

Short Paper

Effect of acid treatment on the consolidation and plasto-elasticity of tapioca powder

Florence E Eichie[†] and Roland S Okor

Department of Pharmaceutics and Pharmaceutical Technology, University of Benin, P.M.B. 1154, Benin City, Nigeria.

Abstract

Purpose: The effects of treating the tapioca (the fibrous residue obtained after up to 90% of the proportion of starch has been removed from the peeled and rasped roots of cassava tubers powder with dilute solutions of hydrochloric acid) in order to find an approach for rendering an otherwise poorly compressible material to a directly compressible powder.

Method: The parameters measured were the degree of consolidation of the powder after compression (i.e. the packing fraction of resulting tablets), the plasto-elasticity of the powders as reflected by the brittle fracture index (BFI) of the tablets made from the powders and the viscosities of mucilages derived from the powders. The influence of time of exposure and the concentration of acid used in the treatment were studied.

Results: The degree of consolidation of the powder increased slightly with increase in duration of exposure to acid (24 – 72 h) but drastically with increase in the acid concentration from 0.1 to 0.4 mol. L⁻¹. On the other hand, the plasto-elasticity of the powders as measured by the BFI values and the viscosities of mucilages derived from the powders decreased slightly with duration of exposure but drastically with increase in acid concentration. A change in acid concentration was therefore the more determinant factor with regards to the consolidation and plasto-elasticity properties of the powders. The decrease in the viscosities of the mucilages following acid treatment of the powders was indicative of a breakdown of polymeric structure in the powder

Conclusion: Acid treatment of tapioca powder imparted plasticity in tapioca powder which became compressible.

Key words: Plasto-elasticity, brittle fracture index, tapioca powder.

[†]To whom correspondence should be addressed. E-mail: eichie@uniben.edu

Introduction

The term 'plasto-elasticity' refers to the relative elastic to the plastic compression property of a material (powder or granules). A material which has a high plasto-elasticity would form crumbly tablets upon ejection from the die due to the high elastic forces. On the other hand, the brittle fracture index (BFI) of tablets is a measure of the tendency of a tablet to cap or laminate during ejection from the die. Invariably, materials with a high plasto-elasticity would give tablets of a high BFI values^{1, 2, 3}. Thus the determination of BFI has been considered as a measure of the plasto-elasticity of the powders from which the tablets are made^{4, 5}. In this study, we have applied the method of previous workers to use BFI values for the estimation of the plasto-elasticity of tapioca powder which is derived from the peeled and rasped roots of cassava tubers from *Manihot utilissima*.

Tapioca is the fibrous residue from which a major proportion of starch (usually up to 90%) has been removed. The use of tapioca powder as a potential tablet base for the formulation of potent drugs which are effective at low dose has been investigated previously⁶. The preliminary finding was that tapioca forms crumbly tablets which is indicative of a high plasto-elasticity. Acid treatment of starch has also been found to reduce the plasto-elasticity of powder which became directly compressible without preliminary granulation⁵. The chemical characterisation of the tapioca was not undertaken in that study. However its hydrophilic swelling and gelling characteristics which were observed in the study suggest that tapioca is essentially a polysaccharide and therefore the plasto-elasticity would be amenable to modification by acid treatment of its powder. This was the hypothesis examined in this study. The changes in viscosities of mucilages derived from the tapioca powder were determined to provide evidence for structural breakdown following acid treatment.

Materials and Methods

Tapioca is a fine, white powder its physical characteristics were determined to include a bulk density 0.344gcm^{-3} , particle density 1.43g cm^{-3} , porosity, 0.36, swelling capacity 1.53 at ambient temperature 25°C . It is a cohesive powder, its angle of repose was determined to be 48° . It forms gels in water which is a basic characteristic of polysaccharides.

The powder was extracted from cassava tubers of a particular specie *Manihot utilissima* and purified by the following procedure: Cassava tubers were peeled, washed and rasped to a pulp. Starch was extracted from the pulp by overnight immersion in excess water followed by straining with a nylon cloth. This process was repeated four times until a large proportion of the starch was removed (up to 90%), as estimated by iodine test on the fresh sample and on the residue. The residue was air-dried for 3 days and then fitz-milled first with the knife edges and finally with the hammer edges of the mill. The powder was again immersed in excess water to float off the fibres. This was repeated for up to three times and allowed to stand overnight. The sediment was strained, air-dried and reduced to a fine powder (particle size $>50\mu\text{m}$) in a ball mill. The fine powder was finally dried to a moisture content of $3.1 \pm 0.5\% \text{ }^{\text{W}}/\text{ }^{\text{W}}$ in a fluid bed dryer at 45°C for 1 h. Moisture content was determined by a balance fitted with a sample heater (Denward Instruments Ltd, model 33H).

Acid treatment of the powder was carried out as follows: A sample of the powder (500g) was equilibrated in excess (2L) HCl (0.1 mol. L^{-1}) at 50°C for various time intervals. At the end of each equilibration period, the acid was decanted and the residue was washed four times with water until the washing was neutral to litmus. The wet mass was stained, air-dried for 3 days and then pulverised in a ball mill. The powder was finally dried in a laboratory fluid bed dryer at 45°C for 1h. The moisture content was determined to be $2.3 \pm 0.1\% \text{ }^{\text{W}}/\text{ }^{\text{W}}$

using the moisture analyser given above. Powder samples were stored in a dessicator before use. The experiment was repeated with acid of various concentrations up to 0.4 mol. L⁻¹. Higher concentrations caused discolouration of the powder, which became brownish.

Formation of tablets with a centre hole

These tablets were made for the measurement of BFI values of the tablets. The Karl Kolb single punch machine (Kilian & Co. Frankfurt, Germany) was adapted with an upper punch with a centre through hole and a lower punch with a centre pin. A sample of the powder (500mg) was placed on the die and compressed at a pressure, 7 (arbitrary units on the load scale). The actual degree of consolidation of the tablets was measured in terms of the packing fractions which were determined by a procedure described earlier^{4,5}. The load was held on the tablet for 30s before it was released. Before each compression the surface of the punches and die were lubricated with a 1% dispersion of magnesium stearate in chloroform to facilitate ejection and detachment of the tablet from the pin. Resulting tablets were of thickness 3.3mm, diameter 12 mm, and a centre hole diameter, 1.6 mm. Tablets without a centre hole were formed by using similar punches but without a centre hole or pin.

Measurement of BFI values - index of plasto-elasticity of the powders

The measurement of BFI values is based on the expression^{7, 8, 9}:

$$BFI = 0.5 \left(\frac{T}{T_0} - 1 \right) \quad (1)$$

where, T₀ and T are the tensile strengths of tablets with and without a centre hole. The centre hole is a built-in model defect to simulate voids which are formed in the compact during tableting. Tablet tensile strengths needed for the estimation of BFI were

obtained by a method described in detail elsewhere^{4, 5}. The values of BFI obtained from equation 1 were taken as measures of plasto-elasticity of the powder samples. Thus a low BFI value indicated a low plasto-elasticity.

Determination of the viscosities of mucilages of the powders

The powders gelled and formed mucilages when they were equilibrated in water overnight at room temperature, 28° ± 2°C. The mucilages (5% w/v) were filtered through a No. 3 Whatman filter paper. Their viscosities were determined by measuring the time of flow(s) through the capillary of a U – tube viscometer (Size F). The viscosity of a fluid is directly proportional to the time of flow. Since this study was of comparative value only, the time of flow (s) was taken as the viscosity index. Each determination was carried out in triplicate and the mean results presented.

Results and Discussion

The packing fraction (P_f) of the tablets depended on the concentration of acid used in the treatment of the powdered samples of the tablet. For instance, after 72 h exposure the P_f values of tablets derived from the acid treated powder were as follows: 0.77 (HCl, 0.1 mol. L⁻¹), 0.77 (HCl, 0.2 mol. L⁻¹), 0.86 (HCl, 0.3mol. L⁻¹), 0.87 (0.4mol. L⁻¹). P_f value of the untreated sample was 0.70. The differences in the P_f values of tablets derived from the untreated powder and those treated with acid concentration of 0.3 or 0.4 mol. L⁻¹ were statistically significant at 95% confidence level. A high packing fraction (i.e. a high degree of consolidation in a tablet is a result of a high degree of plastic deformation of the particles during compaction^{1, 2}. The P_f values obtained here thus showed that acid treatment imparted plasticity to the powders particularly at the higher acid concentration (≥ 0.3 mol. L⁻¹). The decrease in P_f values at the higher acid concentration is also indicative that the extent of elastic relaxation of the tablets upon ejection from the die decreased; i.e. the acid treatment

reduced the elasticity of the powder. This means that the P_f values are not determined only by the extent of plastic deformation during compaction but also by the degree of elastic relaxation upon ejection of the tablet from the machine die.

The BFI value of a tablet is a measure of the plasto-elasticity of the powder from which the tablet was made^{4, 5}. A low BFI value thus indicates a low plasto-elasticity. Such a powder will form compact and coherent tablets^{4, 5}. The results in table 1 showed that increase in acid

Table 1: Effect of acid treatment on the brittle fracture index and packing fractions of the tablets

Time of exposure to acid (h)	BFI	P_f
HCl (0.1 mol. L⁻¹)		
12	–	0.70
24	0.33	0.77
48	0.30	0.76
72	0.30	0.77
HCl (0.2 mol. L⁻¹)		
12	0.29	0.75
24	0.26	0.75
48	0.22	0.76
72	0.19	0.77
HCl (0.3 mol. L⁻¹)		
12	0.19	0.77
24	0.15	0.85
48	0.15	0.85
72	0.13	0.86
HCl (0.4 mol. L⁻¹)		
12	0.15	0.80
24	0.13	0.81
48	0.11	0.84
72	0.11	0.87

The untreated powder formed crumbly tablets hence the BFI value was not measurable

concentration led to a decrease in BFI values, i.e. the plasto-elasticity of the powders decreased. This decrease was more marked at acid concentration ≥ 0.3 mol. L⁻¹.

The viscosities (inferred from flow time) decreased slightly as the duration of acid

treatment increased but sharply as the concentration of acid increased (Figure 1). It is known that branched chain polymers form

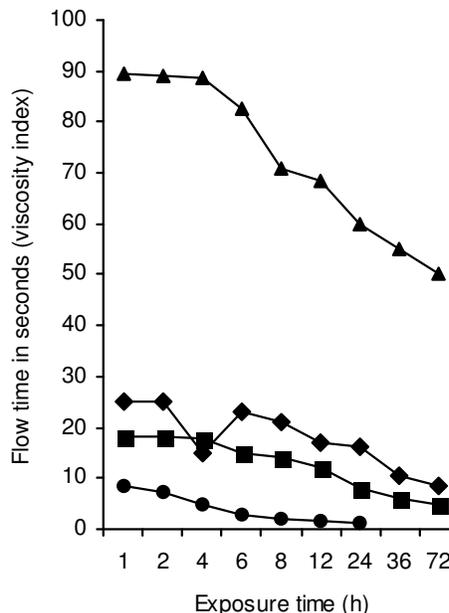


Figure 1: Changes in viscosity of 5% w/v tapioca mucilage with duration of acid treatment. The mucilages were derived from powders previously treated with acid of concentrations (mol. L⁻¹): 0.05 (▲), 0.1 (◆), 0.2 (■) and 0.4 (●). The mucilage of the untreated powder was too viscous to flow through the capillary of the U-tube viscometer.

more viscous mucilages compared to linear polymers because of the greater tendency of branched polymers to form a spongy network in solution. The decrease in viscosity is therefore an indication that the acid treatment probably degraded the branched polymers in the tapioca chemical structure to less elastic linear polymers. The results in Figure 1 suggested that such a conversion in molecular structure can take place rapidly when acid concentration ≥ 0.1 mol. L⁻¹ are used.

The extension of exposure time beyond 24 h did not appear to influence the results in BFI and P_f values markedly at given acid concentration. As can be seen in Table 1 the

results obtained at the 24h point were not markedly different from those exposed for 72h which suggests that 24h, would be the minimum duration of expose to obtain a conversion of the material from elastic to a plastic form.

The viscosities of mucilages derived from the powders decreased slightly with time of exposure but sharply with increase in acid concentration (Figure 1). The decrease in viscosity is an evidence for a breakdown of the polymeric structure. One possibility is that a branched structure was converted to a linear structure which forms less viscous gels. A second possibility is that the polymeric structure was broken down to simpler unit which also gives less viscous gels. Consideration was also given to the denaturing effect of acid on polymers, such as physical transition from crystalline to amorphous form and vice versa. However such a transition would not necessarily be reflected by a change in viscosity. What is certain is that there was a breakdown of the polymer structure following acid treatment, the details of which are yet to be studied.

Conclusion

The study has shown that acid treatment can

cause a breakdown of the polymeric structure in tapioca powder to obtain a less elastic but a more plastic material which is amenable to direct compression. Details of the structural breakdown are yet to be studied.

References

1. Itiola OA, Pipel N. Tableting characteristics of metronidazole formulations. *Int. J. Pharmaceutics* 31: 99 – 105 (1986).
2. Esezobo S, Pipel N. Effect of applied load on the plasto elasticity and tablet strengths of some directly compressible powders. *J. Pharm. Pharmacol.* 39: 303 – 304 (1987).
3. Ejiolori OI, Esezobo S, Pipel N. The plasto elasticity and compressibility of coated powders and tensile strengths of their tablets. *J. Pharm. Pharmacol.* 38: 1 – 7 (1986).
4. Okor RS, Nelson CL. Effect of acid treatment on the plasto-elasticity of certain starch powders. *West Afr. J. Pharm.* 9: 44 – 46 (1995).
5. Okor RS. Plasto elasticity of salicylic acid powders granulated with aqueous coacervated systems of certain acrylate methacrylate copolymers. *West Afr. J. Pharm.* 10: 38 – 40 (1996).
6. Okor RS, Obarisiagbon J. Evaluation of tapioca powder as a tablet base. *Nig. J. Pharm.* 12, 17 – 24 (1981)
7. Hiestand EN, Wells JE, Poet CB, Ochs JF. Physical process of tableting. *J. Pharm. Sci.* 66: 510 – 519 (1977).
8. Okor RS, Eichie FE, Ngwa CN. Correlation between tablet mechanical strength and brittle fracture tendency. *Pharm. Pharmacol. Commun.* 4: 511 – 513 (1998).
9. Itiola OA, Pipel N. Formulation effects on the mechanical properties of metronidazole tablets. *J. Pharm. Pharmacol.* 43: 145 – 147 (1991).