

CORRELATION BETWEEN HYSTERESIS LOOP AREAS OF LOWER PUNCH  
AND OF DIE PRESSURES VERSUS UPPER PUNCH PRESSURES.

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In tablet compression a series of authors (1-11) have employed plots of die wall pressure as a function of applied pressure to elucidate the mechanism of bonding in the compact (Fig. 1). The so-called compression cycles have been the subject of an article by Carstensen and Toure (12,13) in which it was shown that the hysteresis area (rather than the loop itself) was a good parameter of scrutiny for the mechanism of bonding.

Often die wall monitoring is either not possible or not reported, whereas the lower punch pressure is. The authors have, in a compression study of potato starch (95% by weight) wet granulated with cornstarch paste, examined the hysteresis

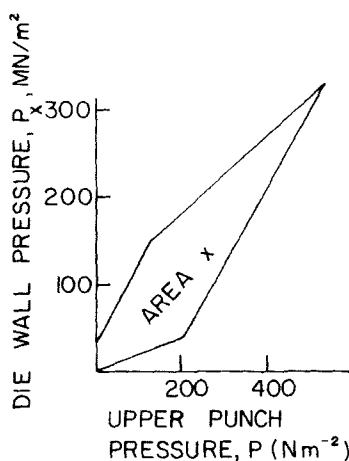


FIGURE 1

Compression cycles of potato starch granulated with corn-starch paste, at  $P^* = 533 \text{ MNm}^{-2}$  and 10% moisture. The ordinate is  $P_x$ , the die wall pressure, but similar plots are obtained, albeit with other slopes, when  $P_y$ , the lower punch pressure is the ordinate. The areas in the loops are denoted  $x$ , when  $P_x$  is plotted versus the applied pressure,  $P$ , and are denoted  $y$ , when  $P_y$  is plotted versus  $P$ , and the area determined. The die diameter is 12 mm.

areas of granules at (a) various maximum pressures and (b) at various moisture contents of the granules. Compression cycles were performed both of ( $P_x$ ) the die wall versus upper punch pressure ( $P$ ) and of ( $P_y$ ) the lower punch pressure as a function of  $P$ . The hysteresis loop areas ( $y$ ) of  $P_y$  versus  $P$  are plotted as a function of the hysteresis loop areas ( $x$ ) of  $P_x$  versus  $P$  in Fig. 2.

It is seen that proportionality appears to apply. This facilitates a great number of interpretations, since  $P_y$

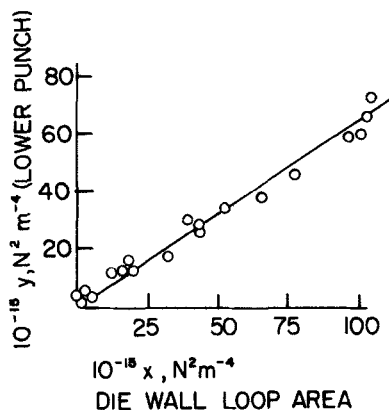


FIGURE 2

Hysteresis areas  $y$  (lower punch) versus hysteresis areas  $x$  (die wall). The 19 points represent the results from one starch granulation at five different moisture levels and four different maximum pressures (except one case with only three different pressures).

versus  $P$  plots (via slopes) do not give information of the behavior of the powder bed (whereas  $P_x$  versus  $P$  plots do). For instance the slope of the latter plot is, at the beginning, equal to  $v/(1-v)$ , where  $v$  is Poisson's ratio. This conclusion cannot be drawn from the  $P_y$  versus  $P$  plot. The area,  $x$ , similarly, gives information regarding mechanical behavior (12) in that if  $x$  is a quadratic function of  $P^*$  (the maximally applied pressure) then the solid should act like a Mohr body. If, on the other hand it is linear, then the compression is by constant yield in stress. These are consequences of Long's model (7), and are being scrutinized in recent literature.

If as suggested by Fig. 2,  $y = \alpha x$ , where  $\alpha$  is a constant, then the same conclusion holds for  $y$ . This would allow scrutiny of the above concepts by use of lower punch pressure cycles, and hence increase the amount of available data for analysis of the Long model (7).

#### REFERENCES

- (1) C.J.deBlaey and J.Polderman, Pharm.Weekblad 105, 57 (197)
- (2) C.J.deBlaey, W.deRijk and J.Polderman, Pharm. Ind., 33, 897 (1971)
- (3) C.Fuhrer, Dtsch.Apotheker Ztg., 105, 1150 (1965)
- (4) T.Higuchi, L.N.Elowe and L.Busse, J.Am.Pharm.Assoc., Sci., Ed., 43, 685 (1954)
- (5) T.Higuchi, T.Shimamoto, S.P.Eriksen and T.Yashiki, J.Pharm.Sci., 54, 111 (1965)
- (6) S.Leigh, J.E.Carless and B.W.Burt, J.Pharm.Sci. 57 888 (1967)
- (7) W.M.Long, Powder Met., 6, 73 (1960)
- (8) E.Nelson, S.M.Naqi, L.W.Busse and T.Higuchi, J.Am. Pharm. Assoc., Sci.Ed., 43, 596 (1954)
- (9) B.A.Obiorah, Int. Journ. Pharmaceutics, 1, 249 (1978)
- (10) E.Shotton and D.Ganderton, J.Pharm.Pharmacol., 12, 93T (1960)
- (11) S.Strijbos, P.J.Rankin, R.J.KleinWassink, J.Bannink and G.J.Oudemans, Powder Technology, 18, 187 (1977)
- (12) J.T.Carstensen and P.Touré, Powder Technology, 26, 199 (1980)
- (13) P.Touré, F.Puisieux, D.Duchêne and J.T.Carstensen, Powder Technology, 26, 213 (1980)