

POTENTIAL OF CARNUBA WAX IN AMELIORATING BRITTLE FRACTURE DURING TABLETING

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ABSTRACT

Carnuba wax (as binder) forms hard tablets even at low compression load attributable to its high plasticity. The aim of the present study is to investigate its potential in ameliorating brittle fracture (i.e., lamination and capping) a problem often encountered during tableting. Granules of paracetamol (test drug) were made by triturating the drug powder with the melted wax or starch mucilage (20%w/v). Resulting granules were separated into different size fractions which were separately compressed into tablets with and without a centre hole (as in-built defect) using different compression loads. The tablets were evaluated for tensile strength and the data used to calculate the brittle fracture index (BFI), using the expression:

$$\text{BFI} = 0.5(T/T_0 - 1)$$

where T_0 and T are the tensile strength of tablets with and without a centre hole respectively. The BFI values were significantly lower ($p < 0.05$) in tablets made with carnuba wax compared with tablets made with maize starch as binders. Increase in particle size of the granules or lowering of the compression load further ameliorated the brittle fracture tendency of the tablets. Using granules with the larger particle size (850 μm) and applying the lowest unit of load (6 arbitrary unit on the load scale of the tableting machine) the BFI values were 0.03 (carnuba wax tablets) and 0.11 (maize starch tablets). When the conditions were reversed (i.e., a highest load, 8 units and the smallest particle size, 212 μm) the BFI values now became 0.17 (carnuba wax tablets) and 0.26 (maize starch tablets). The indication is that the use of large granules and low compression loads to form tablets can further enhance the potential of carnuba wax in ameliorating brittle fracture tendency of tablets during their manufacture.

Keywords: Carnuba wax, granulation, brittle fracture, tableting.

INTRODUCTION

The term brittle fracture refers to the lamination or capping of tablets which occurs at the point of ejection from the die during their manufacture. Basically, the phenomenon is associated with the presence of entrapped air pockets arising from high speed of compression such that the air in the die hardly has time to escape before compaction is effected. Air pockets may also arise from the fracture of brittle materials in the tablet formulation during compression or due to elastic recovery of elastic materials upon withdrawal of the upper punch pressure. Thus, brittle or elastic materials are likely to exhibit a higher brittle fracture tendency than plastic materials (Uhumwangho and Okor, 2004). Interparticulate friction during compaction and consolidation may leave in the tablet low density regions which like the air pockets represent weak points in the tablet from which cracks emanate and propagates when ever stress due to the die wall pressure is exerted on the tablet, as it occurs during decompression (i.e., withdrawal of the upper punch pressure).

Binders which impart plasticity to tablets during compression also ameliorate brittle fracture (Itiola and Pipel, 1986; 1991). On the other hand, excessive

proportion of fines in granules, over drying of granules resulting in embrittlement, insufficient binders and high speed compression are associated with a high brittle fracture tendency.

A quantitative expression for the brittle fracture tendency was provided by Hiestand *et al* (1977) who applying the crack theory of Griffiths considered that cracks will propagate from the edge of a void or low density region in a compact when the stress at the edge of the void is three times over the stress in the bulk of the compact. On this basis, the brittle fracture index (BFI) was given by Hiestand *et al* (1977) as:

$$\text{BFI} = 0.5 (T/T_0 - 1) \quad (1)$$

where T_0 and T are the tensile strength of the tablet with and without a centre hole respectively. The centre hole is a built - in model defect representing the void in actual tablets. In practice, tablets with BFI values ≥ 0.5 are highly prone to brittle fracture (Hiestand *et al.*, 1977).

Carnuba wax is ordinarily not used as binder in conventional tablets. However, in the molten state it has been used as binder to form sustained release tablets (Uhumwangho and Okor, 2006^a). The granules compressed readily forming hard tablets even at low compression loads (Uhumwangho and Okor 2006^b),

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attributable to the high plasticity of the wax. Based on this plastic nature of the wax, the purpose of the present study is to investigate the potential of the wax for ameliorating brittle fracture during tableting and to study how the selection of particle size of the granules and/or the compression load may further influence the fracture tendency.

MATERIALS AND METHODS

Materials

Carnuba wax (Halewood Chemicals Ltd., England) is a fine waxy solid with melting point of 82-88°, yellowish in colour and was used as the granulating agent in the preparation of the matrix granules. Maize starch (BDH, Poole, UK) was also used as binder in the form of mucilage (20% w/v) to produce the conventional granules. The test drug was paracetamol BP (Pharmaceutical grade) and was selected as the test drug because of its well known high tendency to brittle fracture during tableting.

Methods

Granulation with starch mucilage: A sample of the paracetamol powder (80 g) was wet - massed with 25 ml of starch mucilage (20% w/v) to reach the granulation point. Hence, the content of starch binder in the resulting granules was 16.5% w/w. The wet mass was screened through sieve (aperture size, 1000µm) and then dried in a hot air oven (Kotterman, Germany) at 60°C for 0.5h. Moisture content of the resulting granules was 1.92 ± 1.3 %w/w.

Granulation with carnuba wax: The wax material (20 g) was melted in a stainless steel container placed in a water bath at a temperature higher than the melting point of the wax (i.e. 90°). A sample of the paracetamol powder (80 g) was then added to the melted wax and mixed well with a glass rod. The mass was pressed through a sieve of 1000µm aperture size and also dried in a hot air oven (Kotterman, Germany) at 60°C for 0.5h to a moisture content 2.1 ± 1.5 %w/w. The granules were stored in an airtight container before use.

Particle size analysis by sieve method

This was carried out by sieve analysis using test sieves ranging in pore size from 850-212µm and shaking samples of the granules for 5mins with the Endicott sieve shaker (Endicott's Ltd., UK). Fractions retained on each sieves where weight to determine the size distribution. The mean particle size (χ) was calculated using the formula:

$$\bar{\chi} = \frac{\sum f\chi}{\sum f} \quad (2)$$

where f is the frequency of each size χ . The determination was carried out in triplicate and the mean result obtained.

Measurement of bulk density of the granule: The bulk density (BD) was determined using standard procedures (Travers, 1972). In the procedure, a 30g quantity of granule sample was placed into 250ml clean, dry measuring cylinder and the volume, V_0 occupied by the sample without tapping was determined. The BD was calculated using the formula Density = weight of sample/volume occupied by sample.

Flowability: The flowability of the different size granules was determined by measuring the angle of repose formed when a sample of the granules (20g) was allowed to fall freely from the stem of a funnel to a horizontal surface. An angle of repose $\leq 30^\circ$ indicates a free flowing powder (Travers, 1972).

Tableting technique: The different size fractions of the granules were separately used to form tablets Flat faced tablets of mean weight 500±4mg and diameter, 12mm were produced using a single punch machine (Karl Kolb) at different compression loads (arbitrary units 6, 7, 8 on the load scale). Lower compression loads (< 6units) formed crumbly tablets while higher loads (>8 units) formed tablets that were too hard to fracture during hardness tests. The punch and die surfaces were lubricated with a 1% dispersion of magnesium stearate (BDH Chemicals, Poole UK) in chloroform to prevent sticking and hence allow easy ejection of the tablets from the die. To form tablets with a centre hole, upper punches with a centre- through hole (diameter, 0.6mm) and lower punches with a centre pin were used in the compression process. Details of the procedure have been described previously elsewhere (Eichie and Okor, 2002, Uhumwangho and Okor, 2004).

Evaluation of the tablets: The following tableting parameters were determined:

Tensile strength (T): This is the stress needed to fracture a tablet by diametral compression. It is given by the expression (Fell and Newton, 1970):

$$T = 2P/\pi dt \quad (3)$$

where P is the crushing load that causes tensile failure of a tablet of diameter, D and thickness t. The fracture load of ten tablets was determined individually with the Monsanto hardness tester following Brook and Marshal (1968). The mean values of the fracture load were used to calculate the T values for tablets of the various formulations.

Brittle fracture index (BFI): This is a measure of the tablet tendency to laminate or cap during manufacture. It is given by the equation 1 above.

Table 1: Particle size distribution of the granules prepared with carnuba wax (A) and with starch (B) as binders.

Particle size (µm)	Frequency (%) of each size fraction	
	A	B
212	6	15
500	17	26
710	37	38
850	40	21

Table 2: Effect of compression load and particle size on the tableting characteristics of granules made with carnuba wax and starch binders

Compression load (Arbitrary unit on the load scale)	Particle size of the granules (µm)	Tableting characteristics		
		T MN/m ²	T MN/m ²	BFI
Carnuba wax				
	212			
6		1.38	1.22	0.07
7		1.68	1.35	0.12
8		1.89	1.42	0.17
	500			
6		1.25	1.09	0.07
7		1.52	1.29	0.09
8		1.74	1.38	0.13
	710			
6		1.19	1.06	0.06
7		1.39	1.16	0.10
8		1.58	1.23	0.14
	850			
6		1.04	0.98	0.03
7		1.12	1.02	0.05
8		1.32	1.12	0.09
Maize starch				
	212			
6		0.97	0.72	0.17
7		1.36	0.98	0.19
8		1.55	1.02	0.26
	500			
6		0.85	0.68	0.13
7		1.24	0.89	0.19
8		1.42	0.96	0.24
	710			
6		0.76	0.60	0.13
7		1.10	0.82	0.17
8		1.38	0.94	0.23
	850			
6		0.71	0.58	0.11
7		0.99	0.78	0.15
8		1.22	0.89	0.19

RESULTS AND DISCUSSION

Packing and flow properties of the granules

The bulk densities of the granules were $0.63 \pm 0.03 \text{gcm}^{-3}$ (granules made with carnuba wax) and $0.35 \pm 0.02 \text{gcm}^{-3}$ (granules made with maize starch as binder), the latter granules therefore packed more loosely than the former. Nevertheless both types of granules were free flowing with angle of repose $\leq 30^\circ$ in each case. The mean particle

sizes were $821.6 \pm 9.2 \mu\text{m}$ (granules made with carnuba wax) and $646.5 \pm 7.9 \mu\text{m}$ (granules made with starch binder). Carnuba wax thus produced larger granules and therefore has a strong binder effect than the starch binder. The size distribution is given in table 1, where it can be seen that the most common size in the carnuba wax granulation was $850 \mu\text{m}$ but $710 \mu\text{m}$ in the starch granulations.

Tensile strength (T) and Brittle fracture index (BFI) of the tablets

Effect of binder type – Generally, carnuba wax formed harder tablets compared with the starch binder at all compression loads and in granules of various particle sizes (table 2). This observation implies that carnuba wax (compared with the starch binder) promoted a greater plastic deformation of the particles during compaction. Plastic deformation increases the area of particle - particle contact and cohesion leading to the formation of hard tablets (Esezobo and Pipel 1986). Besides, being a thermoplastic, carnuba wax will be expected to form welded bonds due to asperity melting which further accounts for the formation of harder tablets even at the low compression load.

Binders impact plasticity to tablet formulation and thereby ameliorate brittle fracture tendency (Itiola and Pipel, 1986). At all compression loads and using granules of varying particle sizes to form the tablets, carnuba wax persistently gave tablets of lower BFI values compared with the data for tablets made with the starch binder (table 2). This finding suggests that carnuba wax impacted greater plasticity to the tablets. According to the theory of brittle fracture (Hiestand *et al* 1977) plastic deformation due to applied stress will relieve the stress that would otherwise concentrate at the edge of a void (in this case represented by the centre hole), to elicit crack propagation. Hence, systems which deform readily under an applied stress also exhibit a low tendency towards brittle fracture, which explains the fracture-ameliorating effect of carnuba wax.

Effect of particle size of the granules – At a given compression load the smaller granules produced harder tablets compared with the larger granules as shown by the tensile strength values in table 2. This observation agrees with the previous findings using metronidazole as test drug (Itiola and Pipel, 1986, 1991). The increase in tablet hardness is attributable to the large surface area of the smaller particles available for particle – particle bonding. These harder tablets displayed higher BFI values (table 2), because they are not readily deformable to obtain stress relief as discussed above.

Effect of compression load – At all particle sizes of the granules used to form the tablets, slight increases in compression load resulted in significant ($p < 0.05$) increases in tablet hardness and its brittle fracture tendency. This is so because the harder tablets (without additional binder) are less readily deformable.

Thus a high compression load is associated with a high brittle fracture tendency. However, as can be seen in table 2, the carnuba wax granulation of large particle size displayed a relatively low BFI value (0.09) even at the high load.

CONCLUSION

The conclusion is that carnuba wax as a binder forms large granules which deform readily during compaction, thereby producing tablets with a low brittle fracture tendency, even at a high compression load.

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