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LOW-DENSITY CATALYSTS AND CATALYST SUPPORTS

PART II: THE PREPARATION OF STRONG, LOW-DENSITY PELLETS OF ALUMINA

G. T. SHAW and B. I. PARSONS

FUELS RESEARCH CENTRE

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PART II: THE PREPARATION OF STRONG, LOW-DENSITY PELLETS OF ALUMINA

by

G. T. Shaw* and B. I. Parsons*

ABSTRACT

The preparation and properties of pellets from highly porous forms of alumina gel are described. Considerable modification in both apparatus and technique is required to form pellets of low density reproducibly. A continuous-type pelleting press has been adapted to function as a constant-pressure apparatus, and a lubricating system has been developed to minimize friction between the walls of the punches and dies. In general terms, the pellet strength and density are a function of a) the pore volume initially present in the granular gel, and b) the pelleting pressure. Under all the conditions investigated, pellet strength and density increased systematically with the pelleting pressure. At equal pelleting pressures, the larger the pore volume (and the average pore size) present in the granular gel, the lower the pellet density and the greater the pellet strength. It has also been found that the particle size distribution in the powder mixtures increases in importance with increasing porosity. In the case of the gel with the highest porosity, the pellet density decreased as the concentration of fine particles (minus 200 mesh) increased. A mechanism is proposed to explain the improved pelleting characteristics observed with the highly porous aluminas.

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Direction des mines

Rapport de recherches R 230

CATALYSEURS DE FAIBLE DENSITÉ ET SUPPORTS DE CATALYSEUR

PARTIE II: LA PRÉPARATION DE BOULETTES D'ALUMINE RÉSISTANTES ET À FAIBLE DENSITÉ

par

G.T. Shaw* et B.I. Parsons*

RÉSUMÉ

Les auteurs décrivent le mode de préparation et les propriétés des boulettes obtenues à partir de formes très poreuses de gel d'alumine. Il a fallu effectuer des modifications considérables du matériel et des techniques pour former des boulettes de faible densité en régime continu. Une presse de bouletage à alimentation continue a été adaptée de façon à fonctionner sous pression constante, et un système de lubrification a été mis au point afin de réduire au minimum la friction entre les parois des éléments de moulage. En régle générale, la résistance et la densité des boulettes est fonction a) du volume des pores présents au départ dans le gel granulaire, et b) de la pression de bouletage. Dans toutes les conditions expérimentées, la résistance et la densité des boulettes augmentait avec la pression exercée. A pression égale, plus le volume (et la dimension moyenne) des pores sont grands dans le gel granulaire, plus la densité des boulettes est faible et plus grande est leur résistance. On a également constaté que la granulométrie des mélanges de poudres augments avec l'accroissement de la porosité. Dans le cas du gel le plus poreux, la densité des boulettes diminue en proportion inverse de leur concentration en petites particules (traversant le tamis de 200 mailles). Les auteurs proposent une explication des meilleurs propriétés de bouletage observées dans les alumines à forte porosité.

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CONTENTS

FIGURES

TABLE

INTRODUCTION

The following report is the second in a series on the preparation and characterization of low-density catalysts and catalyst supports and their application to typical processes connected with the refining of low-grade oils and tars. In the first report (1) a procedure was described for preparing highly porous forms of alumina gel containing up to 2.5 ml/g pore volume in pores of 50 to 1000 Å radius. (As it is normally prepared, alumina contains a comparatively small pore volume in the range 0.3 to 0.8 ml/g, concentrated in pores of 20 to 100-Å radius.) Marked advantages were observed with the highly porous gels from the viewpoint of compaction and the preparation of strong, attrition-resistant, low-density pellets. The purpose of the present report is to illustrate some of these advantages and to describe the modifications and changes from normal pelleting practice required in the handling of the highly porous powders.

Alumina gels prepared by conventional means, i.e., those with small pores of the order of 100-Å radius, do not pellet easily. The granular material is hard, difficult to grind into powder form, and high pressures are required to form strong pellets. The density of the pellets is invariably high (in the range 1.2 to 1.5 g/ml) and the total pore volume is low, only 0.3 to 0.6 ml/g. Abrasion of the punches and dies is a major problem if the powder mixtures contain appreciable quantities of small particles. Fine particles tend to work in around the sides of the punches, between the walls of the punch and the die, increasing friction and the rate of wear enormously.

In marked contrast, the highly porous aluminas, i.e., those with a large proportion of the pore volume contained in pores of 500 to 1000-Å radius, pellet easily at low pressures to form strong, hard pellets. Tablets ranging in density from 0.7 to 1.5 g/ml can be prepared by regulating the pelleting pressure and the particle size distribution in

the powder mixtures. The large-pore gels are soft in texture and can be ground into powder form simply and quickly. Because of the softer nature of the highly porous aluminas, the small particles do not cause such extensive abrasion of the punch and die system. In many instances, the low-density particles can be effectively prevented from entering the space between the sides of the punch and die by incorporating a liquid seal around the leading edge of the punch.

APPARATUS AND PROCEDURES

The Pellet Press

To the authors' knowledge no pellet press is available commercially which has been designed specifically for the preparation of low-density pellets from soft, fluffy powders. Highly porous powder systems generally pellet easily at quite low pressures. Because the pressures required are so low (and the effects of relatively minor changes in pressure on the pore structure of the pellet are so great), the pelleting pressure must be strictly regulated and all manner of friction around the punch and die assembly minimized.

Most industrial tableting presses operate as "constant volume" systems without regard for the pelleting pressure, i.e., two punches are caused to approach one another in a die until they are a fixed distance apart. The powder mixture is fed to the die by passing a "shoe" containing the powder over the die table at the appropriate point in the pelleting cycle. If there is any variation in the amount of powder placed in the die, the driving mechanism operating the punches merely generates more or less pressure until a pellet of fixed dimensions has been formed. In practice it is difficult to maintain a uniform flow of powder, with the result that there

is frequently quite significant variation in density, porosity, crushing strength and attrition resistance among pellets in a given lot of (uniformly sized) tablets. For many applications some deviation is not important, but in the manufacture of catalysts pellet strength is a critical factor. Weak pellets formed as the result of a gross underfilling of the die are not easily detected and are the cause of numerous operational problems (large pressure drops, plugging, etc.) as they break down in use.

For the present work a standard pharmaceutical tableting press (a Colton Model 21) was modified to function at "constant pressure" by removing the mechanical stop on the lower punch and supporting the punch stem against a hydraulic ram. A schematic diagram of the punch and die assembly is shown in Figure 1, and a photograph of the press is shown in Figure 2. The press is set up such that the punch stem and ram slip slightly against a predetermined hydraulic pressure in the formation of every pellet. The extent of movement, or slip, which the operator must allow in setting up the press depends on how well the particular powder mixture flows in the hopper and shoe.

The pelleting pressure exerted on the powder, in pounds per square inch, is equal to the total press load in pounds divided by the cross-sectional area of the pellet in square inches, i.e.,

$$\text{Pelleting Pressure} = \frac{\text{force on the ram (lb)}}{\text{area of the pellet (sq in.)}}$$

$$= \frac{\text{area of the ram (sq in.)} \times \text{hydraulic pressure (psig)}}{\frac{\pi}{4}\left[\text{diameter of the pellet (in.)}\right]^2}$$

$$= 3.785\ \frac{p}{d^2},$$

where p = hydraulic pressure (psig), d = pellet diameter (in.), and the area of the ram is 2.973 sq in.

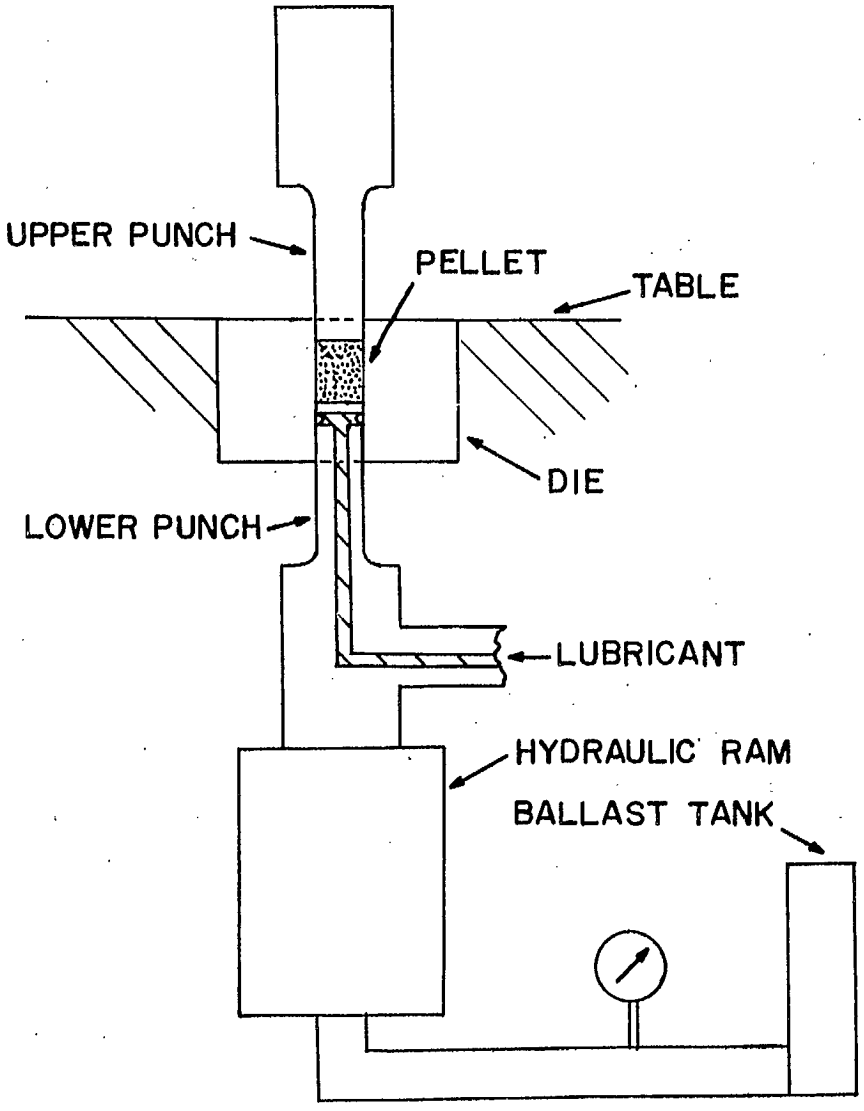

Figure 1 - A schematic diagram of the punch and die assembly used to form pellets at constant pressure.

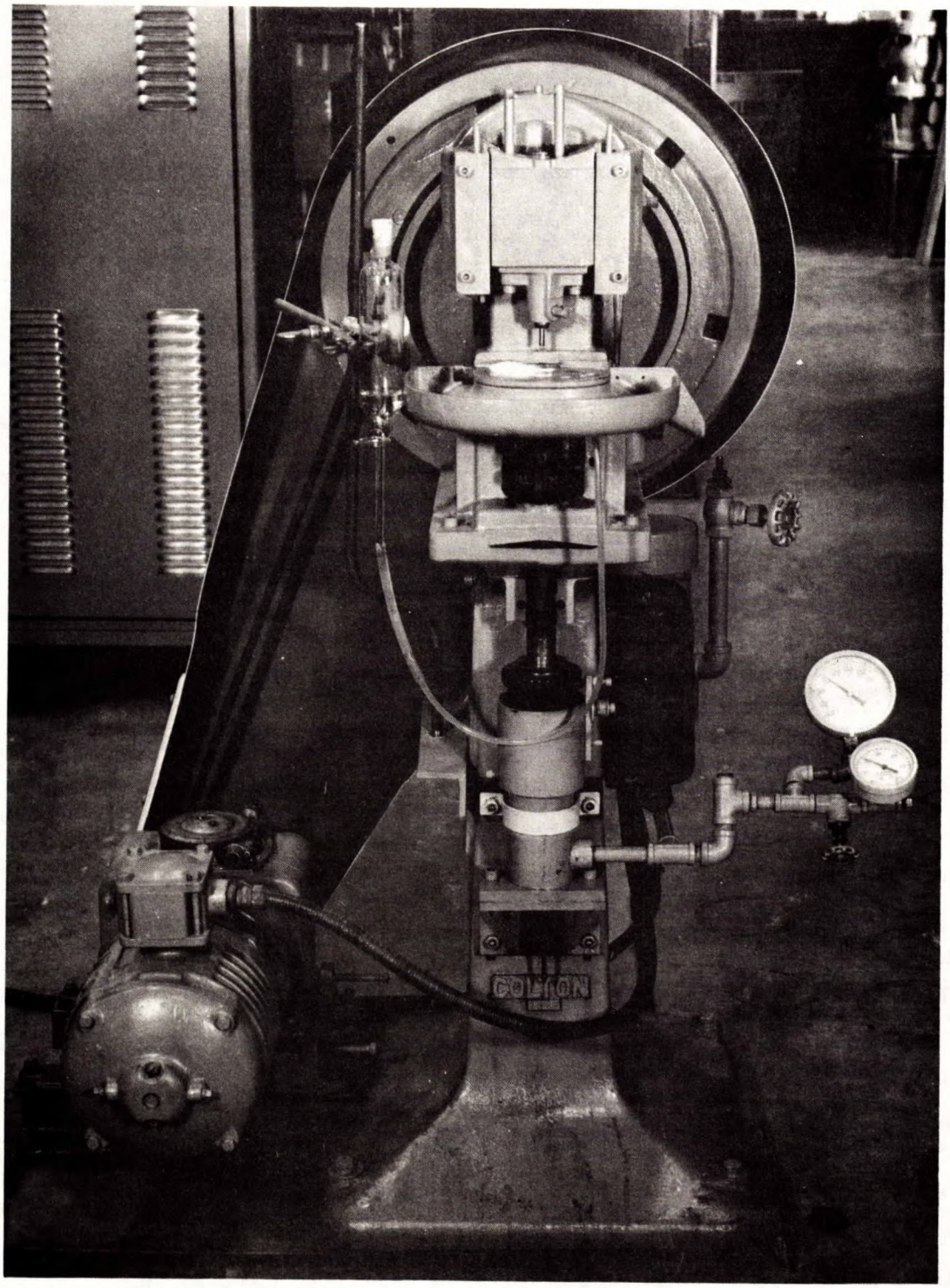

Figure 2 - A photograph of the pelleting press (powder hopper and shoe removed).

To reduce friction (and further improve the control at low pressures), a liquid seal was built into the bottom punch to prevent the ultra-fine particles from entering the space between the punch and die. The seal was accomplished by machining a small groove in the shank of the punch just below the leading edge (see Figure 1) and filling it with a suitable viscous liquid, such as a low-molecular-weight polyethylene glycol, maintained at 4 to 5 inches static (head) pressure. Care must be taken in the selection of the liquid used to make the seal, because small amounts are incorporated in the pellet when this technique is used. It has been our experience that the polyethylene glycol compounds can be easily removed by low-temperature calcination or hydrogenation without significantly affecting pellet strength or density.

As a point of interest, a U.S. patent (2) for a machine for tableting and moulding at "constant pressure" was granted to E. W. Pitzer in 1960. In Pitzer's press, one of the punches is connected by a system of rollers and levers to a compressed-gas cylinder maintained at constant pressure. The arrangement permits effective pressure control at moderate and high pressures but it is doubtful that it would be satisfactory at low pelleting pressure. As far as can be determined, the press is not yet available commercially.

The Preparation of the Alumina Powders

All the aluminas used in the present work were prepared by drying and calcining hydrous aluminum hydroxide gel. A detailed description of the procedures employed to form the aluminas of varying pore volume and pore size distribution is given in Part 1 of the current series of reports (1). The hydrous-gel starting material was purchased from the Reheis Chemical Co. (a Division of Armour Pharmaceutical Co.), Berkeley Heights, N. J. The initial water content was 90% by weight. It should be pointed out that the conversion of hydrous aluminium hydroxide to alumina by dehydration is

not a sharply defined chemical transformation. In the course of drying, the composition of the system gradually approaches that of alumina as represented by the following equation:

$$2Al(OH)_3 \cdot xH_2O \longrightarrow Al_2O_3 + (2x + 3)H_2O$$

At all temperatures below that at which sintering occurs (i.e. 700°C), considerable chemically bonded water is associated with the alumina. Even at 500°C the system retains approximately 5% water.

From the results of preliminary experiments, it was known that the most important factors in the pelleting process were, first, the initial porosity of the alumina gel and, second, the concentration of particle sizes smaller than 200 mesh (U.S. Standard Screen Size) present in the powder mixtures. Accordingly, one lot of alumina was prepared containing a large pore volume (1.8 ml/g) concentrated in pores 1000 Å diameter and greater, another prepared containing a small pore volume (0.3 ml/g) concentrated in pores of 50 to 100-Å diameter, and a third lot prepared of intermediate pore volume and pore size. After the initial drying of the hydrous aluminum hydroxide gel, each sample was ground to pass a 40-mesh screen, then calcined overnight at 300-325°C. The results of cumulative pore volume distribution determinations on the three samples are shown in the top section of Figure 3, on page 12 of this report.

After calcination, each sample of alumina was screened and the particles were separated into various size ranges. Powders containing the same particle size distribution were prepared from each sample lot by combining weighed amounts of the appropriate particle size fractions. In all of the powder mixtures investigated, the relative proportion of particle sizes larger than 200 mesh was maintained approximately constant and the concentration of particles smaller than 200 mesh varied from zero (all particles plus 200 mesh) to 79% by weight. In addition, one powder mixture was made up with particle sizes in the range from 40 to 100 mesh only, and another prepared with all particles smaller than 200 mesh.

Typical results of particle size distribution measurements on the four principal mixtures used in the present study are shown in Table 1.

The Pellleting Procedure

Pellets were made from each powder mixture over a range of pelleting pressures from 2000 to 15,000 psi. The pellets were cylindrical, 3/16-inch diameter with flat faces. Slight variations occurred in the length of pellets because of small variations in the amount of powder placed in the die by the mechanical filling system, but every effort was made to maintain the length of the pellets approximately equal to the diameter. The depth of fill in the die was varied as required to compensate for gross changes in the character of the powder to be pelleted and for changes in the pelleting pressure.

To ensure that none of the effects observed was due to the incorporation of the polyethylene glycol used in the liquid seal system on the bottom punch, small lots of pellets were prepared prior to the connection of the sealing fluid and compared with those prepared with the seal system in operation. Under all the conditions tested in the present study, there were no measurable differences observed. This was not surprising, because substantially less than 1% by weight of the sealing liquid was incorporated into the pellet after the press had been in operation for a few minutes and steady-state conditions had been established.

The Measurement of the Pore Volume Distribution

The pore volume distribution measurements were made by the method of high-pressure mercury porosimetry. The apparatus and techniques used in these measurements have been described previously by the authors (3).

The Measurement of the Pellet Density

The apparent density of each batch of pellets was determined by weighing 10 pellets and calculating the volume from dimensional measurements (diameter and length) estimated with micrometer calipers to the nearest one-thousandth of an inch.

TABLE 1

Typical Particle Size Distributions in the Alumina Powders

Mesh Size (U.S. Standard) Sieve	Mixture No. 1 (wt %)	Mixture No. 2 (wt %)	Mixture No. 3 (wt %)	Mixture No. 4 (wt %)
-40 + 60	59.5	45.8	35.0	29.5
-60 + 80	24.5	18.7	15.0	12.2
-80 + 100	16.3	12.4	9.1	8.0
-100 + 140	nil	15.2	11.0	9.9
-140 + 170		4.2	3.1	2.7
-170 + 200		4.1	3.1	2.7
-200 + 230		nil	5.1	6.7
-230 + 270			4.5	7.0
-270 + 325			5.0	6.2
-325			9.6	15.3
			24.2%	35.2%

The Measurement of the Crushing Strength

The crushing strength of the pellets was measured by placing a pellet (sideways) on the pan of a spring balance and pressing down on it with a small flat steel plate until the pellet broke ..... the force required to crush being the highest recorded weight on the scale. To keep the surfaces parallel and rigid during the measurements, the top plate was mounted in the chuck of a machinist's drill press. The hand-operated mechanism for advancing the drill in the press provided a convenient means for applying force to the pellet and easily compensated for any downward motion occurring in the spring balance itself.

The crushing strength varied with the length of the pellet, and all measurements were corrected to correspond to a nominal length of 3/16 of an inch. Occasionally, low values of the force required to crush individual pellets were observed, due presumably to the occurrence of cracks, or to a gross underfilling of the die cavity. The procedure followed in the present experiments was to measure the crushing strengths of 11 pellets and to report the result as the average of the best 10.

EXPERIMENTAL RESULTS AND DISCUSSION

The objective of the present work was to determine the procedures required to prepare pellets from the highly porous forms of alumina and to demonstrate the advantages to be gained by using the highly porous form if, in fact, any existed. After several trials of a preliminary nature, it became evident that the highly porous aluminas did offer specific advantages for the production of pellets of high strength and low density. It was also found that the particle size distribution in the powder mixtures increased in importance with increasing porosity. A general decrease in pellet density was observed as the concentration of fine particles was increased in the highly porous powder mixtures. This last fact was in marked contrast to the behaviour of the conventional small-pore aluminas where particle size is not an important factor.

The series of experiments described below was undertaken primarily to illustrate the effects of pore volume and particle size in the powder mixtures. Additional experiments were also undertaken to determine qualitatively the effects of the temperature of calcination and of several commonly used pelleting binders.

The Effect of Pore Volume and Average Pore Size

For this phase of the study, two powder mixtures containing essentially identical particle size distributions were prepared from each of the three lots of alumina described previously. (The cumulative pore volume distribution in the high-, low-, and medium- porosity aluminas is shown in the top section of Figure 3.) The concentrations of particles in the various screen fractions are listed in Table 1, mixtures 2 and 4 respectively. In mixture No. 2, no particles were smaller than 200 mesh; in mixture No. 4, approximately 35% by weight of the particles in the powder were smaller than 200 mesh.

The effect of pelleting pressure on the crushing strength and density of the pellets formed from the three aluminas is shown in Figure 4. The results of pore volume distribution measurements on representative samples prepared at high and low pelleting pressures are shown in the bottom sections of Figure 3. Both the pellet density and the pellet strength increased systematically with pelleting pressure. Pellets of lowest density (0.68 to 0.86 g/ml) and greatest strength (13 to 32 lb) were formed from the porous gel powders containing predominantly large pores and pellets of highest density (0.92 to 1.17 g/ml) and lowest strength (1 to 5 lb) were formed from the dense gel powders containing small pores only. As mentioned previously, the density of the pellets prepared from the aluminas of small (and intermediate) pore size was not overly affected by major changes in the particle size distribution in the powder mixtures, whereas appreciably lower pellet densities were observed the greater the concentration of the fines present in the mixtures prepared with the high-porosity alumina. This particular property of the high-porosity aluminas is described in detail in the next section. The results of the pore volume distribution measurements, shown in Figure 3, reflect the same general trend as the pellet density measurements, i.e., pellets formed from the highly porous gel contained a larger pore volume (in pores of larger diameter) than did pellets formed from the dense, small-pore gel.

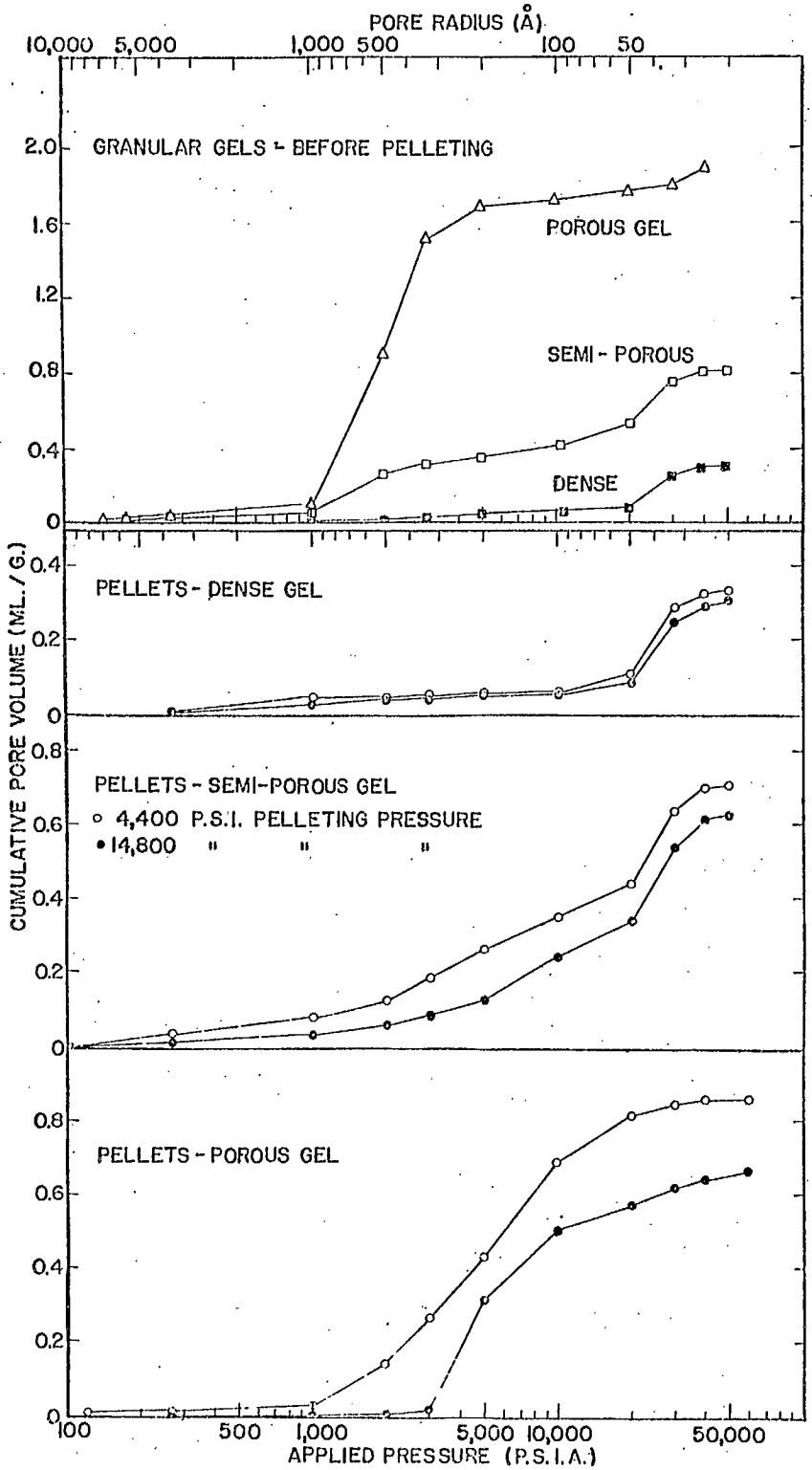

Figure 3 - The cumulative pore volume distribution in the aluminas before and after pelleting.

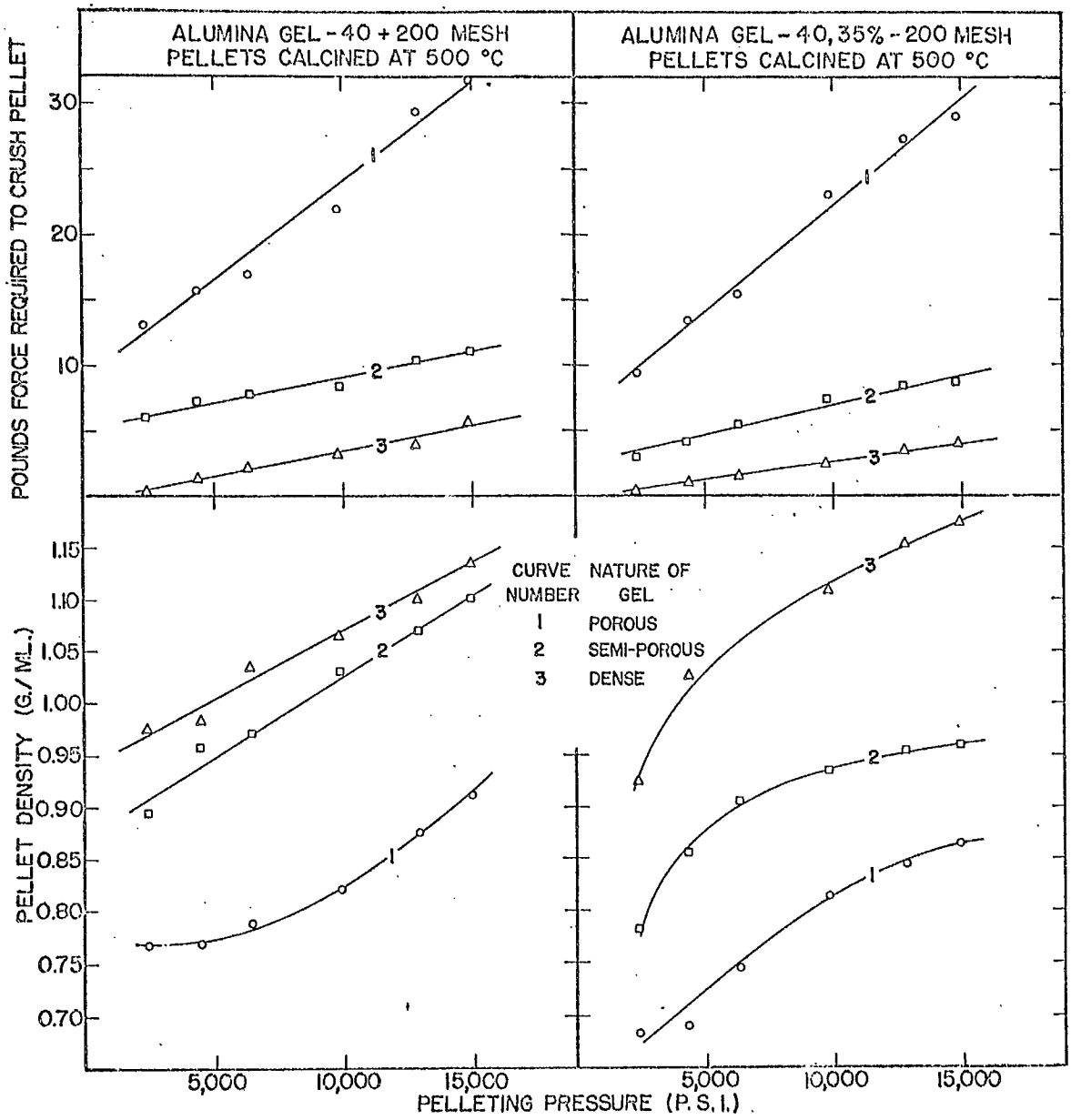

Figure 4 - The effect of pelleting pressure on the crushing strength and density of pellets formed from highly porous, semi-porous, and dense aluminas.

The effect of pore structure on the pelleting characteristics is even more apparent when the crushing strength is compared directly with pellet density (irrespective of the pressure used in the preparation of the pellet). A graph of this type is shown in Figure 5. It must be emphasized that the numerical results used in the preparation of Figures 4 and 5 are the same. The extra line shown in Figure 5 was obtained from tests made on a commercially available alumina manufactured by the Harshaw Chemical Company, Cleveland, Ohio, and is included only to help illustrate the difference between the conventional and the highly porous forms of alumina.

The Effect of the Particle Size Distribution

Preliminary experiments indicated that the most important aspect of the particle size distribution was the concentration of fines (particles smaller than 200 mesh) in the powder mixtures. Accordingly, pellets were made from a series of powder mixtures in which the relative proportions of the particle sizes larger than 200 mesh were held constant and the concentration of fines varied over a wide range. (See page 6 and Table 1 for details of the preparation of the powder mixtures.) Very little change in the pelleting characteristics with particle size occurred with the small-and intermediate-pore aluminas but considerable effect was observed with the high-porosity, large-pore alumina. The results of the experiments with the high-porosity alumina are summarized in Figures 6 and 7.

In general terms (all things being equal), the pellet density decreased dramatically as the percentage of fine particles increased in the powder mixtures. With no fine particles present, the density of the pellets formed at low pelleting pressures was 0.9 to 0.95 g/ml; with 10-17% fines, the density was 0.8 to 0.85 g/ml; and with 24 to 35% fines, the density at low pelleting pressures was in the region of 0.7 g/ml. With the decrease in density there was also a substantial decrease in the crushing strength ..... from between 15 and 20 pounds to between 8 and 12 pounds, depending on the pelleting pressure at which the comparison is made. Considering the low density of the pellet, however, the strength is remarkably good. The only other means of obtaining such low-density forms is by prilling or extruding the powder mixtures.

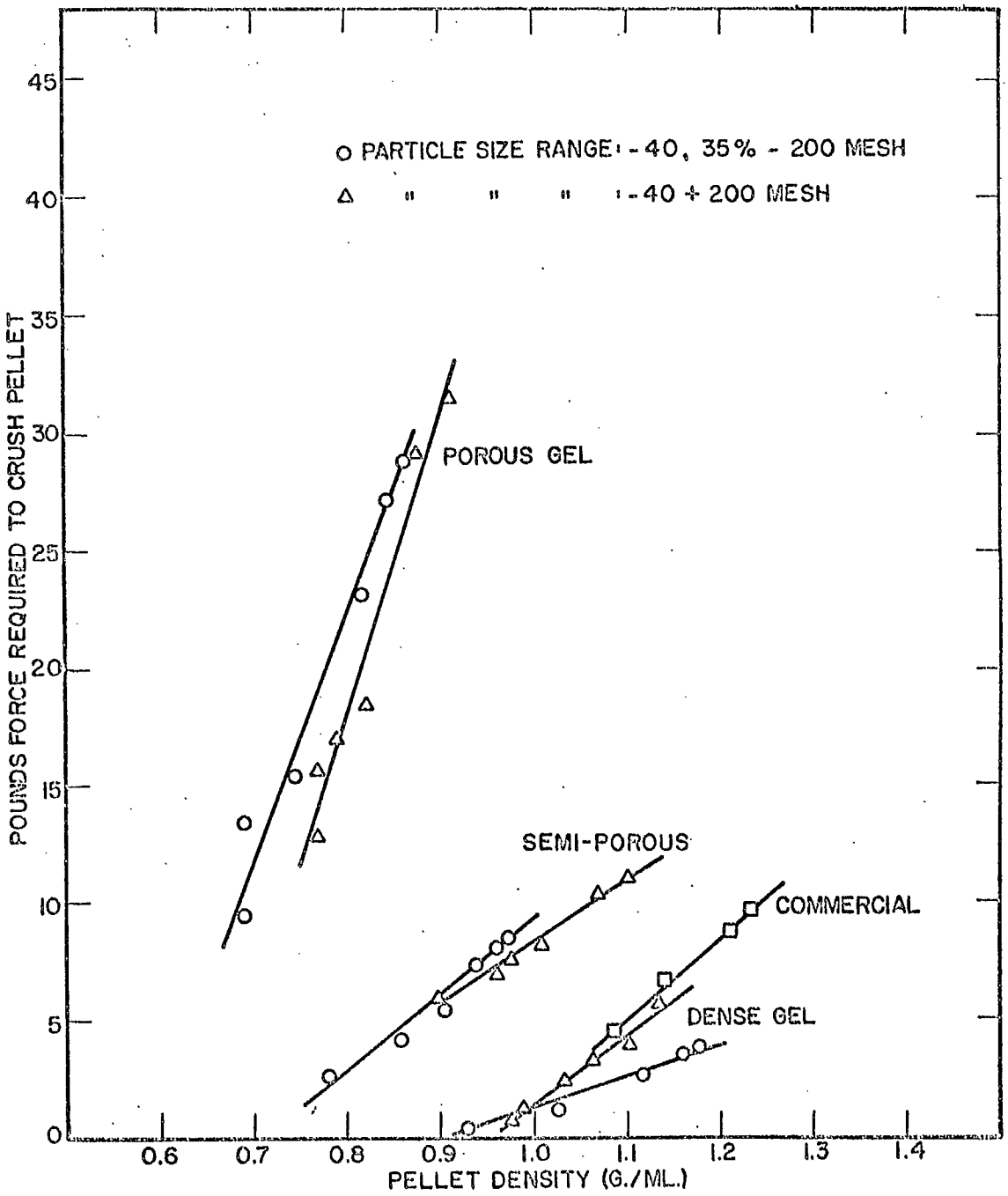

Figure 5 - A graph of the crushing strength as a function of the pellet density.

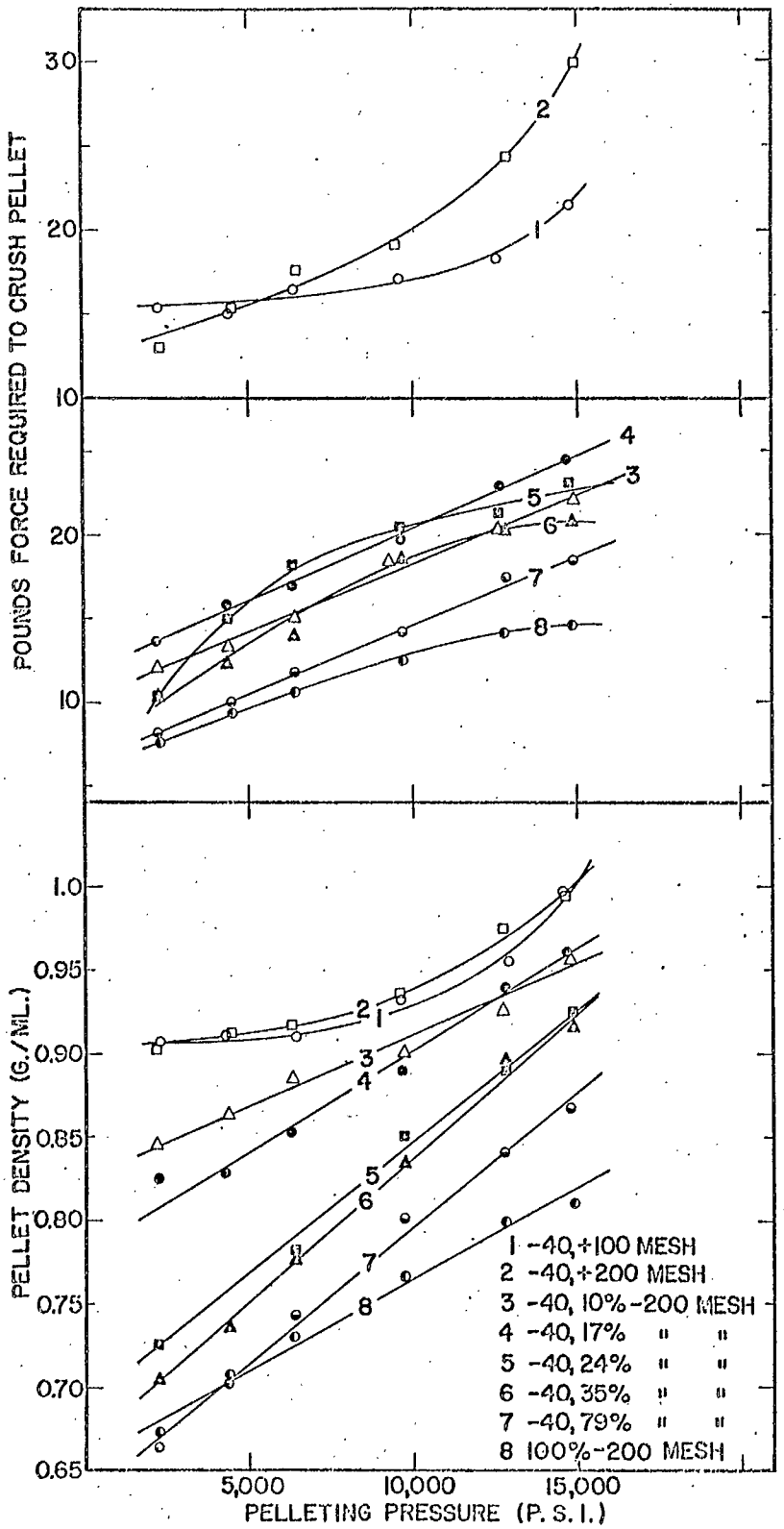

Figure 6 - The effect of the particle size distribution on the crushing strength and density of pellets formed from the highly porous alumina.

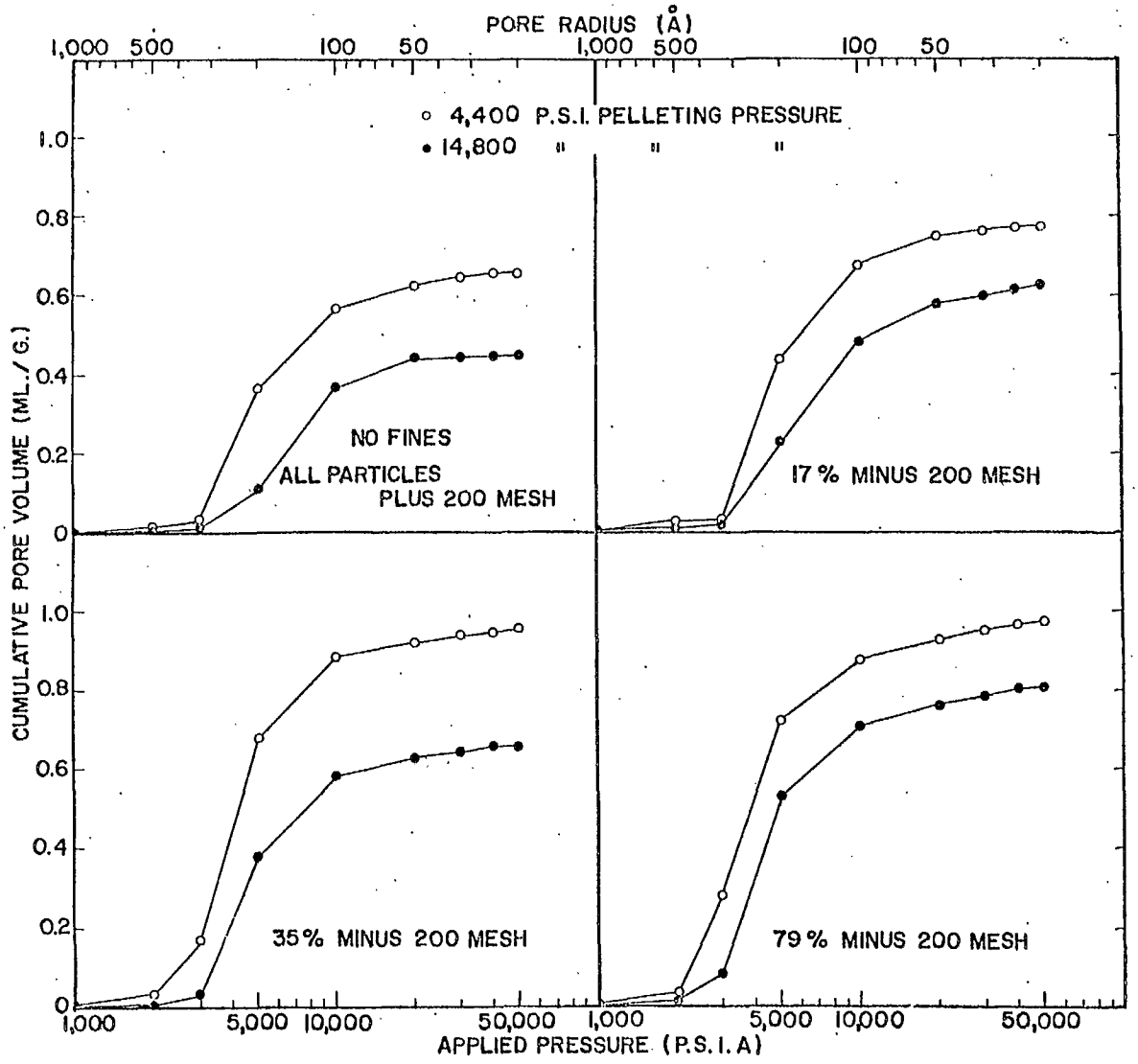

Figure 7 - The effect of the particle size distribution on the cumulative pore volume distribution in pellets formed from the highly porous alumina.

The crushing strength of most commercially available prills or extrudates is an order of magnitude below the 8 to 12-pound pellet strength described here.

As observed previously, the pellet density and crushing strength increased systematically with increasing pelleting pressure. In the case of mixtures 1 and 2, relatively small changes in the pellet density occurred with increasing pressure up to 10,000 psi, but thereafter the rate of change increased considerably. In addition, the results of the measurements of the pore volume distribution (shown in Figure 7) were in keeping with changes observed in the pellet density. At equal pelleting pressures, the pore volume contained in the pellets was generally greater the higher the concentration of fine particles in the powder mixtures.

The Effect of Secondary Variables

a) The temperature of calcination.

b) Pelleting binders.

The experiments undertaken to establish (qualitatively) the effects of secondary variables were made only with the highly porous form of the alumina. Pellets were prepared from two powder mixtures containing particle size distributions similar to mixtures 2 and 4 described in Table 1*. To determine the effect of the temperature of calcination, individual lots of pellet were made from each powder mixture at selected pressures in the range 3000 to 15,000 psi and calcined overnight at 300, 400, 600, and 700°C. To determine the effect of pelleting binders, three commonly used compounds--a) stearic acid, b) a polyethylene glycol of average molecular weight 6000, and c) graphite--were ground to pass a 200-mesh screen and added in varying amounts up to 6% by weight to portions of the powder mixtures, pelleted at pressures from 3000 to 15,000 psi, then calcined overnight at 500°C to remove the binder. The results of density and crushing strength measurements are shown in Figures 8, 9 and 10.

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* Mixture No. 2: all particles present larger than 200 mesh.
Mixture No. 4: 35% of particles present smaller than 200 mesh.

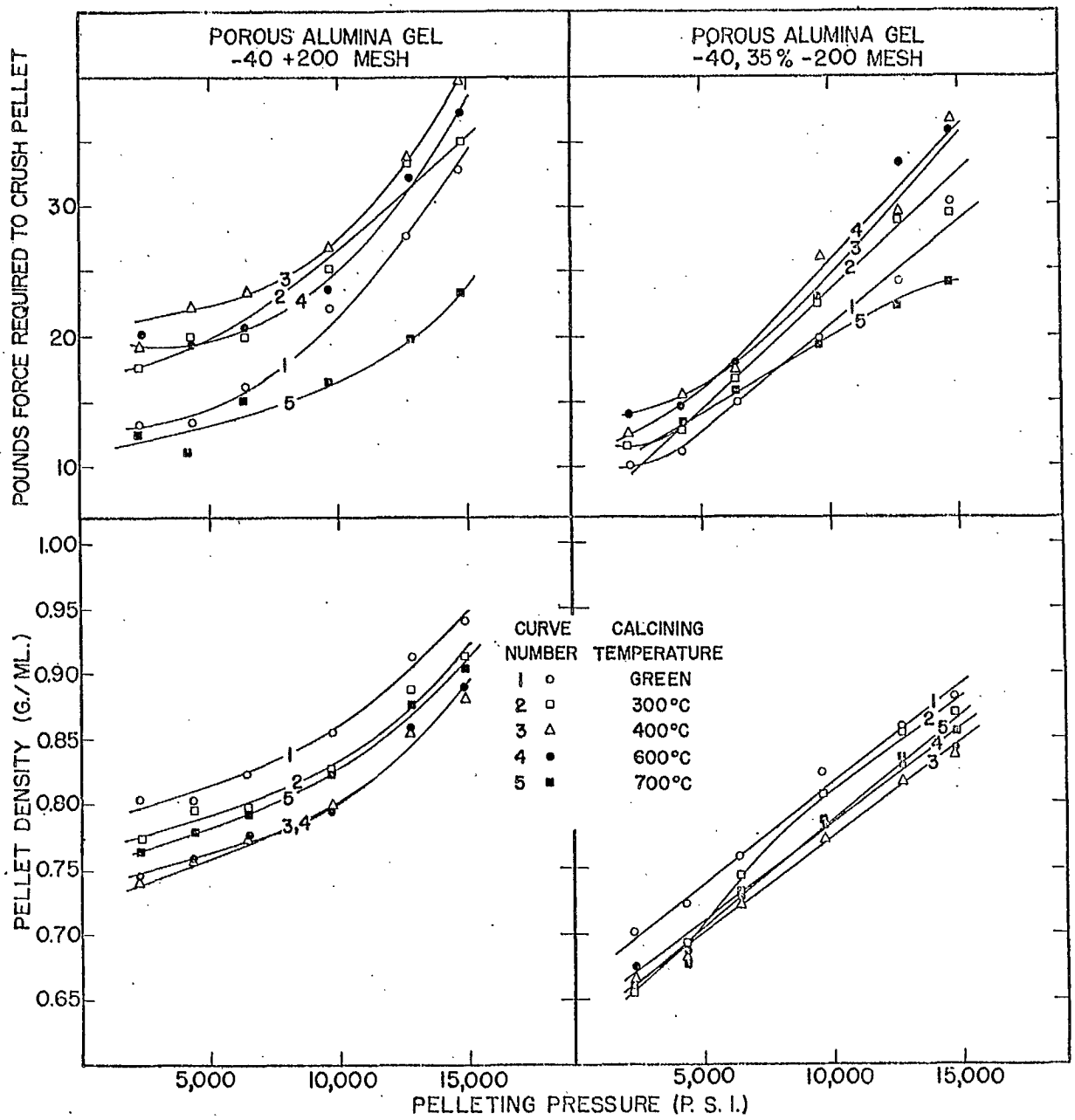

Figure 8 - The effect of the calcining temperature on the crushing strength and density of pellets formed from the highly porous alumina.

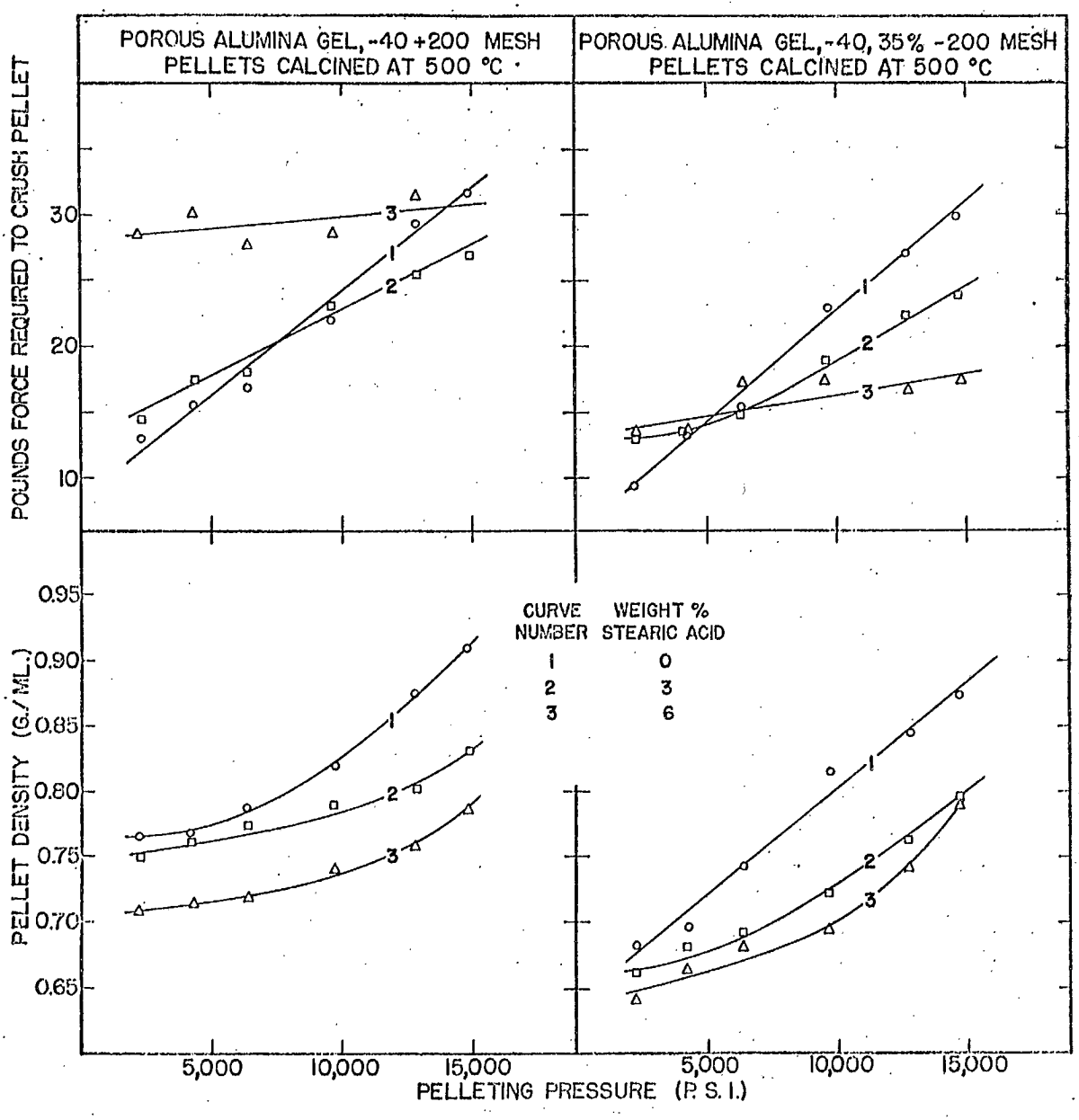

Figure 9 - The effect of stearic acid on the crushing strength and density of pellets formed from the highly porous alumina.

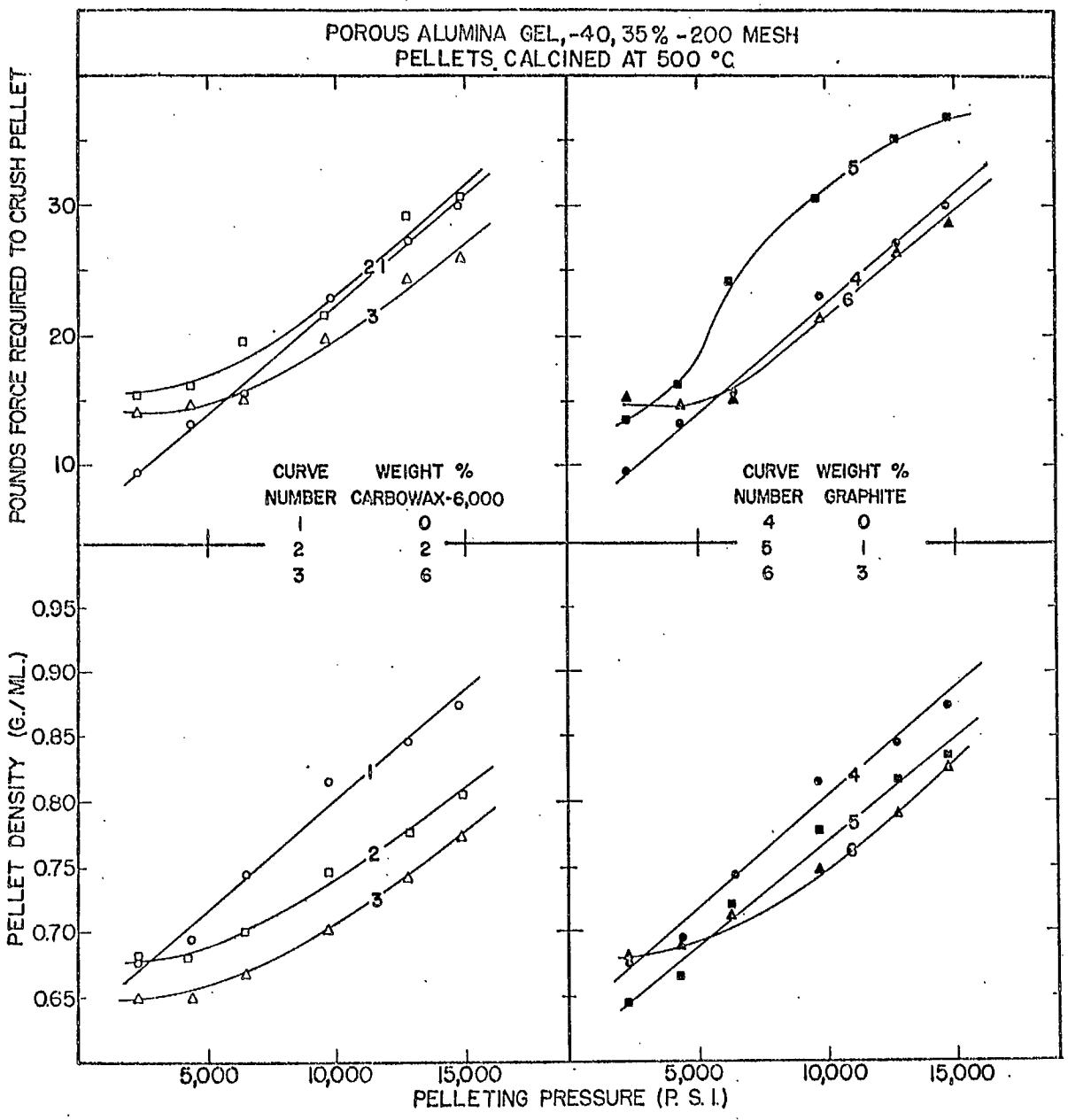

Figure 10 - The effect of polyethylene glycol and graphite on the crushing strength and density of pellets formed from highly porous alumina.

The pellets appear to be quite stable at all calcination temperatures up to 600°C. A slight decrease in pellet density was observed as the temperature of calcination was increased from 300 to 600°C (Figure 8), together with a very slight increase in pellet strength. The effects were greater with the pellets formed from the powder mixtures containing only the 40 to 200-mesh particles than with the mixture containing 35% minus 200-mesh particles. There was a more pronounced change in the region of 700°C ..... pellets calcined at 700°C were measurably weaker and more dense. Alumina gel of high surface area is known to sinter at elevated temperatures (4), and the changes observed at 700°C can most likely be attributed to this factor.

The results obtained with various binder materials were most unpredictable ..... and unexplainable. For example, pellets formed at equal pressures were of lower density the greater the concentration of stearic acid (see Figure 9). The effect was more marked at higher pelleting pressures than at low pressures and was generally independent of the particle size distribution. On the other hand, the crushing strength was found to be highly dependent on the particle size distribution in the powder mixture. In the case of the pellets made from the mixtures containing no small particles, a high concentration of stearic acid increased the crushing strength at low pressures but had comparatively little effect at high pelleting pressures. The strength of pellets made from the powder mixture containing 35% minus 200-mesh particles decreased markedly at high pressures as the concentration of stearic acid was increased. The system obviously becomes very complicated when pelleting binders are used, and no worthwhile comment seems possible at this time.

DISCUSSION

In qualitative terms, the main reason why the pellets made with the highly porous gels are stronger, and more porous, than those made with the dense gels is believed to be that the skeletal structure of the granules in the powder mixtures is much weaker. The difference in strength is readily apparent as one grinds the samples in preparation for pelleting. The dense-gel particles are hard and brittle, and require a ball mill treatment to reduce particle size. The highly porous gels are very much softer and can be broken to 200 mesh easily by using one's fingers or a wooden rolling pin. From the standpoint of the powder, pelleting is a devastating process involving the application of high pressures in the range from 2500 to 40,000 psi. The strength of the pellet is attributed to, and proportional to, the number and area of the contact points (interlocks) among the particles. Structurally weak particles will crush together easily in the course of pelleting, resulting in the formation of many interlocks and a strong pellet. Hard, brittle particles, on the other hand, will tend to shatter to a greater degree in the pelleting process, resulting in the formation of fewer interlocks and a weaker pellet.

The results of the pore volume distribution measurements shown in Figures 3 and 7 are offered as evidence in support of the above mechanism. Very little change is observed in the pore size distribution in pelleting the dense and semi-porous gels ..... compare the distribution curves shown in Figure 3 for the granular gels and the pellets. However, in the case of the highly porous gels, pelleting reduces the size of the pores present. The extent of change in the pore size depends to a large degree on the pelleting pressure. In the original granules of the highly porous gel, the majority of the pores occurred in the range 1000 to 400 Å radius. At low pelleting pressures the pore sizes present in the pellet ranged from 1000 to 50 Å radius; at high pelleting pressures the pore sizes remaining ranged from 500 to 50 Å radius.

The "stronger pellet because of the weaker particle" concept can also be extended to explain why exceptionally low-density (and strong) pellets can be formed from the highly porous gels by increasing the proportion of small-size particles in the powder mixtures. In a well-mixed powder sample, the small particles occupy a substantial part of the interstitial spaces between the larger particles. The small particles will support the larger particles at the time of pelleting, resulting in less actual crushing of the pore volume inherent in the gel granule yet increasing the number and area of contact points providing the strength. In the case of the dense gel, the smaller particles are as hard and brittle as the larger particles and probably shatter in a way little different from the situation where the fines are not initially present.

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