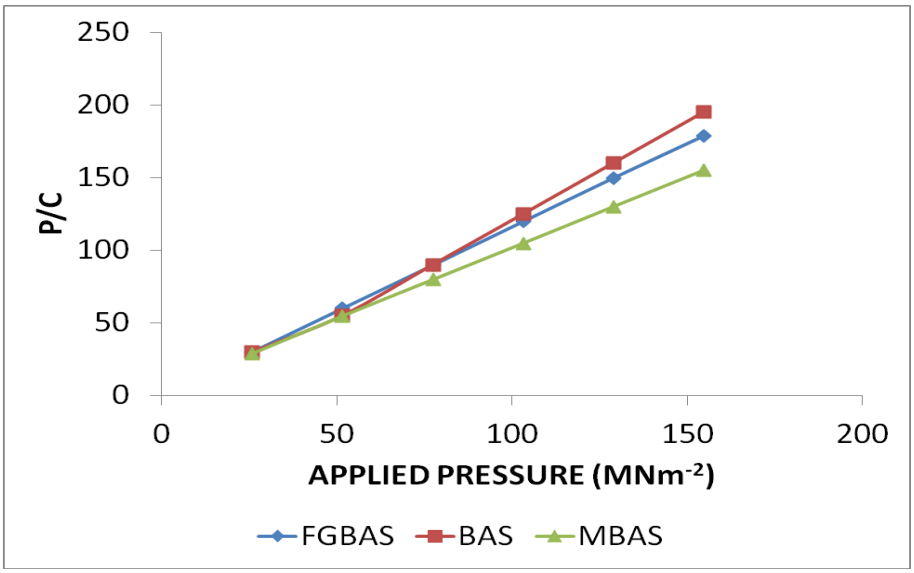

Figure II: Kawakita Plots for the native and modified Borassus Starches

The Heckel plots for the three Borassus starches are presented in Figure I. The mean yield pressure values (P_y) were calculated from region with the highest correlation co efficiency (>0.998). The ranking order for P_y values is BAS> MBAS > FGBAS. FGBAS has the fastest on set of plastic deformation since P_y is inversely proportional to the onset of plastic deformation of materials (Odeku *et al.*, 1998).

The values of D_o, D_A, and D_B for all the starches were as presented in Table II. D_o indicates the degree of initial packing and rearrangement of material at low pressure, and the ranking order is BAS> FGBAS> MBAS. MBAS has the least degree of rearrangement under gravitation forces as usually experience in die filling stage during tablet manufacturing. BAS has the highest value of D_o and correspondingly the highest particle size. Materials with high particle size have less electro static forces that could prevent the rearrangement and packing of

particle in the pre-compression stage.(Odeku *et al*., Odeku and Picker-Freyer 2007)

D_B values can be ranked as MBAS >BAS >FGBAS, these values represent the phase rearrangement and fragmentation of material in the early stage of compression. The total degree of packing achieved by materials are given by D_A values and the ranking order is BAS>FGBAS>MBAS. D_i values were obtained from Kawakita plots. It was however observed that D_i values were higher for all corresponding values of D_o. This is in agreement with earlier studies (Twitchell 2002).

The values of P_K for the three starches are as presented Table III. The ranking order is BAS>FGBAS>MBAS. The P_K is inversely related to the amount of total plastic deformation of a material. MBAS with the lowest P_K gives the highest total plastic deformation during compression. Plastic deformation is generally desirable in pharmaceutical tablet production

The BFI obtained for all the starch compacts is shown in Table III. Tablets with low values of BFI are most unlikely to experience production defects such as capping and lamination, since low value of BFI is corresponding to high mechanical strength. The ranking order for BFI is MBAS < FGBAS < BAS, therefore tablets produced with MBAS will show least tendencies of production defects, such as capping and lamination which is unconnected to the increase plastic deformation associated with increase contact points for inter-particulate bonding. (Alebiowu and Osinoiki 2010; Alebiowu and Itiola 2003; Humbert-Droz *et al.*, 1983).

Conclusion

Modification of *Borassus aethiopum* starch significantly affected the compressibility and mechanical properties of the starches. The acid modified starch showed the fastest on set of plastic deformation, the highest total plastic deformation and the least tendencies to produce tablets with production defects.

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