

## Research Article

# Relationship Between Slugging Pressure and Brittle Fracture Tendency – A Case Study for Aspirin Tablets.

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## Abstract

**Objective** – Slugging is a pre-compression technique for the dry granulation of hydrolysable drugs (e.g. aspirin). The study was carried out to relate the slugging load to the hardness of the granules and the brittle fracture tendency of the final (recompressed) tablets.

**Method** – Varying compression load were applied to aspirin powder to form slugs, which were subsequently broken down to form granules. These were recompressed to give the final tablets. The hardness of the slugs was determined and taken as measure of the hardness of the resulting granules. The following tableting parameters were measured for the final tablets - tensile strength ( $T$ ), packing fraction ( $P_f$ ) and the brittle fracture index (BFI).

**Results** - A high slugging load was associated with the formation of hard slugs and hence hard granules. Upon recompression the hardest granules formed the hardest tablets ( $T = 3.29\text{MN m}^{-2}$ ) while the softest granules formed the softest tablets ( $T=1.09\text{MN m}^{-2}$ ). In turn, the hardest tablets displayed the highest brittle fracture tendency (BFI = 0.59) compared with the softest tablets (BFI= 0.21). A positive linear correlation existed between tablet hardness ( $T$ ) and BFI values ( $r = 0.98$ ).

**Conclusion** – The study showed that excessive slugging load produces hard aspirin granules which in turn yields hard but friable tablets.

**Keywords:** Slugging pressure, aspirin granules, tablet tensile strength, brittle fracture index

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## Introduction

Slugging is a pre-compression process for the formation of extra large tablets (slugs), usually of variable weight, due to poor flow of the drug powder. The resulting slugs are subsequently broken down into granules, which are recompressed to obtain the final tablets. The procedure is applicable to the dry granulation of hydrolysable drugs, such as aspirin, which are not amenable to wet granulation. A previous study<sup>1</sup> highlighted the need to control the slugging pressure, as excessive pressure will produce very hard granules that are difficult to deform during the recompression stage resulting in soft tablets. The present study focuses on the effect of slugging load on the brittle fracture tendency of tablets during recompression of the granules derived from the slugs since excessive compression loads are associated with a high brittle fracture tendency<sup>2</sup>. The objective of the present study, therefore, is to examine the relationship between slugging load and granule hardness on the one hand and the relationship between tablet hardness and their brittle fracture tendency on the other hand.

## Materials and Methods

### Materials

Aspirin powder (BDH, Poole, UK) was selected as the test drug because it is hydrolysable and therefore not amenable to wet granulation. Magnesium stearate (BDH Chemicals, Poole UK) was used as lubricant at a concentration of 1%w/w.

### Method

#### Preparation of the slugs

Aspirin slugs (wt., 1.4±1.2g; thickness, 5.24mm; and diameter, 12mm) were prepared using a single punch-tableting machine (Manesty, Type F3, Liverpool, UK.). The slugs were compressed at various loads (40, 43, 45, 48 and 50) indicated as

arbitrary units on the load scale of the machine. Actual consolidation of the slugs was measured as packing fraction ( $P_f$ ) using the equation (1) below:

$$P_f = W/\pi r^2 t \rho \dots\dots\dots (1)$$

where  $W$  is the mean weight of tablets as determined by a sensitive electronic balance ( $n = 10$ ),  $r$  is the tablet radius (mm),  $t$  is the tablet thickness (mm), and  $\rho$  is the particle density of the aspirin powder which is 1.44g cm<sup>-3</sup> as determined by fluid (liquid paraffin) displacement method<sup>3</sup>. The slugs were broken down to granules using the knife-edge of a hammer mill (Fitz Mill, D6 Manesty Machines Ltd, Liverpool, England) and screened to granular size range, 212 to 850µm; fines below 212µm were rejected.

#### Estimation of hardness of the resulting granules

This was estimated indirectly and in terms of the hardness of the slugs on the basis that a hard slug would produce hard granules. The crushing strengths of ten slugs from each compression load were determined with the Monsanto hardness tester<sup>4</sup> and the mean values of the result are reported.

Another parameter that was taken as index of granule hardness was the friability of the granules, on the basis that hard granules would not crumble easily to dust when subjected to impact stress. In the determination, a sample of the granules (10g) previously sifted (212µm) to remove fines was placed in the drum of the Erweka friabulator which was rotated at 25 rev min<sup>-1</sup> for 5 min, after which the granules sample was sifted (212µm) again to determine the % dust as the index of friability.

*Preparation of the final tablets*

Samples of the granules obtained by crushing the slugs (500mg) were each placed in the die (diameter 12mm) and compressed into tablets at a constant load (45 arbitrary units on the load scale), corresponding to degree of consolidation ( $P_f = 0.98$ ). In each case, the maximum compression load was left on the tablet for at least 30 seconds before releasing it to allow time for consolidation. The punch and the die surface were lubricated with 1% magnesium stearate in chloroform before compaction to facilitate ejection. In another aspect of the study, tablets with a center hole were formed by using an upper punch with a centre through hole (diameter 1.6mm) and a lower punch with a centre pin<sup>2, 5, 6</sup>. These tablets with centre hole were used in the determination of brittle fracture index of the tablet.

*Determination of tablet tensile strength (T)*

The load P, needed to fracture the tablets (n=10) was determined. Tablet tensile strength (T) values were calculated from the expression<sup>6</sup>.

$$T = \frac{2P}{\pi Dt} \dots\dots\dots(2)$$

where t is the thickness, and D the diameter of the tablet. The determination was carried out in triplicate using different batches of tablets and the mean results are reported.

*termination of brittle fracture index (BFI)*

BFI values of the final tablets were obtained from the expression derived by Hiestand et al<sup>7</sup> thus:

$$BFI = 0.5 (T/T_0 - 1) \dots\dots\dots(3)$$

where  $T_0$  and T are the tensile strengths of tablets with and without a centre hole. The centre hole is a model defect built into the

tablet to simulate the void formed (due to air entrapment) during actual tableting.

**Results and Discussion**

*Effect of slugging load*

The effect of slugging load on the hardness and on the degree of consolidation of the slugs is presented in Table 1. As expected, an increase in slugging load was associated with increase in the hardness and the degree of consolidation of the slugs. This is as a result of increased bonding strength with increased compaction load. The effect of slugging load on the friability of the resulting granules (obtained by crushing the slugs) is also shown in Table 1. The friability decreased with increase in slugging load. The lower friability of the granules with increase in slugging load indicates that the resulting granules became harder. This finding relates to greater interparticulate cohesion at the higher slugging load.

*Relationship between hardness and granule compressibility*

It was not possible to measure granule hardness directly with available facilities; hence, granule hardness was expressed here either in terms of hardness of the slugs or the friability of the granules (Table 1). The observed relationship between granule hardness and compressibility is that the softer granules produced softer tablets while the harder granules formed harder tablets (Table 2), contrary to a previous finding<sup>1</sup> that hard granules will yield soft tablets because they are poorly deformable during compression. Plastic deformation of particles is essential for effective particle-particle contact and cohesion during tableting. The reason why the softer granules produced softer tablets, as observed in this study, may be that the softer granules crumbled easily to powder during compression which are less readily compressible compared to granules. This is indicated by the higher degree of

consolidation ( $P_f$  values) of tablets derived from the harder granules (Table 2).

more prone to brittle fracture compared with softer tablets.

#### *Correlation between tablet hardness and brittle fracture tendency*

Tablet hardness (as measured by tensile strength) and the BFI values of the tablets are presented in Table 2. A positive linear

#### **Conclusion**

This study has shown that excessive slugging load yields hard aspirin granules, which in turn produces hard aspirin tablets

**Table 1:** Effect of slugging load on hardness and packing fraction ( $P_f$ ) of the slugs and on the friability of resulting granules.

Parameters	Slugging load (arbitrary unit on the load scale)				
	40	43	45	48	50
Hardness of slug (kg)	7.1	9.8	11.3	13.4	18.2
$P_f$ values	0.92	0.95	0.97	0.98	0.99
Friability of granules (%)	1.29	1.26	1.20	1.18	1.12

**Table 2:** Tableting parameters (T,  $P_f$  and BFI) of the final (recompressed) tablets

Slug hardness, kg:	T ( $\text{MNm}^{-2}$ )	$T_0$ ( $\text{MNm}^{-2}$ )	BFI	$P_f$
7.1	1.09	0.77	0.21	0.92
9.8	1.46	1.06	0.19	0.95
11.3	1.86	1.36	0.18	0.98
13.4	2.45	1.39	0.38	0.98
18.2	3.29	1.51	0.59	0.99

correlation existed between BFI and T values ( $r = 0.98$ ), indicating that the harder tablets were more friable than the softer tablets. The reason is that the harder tablets are less readily deformable by diametral compression compared with softer tablets, which yield more easily to diametral stress (e.g., die wall stress during actual compaction). The theory of brittle fracture<sup>8</sup> assumes that a void or low-density region in a compact is the weak point from which fracture propagates when diametral stress is applied to the compact. The stress is relieved by plastic deformation; otherwise it concentrates at the edge of the void (in this case, the centre hole) to elicit fracture. It is on this basis that the harder tablets are

that are friable. For this reason it is recommended that some amount of dry binder, e.g., PVP be admixed with aspirin powder to permit slugging at moderate pressures.

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