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COMPRESSIONAL AND MECHANICAL PROPERTIES OF NATIVE AND MODIFIED STARCHES OF *Solenostemon rotundifolius* COMPARED WITH GELATIN AND *Zea mays*

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ABSTRACT

The aim of this study is to determine the compressional and mechanical properties of native and modified starches of *Solenostemon rotundifolius* compared with gelatin and *Zea mays*. 500 mg compacts of *Solenostemon rotundifolius* starch (SRS), Water – prepared pregelatinised starch (PGSW), Alcohol – dehydrated pregelatinised starch (PGSA), Gelatin (GLT) and Maize starch (MZS) were made using a hydraulic press at various tableting pressures (56.6-169.9MNm^{-2}) and investigated for deformation characteristics and tensile strength. The ability of a powder bed to be compressed and consequently be reduced in volume is referred to as compression, which depends on the mechanical strength of the material. It plays an important role in the manufacturing of tablets. The ranking order for mean yield pressure, P_Y was PGSA>MZS>GLT>SRS>PGSW; total plastic deformation, D_A, was SRS<GLT<PGSW<MZS<PGSA showing that SRS and PGSW deforms plastically with fast onset of deformation while PGSA has tendency to show fragmentation before plastic deformation. The tensile strength results for SRS (0.22-0.70MNm^{-2}), PGSW (0.29-0.79MNm^{-2}), PGSA (1.76-1.82MNm^{-2}) and MZS (0.71-1.50-1.44MNm^{-2}) which were in agreement with compression studies showed that SRS and the modified starches produced harder and more compact tablets compared with Maize starch B.P. Hence, SRS, PGSW and PGSA could be useful in formulation of tablets with desired mechanical properties.

Keywords: Compressional, Mechanical, *Solenostemon rotundifolius* starch, Maize starch.

INTRODUCTION

Excipients are one of the several components of tablets, assisting in the processing and optimization of drug delivery. Their sources range from natural to synthetic and are simple, organic or inorganic molecules to

highly complex materials (Rudnic and Schwartz, 2000).

Starch, a versatile polymer, is the plant food reserve monopolysaccharide. It is biocompatible, biodegradable, non – toxic pharmaceutical excipient used in tablet production due to its inertness, cheapness and possibility of modification to a variety of useful complex derivatives (Oduola *et al.*, 2013). Starch is altered in a controllable manner to improve its functionality and extend its applications (Light, 1990). This is usually achieved through physical, chemical and enzymatic treatments (Radosta *et al.*, 2004). The physical methods of modifying starch include pregelatinisation, annealing and heat – moisture treatment. Pregelatinisation enhances functionality and applicability of certain starches. The pregelatinised starches (PGS) exhibit good flow, binding and compressibility (Joshi and Neves, 2005). The use of starch as pharmaceutical excipient in industries cannot be over-emphasized. They are used as binders, disintegrants, diluents and glidants.

The properties of pharmaceutical powders are related to the characteristics of their compression under pressure (Odeku, 2007). Compression of powder is defined as the reduction of volume of a powder owing to the application of a force (Alderborn, 2007). It plays an important role in the manufacturing of tablets and granules (Bodga, 2002). Three stages are involved in compression. These are die filling; initial volume reduction due to closer packing of powder particles; and increased load, decreased rearrangement and particle deformation. At the initial stage of compression process, when the powder is filled into the die cavity, the forces that exist between the particles are those that are related to the packing characteristics of the particles, the density of the particles and the total mass of the material that is filled into the die. The packing characteristics of the powder mass will be determined by the packing characteristics of the individual particles (Marshall, 1986). As the load increases, re-arrangement of particles becomes more difficult and further compression results in some form of particle deformation. On removal of load, if the deformation is somehow reversible in which it behaves like rubber, the deformation is said to be elastic. Most solids experience elastic deformation when external force is applied. In some other occasions, when the elastic limit is reached, and loads above this level result in deformation not immediately reversible on the removal of the applied force, the deformation is said to be plastic. This mechanism occurs most frequently in materials in which the shear strength is less than the tensile or breaking strength. Plastic deformation results in clean surfaces and is time-dependent (Armstrong, 1989; Ayorinde *et al.*, 2005). Thus, application of higher rate of force will result in less new clean surfaces, and weaker tablets. A good example is magnesium stearate, which form weak bond and easily wet surfaces. Hence, over-mixing of magnesium stearate may lead to weak tablets (Bodga, 2002).

Materials in which the shear strength is greater than the tensile strength, particles may be preferentially fractured, and the smaller fragments help to fill up the adjacent air

spaces. This is common in hard, brittle particles, and is known as brittle fracture e.g sucrose. The deformity pattern of a particular material depends on the lattice structure. Brittle fracture produces clean surfaces that are brought in intimate contact by applied load.

Studies have been conducted on the compressional characteristics of native and modified forms of various starches (Alebiowu and Itiola, 2002). Review of literature yielded no report on the compressional and mechanical properties of *Solenostemon rotundifolius*. Hence, this study is aimed at conducting the compressional and mechanical properties of native and modified starches obtained from *Solenostemon rotundifolius* in order to ascertain its applicability in formulation of tablets of desired strength, in comparison with standard pharmaceutical excipients, *Zea mays* and gelatin.

MATERIALS AND METHODS

Materials

The materials used were Hausa potato (*Solenostemon rotundifolius)* starch and pregelatinised starches, prepared in the laboratory, maize (*Zea mays*) (BDH Chemicals Ltd, UK), Gelatin (May and Baker Ltd, Dagenham, England).

Methods

Preparation of native and pregelatinised starches

Hausa potato starch was prepared according to the established procedure (Oduola *et al.,* 2013). Also, pregelatinised starches were prepared according to a method described by Oduola *et al.,* 2013. A 500 ml portion of cold distilled water was added to 160 g of dry native starch powder in a bowl. Then 1.5 liters of hot water at 60 ^{0}C was added with continuous stirring and placed on Gallenkamp Regulator Hotplate (England) while the stirring continued until translucent mucilage was formed. The mucilage was dried in the oven at 40 ^{0}C. The resulting flakes were milled in a blender. This was labeled as water-prepared pregelatinised starch (PGSW). The weight of the PGSW (W) was expressed as the percentage weight of the dry starch (W1) used in producing the mucilage to determine % yield (Y). The process was repeated but instead of drying the mucilage, 95% of ethanol was used to precipitate the starch from the mucilage. The precipitate was dried, milled and labeled PGSA.

$$Y = W/W1 \times 100$$

Compression of starches and gelatin

Each material weighing 500 mg were made into compacts by compressing them for 30s with pre-determined loads (56.6 – 169.9 MNm^{-2}) on Apex hydraulic presses (Type 814, Apex Construction Ltd., London W.I and Dartford). The 12 mm die and flat-faced punches were lubricated with 2 % w/v dispersion of magnesium stearate in acetone solution, before each compression.

Following ejection of the compacts, they were stored in a desiccator for 24 hr to allow for elastic recovery and hardening to prevent

false low yield values. The weight, thickness, diameter and crushing strength were then determined. The relative density (D) was calculated using the following equation:

$$D = \frac{W}{VtPs}$$

Where W is the weight (g), Vt, the volume (cm^3) of the tablet and Ps is the particle density (g/cm^3) of the solid material. HeckeI plots of In (1/1-D) versus applied pressure (P) were constructed for all the materials.

Determination of tensile strength

This is the stress needed to fracture a tablet by diametrical compression. It is determined by the expression below:

$$T_S = 2P/\pi Dt$$

Where P is the load that causes tensile failure of a tablet of diameter, D and thickness, t. The fracture load of three tablets was determined individually with the Monsanto hardness tester. The mean values of the fracture load were used to calculate the T_S.

RESULTS

The results in Table 1 indicate parameters for Heckel plots for SRS, PGSW, PGSA, MZS and GLT while Figures 1 and 2 represents the Heckel plots for SRS, PGSW, PGSA compared with MZS and GLT respectively.

Table 1: Parameters for Heckel Plots for SRS, PGSW, PGSA, MZS and GLT

Compression pressure	$In(\frac{1}{1-D})$				
(MN/M^2)	**SRS**	**PGSW**	**PGSA**	**MZS**	**GLT**
56.6	1.288	1.715	1.555	1.592	1.228
84.9	1.289	2.131	2.077	1.510	1.448
113.3	1.502	2.095	1.701	1.701	1.528
141.6	1.519	1.934	1.756	1.952	1.523
169.9	1.506	2.334	1.736	1.923	1.640

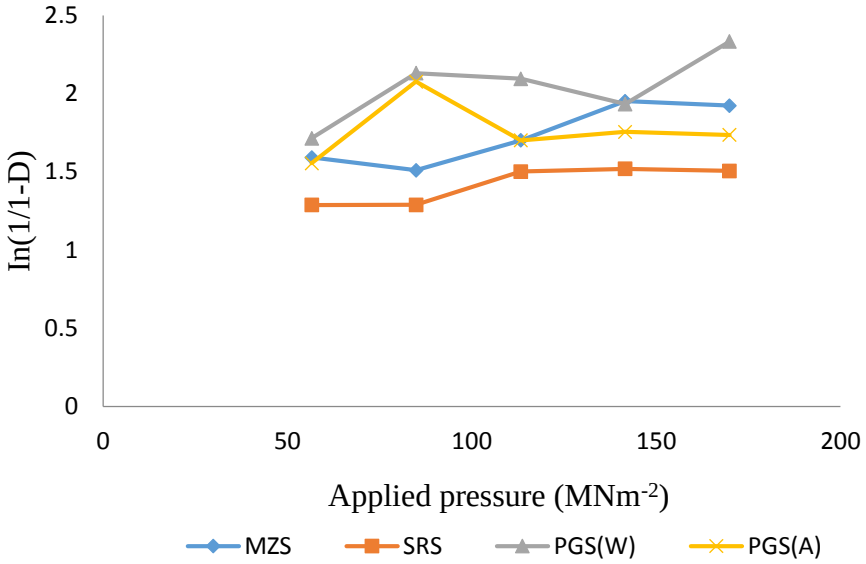

Figure 1: Heckel's plots for MZS,SRS,PGSW and PGSA

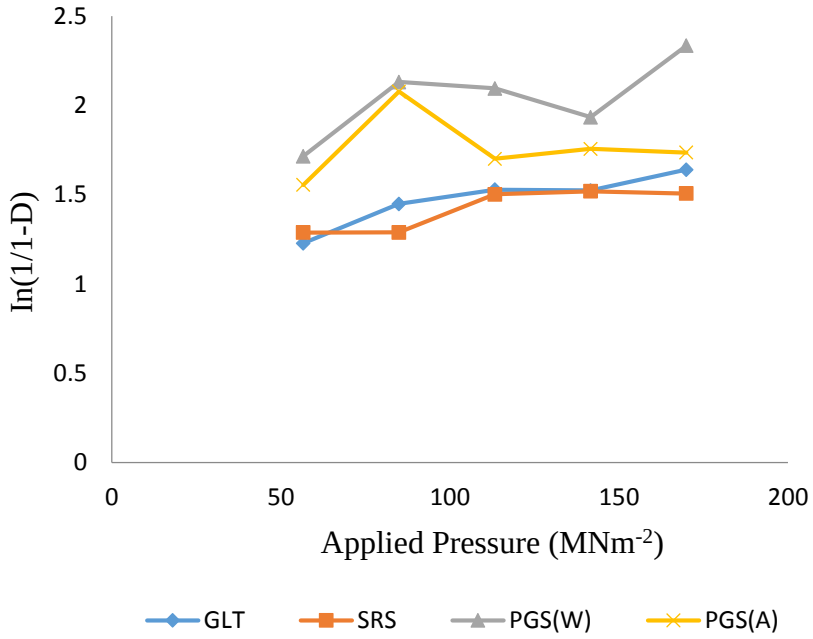

Figure2: Heckel's plots for GLT,SRS,PGSW and PGSA

The results in Tables 2 and 3 represents the parameters from Heckel plots in Figures 1 and 2 for SRS, PGSW, PGSA compared with MZS and GLT respectively

Table 2: Parameters from Heckel Plots for SRS, PGSW, PGSA and MZS

Material	Py	D_0	D_A	D_B
SRS	264.80	0.4200	0.6463	0.2263
PGSW	149.32	0.6103	0.7562	0.1459
PGSA	1617.14	0.4748	0.8067	0.3319
MZS	519.42	0.3786	0.7626	0.3840

Table 3: Parameters from Heckel Plots for SRS, PGSW, PGSA and GLT

Material	Py	D_0	D_A	D_B
SRS	264.80	0.4200	0.6463	0.2263
PGSW	149.32	0.6103	0.7562	0.1459
PGSA	1617.14	0.4748	0.8067	0.3319
GLT	293.69	0.5496	0.6652	0.1156

Table 4: Tensile strengths for selected starches and gelatin powder in compact of 500mg

Applied pressure (MNm^{-2})	*Solenostemon rotundifolius* starch(MNm^{-2})	Maize starch ($MNm^{-2)}$	Gelatin (MNm-2)	Water – prepared pregelatinized starch (MNm^{-2})	Alcohol Dehydrated pregelatinized starch ($MNm^{-2)}$
56.6	0.22	0.71	0.27	0.29	1.76
84.9	0.27	0.96	0.80	0.49	1.91
113.3	0.54	1.28	1.31	0.71	1.81
141.6	0.64	1.50	1.65	0.71	1.79
169.9	0.70	1.44	1.66	0.79	1.82

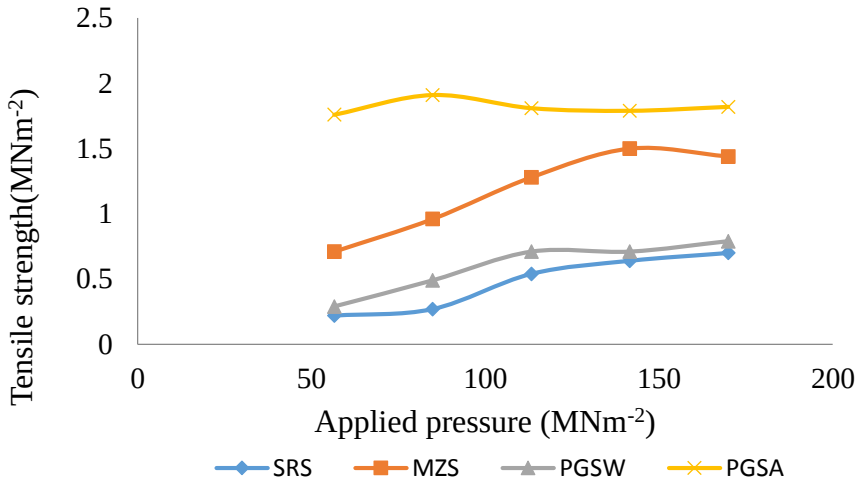

Figure 3: Plot of tensile strength versus applied pressure for SRS, PGSW ,PGSA and MZS

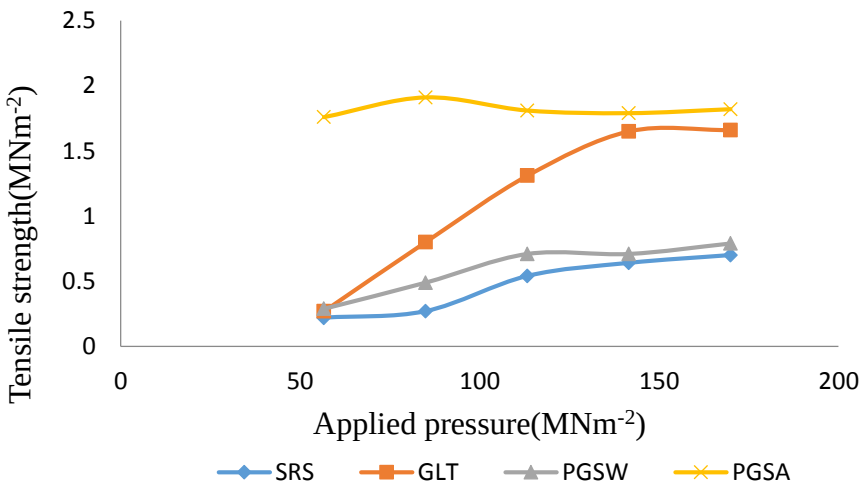

Figure 4: Plot of tensile strength versus applied pressure for SRS,PGSW, PGSA and GLT

DISCUSSION

The Heckel equation is widely used for relating the relative density of a powder bed during compression to the applied pressure (Odeku and Itiola, 2007). Various researches have been carried out on the compression properties of other starches. Alebiowu and Itiola (2002) worked on mechanical properties of pregelatinized plantain and sorghum starches compared with maize starch, and concluded that pregelatinized plantain starch could be useful in formulation of strong tablets. Odeku *et al*

(2005) also discovered that corn starch is better in producing hard tablets than sweet potato and cocoyam starches. Adetunji *et al* (2006) studied compression and mechanical characteristics of trifoliate yam starches. It was found out that trifoliate yam starch is better than maize starch in producing uncoated tablets for which high bond strength is essential. Figures 1 and 2 show the Heckel plots for the three starches compared with maize starch and gelatin respectively. A linear fit was obtained from all the formulations (usually 84.93 – 226.47 MNm^{-2}), indicating deformation mainly by plastic flow (Odeku, 2007). The values of the mean yield pressure, Py, Do, D_A and D_B for the materials are presented in Tables 2 and 3. Py was calculated from the regions of the plots showing the highest linear fit while the intercept was determined from the extrapolation of the line.

The Do value which represents the degree of initial packing in the die as a result of die filling and zero pressure was lowest with MZS followed by SRS. GLT was higher than SRS and MZS. However, modification of SRS by pregelatinisation increased the value. The result shows that modified starches exhibited the highest degree of densification or packing in the die because of die filling due to the denatured particles of the granules, while MZS exhibited the lowest values.

The D_A, which indicates the total degree of packing at zero and low pressures was highest with PGSA and lowest with SRS.
The D_B valued represents the particle rearrangement phase or packing in the early compression stages at low pressure. The value also indicates the extent of particle fragmentation. The D_B value was highest in MZS, and lowest in GLT. PGSA was higher than SRS. This means that modification of starch with ethanol increased densification and fragmentation at low pressures.

The mean yield pressure, Py is inversely related to the ability of the materials to deform plastically under pressure. PGSA had the highest while PGSW had the lowest values. These results indicate that PGSW exhibits a faster onset of plastic deformation than PGSA. This means PGSW is soft and ductile and readily deforms plastically during compression, at low pressures. Generally, the P_Y values were presented in descending order; PGSA>MZS>GLT>SRS>PGSW and D_A thus; PGSA>MZS>PGSW>GLT>SRS, indicating that PGSA is more plastic than other materials under test. This means PGSA produced the hardest tablet. This result is in agreement with the result of tensile strength which showed that PGSA had the highest value (Figures 3 - 4).

The results of Figures 3 - 4 and Table 4 on tensile strength for the selected starches and gelatin powder compacts showed that increase in the pressure applied from 56.6-169.9 MNm^{-2} led to a consistent increase in the tensile strength except maize starch at 169.9 MNm^{-2} that showed a slight reduction instead of an increase. This showed that *Solenostemon rotundifolius* starch and the modified starches produced harder and more compact tablets compared with maize starch. PGSA had the highest tensile strength, probably because of slowest onset of plastic deformation, D_O and highest total plastic

deformation, D_A, since higher amount of plastic deformation would lead to more contact points for interparticulate pores (Odeku and Itiola, 2007).

CONCLUSION

Compression studies have shown that SRS and PGSW deforms plastically during compression, while PGSA has tendency to show fragmentation before plastic deformation. Also, PGSA produced the hardest tablets because of slowest onset of plastic deformation and highest total degree of packing at zero and low pressures. Modification of the starch by pregelatinisation with alcohol (PGSA) increases the mean yield pressure, (Py) drastically, resulting in slow onset of deformation while modification with water (PGSW) results in a reduced mean yield pressure and rapid onset of deformation. Also, total plastic deformation (D_A) increases when modified with ethanol (PGSA) and reduces when modified with water (PGSW). The compression and tensile strength results showed that SRS and the modified starches produced harder and more compact tablets compared with Maize starch B.P. Therefore, SRS, PGSW and PGSA could be useful in formulation of tablets with desired mechanical properties.

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