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EFFECT OF COMPACTION SPEED AND DIE DIAMETER ON ATHY-HECKEL AND HARDNESS PARAMETERS OF COMPRESSED TABLETS

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ABSTRACT

It is shown that within experimental error Athy-Heckel plots of extruded tablets are independent of (a) die fill, (b) die diameter, and (c) punch speed in slow, single-sided compression. Stress at failure is shown to be porosity dependent.

INTRODUCTION

A number of relationships have been described on the subject of compaction behavior of powders in relation to applied force. One of these is the Athy-Heckel equation[1-6]. It states, that if a particulate solid is subject to confinement and pressure (P,Pa), then the porosity (E) follows the equation

$$-\ln E = k\,P + k' \qquad (1)$$

where k and k' are constants.

Substances are usually characterized as compressible, fairly compressible, or poorly compressible depending on the strength (H, kP) as a function of applied pressure. Again, many publications have appeared on this subject[2,7,8].

No publication seems to have examined the effect of machine speed on the Athy-Heckel equation. In fact, the equation is derived on an assumption of a slow compaction process[5]. Since Equation (1) is often used[9,10] to determine the type of bonding (brittle fracture or plastic deformation), an attempt has been made to establish whether, in fact, such a speed dependence exists.

Porosity is, theoretically, considered to be the void fraction of an infinite (boundary free) mass of the particulate system in question. Some investigators[11] have shown the wall effect in tableting, and others[12,13,14] have shown the effect of centrifugal force in packing. An attempt is, therefore, made in the following publication to establish the effect of die size on the quoted parameters.

In the case of the Athy-Heckel equation, there could be a dependence on die size, although its magnitude might be small. In the case of the pressure-hardness profile, there could be a correlation between die diameter and failure.

EXPERIMENTAL

Data were obtained on a hydraulic press (Carver, Menomonee Falls, Wisconsin). Ditab is a recognized direct compression excipient and in the studies was lubricated with 0.5% magnesium stearate. Blending was carried out in a bottle, rolled at 4 rpm on a barrel roller for 2 minutes. The ensuing mixture was passed through a number 14 mesh hand screen.

Tablets of 250 and 500 mg were made on the following punch sizes: 1/4", 5/16", 3/8", 7/16", and 1/2". In the case of the smaller sizes, 500 mg was excessive, and in the larger sizes, 250 mg was insufficient; but an overlap exists in the middle sizes, so that comparison can be made between all sizes. In addition to this, compression at three different compression weights was carried out for one diameter, to establish the effect of load on the profiles.

The powder was weighed out individually, fed into the die with the lower punch in position and compressed with the upper punch on the hydraulic press. The press applied the pressure over a given (but controllable) number of seconds, dwelled for 20 seconds, and was then released.

The punches were flat faced non-bevelled, allowing ready calculation of the volume, V, of the tablet. The apparent density, D', is then given by

$$D' = W/V \tag{2}$$

and the porosity can now be obtained with knowledge of the true density, D, of dicalcium phosphate, i.e.,

$$E = 1-(D'/D) \tag{3}$$

Data obtained in this fashion were plotted according to Equation (1) for the five punch sizes. The results are shown in Table I.

RESULTS AND DISCUSSION

The Athy-Heckel plots of all the data from the hydraulic press experiments are shown in Figure I. The plot shows fair linearity, with a highly significant correlation coefficient (0.98 for 85 data points). These data include all punch diameters.

Figure II shows data for one particular diameter size, in which several compression weights were used. The plot shows no significant die effect. Values for k and k' are shown in Table I. It is seen that k is invariant in d.

k' is the value of -ln{porosity} at P=0, i.e., it relates to the apparent density of the powder in the die. Berg et al.[15] have

TABLE I

Heckel Parameters

Diameter (cm)	k (MPa^{-1})	k'
0.64	0.0021	1.027
0.79	0.0018	1.057
0.95	0.0023	1.099
1.1	0.0022	1.012
1.3	0.0020	1.039

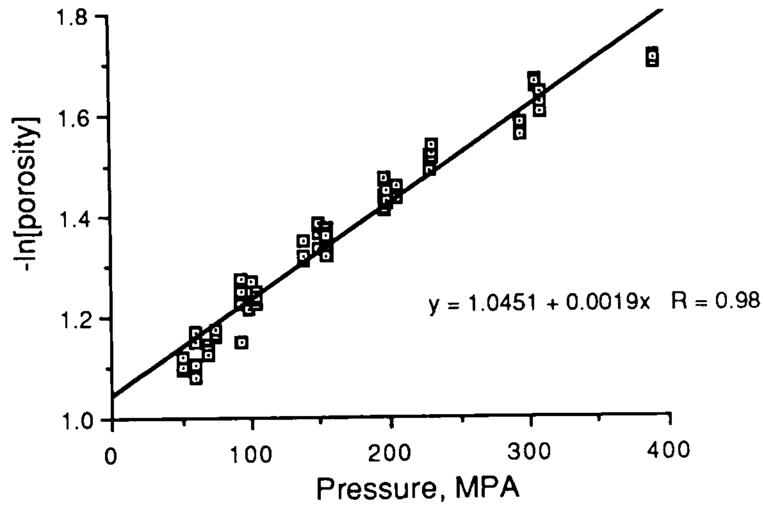

Fig. 1. Heckel plot for all data points

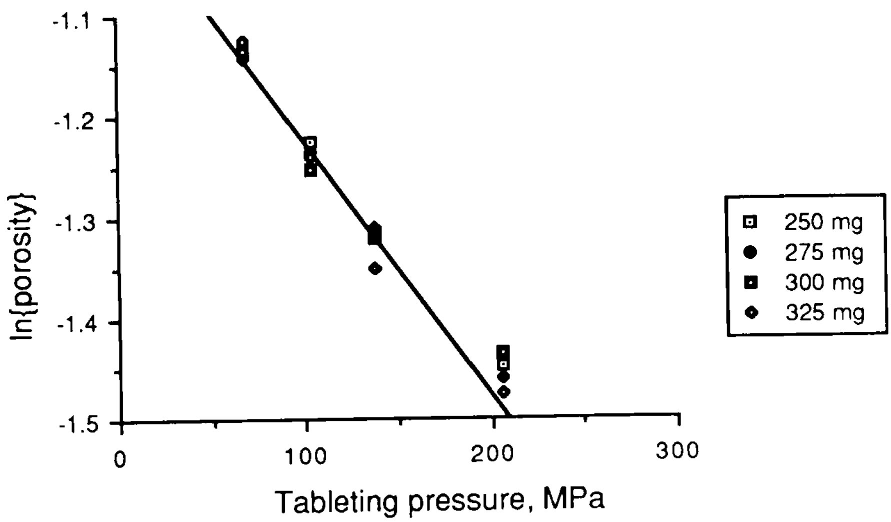

Fig. 2. Lack of effect ot tablet weight on Heckel plot

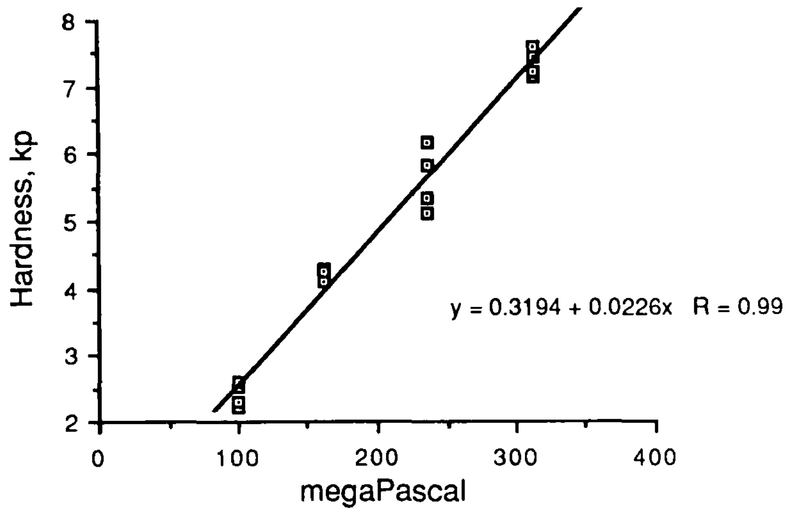

Fig. 3. Hardness vs. pressure for the 0.6356" diameter die

shown this to be a function of confining vessel diameter. However, as shown in Table I, there is no great effect within the diameter variation tested here.

It is seen, therefore, that Heckel plots can be considered fairly independent of the die in which they were carried out. The overlap between tablet weights at a given punch size also shows no discontinuity, so that, in general, data from different punch sizes and with differing amounts of loads are comparable, a fact that makes comparison of data simple.

Hardness values were obtained using a diametrical failure test (Schleuniger Hardness Tester). A typical hardness, (H,(kP),) versus applied compression pressure (P, MPa) plot is shown in Figure III. All such graphs were linear, showing that dicalcium

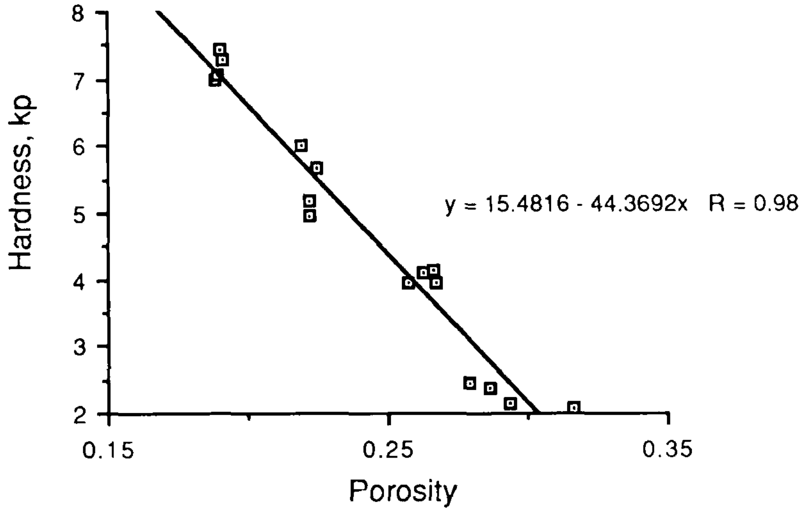

Fig. 4. Hardness versus porosity

phosphate dihydrate per se has no tendency of capping. Otherwise there would be a drop in hardness at a critical applied pressure[16].

The hardness (H, kP) is a function of porosity as shown in Figure IV. If H is converted to stress at failure, σ, by the formula[19]

$$\sigma = 2H/[\pi dL] \tag{4}$$

where d is the diameter of the tablet and L is the thickness of the tablet, then a (log-log) plot as shown in Figure V results.

There has been some dispute in literature[18,19] as to whether to include a porosity term in the denominator of Equation (2).

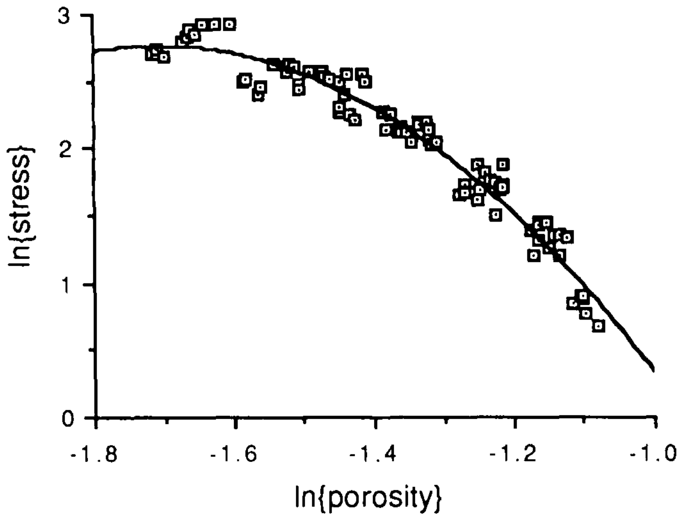

Fig. 5. Non-linear log-log relation between stress and porosity

TABLE II

Effect of Punch Speed

Speed cm/s	Punch Diameter 1/4"	5/16"	3/8"	7/16"	1/2"
0.54	0.222	0.243	0.266	0.297	0.316
1.07	0.222	0.239	0.263	0.292	0.294
2.17	0.225	0.235	0.267	0.291	0.286
4.18	0.219	0.229	0.257	0.289	0.279

Figure V shows that a log-log plot is (a) at best only fairly linear and (b) if so, then the slope is about -3, i.e., there is a strong porosity dependence if the stress at failure is interpreted by the expression in Equation (4).

The porosities of the tablets as a function of punch speed are shown in Table II.

Except for the low speeds at the large die sizes, the porosities attained at a given compression force seem speed independent. This conclusion, however, is only valid for the relatively low speeds employed here, and real differences may well occur at rotary machine speeds.

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