

MECHANICAL ENGINEERING

The Cold Compaction of Polyvinyl Chloride (PVC) Powders

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Abstract. In the polymer industry, during many plastics processing methods, both conventional and novel, polymeric powders or granules are subjected to varying rates of compaction. This has an important effect on the success and efficiency of the process.

Uniaxial strain rates ranging from 10^{-4} to 1.0 per sec., are covered for three different grades of polyvinyl chloride (PVC) powder. Four particle size fractions of these types of 150, 180, 212 and 250 μm are evaluated. The investigation shows that all materials tested exhibit strain rate sensitivity to varying degrees (increased resistance to compaction as the speed is increased). Also, it is found that the values of the mean yield pressure, determined by Heckel relation, are increasing with the speed of compaction, as well as with the particle size, in a non-linear manner. Generally, both ductile and brittle behaviors existed in the compaction process, but at higher speeds (strain rates) the amount of plastic deformation decreased (i.e. brittle behavior dominated), which resulted in smaller contact areas among the compacted particles and increased the final compact 'capping' possibilities. Furthermore, the tensile strength of the final compacts, assessed by the diametral compression 'Brazilian' test, shows a non-linear increase with the speed of compaction. Finally, as the particle size decreased and the particle shape becomes more irregular, the relative tap density increased.

Introduction

The compaction of particulates through a variety of processes like pelleting, forging, extrusion etc., is a common practice in a number of industries. In particular it is widely used with ceramics, metals, catalysts and pharmaceutical materials [1-5], and more recent studies [6-10] have demonstrated the feasibility of applying the cold compaction process to plastic powders. The demand for higher quality products and increase in the rate and volume of turnout by these industries has encouraged detailed research into the mechanism of powder compaction. This aspect of particulate technology has hitherto relied heavily on empirical methods and techniques, which have served the early industries very well over the years. However, such

empirical methods have been so well refined that a quantum leap forward in the compaction technology requires a new approach; one which is based on more detailed and systematic study of the mechanics of the process and the properties of the particulate matter. This is particularly important in the plastic industries, as the demand on the ever increasing number of polymeric materials and products, is progressing very rapidly.

The initial attraction of solid phase compaction of polymeric materials is that it provides a method of manufacturing articles in the speciality plastics such as ultra-high molecular weight polyethylene, polytetrafluoroethylene or poly (vinylidene chloride). These plastics have unique physical, mechanical and thermal properties but are severely limited in application because they do not lend themselves to shaping by the conventional melt processing methods such as injection molding or extrusion. However, it is now being recognized that solid phase compaction need not be restricted to these "difficult" materials and indeed, when used with the more common plastics, there are special advantages because it is more energy efficient, it is not limited to thin wall sections, it permits higher levels of fillers to be incorporated and can give faster cycle times since the relatively slow heating and cooling stages are removed from the cycle [11].

The interest in the behavior of plastic powders and granules under pressure is not restricted to novel processing methods. Many of the conventional forming methods involve, at some stage, the application of pressure to solid relatively cold granules of plastic. For example, the operation of an extruder or injection molding machine depends on the positive pressure at the inlet port to the barrel, which is caused by the head of powder in the feed hopper. Also, in the compression and transfer molding processes the compaction behavior of the polymer is an important factor for the process success. Thus, it is of considerable interest to know how plastic granules and powder will behave when subjected to pressure within a restricted section or rigid container.

During the cold forming of plastics, as is the case with all powders, the most important effect is the pressure loss within the plastic powder/granules as a result of inter-particle friction and frictional effects between the particles and the container wall [2,9,12]. This has particular relevance in the field of solid phase compaction because it has a major influence on the density distribution within the powder mass and hence the quality of the compacted solid [2-10]. Therefore, it is necessary to be able to predict the magnitude of the density variation in a particular molded compact based on a knowledge of the die geometry, powder characteristics and applied forces. There have been numerous studies in this field for metals, ceramics and pharmaceutical powders [1-5,13,14], but very little information is available for plastic powders. Most of the studies that have been made [6-11] have concentrated on identifying the significant parameters which govern the process, including the general

effects of applied pressure, dwell time under pressure, particle size distribution, etc. Many aspects, however, are still obscure. One most important aspect, which to date has not been carefully and systematically investigated, is the influence of loading and unloading rates on the mechanical properties of the formed compact. To the author's knowledge only a few attempts to take into account the speed of pressing are reported [9,15-17]. Despite that, it is well known that polymers are viscoelastic materials and therefore the rate effects cannot be ignored.

Rees and Rue [18] and recently Es-Saheb *et al* [13] have highlighted the importance of strain rate [i.e. speed] during the compaction process as well as during the process of testing the final compact strength in pharmaceutical industry. Also, it has become increasingly important, as a result of the work of Amidon *et al* [19], that the rate of axial and radial pressure removal have a paramount influence on the instantaneous stress distribution in both the preform and the die. Furthermore, the resilience of the formed compact is believed to be heavily dependent on the stress distribution history during the whole compression cycle [5]. Accurate studies of the rate effect are essential in order to complete the total picture of the material behavior. In addition, knowledge of rate-dependent behavior over a wide range of strain rates is necessary in formulating constitutive relations as well as in determining the predominant mechanisms responsible for material behavior.

However, to describe the compaction process, there have been numerous attempts made to relate the applied pressure and the material deformation. Among these are due to, Cooper and Eaton [20] and Kawakita [21], also, it was notable that Kinopickey [22], Shapiro [23] and Heckel [24] arrived at a fairly similar constitutive equation using different assumptions. Now generally known as the Heckel equation, it implies a linear relationship between pressure, P , and $\ln [1/(1-D)]$, and takes the form:

$$\ln [1/(1-D)] = KP + A \quad (1)$$

Where, D is the relative density (= actual density / pore-free density), K and A are constants. The gradient, K , is a measure of the ability of the compact to densify by plastic deformation. The reciprocal of the gradient is equal to the mean yield pressure of the powder. Generally, it is found that materials which deform plastically tend to give lower values for the mean yield pressure than materials which deform by brittle fracture. This equation became generally accepted and was widely used in metallurgy and pharmaceutical industries and, now, it is applied to polymeric powders.

In this paper a systematic investigation on the influence of axial compression rate as well as the particle size distribution, on the compaction characteristics and final compact strength of three polyvinyl chloride (PVC) powders is presented. The strain rate range covered is 10^{-4} to 1.0 per sec.

Materials

The initial program of work was carried out for three selected types of polyvinyl chloride (PVC) powders, made available by SABIC (Saudi Arabia Basic Industrial Company), namely PVC/67SG, PVC/70S and PVC/57S. At the first stage of this investigation, these materials were characterized. For each of the three polymers, the differential scanning calorimetry (DSC) tests were performed and the glass transition temperature (T_g) was found to be about 88°C. Also, the particle size and shape distribution for all materials were obtained, by applying both the sieve analysis and microscopical techniques. In this analysis, standard sieving tests were carried out for the three powders and the usual plots of the powder retained weight in each sieve (WT%) versus the sieve size (or mesh number) were obtained. Following this, powder samples from each sieve were examined under the microscope and photographs were taken. From these photographs measurements of the area, A , of about 100 particles (i.e. n particles), using a planimeter device were recorded. These data were processed, assuming an equivalent spherical shape particle (i.e. circular projected area), and the particle equivalent diameter (d_{eq}) is calculated according to the equation: $d_{eq} = \sqrt{(4A)/\pi}$. For each sieve the arithmetic particles mean diameter (\bar{d}) is then obtained using the equation: $\bar{d} = \sum d_{eq}/n$. This mean diameter of the particles is assumed to represent the average particles size for each sieve and it is plotted versus the powder retained weight (WT%) for each material. The results are then displayed on common axes for the three investigated powders as shown in Fig. 1. The tests showed that the materials consisted of particles of varying size and irregular shape.

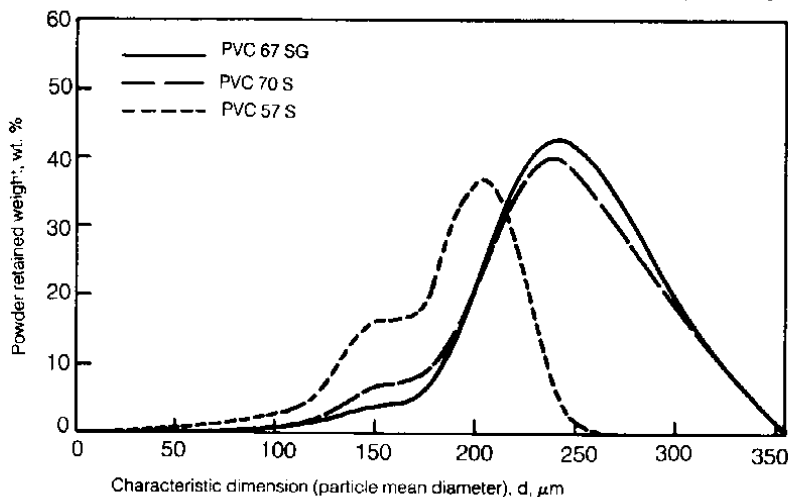


Fig. 1. Particle size distribution analysis.

Furthermore, bulk density tests were made for all materials according to ASTM D/895 method. The densities were found to be 541, 522 and 538 kg/m³ for PVC/67SG, PVC/70S and PVC/57S respectively. It is known that the bulk density, apparent density and tap density are very strongly influenced by the particle size and shape and their distribution. However, the investigation of these effects in polymeric powders as well as the discussion of the particle size and shape measurement techniques employed will be the subject of another work.

Apparatus and Compaction Techniques

The powders were compacted in a standard (Manesty, U.K.) hardened steel cylindrical die of 10.0 mm diameter. Flat cylindrical samples of 6.5 mm final height were produced using single end compaction. The compaction force was applied uniaxially at constant speed by an Instron type universal testing machine of 100KN capacity. The constant speeds of compaction used were: 0.5, 2.0, 20, 100 and 200 mm/min., which correspond to average strain rates obtained of 0.00056, 0.00225, 0.0225, 0.1125 and 0.225/sec. respectively. These average strain rates are calculated, simply by dividing the compaction speed, V , at each case by the initial compact height, $h = 14.81$ mm, (i.e. $\dot{\epsilon} = V/h$). Also, the mean particle sizes used from each material were: 150, 180, 212 and 250 μm . During each test the axial load was monitored via load cell, and the axial displacement of the punch by means of LVDT. Load-displacement curves were obtained on charts and later processed. A typical load displacement curve obtained during the compaction of PVC/67SG powder at 0.5 mm/min constant speed of compaction is shown in Fig. 2.

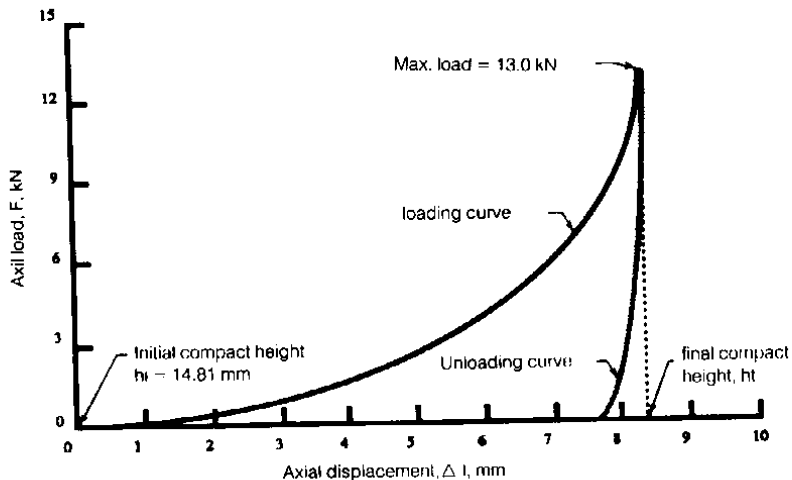


Fig. 2. Typical load-displacement curve for PVC/67SG powder compacted at constant speed of 0.5 mm/min.

In all tests, compacts were ejected from the die in the same direction as pressing. Then, the compacts were tested for strength, at speed of 0.1 mm/sec. on the Instron machine, by employing the traditional diametral compression 'Brazilian' test [5]. From the tests, the tensile strength, σ_t , for each green compact was then calculated using the equation:

$$\sigma_t = (2F)/(\pi dT) \quad (2)$$

Where, F is the breaking load, d and T are the diameter and thickness of the compact respectively. The tensile strength obtained from the diametral compression test, was originally used for testing the strength of concrete in the early fifties by civil engineers [25], and has since been used by many investigators to determine the tensile strength of various materials, such as coal [26], ceramics [27] and pharmaceutical compacts [5,13,28].

Experimental Results

The measurements in each test were processed for graphical presentation. The axial load is converted to axial pressure P , the punch displacement to axial strain ϵ_x and strain rate $\dot{\epsilon}_x$. The average strain rate was obtained from the displacement-time curves. The relative density D and tap density D_t were calculated from the displacement, mass of the powder and the measured true density. The tap density D_t is equal to the maximum initial density divided by the true density, where the maximum initial density, here, is the maximum density achieved by the powder mass within the die before it starts to resist the applied axial loading. Then, from the obtained Heckel plots for each case, and by the aid of a special software to check for the straight portion of the plot, the mean yield pressure, $1/K$, was calculated [5,13]. From these data, typical characteristic plots were made for each powder to show the effect of strain rate and particle size and shape on the powder behavior during compaction.

Typical Heckel plots at various speeds (strain rates) up to 200 mm/min., and particle sizes, for PVC/67SG powder are shown in Figs. 3 and 4 respectively. Each curve is obtained from a large number of experimental points and from more than one test. Meanwhile the variations of the mean yield pressure, as well as the tensile strength, for all materials and particle sizes, with the axial strain rate are displayed in Figs. 5 and 6. An interesting plot giving the variation of the relative tap density for all powders with the particle sizes is shown in Fig. 7.

A typical plot showing the variation of the axial compaction pressure with the axial compression strain rates at different constant axial strains of compaction, for PVC/70S powder of 250 μm particle mean size is shown in Fig. 8. It is, however, interesting to examine the axial pressure variation with the strain rate for the three materials considered over the whole range of strain rate. These are given in Fig. 9 all at an axial strain of 35% and particle mean size of 250 μm .

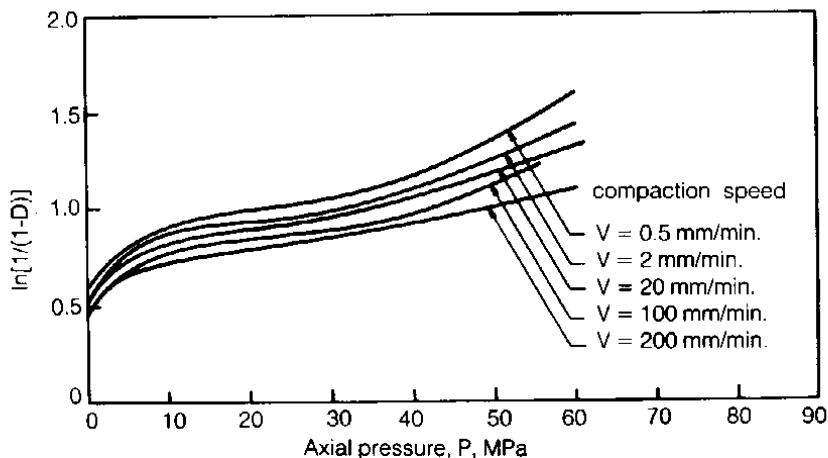


Fig. 3. Typical Heckel plots for polyvinyl chloride powder (PVC/67SG), of 250 μm particle mean size, compacted at different constant speeds.

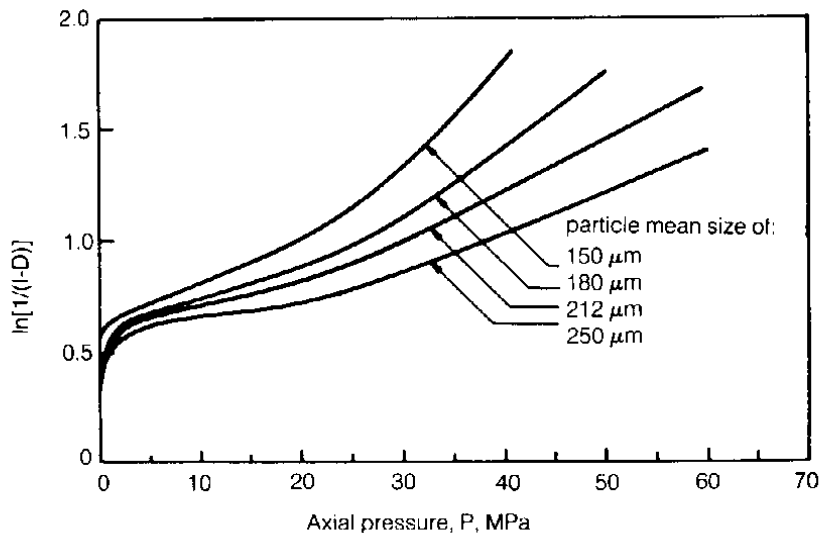


Fig. 4. Typical Heckel plots for polyvinyl chloride, (PVC/67SG), powder, compacted at constant speed of 100 mm/min.

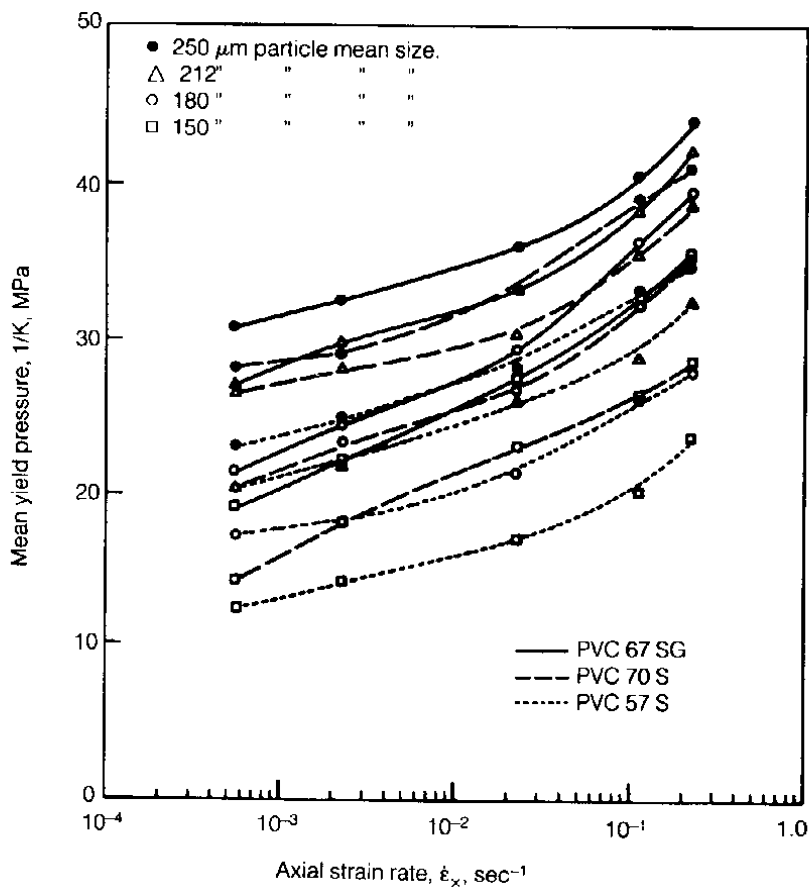


Fig. 5. The variation of the mean yield pressure, $1/K$, with the axial strain rate for all polyvinyl chloride powders.

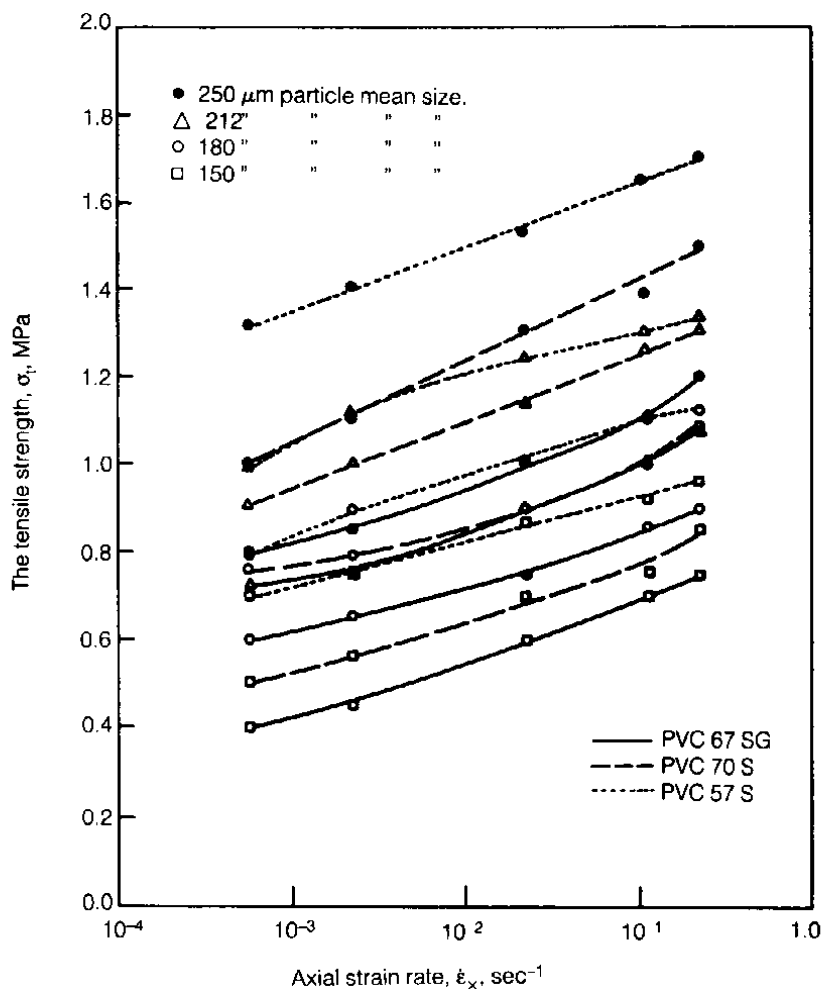


Fig. 6. The variation of the tensile strength, σ_t , of all polyvinyl chloride compacts with the axial strain rate of compaction.

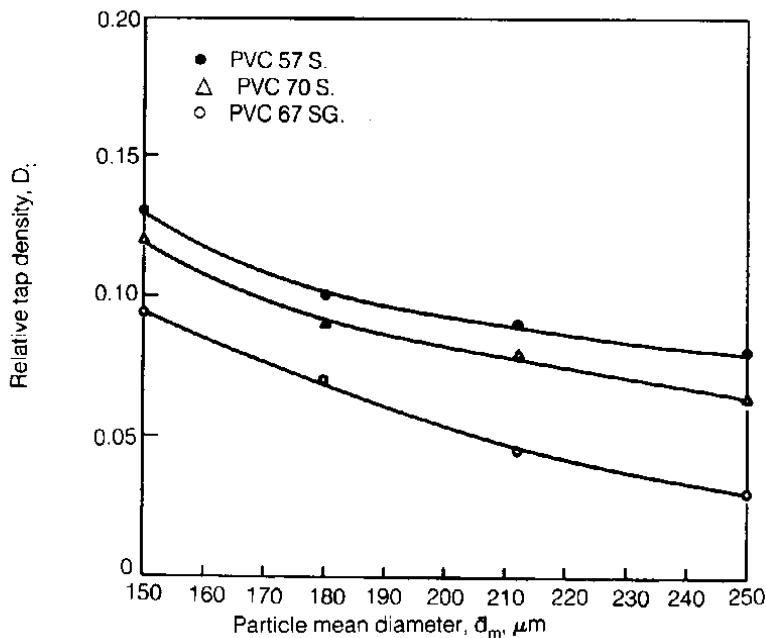


Fig. 7. The variation of the relative tap density with the particle mean dimension for all PVC powders tested.

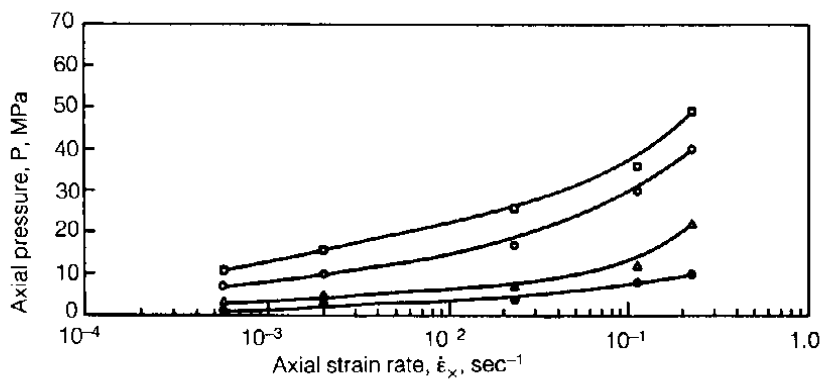


Fig. 8. Typical axial pressure variation with axial compression rates at constant axial degree of compaction, ϵ_x , of \bullet 15, Δ 25, \circ 35 and \square 45%, for polyvinyl chloride powder, PVC/70S, of 250 μm particle mean size.

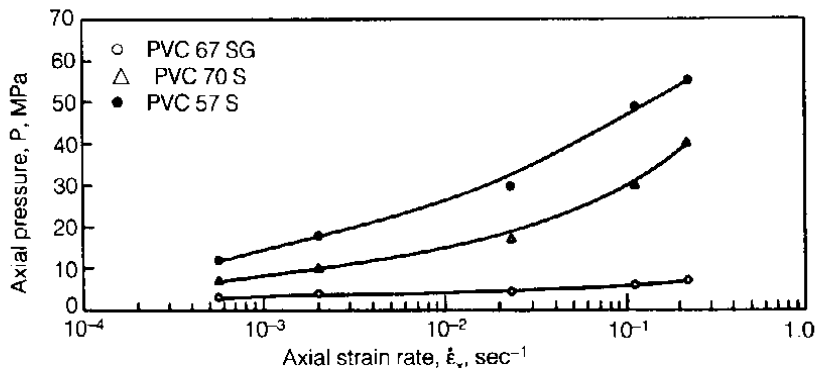


Fig. 9. Axial pressure variation with axial compression rates at constant axial degree of compression, ϵ_x of 35% for all PVC powders, and 250 μm particle mean size.

Discussion

In this series of tests the bulk density, average particle size, particle shape, etc. were determined for a selection of plastic PVC loose powders before cold compacted in a solid metal die. The objective was to identify the powder characteristics which enhance the compactability of the powders so that those which do not compact well in their current state could have their characteristics suitably modified.

In general, however, the polymeric materials considered exhibit sensitivity to the rate of loading. In the advent of high speed molding and production machines, it becomes essential to recognize the degree of this sensitivity. From Fig. 3 it can be seen that, at about 45% strain, which corresponds to $\ln[1/(1-D)]$ of ~ 1.05 (*i.e.* $\sim 65\%$ relative density, D), the axial pressure at 0.225 per sec (200 mm/min) exceeds that at 0.00056 per sec (0.5 mm/min) by about 50%. All materials show an increase of pressure with strain rate at the range studied (*i.e.* $10^{-4} - 1.0$ per sec). Also, for all materials tested, non-linearity is observed in the Heckel plots over the whole pressure and speed ranges investigated, as well as for all particle sizes, as can be seen in Figs. 3 and 4. This indicates that all the materials examined do not deform exclusively by plastic deformation mechanism. Here, it is important to mention that the heckel plot has typically been applied to materials, such like metals and pharmaceuticals, where the deformation involved in these materials is mainly occurred via the motion of dislocations. This is somewhat different to the deformation mechanisms involved in the polymers which have viscoelastic nature. Consequently it will affect the non-linearity of the Heckel plot.

It is noticed that as the speed of compaction and particle size are increased the non-linearity of the plots increases. This in fact supports the deformation mechanism suggested by Es-Saheb [5] and Al-Hassani and Es-Saheb [3]; which is, as the speed of compaction increases the plastic deformation of the material decreases and a fragmentation mechanism becomes increasingly dominant (i.e. the material tends to behave in a more brittle fashion), hence the increasing non-linearity of Heckel plots and the higher values of the mean yield pressure, see also Fig. 5. That is why most materials exhibit end capping at high speeds. The phenomenon is usually associated with brittle materials such as ceramics as reported by Thompson [14]. The interaction between ductile and brittle behavior affects the degree of particle crushing and bonding and hence the final compact strength as shown in Fig. 6. However, from all the tests carried out in this work, it is clear that all materials and particle sizes tested, displayed rate effects at varying degrees. All powders compacted offered increasing resistance to the applied pressure at higher compaction rates, as it is shown in Figs. 8 and 9. The strain rate sensitivity, in descending order, of the materials tested is, PVC/57S, PVC/70S and PVC/67SG respectively as shown in Fig. 9.

One important feature worth noting is that at low rates the internal time-dependent mechanisms which cause relaxation are more significant. Alongside the increase in pressure due to the resistance of powder to the imposed reduction of volume, there is also a tendency for the pressure to decrease with time. That is why an average strain rate rather than an instantaneous strain rate was used.

Ideally, a constitutive equation which allows for strain rate should be fitted to the results. But, this would require vigorous and complicated mathematical considerations. It is difficult as it is, without the influence of strain rate, to incorporate large strains in the non-associative plasticity theory [4].

It is known that the amount of bonding (which is mainly due to the amount of plastic deformation involved in the process) and the tensile strength of the formed compact are functions of the bonding created by the main compaction pressure. Thus as the speed of compaction is increased the final compacts are, apparently, showing higher tensile strength, see Fig. 6. This is actually due to the higher resistance of the material to compaction which resulted in higher axial compaction pressure to be attained. But, as discussed above and shown in Figs. 3 and 4, for the different speeds and the same particle size powder, the axial pressure required to achieve the same amount of compaction (strain) is higher as the speed of compaction is increased, see Figs. 8 and 9, and hence more bonding is expected and consequently a relatively stronger compact is obtained, as displayed in Fig. 6. Also, the strength of the formed compact is noticed to increase as the particle size increases.

As far as the relative tap density is concerned, we note that for all materials tested the relative tap density is increasing as the particle size decreases, see Fig. 7.

This is true because, as the relative dimensions of particles to the die are increased, much better packing is expected, and hence more resistance to compaction is encountered. However, PVC/57S powder is noted to give the higher tap density. This would be attributed to the facts that it has the finest particle size distribution and the more irregular particle shape (see Fig. 1).

Conclusion

There is general tendency for all the powders tested to exhibit increased resistance to compaction with strain rate. At high strain rates the brittle behavior dominates the process and compact 'capping' becomes more prominent. The powder with relatively lower bulk density and fine irregular particles has the best combination of characteristics to facilitate good compaction.

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اندماج مساحيق كلوريد عديد الفينيل (بي في سي) على البارد

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ملخص البحث. تتعرض مساحيق وحببيات اللدائن في الصناعة أثناء العديد من طرق وأساليب تصنيع البلاستيك التقليدية منها والجديد لمعدلات متفاوتة من الدمج. وهذا له تأثير مهم على نجاح وكفاءة الأسلوب.

تمت في هذه الدراسة تغطية معدلات انفعال أحادية المحور تتراوح بين (١٠)° إلى ١/١ ثلث لثلاث درجات مختلفة من مساحيق كلوريد عديد الفينيل (بي في سي). وقد تم تقويم أربعة أحجام من الحبيبات، لهذه الأنواع الثلاثة، وهي ١٥٠، ١٨٠، ٢١٢ و ٢٥٠ ميكرومتر. وقد بينت الدراسة بأن جميع هذه المواد قد أظهرت حساسية لمعدلات الانفعال بدرجات متفاوتة (تزداد المقاومة للاندماج كلما زادت السرعة). وكذلك فقد وجد أن قيم ضغط أو جهد الإذعان المتوسط، والذي حُدّد من علاقة هكل، تزداد بطريقة لاخطية مع زيادة سرعة الاندماج وزيادة حجم الحبيبة. ويوجد عموماً في أسلوب الدمج كل من أسلوب التشوه اللدن والتقصفي ولكن في السرعات العالية (أو معدلات الانفعال العالية) يقل مقدار التشوه اللدن (ويغلب السلوك التقصفي). وبالتالي تقل مساحات الاتصال بين الحبيبات المندمجة مما يزيد من إمكانيات (تكسر) المدمج النهائي. علاوة على ذلك فقد أظهرت قوى تحمل الشد للمدمجات النهائية والتي تم تقديرها بواسطة اختبار الضغط القطري (الاختبار البرازيلي) زيادة لاخطية مع زيادة سرعة الاندماج. وأخيراً فإن كثافة النقر النسبية قد ازدادت كلما صغر حجم الحبيبات وأصبحت أشكالها غير منتظمة أكثر.