Particle Size Segregation during Hand Packing of Coarse Granular Materials and Impacts on Local Pore-Scale Structure

I. Lebron* and D. A. Robinson

ABSTRACT

Soils and sediments consist of granular particles with an intricate network of pores between them. The structure and orientation of these pores will determine how the material transports fluids and contaminants. A common practice in soil science to simplify experiments and to achieve a homogeneous medium, against which to test transport equations, is to repack a quasi two-dimensional (2-D) Hele Shaw cell or a column. Soil is broken up and sieved to remove large particles that could cause anomalous measurements; then it is repacked into the column. However, this procedure destroys the natural structure and imparts a new structural arrangement. The material may appear to have similar bulk properties such as porosity and bulk density, but as we aim to demonstrate, the structural properties will be a function of the method used to repack, and it is unlikely that one can achieve a uniform distribution at the micro-scale. We present results of experiments using granular materials, demonstrating how mixtures of particles of different sizes segregate when poured, forming banded structures. The rate at which a material is poured will determine the uniformity of the packed sample.

We are in an age that has seen a tremendous growth in the number and variety of sensors available to probe earth materials compared with 50 yr ago. To develop and test new techniques as well as study phenomena at greater spatial and temporal resolution, laboratory methods such as column experiments and quasi 2-D Hele Shaw cells (slabs) are commonly used. The rush to test new equipment and describe new phenomena, as well as excessive reliance on automated systems, often takes precedence over the care needed to obtain a representatively packed granular material at the scale at which we want to work. Reference and textbooks dealing with soil physical methods often overlook the basic issue of how to achieve relatively homogeneous packing. In this paper we seek to demonstrate some of the pitfalls associated with packing a column of granular material by hand, which is by far the most common method used in research and commercial laboratories. We go on to demonstrate how relatively uniform packing can be achieved with a granular material.

The simple statement, “sand was carefully poured into the column to achieve a uniform homogeneous packing” hides many subtleties. Questions arise such as, how was the material poured? Was it poured quickly or slowly? Was it poured from high up or near the surface? Was it mixed after it was poured, and if so, how? All these questions appear trivial, and yet, they are in fact crucial to determining whether the packing of a column with a granular material is approaching homogeneity.

Homogeneity in a hand packed granular material is defined as having a uniform distribution of particle domains throughout the characteristic length of a macroscopic sample. A particle domain is the minimum amount of particles (much greater in size than the molecular scale) that exhibit macroscopic properties but on a much smaller scale than the characteristic length of the macroscopic sample (Torquato, 2001). The domain might be considered the basic unit of an “effective medium”. As an example, a cube of monosize material consisting of 40 particle diameters in its respective directions for all its dimensions might be considered a domain.

A literature survey shows that quasi 2-D Hele-Shaw cells, or slab, experiments are widely used to study 2-D flow phenomena of all kinds (Makse et al., 1997; Baxter et al., 1998; Wang et al., 2002; Friedman and Robinson, 2002). Although methods have been developed which claim to achieve uniform packing, such as the use of randomizing screens (Bauters et al., 1998), hand pouring still dominates as a method. This occurs partly because many labs lack more sophisticated equipment, but mostly because there is a lack of appreciation of the undesirable microstructural segregation phenomena.

Quasi 2-D vertical Hele Shaw cells or slabs have been particularly useful in the study of fluid fingering in sands. Instability of wetting fronts and finger formation in soil science has been studied for the last 50 yr (Hill, 1952; Raats, 1973; White et al., 1976; Bauters et al. 1998; Nieber et al., 2000), and it is here that the microstructure of the granular material is perhaps of most importance. Slab experiments provide a convenient way to study these phenomena experimentally, and great care has often been used to ensure uniform structure. Glass et al. (1989) presented fingering results for 2-D slabs that were reportedly packed with great care. They used randomizing screens to pack the sand and then checked the microstructural arrangement using a light transmission technique (Hoa, 1981). Such care and attention to detail can often be overlooked with transport experiments.

We begin by reviewing and describing some of the microstructural arrangements of particles that develop due to pouring. We then go on to present some new results that are pertinent to packing issues related to the study of transport phenomena. We finish by presenting a method that can be used to attain reasonable uniformity when packing a column by hand.

Abbreviations: 2-D, two-dimensional.
Table 1. Physical characteristics of the materials used in the study.

<table>
<thead>
<tr>
<th></th>
<th>Glass beads</th>
<th>Quartz grains</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle density, g cm$^{-3}$</td>
<td>2.5</td>
<td>2.65</td>
</tr>
<tr>
<td>Particle size classes, μm</td>
<td>800–900</td>
<td>1100–1000</td>
</tr>
<tr>
<td>Large</td>
<td>800–900</td>
<td>1100–1000</td>
</tr>
<tr>
<td>Small</td>
<td>300–400</td>
<td>250–100</td>
</tr>
</tbody>
</table>

MATERIALS AND METHODS

Granular Materials

Experiments were conducted using granular media in the form of glass beads and quartz sand. The glass beads were highly spherical soda-lime silicate glass beads (Fox Industries, Inc., Baltimore, MD). The quartz sand was high quality, graded silica sand (Norco, CA). Both materials were sieved to the appropriate size classes and were further characterized with a scanning electron microscope (AMRAY 3200, AMRAY Inc., Bedford, MA). The quantification of the grain size was performed using an image analysis software package (Princeton Gamma-Tech Inc., Princeton, NJ). The physical properties of the granular materials are displayed in Table 1, and micrographs of the materials used are displayed in Fig. 1. To differentiate visually between the small and large grains in the pouring experiments, both the small quartz grains and the small glass beads were colored with black permanent ink.

Quasi Two-Dimensional Vertical Hele-Shaw cell

A 2-D cell was constructed from Plexiglas and used in two of the three series of experiments (Fig. 2). The cell had a delivery slope with an angle of 30° that had an adjustable aperture so that the grain flow rate could be regulated. The cell was 3 cm wide and 30 cm long, and the drop height from the delivery slope aperture to the base was 10 cm. The base of the cell was roughened to create a nonslip surface. The materials were poured into the 2-D cell via the delivery slope (Fig. 2). The size ranges of the materials used in the experiments are presented in Table 1.

Experiments with Poured Granular Materials

Three separate series of experiments were run using binary mixtures of glass beads (spherical) and quartz sand grains (angular). The first series were designed to demonstrate segregation effects during the pouring of binary mixtures, the second to show how the pouring rate affects the grain size distribution in a pile formed from a binary mixture, and the third to demonstrate how to achieve homogeneous packing.

In the first group of experiments a binary mixture of glass spheres of 50% large and 50% small by mass was poured into the 2-D cell using the delivery slope to form a pile with a single slope. This procedure was also followed in subsequent experiments using quartz sand binary mixtures; however, in that case the binary mixtures were composed of 20, 40, and 60% small grains by mass with their respective proportions of large grains to make 100% mass fraction. The flow rate for the glass spheres was 2.4 g s$^{-1}$ and for the quartz sand was about 12 g s$^{-1}$. The velocities were determined using the time measured with a stopwatch for a known mass to be deposited completely.

In the second series of experiments, an 800-g mixture of glass spheres (47% large, 53% small by mass) was poured from a beaker by hand into the 2-D cell. The glass spheres were poured into the center of the 2-D cell through a funnel, forming a triangular pile with two slopes. The speed of pouring was altered from 50 g s$^{-1}$, roughly equivalent to what might be considered a normal pouring rate by hand, to 3.7 g s$^{-1}$, and finally 0.83 g s$^{-1}$. After each experiment the cell was photographed using a digital camera with 3.2 megapixel resolution.

The third experiment was performed in a 2-L beaker using sand, 50% large grains and 50% small grains by mass. The

---

1 Trade names are provided for the benefit of the reader and do not imply any endorsement by the USDA.
material, which was initially segregated, was stirred at different water contents for at least 1 min with a rod. The water was sprayed from the top and allowed to percolate by gravity. The amount of added water was quantified by weight. The effectiveness of the mixing process was recorded each time with the digital camera.

**Phenomena Observed in Poured Granular Material**

The 2-D cell is commonly used to visualize segregation phenomena with granular materials. Although segregation effects have been observed in the geosciences, especially in sedimentology (Bagnold, 1941; Carrigy, 1970; Statham 1974), there is a new thrust to obtain a better physical description of the phenomena at the grain scale, especially with the use of new image-based techniques. Soils and sediments are granular materials, and as such they are neither solid nor fluid but exhibit characteristics of both. For instance, they flow when poured and act like solids when stationary and confined. It is this somewhat intermediate character that makes granular materials so fascinating. It’s mostly since the last decade that the study of granular flow, avalanching, and packing has drawn substantial attention in the physics literature (Jaeger et al. 1989, 1997; Mehta, 1994; Liu et al., 1995; Wittmer et al. 1997; de Gennes, 1998; Koepp et al., 1998; Daerr and Douady, 1999; Herrmann, 1999; Makse, 2000; Ristow, 2000). In soil engineering the issue of homogeneous packing of soil has become more relevant with the use of soil as a capping material for waste sites. Methods using hoppers have been developed in attempts to achieve uniform repacking of soil in constructing engineered caps (Aguiby et al., 1996).

An area that has also caught much attention is the flow and avalanching behavior of granular materials (Jaeger et al., 1989; Boutreux et al., 1998; Daerr and Douady, 1999). This has many important industrial applications, including granular flow and clogging in grain silos or sand hoppers (Vanel et al., 2000). It also has the more obvious application in earth sciences for understanding landslides and sand dune formation (Bagnold, 1941; Makse, 2000). Many of the recent findings on granular flow have particular application for soils and sediments, especially when considering microstructural effects on transport phenomena.

The simplification of considering soils and sediments as homogeneous materials is often an assumption made when measuring and modeling transport properties of porous media. Many experiments are constructed using “uniformly packed sand” or “sieved, repacked soil” to achieve a homogeneous material. However, as we will demonstrate, the simple act of pouring sand into a container does not create a homogeneously packed material, and thus the initial assumption of a homogeneous medium is often invalid.

**RESULTS AND DISCUSSION**

**Stratification with Poured Binary Mixtures—Particle Size Effects with Glass Spheres**

Stratification occurs when binary mixtures or those with a broad particle size distribution are poured into the cell (Makse et al., 1997; Baxter et al., 1998). This was demonstrated using a 50:50 mixture of glass spheres (300 and 800 μm) poured at 2.4 g s⁻¹ via the delivery slope (Fig. 4). The upper figure is a photograph of the actual pile. The segregation lines appear faint due to poor contrast between the colors of the beads. However, a black and white embossed image is presented below and highlights the banding. Similar phenomena were presented by Makse et al. (1997), also for materials with differing shapes. Avalanching occurs along the surface of the pile, with periodic accumulation and collapse. As small grains land at the top of the pile they become trapped in the crevices formed by the larger grains. During each avalanche sequence the grains are sheared together, causing the larger grains to migrate to the zone of least shear strain (i.e., the surface) and the smaller grains to migrate toward the zone of greatest shear strain (i.e., the base of the flow) (Bagnold, 1954). This results in banding and a distribution of particle sizes with the smallest at the base grading up to the largest at the surface. These bands form a periodic structure with each subsequent avalanche event.
layering did not occur so clearly, except for the case shown in Fig. 5 (left). The piles contained most of the small grains close to the apex of the pile, indicating that the small particles, once they are poured onto the pile, get caught in the crevices formed by the bigger particles. When avalanching occurs, these small particles are easily caught in the crevices and tend not to avalanche all the way to the base of the pile. This phenomenon is attributable in part to shape and in part to the size ratio between the grains and the velocity of flow. Robinson and Friedman (2002) observed that when the mass fraction of the small particles is less than about one-third, or less than the porosity formed between the large particles, the small grains migrate or percolate through the pore space. This assumes that the “small” particles are sufficiently small to fit between the pores created by the large particles. The small grains percolate down through the pile, resulting in less banding and an accumulation of fine particles at the base of the pile. Figure 5 clearly demonstrates that simply pouring an initially mixed binary mixture into a container does not lead to a homogeneous mixture but quite the opposite, a strongly segregated mixture.

**Effect of Pouring Rate on Macroscopic Porosity**

An important aspect of pouring a granular material is the rate of pouring. A set of experiments was conducted to mimic different packing conditions that might be used in the laboratory. A mixture of glass spheres with a ratio of 47% small and 53% large by mass was hand poured into the 2-D cell at three different rates (Fig. 6). The photographs on the left are the actual images, with the small beads appearing darker. The diagrams on the right are negative images that help to enhance the view of the structural arrangement. The photographs demonstrate that as the rate of pouring is slowed (top to bottom) more banding occurs (Fig. 6c), rather than the accumulation of small beads under the pile apex (Fig. 6a). Figure 6a shows that when poured rapidly, small particles accumulate under the apex of the pile. The speed of delivery and the fact that there was no periodic avalanching means many small particles are trapped in the center of the poured pile. As we reduced the velocity of pouring, we observed an initial accumulation of big particles in the apex of the pile (Fig. 6b). This opposite effect to what was observed in...
Fig. 6a may be caused by particle segregation in the pouring container. Big particles tend to move to the fastest part of the granular flow despite the fact that initially the mixture was thoroughly mixed.

There has been some controversy in the literature about the effect of pouring rate on the stratification of granular materials. Makse et al. (1997) suggested that segregation occurs primarily because of the difference in shape between large and small grains. Baxter et al. (1998), however, demonstrated that this was not the case and identified rate as a crucial parameter. Some of the difficulties in comparing results from experiments performed by different authors are that some of the details of the experimental set up are missing or overlooked. The starting point of the pouring is very important, as grains in a hopper might segregate within the hopper due to the smaller grains percolating downwards toward the outflow. In the case of using a beaker as a pouring container, the small grains may again percolate downwards, congregating at the base of the beaker, which would tend to make them the last grains to be poured out. Baxter et al. (1998) used 20 kg of borax pentahydrate with a particle size distribution between 100 and 1400 μm and flow rates of 7 and 800 g s⁻¹. In their results, the fine particles were under the apex of the pile in both cases, both when the delivery speed was 800 g s⁻¹ (no stratification) and at 7 g s⁻¹ (with stratification). This segregation might have been due to the particle size distribution and the shape of the granular material.

A difference in, or distribution of, particle sizes is necessary for nonpercolating granular materials to produce stratification (i.e., materials with a broad particle size distribution or binary mixtures with >35% small grains). Velocity of flow is a major factor. It appears that both grain shape and the difference in grain shape between the particles of different sizes are also important. Intuitively, shape should be important, as the more rounded the grains are, the more easily they will roll past one another and shear together. Since this shearing appears to be the principal mechanical mechanism to account for segregation (Bagnold, 1954), the grain shape and its resistance to flow are likely to affect the phenomena.

**Impact of Packing on Bulk and Local Properties**

All the information presented above indicates that one needs to be aware that bulk properties may not be representative of the properties in different locations within the repacked cell. Assuming that a heterogeneous media is homogeneous will lead to erroneous
conclusions when studying phenomena at the sample scale. To evaluate the effect that the different packing had on the physical properties of the poured material we proceeded to measure the bulk density and porosity in three locations of the 2-D slab.

Following the pouring of the piles in Fig. 6a and 6c, the piles were divided into Sections 1, 2, and 3, corresponding with the lines on Fig. 6. The beads were removed from the cell, weighed and sieved to determine the ratio of small grains to large in the respective sections. The bulk density and porosity were determined by measuring the volume that they had occupied. The ratio of small beads relative to large on a mass basis is presented in Fig. 7 (upper). Shown in 7 (lower) are the respective porosities determined using a particle density of 2.50 g cm$^{-3}$. The dashed line in the upper panel of Fig. 7 shows the expected mean ratio of small grains to large, assuming an even distribution of the small and large grains in the 2-D slab.

The data are seen to diverge from this expected ratio depending on whether they were poured quickly or slowly. When poured quickly, more small grains tended to accumulate toward the center of the pile. When poured slowly, the opposite happened, and more small grains ended up in the lower slopes of the pile. These relatively small differences in the ratio of small and large grain distribution led to differences of up to 10% in the local porosity (Fig. 7, lower) in the different sections of the piles. Most remarkably, the porosity in the center section of the pile poured slowly was 0.45, which is about 0.2 greater than that found by Robinson and Friedman (2001) for the same size ratio when aiming at a tight packing. This demonstrates the importance of the packing methodology in determining the porosity. The methodology of Robinson and Friedman (2001) was to use gentle tapping to redistribute the particles. The higher porosities observed by pouring are indicative of the phenomena of arching (Wittmer et al., 1997). This can be caused by small grains acting as wedges between large grains, and it results in a structure with high potential energy, especially when the fraction of small grains is lower than required to fill the voids between the large grains. Instabilities in pile structure due to particle size distribution and arching were presented by Robinson and Friedman (2002).

The impact of these observed differences in porosity and pore structure for water flow and other transport properties could be marked. The fact that arching is likely to be occurring means that the structure has high potential energy and may become unstable during infiltration and thus bias transport experiments. If the flow of the water dislodges small grains, it may cause crumbling of the local structure, making the matrix change dynamically as the water flows, causing percolation of the small particles through the column. It would be well worth investigating if the small fraction moves and thus porosity changes as water flows through a pile.

A Method of Achieving Uniform Packing

The critical question is whether homogeneous packing can be achieved by hand pouring and mixing. Simply stirring the grains as a dry mixture is not effective, but stirring the grains with a small amount of water radically improves the mixing. The attractive cohesive force between the water molecules and the attractive adhesive force between the water molecules and solid surfaces makes the grains sticky. If a porous media manifests cohesiveness, it derives that cohesion from the functioning of adhesive water bridges.

The cohesion of granular materials has intrigued scientists for a long time (Keen, 1924; Haines, 1925; Fisher, 1926). It is a problem many of us will have encountered practically in the construction of sandcastles at a beach. If the sand is too wet or too dry the castle won’t stand; only at intermediate water contents does the water provide adhesive water bridges between the grains (Hornbaker et al., 1997; Bocquet et al., 1998). This adhesion we believe, provides the key to thorough mixing and uniform packing. Data (Fig. 8) presented by Keen (1924) illustrates how cohesion between soil grains for a silty soil increases as a function of water content, until rapidly declining as saturation is approached.

This point is illustrated in Fig. 9, where colored sand grains of different sizes were placed in two layers. The
coarse sand (800–850 μm) and fine sand (350–450 μm) were initially mixed dry, but uniform mixing could not be achieved even after 5 min. Banding is visible and many of the fine particles migrate to the base of the container. Also included is a photo of the results with the same materials having had 0.00175 g<sub>water</sub> g<sub>solid</sub>⁻¹ sprayed into the sand and mixed for 1 min. For this case, the uniformity of the mixing appears greatly improved. Also shown are the results for the same materials with 0.0296 g<sub>water</sub> g<sub>solid</sub>⁻¹ sprayed into the sand and mixed for 1 min. The change in color and its uniformity reflect the uniform distribution of the grains throughout the media assisted by the adhesion of the water bridges between the grains. As a final example, the same experiment was conducted with the materials saturated. Again the materials were mixed for 1 min, and a reasonable visual mixing is observed; however, separation of the grains began to occur. We found that the best way to mix the material under saturated conditions was to use a gentle lifting motion. This method was previously used by Robinson and Friedman (2001).

We would suggest that to obtain the most uniform packing of a dry material the addition of a little water and subsequent mixing for at least 1 min is the best way to obtain relatively uniform packing. This ensures that the effective properties of the particle domain are the same as those measured at the macroscopic scale. The column should then be gently tapped on a bench to achieve tight packing. The column may then be dried if dry packing is required. If the opposite is required and a saturated packing is needed, it would appear that the method described above of gently lifting particles in a rotational manner provides reasonable results and prevents air entrapment, which can cause error in results.

**CONCLUSIONS**

Photographs and data are presented that demonstrate that the rate and method of pouring of binary mixtures of granular material leads to different structural packing arrangements in a 2-D slab cell. In laboratories, scientific or commercial, hand pouring is the general method used for filling disturbed soil columns or 2-D slabs. When hand pouring, standardization among laboratories or even among technicians is difficult. The rate at which a granular material is poured into a cell is
crucial in determining the structural distribution of the grains. This was demonstrated to impact the local porosity by as much as 10%. These differences in local porosity may translate into “anomalous” transport measurements when considered in relation to modeled results. Interpretation of such measurements may lead to incorrect criticism of models designed with uniform effective properties as input parameters. For the same pouring rate, particle size distribution and particle shape have been shown to cause structural segregation. Avalanching processes produce layering of different size grains; the magnitude of the layering is affected by the angularity of the grains. A method is presented that we believe will overcome most of the potential errors in obtaining a uniform distribution of grains. This method uses the cohesiveness of the particles when partially wet to avoid segregation.

ACKNOWLEDGMENTS

The authors would like to acknowledge funding provided in part by a NSF grant, EAR-0074841 and a USDA-NRI grant 2002-35107-12507.

REFERENCES


Statham, I. 1974. The relationship of porosity and angle of repose to variability when considered in relation to modeled results. Interpretation of such measurements may lead to incorrect criticism of models designed with uniform effective properties as input parameters. For the same pouring rate, particle size distribution and particle shape have been shown to cause structural segregation. Avalanching processes produce layering of different size grains; the magnitude of the layering is affected by the angularity of the grains. A method is presented that we believe will overcome most of the potential errors in obtaining a uniform distribution of grains. This method uses the cohesiveness of the particles when partially wet to avoid segregation.