Measurement of Pore-Size Density Function in Sand

C. H. JUANG AND C. W. LOVELL

A study is presented on characterization of sand fabric by means of the pore-size distribution, which allows the engineer to indirectly but quantitatively examine the sand fabric. The term "pore-size distribution" is clarified so that a better link is established between the terminology used in the literature of mercury porosimetry and that used in the conventional probabilistic approaches in geotechnical engineering. A technique has been verified satisfactory for preparation of sand specimens for mercury intrusion tests, which make possible the measurement and determination of the pore-size distribution and the density function in sand. Data deduction and typical pore-size distributions and density functions for the sands studied are presented and discussed.

The characterization of soil fabric is an important aspect of soil engineering. It can lead to better understanding and prediction of engineering properties of soils. The term "soil fabric" is generally understood as "the physical constitution of a soil material as expressed by the special arrangement of the solid particles and associated voids" (*1*). Past research has shown that the fabrics of compacted clayey soils can be characterized by their pore-size distribution characteristics, and some engineering properties of clayey soils can be quantitatively correlated with their pore-size distribution parameters (*2*; *3*; *4*, pp. 839–856; *5–12*). Although engineering properties of sands are also believed to be strongly affected by their fabric characteristics (*13–16*, p. 59) a simple and rational technique to characterize the sand fabric has not yet been reported. A study is presented on characterization of the sand fabric by means of pore-size distribution. The pore-size distributions of the compacted sands studied were measured and determined by using mercury intrusion porosimetry (MIP).

MERCURY INTRUSION POROSIMETRY

The pore systems of all but the most uniform soils have an extremely complex geometry. Therefore, to characterize soil fabric by means of pore size and its distribution is a rather abstract concept. There are, however, several advantages to the use of pore-size distribution in soil engineering. For example, it allows the engineer to quantitatively examine the soil fabric; it can be measured and determined by a simple, routine laboratory test; good correlations have been established between some engineering properties and pore-size distribution parameters (*4*, *6*, *12*); it provides an equally good or even better measure to characterize the soil fabric than its counterpart—grain-size distribution—does.

C. H. Juang, Department of Civil Engineering, Clemson University, Clemson, S.C. 29634-0911. C. W. Lovell, School of Civil Engineering, Purdue University, West Lafayette, Ind. 47907.

Over the past decade MIP has been shown to be the most suitable method to measure and determine the pore-size distributions of the soils. The mercury intrusion technique is based on the Washburn model (*17*):

$$P = -(4T \cos \theta / x) \quad (1)$$

where

P = absolute pressure being applied,
T = surface tension of mercury,
θ = contact angle between mercury and the pore wall, and
x = size of pores that can be intruded at pressure P, usually called the apparent pore diameter.

For any pore size that is determined by Equation 1, a corresponding pore volume $[F_v(x)]$ is intruded and recorded. Hence, a pore-size distribution [i.e., $F_v(x)$ versus x] can be readily generated in a mercury intrusion test. The measurement of pore-size distribution with MIP may be as follows. A pore-size distribution function may be established by repeatedly determining what fraction of the total pore volume the pores have for pore sizes ranging between x and infinity. Mathematically the complementary cumulative distribution function of the pore size $[F_X(x)]$ may be expressed as

$$F_X(x) = \int_x^\infty f_X(x)dx \quad (2)$$

where f_X is the probability density function of pore size, and

$$\int_0^\infty f_X(x)dx = 1 \quad (3)$$

It may be noted that F_X, defined in Equation 2, expresses the probability that the random variable takes on realizations x equal to or greater than X. Although this is in contrast to the conventional use of the cumulative distribution function in geotechnical engineering, it is better for interpreting the measured pore-size data, and it does serve the purpose of defining the probability density function. With this viewpoint, Juang and Holtz (*18*) have derived a volumetric pore-size density function $[f_V(x)]$ as follows:

$$f_V(x) = -[d(V_t - V)/dx] \quad (4)$$

where V_t is the total volume of pores in a soil, which is a constant, and $V_t - V$ is the volume of pores with pore sizes equal to or greater than x. Integrating Equation 4 over the range from x to infinity yields

$$F_V(x) = V_t - V \quad (5)$$

The function $F_V(x)$ is a complementary cumulative distribution of the pore size in terms of pore volume, but it is often simply called the pore-size distribution in the literature of MIP. Because $F_V(x)$ is generally expressed in terms of pore volume, it is sometimes called the pore-volume distribution (ASTM D4404). It is believed, however, that the term "pore-volume distribution" could be misleading for most engineers, who have become increasingly familiar with the language of probability theory used in geotechnical engineering: the term could be misunderstood as a distribution of pore volume when in fact pore size is the random variable considered. To be consistent with the current probability terminology used in geotechnical engineering, it is suggested that the probability density function of the pore size or, simply, the pore-size density function be used and reported in the MIP test. The pore-size density function may be obtained by dividing Equation 4 by the total pore volume (V_t), which yields

$$f_X(x) = (1/V_t) \cdot \{- [d(V_t - V)/dx]\} \tag{6}$$

Because Equation 6 can be deduced directly from the mercury intrusion data and is consistent with the language of probability theory used in geotechnical engineering, it is suggested that it be used in the presentation of the MIP test data.

PORE-SIZE DENSITY FUNCTION OF SAND

Specimen Preparation

The sand tested is a uniform Ottawa sand. The grain-size distribution is shown in Figure 1. The soil classification of the sand is SP and A-1-b(0) according to the Unified Soil Classification and AASHTO systems, respectively.

Perhaps the most difficult problem in measuring and determining the pore-size distribution and pore-size density function in sand is the "sampling," that is, how to mold the soil specimen for the MIP test without destroying the sand fabric. In this study, the problem was solved by using a technique developed by Juang and Holtz (*19*). The technique involves using a resin as a bonding material. A small amount of Varcum 1364 powdered phenolic resin made by Reichold Chemicals, Inc., was mixed with sand. Pluviation was used to compact the sand sample in a standard Proctor compaction mold (ASTM D698) 102 mm in diameter and 116 mm high. After compaction the entire sample and the mold were carefully placed in the oven at a temperature of 170°C for about 1 hr. This time and temperature were sufficient to melt the resin, which surface coated the sand grains. The sample was then removed and placed in a desiccator for several hours until it cooled. The specimens for the MIP test were then trimmed from resin-bonded compacted samples. The dimensions of individual specimens were approximately 12 × 8 × 8 mm. After trimming, the specimens were placed in an oven for about 5 to 10 min in order to get rid of moisture, if any, that might have been absorbed by the specimens during trimming. The specimens were then kept in glass bottles and placed in the desiccator.

Different states of compaction of the sand studied were obtained by pluvial compaction (*14*). Figure 2 shows the pluviation scheme. The sand-resin mixture was poured from a 1000-ml flask through a nozzle made from a rubber stopper containing one hole either 6.8 or 3.8 mm in diameter. The sand-resin mixture was free to fall to the surface of the sample while

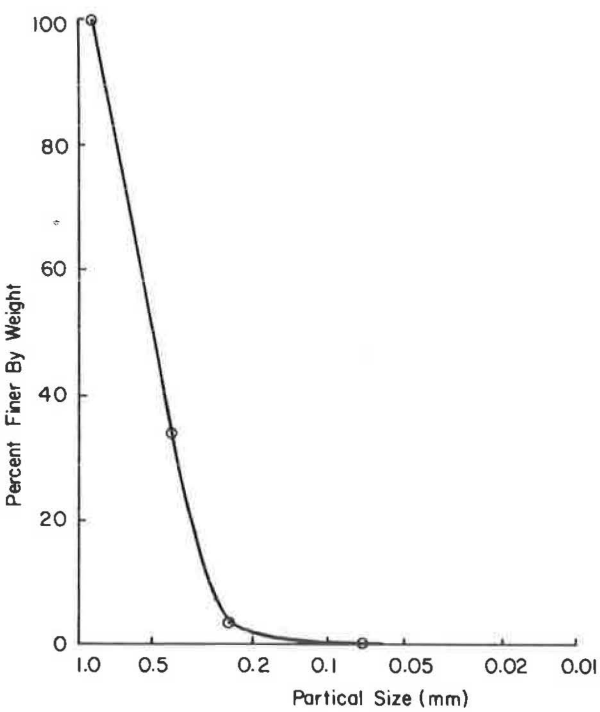

FIGURE 1 Grain-size distribution of sand tested.

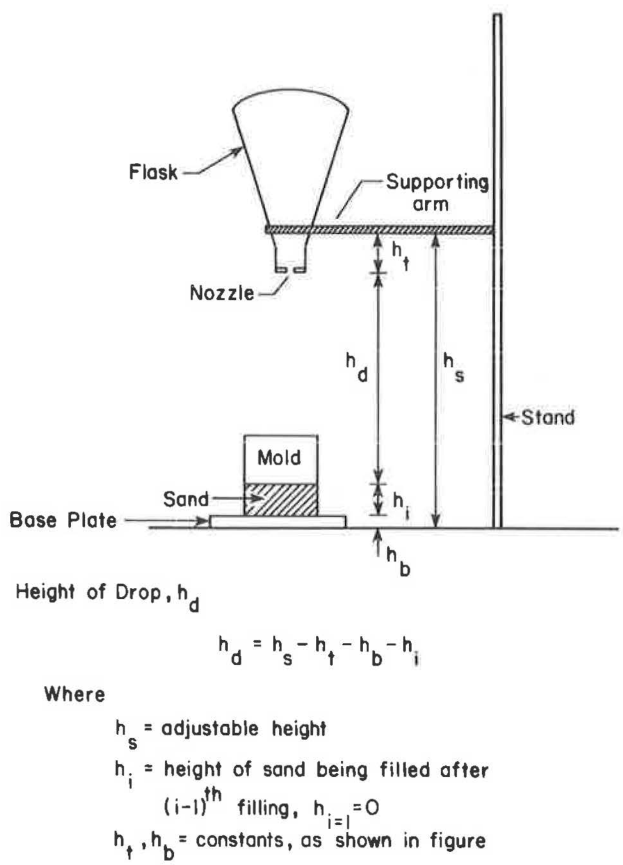

FIGURE 2 Schematic diagram of pluviation system.

the flask was rotated at a rate of approximately 15 rpm. The density of the sample obtained depended on the hole size and the drop height. In this study, the samples were made in five layers with drop height for each layer of 108 mm. In order to keep the same drop height, the flask position was adjusted for each layer. The choice of the drop height and hole size depended on the density of the compacted sample desired. For this study, they were selected by trial and error to produce a relatively dense and a relatively loose state, respectively. The results of the pluvial compaction of the sand-resin mixture are as follows (the results indicate that the desired density can be replicated by controlling the diameter of the hole and the drop height):

	State of Compaction	
Parameter	*Relatively Loose*	*Relatively Dense*
Sample no.	1–6	1–6
Diameter of hole (mm)	6.8	3.8
Drop height (mm)	108	108
No. of layers	5	5
Achieved dry density (Mg/m^3)	1.62	1.72

The minimum amount of resin that provides sufficient bonding and workability was found to be 0.5 percent by weight of sand (*18*). This percentage was used throughout the entire testing program. It must be noted that after oven drying, the compacted samples experienced about 5 percent shrinkage in volume but virtually no change in total weight. The powdered phenolic resin acted as a glue when the compacted sand was placed in the oven, and no chemical reaction between sand and resin occurred during oven drying (*18*).

The foregoing procedure has been found to be satisfactory. The fact that the results of the intrusion tests on specimens trimmed from the oven-dried samples are reproducible indicates that the procedure is an effective technique of obtaining sand specimens for the MIP test.

MIP Test

The apparatus and procedures used in this study were similar to those used by Garcia-Bengochea et al. (*4*) and ASTM D4404. However, the freeze-drying procedure was not used because the specimens had already been dried by using the specimen preparation technique described in the foregoing section. In addition, the penetrometer with intrusion capacity of 1.2 ml instead of 0.2 ml was used during the low-pressure intrusion period, because it was expected that a larger pore volume exists in sand than in clay. The low-pressure intrusion was mainly to measure the distribution of the large pores (20 to 500 m). The use of a large penetrometer (with a capacity of 1.2 mL) enhances the precision of the measurement of the distribution of the large pores. To extend the distribution curve to the range of the smaller pore size, a small penetrometer (with a capacity of 0.2 mL) was also used and the high-pressure intrusion was carried out for a separate specimen of the same sand. Thus, a combination of data measured by both penetrometers was used to obtain the distribution of the entire range of pore sizes.

RESULTS AND DISCUSSION

As mentioned earlier, the absolute applied pressures recorded in the MIP test can be used to calculate the apparent pore diameters on the basis of Equation 1. However, this step requires that the surface tension and contact angle be known. For the sand tested, the surface tension was judged to be 484 dynes/cm on the basis of Kemball's data (*20*). The contact angle of mercury on melted phenolic resin was determined to be 154 degrees following the method suggested by Winslow (*21*). Because the surface tension of phenolic resin is much lower than that of sand, the contact angle of mercury on sand was judged to be 154 degrees (D. N. Winslow, private communication). Once the two foregoing parameters have been determined, the rest of the data reduction procedures are more or less routine computations (e.g., *2*, *3*, *4*, ASTM D4404).

A graphical presentation of the complementary cumulative distribution with the intruded pore volumes per gram of specimen on the ordinate to an arithmetic scale and the apparent pore diameters on the abscissa to a logarithmic scale is a conventional way to present the MIP test data (*2*, ASTM D4404). Figure 3 shows the measured pore-size distributions of the sands at different states of compaction.

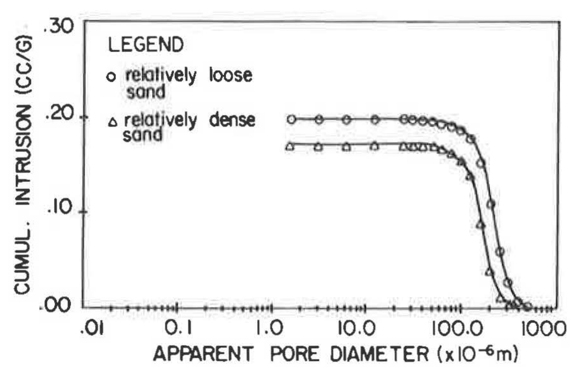

FIGURE 3 **Pore-size distributions of sands tested in terms of cumulative intrusion.**

As mentioned earlier, it may be desirable to present the measured data in terms of pore-size density function according to Equation 6. The pore-size density function may be obtained by using the finite difference approximation method (*19*) or by direct differentiation of a curve-fitted cumulative distribution function. The two methods generally yield approximately the same results (*6*). Figure 4 shows the pore-size density function of the sands tested.

In general, the replication of the MIP tests was satisfactory. Because of the relatively insignificant variation in the test results, Figure 4 actually represents an average of 12 data sets measured for each sand.

Finally, it has to be pointed out that in Equation 4 or 5, the term V_t implies the total pore volume that can be intruded by pressured mercury. Theoretically, it is equal to the total pore volume that exists in the soil specimen. In reality, however, a significant unintruded pore space is not unusual in an MIP test (*22*, *23*). For the sand specimens tested, however, the unintruded pore space is generally less than 5 percent. Thus, no correction measure (*24*) was taken, and the former definition

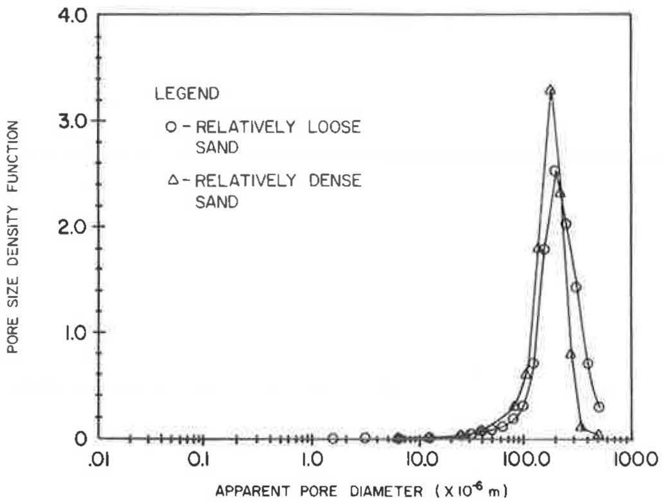

FIGURE 4 Pore-size density functions of sands tested.

was assumed in the calculation of the pore-size density function for the sands tested.

The pore-size density function curve provides some useful qualitative characteristics of the sand fabric. The curves in Figure 4 show a single mode characteristic around 200 m. As the state of compaction changes, so do the mode and its probability density (see Figure 4). This indicates that the pore-size density function may be used to characterize the soil fabric. In addition, the pore-size density function has been successfully used in, and is the core of the development of, a theoretical permeability model (*19*), which indicates that the pore-size density function is a useful quantitative measure of the sand fabric.

CONCLUSIONS

1. A technique has been verified to be satisfactory for the preparation of sand specimens for the MIP tests, which enables the measurement of the pore-size distribution and pore-size density function in sand.

2. A clarification of "pore-size distribution" has been given to provide a better link between the terminology used in the literature of mercury porosimetry and that used in the conventional probabilistic approaches in geotechnical engineering.

3. The pore-size density functions of the sands studied were found to be singly modal on a log-diameter scale. The mode occurred around 200 m, but the mode and its probability density changed as the compactive effort varied.

4. The results of this study have provided a basis for further research to investigate the sand fabric through variation in the pore-size density function. It is believed that such study could lead to better understanding or prediction of some engineering properties (compressibility, liquefaction potential, etc.) of sands.

ACKNOWLEDGMENT

The study on which this paper is based was supported by the Federal Highway Administration and the Indiana State Highway Commission through the Joint Highway Research Project at Purdue University. Also, the authors wish to thank R. D. Holtz, D. N. Winslow, and J. Lovell for their assistance in various aspects of this study.

REFERENCES

1. R. Brewer. *Fabric and Mineral Analysis of Soils*. John Wiley and Sons, Inc., New York, 1964, 482 pp.
2. S. Diamond. Pore Size Distributions in Clays. *Clays and Clay Minerals*, Vol. 18, 1970, pp. 7–23.
3. S. Diamond. Microstructure and Pore Structure of Impact Compacted Clays. *Clays and Clay Minerals*, Vol. 19, 1971, pp. 239–249.
4. I. Garcia-Bengochea, C. W. Lovell, and A. G. Altschaeffl. Relation Between Pore Size Distribution and Permeability of Silty Clay. *Journal of the Geotechnical Engineering Division, ASCE*, Vol. 105, No. GT7, 1979, pp. 839–856.
5. P. N. Gaskin and G. P. Raymond. "Pore Size Distribution as a Frost Susceptibility Criterion." *Proc., Symposium on Frost Action on Roads*, Vol. 1, Norwegian Road Research Laboratory, Oslo, 1973.
6. C. H. Juang. *Pore Size Distribution of Sandy Soils and Prediction of Permeability*. FHWA/IN/JHRP Report 81-15. Purdue University, West Lafayette, Ind., 1982.
7. T. C. Kenney. Discussion: Frost-Heaving Rate Predicted from Pore-Size Distributions. *Canadian Geotechnical Journal*, Vol. 17, No. 2, 1980, pp. 332–333.
8. G. O. Klock, L. Boersma, and L. W. DeBacker. Pore Size Distributions as Measured by the Mercury Intrusion Method and Their Use in Predicting Permeability. *Proc., American Society of Soil Science*, Vol. 33, No. 1, 1969, pp. 12–15.
9. T. J. Marshall. A Relation Between Permeability and Size Distribution of Pores. *Journal of Soil Science*, Vol. 9, 1958, No. 1, pp. 1–8.
10. R. J. Millington and J. P. Quirk. Permeability of Porous Media. *Nature*, Vol. 183, Feb. 1959, pp. 387–388.
11. A. Murota and K. Sato. Statistical Determination of Permeability by the Pore Size Distribution in Porous Media. *Proc., 13th Congress of the International Association for Hydraulic Research, Japan*, Vol. 4, Sept. 1969, pp. 183–190.
12. M. A. Reed, C. W. Lovell, A. G. Altschaeffl, and L. E. Wood. Frost-Heaving Rate Predicted from Pore Size Distribution. *Canadian Geotechnical Journal*, Vol. 16, No. 3, Aug. 1979, pp. 463–472.
13. A. Mahmood. Fabric-Mechanical Property Relationships in Fine Granular Materials. *Clays and Clay Minerals*, Vol. 22, No. 516, 1973, pp. 397–408.
14. J. K. Mitchell, J. M. Chatoian, and G. C. Carpenter. *The Influences of Sand Fabric on Liquefaction Behavior*. Report TE76-1. Department of Civil Engineering, University of California, Berkeley, June 1976.
15. M. Oda. Initial Fabrics and Their Relations to Mechanical Properties of Granular Materials. *Soils and Foundations*, Vol. 12, No. 1, 1972, pp. 1–18.
16. M. Oda. *Fabrics and Their Effects on the Deformation Behavior of Sand*. Department of Foundation Engineering, Saitama University, Japan, 1976.
17. E. W. Washburn. Note on a Method of Determining the Distribution of Pore Sizes in a Porous Material, *Proc., National Academy of Sciences*, Vol. 7, 1921, pp. 115–116.
18. C. H. Juang and R. D. Holtz. A Note on Preparation of Specimens of Noncohesive Material for Mercury Intrusion Porosimetry. *Geotechnical Testing Journal, ASTM*, Sept. 1986.
19. C. H. Juang and R. D. Holtz. A Probabilistic Permeability Model and the Pore Size Density Function. *International Journal for Numerical and Analytical Methods in Geomechanics*, Vol. 10, 1986.
20. C. Kemball. On the Surface Tension of Mercury. *Transactions of the Faraday Society*, Vol. 42, 1946, pp. 526–537.

21. D. N. Winslow. *The Pore Size Distribution of Portland Cement Paste.* M.S.C.E. thesis. Purdue University, West Lafayette, Inc., Jan. 1969.
22. R. A. Lohnes, E. R. Tuncer, and T. Demirel. "Pore Structures of Selected Hawaiian Soils." In *Transportation Research Record 512*, TRB, NRC, Washington, D.C., 1976, pp. 76–69.
23. S. Diamond and W. L. Dolch. Generalized Log-Normal Distribution of Pore Sizes in Hydrated Cement Paste. *Journal of Colloid and Interface Science*, Vol. 34, No. 1, Jan. 1972, pp. 234–244.
24. H. I. Meyer. Pore Distribution in Porous Media. *Journal of Applied Physics*, Vol. 24, 1953, pp. 510–512.

Publication of this paper sponsored by Committee on Physicochemical Phenomena in Soils.