

## 2.2 Particle Density

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### 2.2.1 Introduction

The *density* of a material is defined as the mass of a quantity divided by the volume of the same quantity. Conventional units for density are megagrams per cubic meter, or its numeric equivalent, grams per cubic centimeter. *Particle density* refers to the density of the solid particles collectively. In contrast, *grain density* refers to the density of specific grains. A bulk soil containing individual quartz or feldspar, each having its own grain density, would have a collective particle density, which is the weighted density of all individual grains. *Bulk density* includes the volume of the pores created between particles and pores that exist within individual particles. *Specific gravity*, sometimes referred to in reference to particle density, is the ratio of particle density to that of water at 3.98°C (1.0000 g cm<sup>-3</sup>) or other specified temperature, and thus is unitless.

Particle density is required for heat-capacity calculations, sedimentation analysis, or calculations involving volumes or mass of soil. In addition, particle density is required for mathematically correcting bulk soil samples that contain a significant amount of gravel or rock fragments. These corrections need to be applied to determine fine-soil density, water content, cation-exchange capacity, or other soil properties affected by volume displacement of rocks (Flint & Childs, 1984; Childs & Flint, 1990). The following discussion includes methods to measure both soil and rock samples. Understanding the role of rock fragments in soils or the hydrologic properties of rock have become a significant component of research for many soil physicists. Characterization methods have advanced in the last decade and will be discussed in this section.

### 2.2.2 Principles

Determining the particle density of a soil or rock sample requires that the mass and the volume of the sample be carefully measured. Mass is determined by weighing, which can be done precisely and accurately on an analytical balance. Volume is determined by liquid or gas displacement methods (pycnometry). Other methods can be employed, such as calculation from known porosity and bulk density. Estimation techniques can also be used when constituent properties are known (e.g., percentage quartz or feldspar), when their grain densities are known, such as from standard handbooks. The method chosen depends on available sample materials, equipment, and the accuracy needed.

## 2.2.3 Methods

### 2.2.3.1 Calculation from Porosity and Bulk Density

Particle density, the density of the solid particles in soil and rock, can be easily calculated from the porosity and bulk density. The ratio of the bulk density,  $\rho_b$ , to the particle density,  $\rho_p$ , describes the fraction of the total volume occupied by solids. One minus that ratio is the porosity,  $\phi$ . Thus, the particle density is the ratio of the bulk density divided by one minus the porosity:

$$\rho_p = \rho_b / (1 - \phi) \quad [2.2-1]$$

A variety of methods that can be employed to determine bulk density and porosity are outlined in detail in Sections 2.1 and 2.3. The procedure chosen for those measurements is based on the application of the particle density value, the type and size of the sample, and the desired accuracy.

Consideration of the degree to which water is removed from the pores during drying to determine bulk density is relevant to the application of the particle density value. For example, clays, which often line the flow channels of soils and rocks, contain water, which causes them to expand, thus narrowing the pore channels. A common practice is to dry a sample in an ambient humidity oven at 105°C for 24 h, which removes free and bound water from the pores and clays, but does not remove structural water. A lower temperature and elevated relative humidity, approximating a water potential of -70 MPa, will remove the free water from the flow channels yet maintain the bound water in the clays (Flint, 1998). A calculation of particle density using elevated relative humidity drying will be smaller.

### 2.2.3.2 Liquid Displacement

There are generally two liquid-displacement methods used for calculating particle density, both being a form of pycnometer (*pycn* meaning “dense”). One method is generally referred to as the *pycnometer method* because it uses a specific type of commercial pycnometer. The other method is referred to as *Archimedes' displacement method* because it is based on Archimedes' original method (Webb & Orr, 1997). Both use weight and volume displaced by water (or other liquid chosen by the user).

#### 2.2.3.2.a Pycnometer Method

Typically, one of two pycnometer methods can be used, depending on sample size and accuracy needs. Commercially available stoppered bottles are used for small samples when higher degrees of accuracy are needed ( $\geq$  three significant figures). Volumetric flasks can be used for large samples if a high degree of accuracy is not required ( $<$  three significant figures).

**Stoppered Bottle.** A variety of commercially available stoppered bottle pycnometers (Fig. 2.2-1) have volumes generally ranging from 10 to 100 mL, but up to 1000 mL for pycnometers used for larger samples. Some pycnometers are avail-

able with a thermometer as part of the apparatus. For determining particle density for fine-grained soils, the Gay-Lussac specific-gravity bottles are typically used. These generally have serial numbers on the bottle and stopper with the glass stopper having a capillary for overflow with a ground flat top to remove excess water. The following method describes the use of a standard 50-mL bottle.

Weigh a clean, dry pycnometer in air. Add about 10 g of air-dry sample material (sieved through a 2-mm sieve to determine the density of fine soil particles). Clean the outside and neck of the pycnometer of any soil that may have spilled during transfer. Weigh the pycnometer (including stopper) and its contents. Determine the water content of a duplicate soil sample by drying it at 105°C for 24 h or other specified temperature (see Sections 2.2.3.2.b, 3.1.1, 3.1.3.1, and 3.1.3.2 in reference to oven dryness and water content determination using ovens).

Fill the pycnometer about one-half full with de-aired distilled water, washing into the pycnometer any soil adhering to the inside of the neck. Remove entrapped air by placing the pycnometer into a vacuum chamber and slowly apply a vacuum while being careful not to let the bubbles remove any soil from the bottle. Add enough de-aired water to fill the pycnometer, and then insert the stopper and seat it carefully, forcing the excess water out of the capillary. Thoroughly dry and clean the outside of the bottle with a dry cloth, using care to avoid drawing water out of the capillary. Weigh the pycnometer and its contents and determine the temperature of the water.

Finally, remove the soil, gravel, or rock fragments from the pycnometer and thoroughly wash it. Fill the pycnometer with de-aired distilled water at the same temperature as before. Insert the stopper, thoroughly dry the outside with a cloth, and weigh the pycnometer and water as before.

Calculate the particle density ( $\rho_p$ ) as follows:

$$\rho_p = [\rho_w(W_s - W_a)] / [(W_s - W_a) - (W_{sw} - W_w)] \quad [2.2-2]$$



Fig. 2.2-1. Stoppered bottles used in the pycnometer method to determine particle density.

where  $\rho_w$  is the density of water ( $\text{g cm}^{-3}$ ) at the temperature observed,  $W_s$  is the weight of the pycnometer plus soil sample corrected to oven-dry water content,  $W_a$  is the weight of the pycnometer filled with air,  $W_{sw}$  is the weight of the pycnometer filled with soil and water, and  $W_w$  is the weight of the pycnometer filled with water at temperature observed.

**Volumetric Flask.** Volumetric flasks are commonly used for determining particle density for rock fragments or gravel (see Fig. 2.2–2). There is a variety of commercially available volumetric flasks and specific gravity flasks with volumes ranging from 100 to 500 mL. The advantage over the generally smaller stoppered bottle pycnometer is the accommodation of a larger sample size and a larger opening for the sample. If an even larger opening is needed for the sample, a graduated cylinder can be substituted. The following method describes the use of a standard 500-mL volumetric flask.

Weigh a clean, dry volumetric flask in air. Add about 100 g of air-dry soil, gravel, or rock fragments. Weigh the volumetric flask and its contents. Determine the water content of a duplicate soil sample by drying it at  $105^\circ\text{C}$  for 24 h or other specified temperature (see Section 2.2.3.1–2.2.3.3 for discussion of oven dryness).

Fill the volumetric flask about one-half full with de-aired distilled water, washing into the flask any particles adhering to the inside of the neck. Remove entrapped air by placing the flask into a vacuum chamber, and slowly apply a vacuum while being careful not to let the bubbles remove any particles from the flask. Add enough de-aired, distilled water to fill the volumetric flask up to the filled mark.

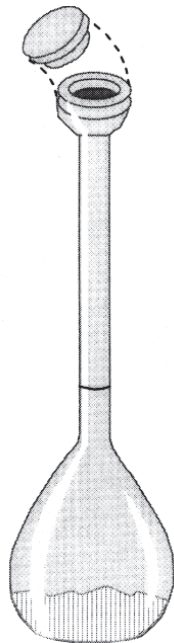


Fig. 2.2–2. Standard 500-mL volumetric flask used for determining particle density.

Thoroughly dry and clean the outside of the flask with a dry cloth. Weigh the flask and its contents and determine the temperature of the water.

Finally, remove the soil, gravel, or rock fragments from the flask and thoroughly wash it. Fill the flask with de-aired distilled water at the same temperature as before, thoroughly dry the outside with a cloth, and weigh the flask and water as before.

Calculate the particle density ( $\rho_p$ ) as follows:

$$\rho_p = [\rho_w(W_r - W_a)] / [(W_r - W_a) - (W_{rw} - W_w)] \quad [2.2-3]$$

where  $\rho_w$  is the density of water ( $\text{g cm}^{-3}$ ) at the temperature observed,  $W_r$  is the weight of the volumetric flask plus rock fragments or gravel corrected to oven-dry water content,  $W_a$  is the weight of the volumetric flask filled with air,  $W_{rw}$  is the weight of volumetric flask filled with gravel or rock fragments and water, and  $W_w$  is the weight of volumetric flask filled with water at the temperature observed.

### 2.2.3.2.b Archimedes' Displacement Method

The following method is used to determine particle density for large irregularly shaped rock fragments, gravel, rock cores, or soil cores. The advantage of this method over the pycnometer method is that enough data are collected to calculate particle density, bulk density, and porosity simultaneously. If an initial weight of the sample is taken, then gravimetric and volumetric water content can also be determined.

The gravimetric and volumetric water content, particle density, bulk density, and porosity, which are determined using a saturation chamber capable of sustaining a vacuum, drying oven, calibrated balance, and Archimedes' method, are derived as follows:

$$\rho_b = W_d/V_b \quad [2.2-4]$$

$$\phi = (W_s - W_d)/V_b \quad [2.2-5]$$

$$\rho_p = W_d/(V_b - \phi) \quad [2.2-6]$$

$$\theta_w = (W_i - W_d)/W_d \quad [2.2-7]$$

$$\theta_v = \theta_w \rho_b \quad [2.2-8]$$

where  $\rho_b$  is the bulk density of the sample,  $W_d$  is the dry weight of the sample,  $V_b$  is the bulk volume of the sample,  $\phi$  is the porosity of the sample,  $W_s$  is the saturated weight of the sample,  $\rho_p$  is the particle density of the sample,  $\theta_w$  is the gravimetric water content,  $W_i$  is the initial weight of the sample,  $\theta_v$  is the volumetric water content.

In order to fill all sample pores, especially for samples with high clay content, small pores, or pore structures with dead-end pore spaces, an apparatus to alternately vacuum saturate and  $\text{CO}_2$ -flush the sample is necessary (Fig. 2.2-3). Carbon dioxide fills the pores after the sample is evacuated under a vacuum, and

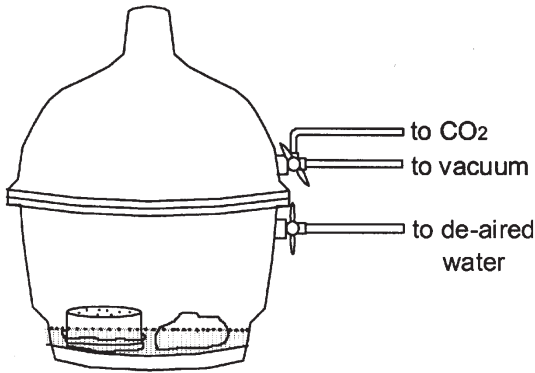


Fig. 2.2-3. Apparatus for vacuum saturating samples.

water is then drawn into small or dead-end pores as the  $\text{CO}_2$  more readily dissolves in the water than in air. A useful chamber is a plastic desiccation chamber (size appropriate for number and size of samples) with a three-way valve and a toggle valve.

Place rock fragments or rock core directly in the chamber. If samples are unconsolidated, prepare the soil core sample in brass rings (5–10 cm [2–4 in.] in diameter is most commonly used, and 2.5–5 cm [1–2 in.] in height is the most commonly used size) with double-layered cheesecloth attached to the bottom with a rubber band. Place the samples in the vacuum-saturation apparatus. Evacuate the chamber for 5 to 10 min. Then turn off the vacuum, flush the chamber with  $\text{CO}_2$ , and let sit for 5 to 10 min. (If the sample has very small pores, repeat these two steps.) Evacuate the chamber again for several minutes, and then, while maintaining a vacuum, slowly fill the chamber with de-aired water. Once the samples are covered with water, leave the samples under a vacuum overnight.

The saturated core sample can now be processed to determine bulk density, porosity, and particle density. Remove a sample from the vacuum chamber (one at a time to maintain saturation) and wipe it gently with a damp cloth to remove excess moisture or “glisten”, being very careful with the unconsolidated samples. Weigh the sample on a balance to obtain the saturated weight.

To determine the volume of the unconsolidated sample in a ring, measure the diameter and height. To determine the volume of an irregularly shaped or consolidated sample, follow the Archimedes’ submersion–displacement method described below.

The Archimedes’ method employs an apparatus consisting of a wire basket suspended by an external support over a beaker of water, which is sitting on a top-loading balance (Fig. 2.2-4). The wire basket should not contact the balance or any part of the beaker, only the water within. Record the temperature of the water. Before a sample is measured, the balance should be zeroed with the empty basket submerged. The basket is then raised from the water, taking care not to displace any drops of water, and the saturated core is placed in the basket and submerged. This process is done quickly to minimize evaporation from the sample. The sample should be fully immersed. The balance will measure an increase in mass equivalent to the mass of water displaced by the sample in the beaker. The water displaced includes

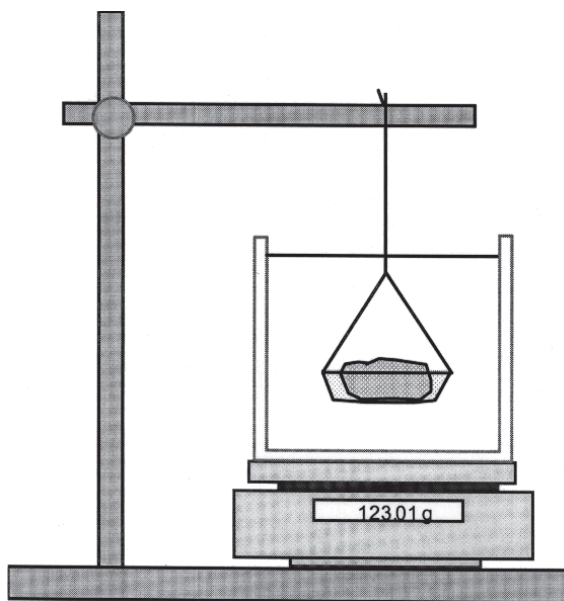


Fig. 2.2-4. Volume displacement using Archimedes' method.

the solids and voids in the saturated sample and therefore provides for the determination of the bulk volume of the sample. The sample is then dried.

Oven-dry the sample (to the desired dryness, either at an elevated relative humidity or at standard 105°C). Record the sample weight and correct for the weight of the ring and cheesecloth, if necessary. Calculate sample parameters using Eq. [2.2-4] through [2.2-8].

### 2.2.3.3 Gas Displacement

Gas displacement methods may be the fastest, easiest, and most accurate methods currently used to measure particle density. These methods employ a variant of the ideal gas law known as Boyle's law. The ideal gas law states that:

$$PV = nRT \quad [2.2-9]$$

where  $P$  is pressure,  $V$  is volume,  $n$  is the number of moles of gas,  $R$  is the Universal Gas Constant,  $T$  is temperature.

Boyle's law states that the volume of a gas is inversely proportional to the pressure applied when the temperature is held constant ( $V \propto 1/P$ ). Boyle's law can also be written, maintaining  $nRT$  as constant, as:

$$P_i V_i = P_f V_f \quad [2.2-10]$$

where  $P_i$  is the initial pressure,  $V_i$  is the initial volume,  $P_f$  is the final pressure, and  $V_f$  is the final volume.

The method typically involves a device known as a *gas pycnometer* with two chambers of known volume (Fig. 2.2-5). Once  $V_i$  of the empty chamber is known,

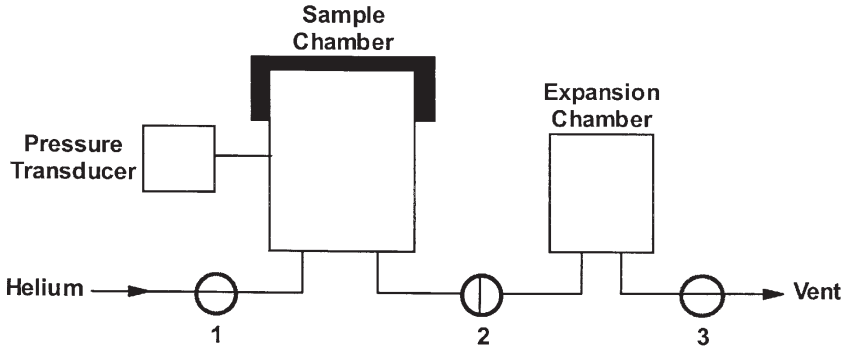


Fig. 2.2–5. Schematic of He pycnometer.

a sample is placed in the sample chamber, and the sample chamber is pressurized to a specified pressure. A valve then allows the gas to move from the pressurized sample chamber to the unpressurized expansion chamber, resulting in a pressure drop in the sample chamber that is directly related to the volume of the sample. The final equilibrium pressure,  $P_f$ , is used to calculate the total volume. In this case, the final volume,  $V_f$ , is a combination of the initial volume,  $V_i$ , the volume of the expansion chamber,  $V_e$ , and the volume of the sample,  $V_s$ . The sample volume is calculated as the combined precalibrated empty volume of the two chambers minus the final volume with the sample in place. The formulation is  $P_i(V_i - V_s) = P_f(V_i + V_e - V_s)$  or rewritten for the volume of the sample as:

$$V_s = V_i - \{V_e/[1 - (P_f/P_i)]\} \tag{2.2-11}$$

where  $V_i$  and  $V_e$  are determined from calibration.

Many homemade gas pycnometers have been constructed using a variety of gases and methods for measuring pressure. Details on how to construct and employ gas pycnometers may be found in Danielson and Sutherland (1986). Commercially available pycnometers, however, are extremely easy to use, provide high measurement accuracy for a variety of sample types, and are fully automated. Sample containers within available pycnometers vary in size from 1 to 135 cm<sup>3</sup>. Instruments typically use research-grade He, requiring a tank capable of storing the gas and associated pressure regulator(s) required to deliver the gas at the pressure specified by the manufacturer. Helium gas is typically used because it does not absorb on most solids, and it more closely obeys the ideal gas law than air or other gases.

Pycnometers require calibration to determine the volume of the sample chamber and expansion chamber. This is typically done during the determination of a sample with a known volume of nonadsorbing material, such as a stainless steel sphere. Follow instructions provided by the manufacturer.

Sample dryness is important to consider when using He pycnometers. Samples dried to  $-70$  MPa, equivalent to drying at 60°C and 65% relative humidity, maintain saturation of the hydrated minerals, and leave a monomolecular layer of water on mineral surfaces (Bush & Jenkins, 1970). Samples wetter or significantly drier than this value will lead to errors in the calculated volumes. Typical 105°C



oven-dry samples ( $-1000$  MPa) are used to maintain a standard for comparison with other techniques and other methods. As the dry He is allowed to come into equilibrium with wet samples, water molecules will evaporate and enter the gas phase, creating an additional partial pressure. This violates the requirement that  $nRT$  remains constant, as the number of moles of gas,  $n$  (Eq. [2.3–9]), increases. Webb and Orr (1997) suggest that as water vapor increases, the apparent pressure will increase, causing an increase in calculated volume. Experimental data, however, suggest the opposite result. Figure 2.2–6 presents sample volumes calculated using Archimedes' method and a commercial, fully automatic, He pycnometer using two samples with different amounts of altered minerals for increasingly drier states. The volume at  $105^{\circ}\text{C}$  is taken as the standard in this case. These experimental data (Fig. 2.2–6) and experiments on distilled, de-aired water consistently yield an underestimate of volume due to an underestimate of pressure. This underestimate is possibly due to absorption of He by the water (which also violates the requirement that  $nRT$  remains constant, and may be a stronger effect than vaporization of small amounts of water). As the sample is dried, additional water is driven off, changing the mass and calculated volumes, with the offset between the two methods being fairly constant. As the sample is dried between  $-70$  and  $-1000$  MPa, the He pycnometer calculates a constant volume, even though additional water is removed (the equivalent saturations at these water potentials are  $0.1$  and  $0.0\text{ g cm}^{-3}$  in Fig. 2.2–6a and  $0.05$  and  $0.0\text{ g cm}^{-3}$  in Fig 2.2–6b, respectively). The monomolecular layer of water is being removed, and the surface area for absorption is being greatly reduced.

Further drying beyond  $-1000$  MPa removes tightly bound and structural water, causing the volume calculated to decrease, but at a lower rate, as the He cannot enter all of the evacuated pore space leading to an overestimate of volume. This effect is most obvious for a sample with a larger relative amount of altered minerals that contain significant bound and structural water (Fig. 2.2–2b).

Because of the temperature sensitivity of pressure-volume relations, it is recommended that the temperature be relatively constant during the measurement. While temperature has a negligible effect on the volume of soil solids, it may have a significant effect on the performance of the gas pycnometer, and testing should be conducted within the specified operating temperature range of the apparatus.

#### 2.2.3.4. Estimation from Constituent Properties

A relatively simple method for estimating particle density is to look at the materials being studied and compare them with published standards for the same materials. There are two standard ways to make the estimation. Knowledge of the parent material or bedrock allows particle density to be estimated using a look-up table. Another approach is to evaluate the mineral composition by percentage and apply the known mineral particle densities weighted by the same percentage.

##### 2.2.3.4.a Material Type

Standards for known materials are used as a substitute for direct measurement if high degrees of accuracy are not required. For example, if you require an estimate of the particle density of granite, the literature values range from  $2.64$  to  $2.76\text{ g cm}^{-3}$  (Lide, 1999), which may be an adequate estimate for most purposes. Sim-

ilar estimates are provided by CRC Press (Lide, 1999) for common material types (Table 2.2-1).

2.2.3.4.b Mineral Composition

Similar to using material type, knowledge of the mineral composition of a sample is another simple way to estimate particle density. Simply sum the particle density for each constituent times its percentage contribution to the whole sample:

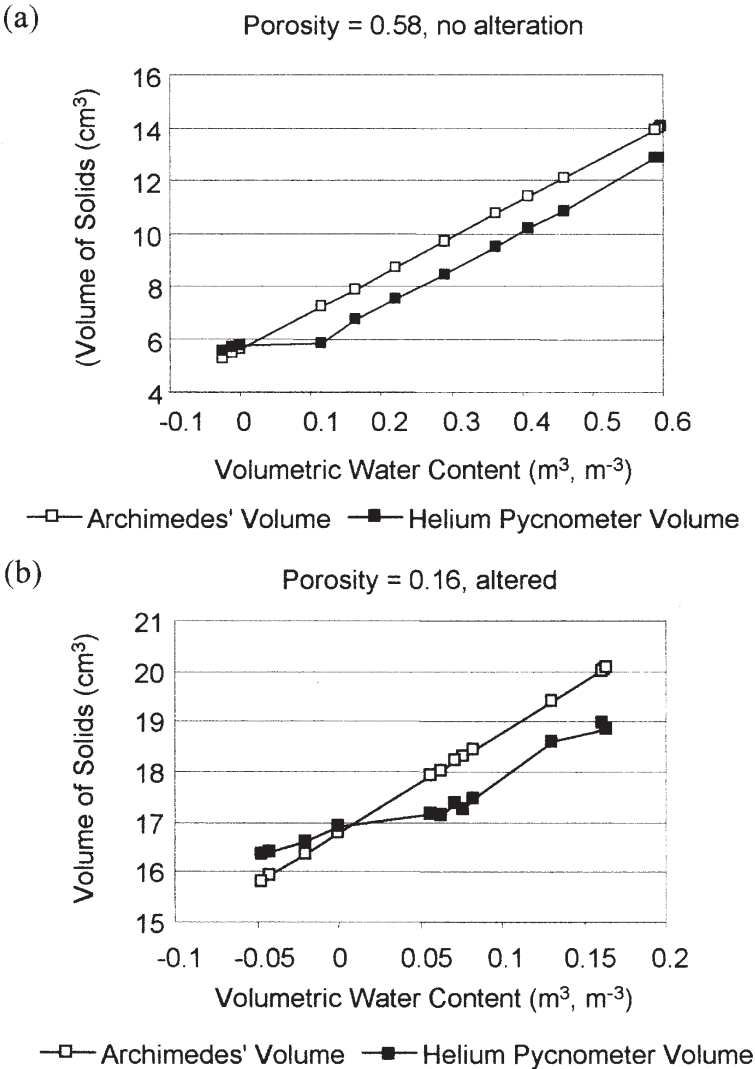


Fig. 2.2-6. Measurement of particle volume at various volumetric water contents using Archimedes' volume displacement and helium pycnometry methods, for a sample (a) with no altered minerals and (b) with a large portion of altered minerals.

Table 2.2-1. Particle densities for various parent materials.

Material	Particle density
	g cm <sup>-3</sup>
Agate	2.5–2.7
Basalt	2.4–3.1
Dolomite	2.84
Flint	2.63
Granite	2.64–2.76
Humus	1.5
Limestone	2.68–2.76
Marble	2.6–2.84
Sandstone	2.14–2.36
Serpentine	2.5–2.65
Slate	2.6–3.3

$$X_1P_1 + X_2P_2 + \dots + X_nP_n \quad [2.2-12]$$

where  $X_n$  is the constituent mineral, and  $P_n$  is the contribution percentage of that mineral.

For example, quartz has a published particle density of 2.65 g cm<sup>-3</sup>, and feldspar has an estimated range of 2.5 to 2.8 g cm<sup>-3</sup> (Lide, 1999). A reasonable estimate of the particle density of quartz sand with 10% feldspar would range from 2.63 to 2.67 g cm<sup>-3</sup>. Similar estimates are provided by Blake and Hartge (1986) and Lide (1999) for common material types (Table 2.2-2).

### 2.2.4 Comments

Several methods have been described to measure particle density. Each has advantages and disadvantages, depending on cost, accuracy, and time. The method chosen should depend on the resources available, the application, and the accuracy

Table 2.2-2. Particle densities for various minerals.

Material	Particle density
	g cm <sup>-3</sup>
Apatite	3.2
Calcite	2.21
Clay	1.8–3.1
Illite	2.8
Kaolinite	2.65
Montmorillonite	2.5
Chlorite	3.0
Feldspar	2.5–2.8
Orthoclase	2.56
Glass	2.4–2.75
Gypsum	2.31–2.33
Mica	2.6–3.2
Biotite	2.7–3.1
Muscovite	2.83
Mordenite	2.13
Opal	1.9
Pyrite	5.02
Quartz	2.65

requirements for the information. Archimedes' method provides the most information for the amount of effort required and is reasonably accurate with large samples. In addition, since particle density is seldom measured as the only property of a sample, the advantage of using Archimedes' method is that it includes a measure of bulk density and porosity and possibly gravimetric and volumetric water content. For particle density alone, the simplest and most accurate method is the commercial He pycnometer. Although the sample needs to be relatively dry, the method works well on soil, gravel, and rock fragments. Even if Archimedes' method were used, the additional measure of particle density using a He pycnometer would provide a check to ensure that the sample was fully saturated during the displacement of water. For economy, the simple water pycnometer provides very good results, but it can be time consuming if many samples need to be processed.

### 2.2.5 References

- Blake, G.R., and K.H. Hartge. 1986. Particle density. p. 377–382. *In* A. Klute (ed.) *Methods of soil analysis*. Part 1. Agron. Monogr. 9. 2nd ed. ASA and SSSA, Madison, WI.
- Bush, D.C., and R.E. Jenkins. 1970. Proper hydration of clays for rock property determination. *J. Petrol. Technol.* 22:800–804.
- Childs, S.W., and A.L. Flint. 1990. Nature of forest soils with rock fragments. p. 95–121. *In* Stan Gesel (ed.) *Proceedings, North American Forest Soils Conference*. Edmonton, Alberta. Fac. of Forestry Publication. Univ. British Columbia, Vancouver, BC, Canada.
- Danielson, R.E., and P.L. Sutherland. 1986. Porosity. p. 443–461. *In* A. Klute (ed.) *Methods of soil analysis*. Part 1. Agron. Monogr. 9. 2nd ed. ASA and SSSA, Madison, WI.
- Flint, A.L., and S.W. Childs. 1984. Physical properties of rock fragments and their effect on available water in skeletal soils. p. 91–103. *In* J.E. Box, Jr. (ed.) *Erosion and productivity of soils containing rock fragments*. SSSA Spec. Publ. 13. SSSA, Madison, WI.
- Flint, L.E. 1998. Characterization of hydrogeologic units using matrix properties. U.S. Geological Survey Water-Resources Investigations Report 98-4243.
- Lide, D.R. (ed.) 1999. *CRC handbook of chemistry and physics*. CRC Press, Washington, DC.
- Webb, P.A., and Orr, C. 1997. *Analytical methods in fine particle technology*. Micromeritics Instrumentation Corporation, Norcross, GA.