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RESEARCH ARTICLE

COMPRESSIONAL PROPERTY STUDY OF DIGITARIA IBURUA (POACEAE) STARCHES

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ABSTRACT

Objective: This research work was aimed at determining the compression behavior of Digitaria iburua starch (DS) and acid hydrolyzed starch (MCS) using gelatin (GEL) as a standard compressible excipient. **Methods:** DS was extracted from the grains and then acid hydrolyzed to produce MCS. The particle density of DS, MCS and GEL were determined. Compacts (500 mg) of DS, MCS and GEL were produced by compressing the powder at various pre-determined pressures (56.625-311.438 MNm^{-2}). The compression properties of the compacts were evaluated using the Heckel equation. **Results:** The Heckel plots for DS, MCS and GEL possessed nonlinear curves at the lower and higher pressures and linear curves in between. The mean yield pressure (P_Y) was high which is indicative of higher resistance against plastic deformation. Modification of DS to MCS increased P_Y for deformation with corresponding decline in onset of deformation and lesser total deformation. The P_Y, the relative density at zero pressure (D_O) and the total deformation occurring in the powder (D_A) values ranked in the following order; GEL>MCS>DS. The fragmentation tendency of the powder (D_B) ranked in this order; MCS>GEL>DS. **Conclusion:** DS and MCS fragmented on compaction. DS and MCS are not suitable direct compressible excipient.

Key words: Acid hydrolyzed starch, Digitaria iburua, Heckel equation, Mean yield pressure.

INTRODUCTION

Compaction is defined as the formation of a porous specimen of defined geometry by powder compression [1]. Bonds are formed between particles which provides coherency to the powder (compact) due to increased proximity of particle surfaces accomplished during compression. Granules enhance compaction of tablet materials due to their porosity and the presence of inter and intra-granular pores. A sequence of events occurs during compaction in tablet production. This include: (a) transition or particles rearrangement; (b) deformation at the point of contact; (c) fragmentation; (d) bonding; (e) deformation of solid body; (f) decompression; and (g) ejection of tablet.

Models describing compression of powders

Heckel equation

Heckel equation is one of the most widely used equations for describing the compression properties of powders [2-5]. The Heckel plot is used to classify the compaction behavior of materials. The plot usually has a linear part, but it is curved at the low and high pressure ends. The Heckel plot relates the density of the compact to the applied pressure. According to the Heckel equation, powder compression follows first – order kinetics, where the pores are the reactant and the densification of the product. Based on this assumption, the Heckel equation can be rendered as:

$$l_n\left(\frac{1}{1-D}\right) = KP + A \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where ***D*** is the relative density and ***(1 – D)*** denotes the pore fraction or tablet porosity; ***P*** the applied pressure, ***K*** the slope of the straight linear portion of the plot which is suggested to reflect the deformation of particles during compression. The reciprocal of ***K*** is the yield pressure (P_Y). ***A*** is a constant is the intercept of the prolonged linear portion of the plot with the y axis. It reflects particle rearrangement and fragmentation.

The yield pressure is defined as the stress at which plastic deformation of the particles is initiated which can be derived from the linear part of the Heckel plot [1]. The non-linear parts of the curve has an initial curvature at low pressure which reflects particle fragmentation and rearrangement, while the deviation from a straight line at higher pressure may be as a result of capping and lamination of the powder. The Heckel plot and the derived parameters are very sensitive to variations in the experimental conditions, such as the maximum compression pressure applied [6].

Another model describing the compression of powders was described in 1971 [7] then Adams equation was derived in order to estimate the fracture strength of single granules from in-die compression data. The bed granule in the die is modeled as a series of parallel load bearing columns [8].

Digitaria iburua (Poaceae) is grown and harvested in the savannas of West Africa. *Digitaria iburua* has continued to be important locally because it is both nutritious and one of the world's fastest growing cereals reaching maturity in as little as six to eight weeks. The common names of *Digitaria iburua* is Black fonio – English; Black acha – English; Fonio noir – French; Manne noir – French; Iburu – German and Iburu – Nigeria (Hausa).

Figure 1: Photograph of *Digitaria iburua* grain (*American Association of Cereal Chemist*).

The aim of this study was to modify pure DS by acid hydrolysis to produce MCS and to conduct a compression study on DS and MCS using gelatin powder (GEL) as a standard to ascertain its suitability as a direct compressible pharmaceutical excipient.

MATERIALS AND METHODS

Botanical Material

Digitaria iburua grains were obtained from a commercial source in Samaru village, Zaria Nigeria.

Chemicals

Magnesium stearate (BDH chemicals Ltd., Poole-England), Hydrochloric acid (May & Baker, Lagos-Nigeria), Xylene (May and Baker, Lagos-Nigeria), Talc powder (BDH chemicals Ltd., Poole-England), Sodium Hydroxide Pellets (Avondale Laboratories Ltd., Bradbury-England)

Methodology

Collection and Identification of *Digitaria iburua* grains

Digitaria iburua grains were obtained from a commercial source in Samaru, Zaria, Nigeria. The grains were identified with Voucher No. 1315 by the Herbarium unit of Department of Biological Sciences, Ahmadu Bello University, Zaria, Nigeria.

Extraction of Starch from *Digitaria iburua* grains

The *Digitaria iburua* grains were inspected and all foreign material was removed. 8 kg of the grains was weighed washed with clean water then steeped in water for 24 h after which water was removed and the soaked grains milled (Philips Cucina HR 1757, Japan). The grinded mixture was mixed with distilled water and sieved using a calico cloth to obtain the starch. The starch was treated with 0.1 N NaOH to neutralize the acidity and also separate gluten from the starch. It was then allowed to settle over a period of time and the supernatant decanted leaving the sediment which was centrifuged for 10 min at 2,500 revolutions per min (rpm) (General Laboratory centrifuge– 2, Japan). The starch was obtained after scooping off the upper brown layers. The tightly packed starch was then dried in the oven (Gallenkamp oven BS size 3) at 40 ^{o}C and milled to fine powder using a blender. The starch was then packed in amber-coloured air-tight glassware and stored for further use.

Determination of Percentage Yield of Starch

The yield of starch (percent) was calculated as the ratio of the mass of starch obtained to the mass of the grains used as starting material.

Determination of Gelatinization Temperature

A 25 mL starch suspension of 0.2 % w/v concentration was prepared. The suspension was heated in a water bath at 40 ^{o}C. Sample was withdrawn and viewed under a light microscope (Ceti, Belgium). This was repeated after every 2 ^{o}C increments until the temperature at which granules lost their polarization crosses completely was attained. This temperature was recorded as the gelatinization temperature [9].

Production of Microcrystalline Starch (MCS)

A 200 g suspension of 36 % w/v starch was prepared and 12.5 mL of 6 N HCl added and the reaction was allowed to proceed at 48 ^{o}C. This reaction was allowed to run for 6 h after which starch produced was withdrawn for processing and examination. Processing of the microcrystalline starch involved vacuum filtration and suspension of the starch in 250 mL water followed by adjustment to pH 6 with NaOH, then vacuum filtration with 750 mL water and dehydration with ethanol. This procedure was repeated for 12 h, 18 h and 24 h.

Microscopy

A little quantity of the powder sample was mounted on a slide in glycerol on a previously calibrated microscope. A photograph of the powder sample was captured using a compound microscope (Fisher, Rochester – New York), at 400 x magnification.

Determination of Percentage Yield and Organoleptic properties of MCS

The percentage yield of the microcrystalline starch was calculated. Organoleptic properties such as taste, colour, odour and texture at various hydrolysing conditions were determined.

Iodine Test

A 50 mL volume of 2 % w/v starch suspension was prepared. A volume (10 mL) was then measured, boiled and cooled. Two drops of 0.1 N iodine solution was added and shaken. The resulting colour was examined and noted.

Particle Density: An empty 50 mL pycnometer bottle was weighed (W) and filled with xylene. The filled bottle was weighed (W_1) and the difference between W_1 and W obtained as W_2. A 2 g quantity of the powder was weighed (W_3) and transferred into the bottle. The excess solvent was wiped off and the bottle weighed again (W_4). The particle density, ρ (g/cm^3), was then calculated from the equation given below:

$$\rho = \frac{(W_2 \times W_3)}{50(W_3 - W_4 + W_2 + W)} \quad \text{.....(7)}$$

Where ρ is particle density, W is weight of empty bottle, W_2 is weight of xylene, W_3 is weight of powder and W_4 is weight of bottle plus sample plus xylene [10].

Preparation of Compacts

Compacts (500 mg) were produced by compressing the powder with the various predetermined pressures (56.625, 84.938, 113.250, 141.563, 169.875, 198.188, 226.500, 254.813, 283.125 and 311.438 MNm^{-2}) for 30 s (dwell time) using Carver hydraulic hand press (Model A14, Apex Co. Kent — U.K.). Before each compression, the 12.5 mm die and the flat-faced punches were lubricated with a 2 % w/v dispersion of magnesium stearate in ethanol. After ejection, the tablets were stored over silica gel in a dessicator for 24 h to allow for elastic recovery and hardening preventing false low yield values [11].

Evaluation of Compression Properties

The diameter and thickness of the compacts were determined using a digital caliper (Z 540-1, USA) to the nearest 0.01 mm. Individual weights of the compacts were also determined to the nearest 1 mg using Mettler analytical balance. Their relative density (D) was calculated using the equation:

$$D = \frac{W}{V}\rho \text{-----(8)}$$

Where W = compact's weight, V= compacts volume and ρ = particle density.

Heckel plot of ln [1 / (1 - D)] versus applied pressure (P) for all formulations were drawn. The mean yield pressure P_Y, was determined as the inverse of the slope k.

Statistical Analysis

Statistical analysis was carried out to compare the compression property of DS/MCS/GEL tablets using correlation analysis as a statistical tool. Correlations were considered significant at the 0.01 level.

RESULTS

The results of preliminary investigation on *Digitaria iburua* starch (DS) revealed that on examination, DS was odourless, off-white and tasteless with a smooth texture when felt between the fingers. The yield of DS extracted from the *Digitaria iburua* grains was 62.88 % w/w. The DS gave a black-blue coloration when treated with iodine solution confirming the presence of starch. The gelatinization temperature was 56.0 ^{o}C.

Production of Microcrystalline Starch

After subjecting *Digitaria iburua* starch to acid hydrolysis at 48.0 ^{o}C for 6 h, 12 h, 18 h and 24 h the MCS obtained, gave an impressive yield of 87.52 % w/w after 6 h but the yield decreased to 84.14 % w/w after 24 h. The change in yield decreased as the hydrolyzing time increased. A colour change was noticed at 24 h as the off-white colour turned light brown.

Microscopy

The photomicrographs of the DS and MCS are shown on Plates 2 and 3 respectively when viewed under the microscope at 400 x magnification. On examination, DS and MCS appeared to be spherical in shape with a mean particle size range of 10.5 µm for DS and 9.58-9.61 µm for MCS.

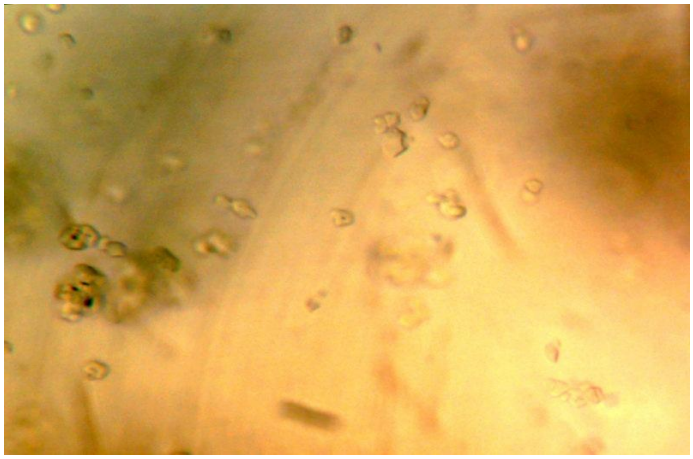


Figure 2: Photomicrograph of *Digitaria iburua* starch (Mag. X 400) (Fisher, Rochester – New York).

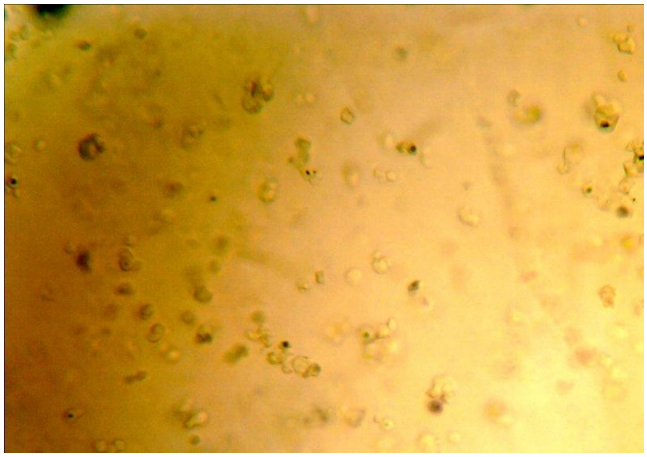


Figure 3: Photomicrograph of *Digitaria iburua M*icrocrystalline Starch (Mag. X 400) (Fisher, Rochester – New York).

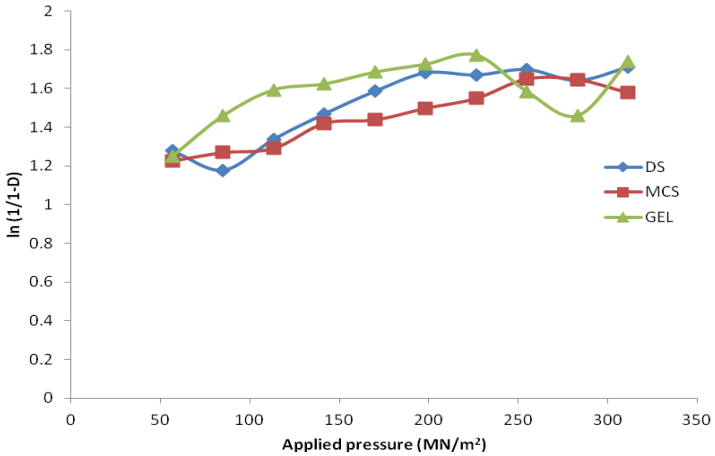
The results of the compression property study for the pure powder of DS and MCS was computed and presented in Table 1 and 2 below. The P_Y, D_O and D_A values ranked in the following order GEL>MCS>DS. The D_B value ranked in this order MCS>GEL>DS. The Heckel plots in Figure 1 show that DS, MCS and GEL possess non-linear curves at lower and higher pressures and linear curves in between.

Table 1: Compression Pressures for DS, MCS and GEL to obtain 1/1-D values.

Compression pressure (MN/m2)	Relative Density g/cm^3	1 – D	$\frac{1}{1-D}$	$ln\left(\frac{1}{1-D}\right)$
(DS)				
56.625	0.7213	0.2787	3.6880	1.2776
84.938	0.6917	0.3083	3.2457	1.1773
113.250	0.7376	0.2624	3.8109	1.3379
141.563	0.7699	0.2301	4.3459	1.4692
169.875	0.7953	0.2047	4.8851	1.5862
198.188	0.8139	0.1861	5.3735	1.6815
226.500	0.8119	0.1881	5.3163	1.6708
254.813	0.8171	0.1828	5.4705	1.6993
283.125	0.8065	0.1935	5.1679	1.6425
311.438	0.8194	0.1806	5.5371	1.7115
(MCS)				
56.625	0.7068	0.2932	3.4106	1.2269
84.938	0.7199	0.2801	3.5599	1.2698
113.250	0.7251	0.2749	3.6377	1.2913
141.563	0.7583	0.2417	4.1373	1.4201
169.875	0.7625	0.2375	4.2105	1.4376
198.188	0.7725	0.2375	4.2105	1.4376
226.500	0.7874	0.2126	4.7037	1.5483
254.813	0.8076	0.1924	5.1975	1.6482
283.125	0.8071	0.1929	5.1840	1.6456
311.438	0.7935	0.2065	4.8426	1.5776
(GEL)				
56.625	0.7136	0.2864	3.4916	1.2504
84.938	0.7677	0.2323	4.3048	1.4597
113.25	0.7966	0.2034	4.9164	1.5926
141.563	0.8027	0.1973	5.0684	1.6230
169.875	0.8144	0.1850	5.3876	1.6842
198.188	0.8218	0.1782	5.6117	1.7248
226.500	0.8230	0.1699	5.8858	1.7725
254.813	0.7950	0.2050	4.8780	1.5847
283.125	0.7677	0.2323	4.3048	1.4597
311.438	0.1759	0.1759	5.6850	1.7378

Table 2: Parameters from Heckel plots

Material	P_Y	D_O	D_A	D_B
DS	225.29	0.2797	0.5605	0.2808
MCS	499.43	0.3706	0.6716	0.3010
GEL	613.36	0.4615	0.7541	0.2926



DS = *Digitaria iburua* Starch, MCS = Acid hydrolyzed starch, GEL = Gelatin

Figure 1: Graph of ln (1/1-D) against pressure (MN/m^2) for DS, MCS and GEL compacts.

DISCUSSION

The mode of fragmentation or deformation of the powder was assessed by Heckel analysis. According to Heckel equation, powder compression follows first order kinetics, with the inter-particulate pores as the product and the compactibility of the powder bed as the product. The mean yield pressure P_Y is the pressure at which plastic deformation of a particle is initiated. The D_O value refers to the relative density at zero pressure. D_A value reflects the total deformation occurring in the powder while D_B values are an indication of the fragmentation tendency of the material. The Heckel plots show that DS, MCS and GEL possess non-linear curves at lower and higher pressures. The non – linear parts of the plot had an initial curvature at low pressure which reflects particle fragmentation and rearrangement while the deviation from a straight line at higher pressure was as a result of capping of the powder [1]. The linear curve for the compression pressures suggests the materials compacted by plastic deformation. A linear curve obtained from the results indicates deformation mainly by plastic flow [2]. The linear plot from which the Heckel equation is derived appeared slightly between low and high pressures. P_Y value is ranked in this order: GEL>MCS>DS. D_O and D_A is in this order: GEL>MCS>DS. D_B value is in the order: MCS>GEL>DS implying that MCS had the greatest tendency to fragment compared to GEL or DS. The P_Y values derived from the part of the Heckel plot rank in this order: DS>MCS>GEL. This implies that DS easily undergoes deformation or that the onset of plastic deformation is faster compared to MCS and GEL. The slope of the Heckel plot was calculated from the compression part of the plot and the slope provides information on the total deformation of the powder during the compression phase. A lower Heckel slope corresponds to a higher mean yield pressure and thus a higher resistance against deformation while a higher Heckel slope corresponds to a low mean yield pressure and thus a higher and easier deformation [12]. The results show that mean yield pressure $P_{Y,}$ values are all high and rank in this order GEL>MCS>DS. This implies that a higher resistance against pressure is required. DS and MCS do not deform readily on compression at low pressures. The mean yield pressure was high which is indicative of higher resistance against plastic deformation.

The difference in the properties presented in table 2 when GEL is compared with DS/MCS was significant at $p \leq 0.05$ at the 95 % confidence interval. There is a strong positive correlation when DS is compared with MCS.

CONCLUSION

DS and MCS fragmented on compaction. DS and MCS are not suitable direct compressible excipient. DS and MCS can be recommended as good disintegrants.

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