Deposition of Particulate Materials into Confined Spaces
— New Tester Development and Experimental Results —

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Abstract

The filling of particulate material into confined spaces such as dies (typical volume a few cubic centimeters) presents problems that affect the quality of the final product. Some researchers have studied the effects of filling method on fundamental particulate properties and load distribution in large storage systems such as bins and silos. However, there is a lack of quantitative analysis of the process of deposition of particulates into small storage systems such as dies. A low-cost tester capable of acquiring real-time data has been developed for experimental analysis of the process of filling of dies. A literature review and preliminary study of the effect of die filling methods (funnel fill and sieve fill) impact on spatial fill distribution (for spray-dried Alumina) in a cylindrical die have been carried out. Cumulative mass profile plots show that the load cells record a flatter profile in the case of sieve fill than the funnel fill case. This suggests a more uniform powder fill for the sieve filling method compared to the funnel fill method.

1. Introduction

A wide variety of products such as feed pellets, tablets, tool inserts, electronic components and automobile parts are produced by subjecting a fixed mass or volume of dry cohesive particulate material, poured into a die, to high pressures. This manufacturing technique is referred to as compaction or pelletization and the pressed part is referred to as a compact. The quality of the product made by pelletizing is recognized to be dependent upon many factors such as intrinsic material properties, particulate material properties, nature of applied load, and die geometry. Many pelletization defects such as lamination, encapspping, and stress cracking etc. are caused by anisotropic compaction of the particulate material. It is further recognized by researchers that the anisotropic compaction, among other factors, is attributable to non-uniform pre-compaction fill-density of the particulate material in the die. Filling (deposition) of powders in confined spaces (e.g., dies) is the required first step in the process of manufacture of pressed parts/products. Since errors get compounded in any manufacturing process, it stands to reason that ensuring a pre-compaction uniform particulate deposition in the die or mold would be an effective method of ensuring and enhancing quality of the pressed compacts.

A review of literature revealed that researchers have studied the effect of filling methods on fundamental particulate properties and load distribution in large storage systems but there exists a significant lack of qualitative and quantitative analysis of the process of die filling vis-à-vis particulate material's pre-compaction density distribution within the die volume. This study is a systematic attempt to deal with the issue of effect of die filling methods and other related factors such as die cross-section, die aspect ratio, and rate of die filling on the spatial, pre-compaction powder (fill) density distribution within the die volume. This is a work-in-progress and only preliminary results of the newly developed tester’s capability to determine the effect of die fill method is discussed herein.

2. Literature Review

The literature review is divided into two sections. The first section deals with the review of literature pertinent to die filling. In the second section, literature review about the determination of in-situ spatial density distribution is presented.

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2.1 Die filling:

Molenda et al. (1994) have reported that the spatial arrangement of individual wheat grain kernels, forming a granular bed using three different fill methods, significantly influenced the loads on the walls and the radial distribution of vertical pressure on the floor of the flat-bottom model bin. The three methods of bin filling used by Molenda et al. (1994) are shown schematically in Figure 1. The filling methods were: central, circumferential, and sprinkle. Measured data showed that the highest mean vertical pressure on the floor was caused by the sprinkle filling method. Circumferential filling of the smooth wall container resulted in the highest vertical pressure at the center of the container, while the highest pressure for central filling was on the second ring from the center. For the rough wall and all filling methods, the minimum vertical pressure was observed on the ring second from the wall. An increase in the vertical pressure on the ring nearest to the wall was observed for all tests except for the smooth wall bin and central filling where the pressure on the ring nearest to the wall decreased.

Moysey (1984) investigated the effect of the use of grain spreaders on the friction properties of grains and on bin wall pressures. Two filling methods were employed — sprinkle filling (achieved using a grain spreader) and stream filling (grains flowing in a stream into the bin). For both bins, sprinkle filling resulted in lateral pressures that were appreciably lower than stream filling. Sprinkle filling also resulted in a 7-8% higher bulk density than stream filling. In the same study, Moysey has also shown that in shear box tests to determine the internal friction angle in some cereal grains, the method of filling the box has a significant effect on the bulk density of the sample and on the amount of dilation which occurs during shear.

Moriyama et al. (1985), measured the wall pressure ratio after filling and during discharge, with the pressure measurements taken at the hopper gate zone near the transition in the bin for two different filling methods, central and peripheral feeding, and for six different types of bulk solids. They found that the wall pressure ratio was different for the two different filling methods (pressure ratios were generally higher for central fill vs. peripheral fill) and was also affected by different hopper angles. That the method of bin filling influences the pressure distribution has also been reported by Aoki (1976), Nielson (1983), Munch-Anderson et al. (1990), and Kwade et al. (1994).

Large capacity reactors with packed bed of catalysts find important applications in the chemical industry. One of the problems associated with such reactors is the local preheating of the catalyst, an exothermic reaction, preferentially occurring at the inhomogeneities of the packed bed structure. The packed bed structure is a function of the method of filling the reactor with the catalyst. Since local preheating has an adverse effect on reactor efficiency, researchers have investigated various methods of filling reactors with catalysts. Smid et al. (1993) have studied the effect of three different filling methods on the bulk density of the resultant packed bed structure. The three filling methods used were: the free pouring method, which is characterized by the angle of repose of the material and two so-called "rainy" filling methods with horizontal top surfaces which were implemented with a special distributing grid, located either close to bed surface or higher up. It was found that a more homogenous distribution of the catalyst was obtained in the case of the "rainy" filling method as compared to the free pouring method. Several methods of producing catalyst layers with high bulk densities have been patented of which Johnson et al. (1976), Baillie (1977), and Cermak et al. (1989) are but three examples.

Bocchini (1987) has conducted experiments to try to establish the influence of small die openings on the powder fill bulk density. Using rectangular dies of various widths and copper and mixed elemental bronze powder, Bocchini showed that filling bulk density decreases with decreasing die opening. Bocchini proposes that the decrease of filling density caused by decrease in die thickness can be explained by introducing the concept of boundary layers existing at the die walls.

Faikin et al. (1976) have studied the influence of the rate of die filling on the bulk density of the powder fill. They varied the intensity of filling the same die with powder by employing hoppers with different discharge orifice diameters using different powders. They determined that with increase in pouring rate, the bulk density of the powder fill falls to a certain limit, beyond which further increase in filling inten-
sity produces no further decrease in density, and, conversely, decreasing the intensity of filling increases the bulk density of the powder in the die. The variation in pouring intensity was found to change powder fill density by 7-10%. They explain this phenomenon by observing that at a sufficiently low intensity of powder filling, particles dropping onto the free surface of the powder can move unhindered to sites corresponding to minimum potential energy, as a result of which their arrangement will approximate one of the regular systems of stacking and that by enabling particles to perform free, optimum movements in the course of filling, a decrease in powder pouring intensity reduces the probability of bridge formation or even completely prevents it and consequently increases the density of powder fill.

Similar results have been obtained by Readey et al. (1995) in their study on the pressure-compaction response of a spray-dried, 94% alumina powder containing a polymeric binder. They have reported that fill bulk density is a strong function of both die diameter and aspect ratio (compact thickness-to-die diameter).

All the studies reviewed above are those in which researchers have investigated the effects of different particulate filling methods in grain bins and hoppers, formation of catalyst beds, and the material discharge characteristics from model bins and hoppers. No study could be located in the literature that expressly addresses the issue of experimental analysis of the process of deposition of particulate materials into confined spaces achieved by different deposition methods vis-à-vis the fill density distribution.

2.2 Density distribution measurement:

Smid et al. (1993) subjected a filled packed catalyst bed to the passage of gamma radiation emanating from an external radioactive source located on one side of the reactor model. After passing through the bed, attenuation of radiation was detected by a scintillation probe situated outside the second wall. This so-called radiogaging technique is based on the assumption that the attenuation of a beam of gamma photons is proportional to the material’s bulk density.

A similar, and more refined technique, is Computerized Axial Tomography (CAT) scanning using x-rays (wavelength 10^-4 to 10 nm). In x-ray CAT scanning, a source delivers a series of short pulses of x-ray radiation as it and an electronic detector are rotated around the object being tested. The attenuated x-ray responses of the detector are fed to a computer that analyzes and integrates the x-ray data from the numerous scans to construct a detailed cross-sectional image of the object. The most well known and wide spread use of CAT scanning is for medical purposes for scanning of the human body. Apart from its application in the medical field, CAT scanning has been found in many other areas with most of the non-medical application of CAT scanning being in the areas of petroleum engineering where extensive analysis of porous media is required. For example, Coshell et al. (1994), have successfully imaged, by x-ray CAT scan, oil shales in Australia. Phogat et al. (1991), have applied CAT scanning to dual-source gamma-ray attenuation in soil columns to determine nondestructively the spatial distribution of volumetric water content and bulk density. Tollner et al. (1992), have used x-ray CAT scanning to image interior regions of ‘Red Delicious’ apples under varying moisture and, to a limited extent, density states. Kantzas (1994) has used x-ray CAT scanning to study fluidized and trickle beds in chemical reactors using glass beads and nitrogen.

Process Tomography is a technique very much similar to CAT scanning except for the spatial relationship between the object under investigation and the source and detector array. In CAT scanning, the test object is kept stationary and the source and detector array are in motion. In Process Tomography, the test object is in motion and the source and detector array are kept stationary. Similar to CAT scanning, in Process Tomography, the basic idea is to install a number of sensors around the pipe or vessel to be imaged. Although the applications of Process Tomography lie in analysis of flowing mass, it is conceivable that if the stationary particulate mass in a die or mold were analyzed by a source and detector array being moved along the outside of the die cavity, it might be possible to analyze the internal structure, i.e., mass distribution, of the particulate deposited in the die. Toward this end, literature review in the area of Process Tomography was also carried out.

Hosseini-Ashrafi and Tuzun (1993) have worked on the tomographic study of voidage profiles in axially symmetric granular flows. The state-of-the-art literature about process tomography has been compiled by Williams and Beck (1995).

Kondoh et al. (1996) carried out a study aimed at clarifying the powder deposition process in filling the die cavity. They observed the deposition process with the help of a videographic imaging apparatus. They have shown that particle size segregation in filling occurs independently of the shoe speed and die cavity shape. Further, coarse particles gather in places where powder flows along the angle of repose, while
fine particles gather in places where empty spaces within the die cavity are initially produced and which gradually get filled.

The ability to measure the in-situ density distribution within the particulate mass inside the die volume is of paramount importance to the objectives of the proposed research work. The literature review was carried out to locate instances where researchers might have carried out such an in-situ density distribution measurement of particulates in dies or molds. No such study could be found but the literature review provided a starting point with which to evaluate the different techniques for the purposes of this study.

3. Methodology

Based upon findings from the literature review, two potential techniques, x-ray CAT scanning and MRI were investigated. After considering the pros and cons of these and other lesser known techniques such as vacuum assisted epoxy impregnation, radiogaging and process tomography, a new tester — the real-time spatial particulate mass deposition tester — has been invented (for which a provisional patent has been obtained, Dhanoa and Puri, 1997), to determine the in-situ pre-compaction particulate density distribution. Details about the new real-time spatial particulate mass deposition tester that has been invented are given in the following sections.

3.1 Real-time Cumulative Mass Distribution Determination Load Cell Tester

Principle of Operation

The tester is based on the principle that an anisotropic fill density distribution would result in an uneven particulate vertical pressure distribution on the inside surface at the bottom of the die. Further, this uneven vertical pressure distribution can be detected by placing multiple, sensitive enough load cells on the inside surface at the bottom of the die and analyzing the output signals acquired from the load cells using any suitable data acquisition system.

Construction and Operation

The tester consists of a tiered arrangement of load cells such that the load cell load application points (tops of the custom fitted buttons) are just about flush with the uppermost plate top surface as shown in Figure 2. The dimensions of the die used in the tests are also shown in Figure 2. The die was cut from a clear acrylic pipe. The load range of the load cells is 0-4 grams. Combined with a state-of-the-art data acquisition system (model #AT-AI-16XE-10 by National Instruments Corp.), resolution of 0.002 gram can be obtained from the load cells. Figure 3 shows the schematic of the operation of the tester for the two filling methods used in this study. The tester is placed on a level horizontal surface. A thin plastic film (such as those used in wrapping foods) is placed over the tester to isolate the fill particulate material from the load cells. The open-ended die is then placed over the tester. The output voltage signals from the five load cells during the die filling process are recorded using the data acquisition system. The resultant data captured by the data acquisition system can then be subjected to statistical analysis to determine the fill distribution. The vibratory feeder (model #F-T01-A by FMC Corp.) was used in this study to obtain precise control over the rate of particulate flow into the die as well as to achieve different rates of die fill to analyze the effect of flow rate on the fill density distribution as mentioned earlier in the introduction section.
4. Results

The first phase of experimental analysis was to carry out proof-of-concept verification tests of the designed and fabricated real-time spatial cumulative mass distribution determination load cell tester (tester). The next step was to carry out the preliminary experimental analysis. Since this is still a work-in-progress, results and qualitative data interpretation for only one powder, spray-dried Alumina ($d_{50}=100$ microns, 4% PolyVinyl Alcohol binder), and one fill rate (18 g/s) are reported here. Each test was repeated five times. The powder was allowed to fall into the die from a fixed height of 300 mm.

4.1 Proof-of-concept verification of test method

Due to its inherent nature whereby a liquid exerts uniform pressure in all directions at any depth, water was considered to be an acceptable material with which to determine the functioning of the tester. The die was filled with water to various depths (quarter, half, three-fourths and full) and the output from a load cells was recorded. A waterproof seal between the die wall bottom and the plastic film was obtained by applying a layer of high vacuum silicone grease (made by Dow Corning) between the two. The test results are shown in Figure 4. The plots show the load cell readings at any depth are within a ±5% range of the average reading from the five load cells at that fill depth. Also, the average readings at different depths are linear ($r^2=0.99$) with negligible intercept (~0.0032 g) thus demonstrating the location independence of the load cells and linearity with fill height. Calculations showed that the ratio of measured to theoretical values for the load at different depths of fill ranged from 2.14 to 2.19. As expected, measured values were higher than theoretical values. This is most likely due to the fact that the load cell extensions (2 mm diameter) are seated in a hole which is covered with a thin plastic wrap which would contribute to increased effective area (compared to the load cell extension’s cross-sectional area) and hence the measured load.

4.2 Experimental results

Figure 5 shows typical data for the case where the die was filled by the funnel fill method. The figure shows plots of the real-time cumulative mass vs. time data captured at the rate of 10 per second at each of five load cell locations as shown in Figure 2. From the plots it can be seen that maximum cumulative mass at the end of the filling process is recorded at the center of the die (load cell #3). The two load cells on either
side (±0.46r, load cell #2 and #4) recorded the next two highest readings. The two load cells near the walls of the die (±0.92r, load cell #1 and #2) recorded the lowest readings. In the vicinity of the die wall, the powder wall interaction and wall friction is expected to reduce the loads at the bottom of the die. This finding is consistent with Bochinni's (1987) and others' observations.

Figure 6 shows plots of cumulative mass profile across the load cell locations at the end of successively increasing time periods from the start of the die fill process. The load cells indicate a powder fill, and hence density, distribution that decreases radially outwards from the center of the die towards the walls.

Figure 7 shows a typical dataset for the case of sieve filling method of Alumina. Figure 8 shows plots of cumulative mass profile across the load cell locations at the end of successively increasing time periods from the start of the die fill process. Again, similar observation can be made regarding the particulate deposition as in the funnel fill case. The load cells near the walls record less load than the three in the middle. A comparison of data in Figures 6 and 8 shows that the load cells record a more flatter profile in the case of sieve fill than the funnel fill case. This suggests a more uniform powder fill for the sieve filling method compared to the funnel fill method.

Data in Figures 5 and 6 include the effect of heap formation as the surcharge on top of the deposited material. To discount the effect of heap formation, Figure 9 shows the plot of load cells readings for the two fill methods at the end of the die fill process wherein the loadcell readings for the funnel fill case have been reduced in proportion of the surcharge height above the load cell location. The surcharge corresponds to the heap that had an angle of 27° (measured from digitized video still frames) for the funnel fill method. For the sieve fill method, the free surface was nearly horizontal. In Figure 9, the slopes of the profile plots on either side of the center of the die (on both sides of load cell #3) are shown. The slope values shown in Figure 9 were obtained by regressing the three data points in each case. It can be seen that the sieve fill plots are more flatter than
the funnel fill case thus reflecting the ability of the developed tester to distinguish between the two filling methods. For both methods, the powder fill is asymmetric as indicated by the slope values in Figure 9. This asymmetry is expected to influence the subsequent processing of the powder although to a lesser extent in the sieve filling case than the funnel fill case.

5. Future Activities

Based upon the results and experiences gained from this preliminary experimental analysis, a comprehensive design of experiments has been designed. In addition to spray-dried Alumina, four more powders will be tested. These are, 1) microcrystalline cellulose, 2) wheat flour, 3) silicon nitride powder, and 4) glass beads. In addition to the die filling factor, all powders will be tested in a randomized sequence for other factors mentioned earlier in the introduction section, namely, die cross-section, die aspect ratio, and rate of die filling. Statistical analysis will then be applied to the collected data to determine whether any of the factors under consideration significantly effect spatial die fill density distribution. Validation of the tester will be done by the CAT Scan experimental analysis technique.

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References

1) Aoki R. 1976. Stresses of powders and granular materials in bins and hoppers. Theoretical and Applied Mechanics 26:9-24.
2) Baillie L.A. 1977. US-Pat. 4 039 431, Atlantic Richfield Comp.
3) Bocchini G.F. 1987. Influence of small die width on filling and compacting densities. Powder Metallurgy 30(4): 261-266
4) Cermak J., J. Novosad, J Smid, O.P. Klenov and V.S. Lachmotskov, J.S. Matros. 1989. Czech Pat. 257 561.
5) Cosnell L., R.G. McIver, R. Chang. 1994. X-ray computed tomography of Australian oil shales: nondestructive visualization and density determination. Fuel 73(8): 1317-1321.
6) Dhanoa P.S., V.M. Puri. 1997. Provisional Serial #60/054,981.
7) Falkin V.I., V.P. Levin, Y.N. Gribenyuk. 1976. Influence of the rate of die filling on the density of the powder. Soviet Powder Metallurgy and Metal Ceramics 15(8): 590-592.
8) Hosseini-Ashrafi M.E. and U. Tuzun. 1993. Tomographic study of voidage profiles in axially symmetric granular flows. Chemical Engineering Science 48(1): 53-57
9) Johnson J.A. and H.R. Wesler. 1976. US-Pat. 3 972 686, Universal Oil Products Comp.
10) Kanzas A. Computation of hold-ups in fluidized and trickle beds by computer assisted tomography. AIChE Journal 40(7): 1254-1261.
11) Kondoh M. And S. Takemoto. 1996. Visualization of powder behaviour for gravity filling. Toyota Central R&D Labs, Inc.
12) Kwade, A., D. Schulze and J. Schwedes. 1994. Determination of the stress ratio in uniaxial compression tests, Part 1. Powder Handling and Processing. 6(1): 61-65.
13) Molenda M., J. Horabik and I.J. Ross. 1994. Effect of filling method on load distribution in model grain bins. ASAE Paper no. 94-4517. St. Joseph, MI.
14) Moriyama R. and J Genji. 1985. Effect of filling methods on the wall pressure near the transition in a bin. Bulk Solids Handling 5(3): 603-609.
15) Moysey E.B. 1984. The effect of grain spreaders on grain friction and bin wall pressures. J. Agric. Engng. Res. 30: 149-156.
16) Munch-Anderson, J. and J. Nielsen. 1990. Pressures in slender grain silos. Measurements in three silos of different sizes. In Proc. 2nd European Symposium on the stress and strain behavior of particulate solids — Silo Stresses, Praha, Czechoslovakia.
17) Nielsen, J. 1983. Load distribution in silos influenced by anisotropic grain behavior. International Conference on Bulk materials Storage, Handling and Transportation, Newcastle, NSW, Australia. August 1983.
18) Phogat V.K., L.A.G. Aylmore and R.D. Schuller. 1991. Simultaneous measurement of the spatial distribution of soil water content and bulk density. Soil science Society of America Journal 55(4) 908-915.
20) Smid J., P.V. Xuan and J. Thyn. 1993. Effect of filling method on the packing distribution of a catalyst bed. Chem. Eng. Technol. 16: 114-118.

21) Tollner E.W., Y.C. Hung, B.L. Upchurch and S.E. Prussia. 1992. Relating x-ray absorption to density and water content in apples. Transactions of the ASAE 35(6): 1921-1928.

22) Williams R.A. and M.S. Beck. 1995. Process Tomography – Principles, Techniques and Applications. Butterworth Heinemann Ltd., Oxford.

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