

## **PORE SIZE MEASUREMENT IN SOIL: A REVIEW OF METHODOLOGY**

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(Received 14 October 1999; accepted 7 July 2000)

An accurate and complete analysis of pore size distribution of a soil is useful for interpreting structural changes and for predicting water flow rate, water storage capacity, water availability to plant and rate of diffusion of gases. Water release curve (relationship between soil water content and suction) is widely used to obtain pore size distributions but is not dependable in fine-textured soils that shrink on drying. There are other methods (nitrogen sorption, mercury intrusion porosimetry, non-polar liquid desorption and image analysis) to overcome this problem. In analysing the pore size distribution from the methods (except image analysis) generally capillary model is used assuming the pores are cylindrical or slit-shaped. Like water desorption, other methods also have disadvantage of pre-drying of soil samples without changes in the pore system along others. Stereological image analysis (of a soil) can provide quantitative three-dimensional information about its structural parameters and also used to identify patterns of water and solute movement. These methods of pore size measurements are discussed, compared and their use in soil studies is discussed.

**Key words:** Pore size distribution, Water release curve, Nitrogen sorption, Mercury intrusion porosimetry, Non-polar liquid desorption.

### **Introduction**

Soil structure may be defined either in terms of the combination of primary (individual) soil particles into secondary particles as aggregates, clusters or peds of different sizes, shapes and arrangement or in terms of porosity and pore size distribution (Marshall 1962). The evaluation of soil structure through the knowledge of the amount, size, configuration and distribution of soil pores is perhaps most meaningful because it affects many important soil physical properties, especially those pertaining to water retention and transport of solutions, gases and heat. Therefore, information concerning soil porosity and pore size distribution is more useful in characterising soil as a medium for plant growth rather than the soil particles. A number of scientists have reported the studies of pore size distribution as it affects plant growth and as a general method for defining the structure of porous materials (Barden and Pavlakis 1971; Hall *et al* 1977; Guidi *et al* 1985; Wu *et al* 1990; Pagliai *et al* 1993). It affects the environment for the plant roots through its effects on water and oxygen supply and penetration of roots (Bowen 1981). Roots commonly do not penetrate into the soil peds where rigid pores are smaller than the diameter of the root tips or root hairs (Aubertin and Kardos 1965).

The information regarding the pore size distribution of a soil is also useful for predicting water infiltration rate, water availability to plants, water-storage capacity, drainage and aeration status. As far as the infiltration or drainage is concerned,

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a soil must have enough large pores to allow rapid infiltration of surface water followed by early drainage so that oxygen does not become limiting. At the same time the soil should have an extensive and comprehensive system of pores small enough to resist gravitational drainage. Yet large enough to release significant quantities of water to plant roots without causing the water in the roots to fall to low energy levels (Cary and Hayden 1973). This pore size group which restrains water from drainage it readily releases to plant roots is responsible for the amount of "available water" in the soil. Ideally, this quantity of water should be as large as possible without sacrificing adequate infiltration and aeration.

*Soil pore size classification.* Soil pore size may range from less than  $10^{-2}$   $\mu\text{m}$  for clay materials to several hundred  $\mu\text{m}$  for sand (Sills *et al* 1973). Thus there is need to group them into different classes according to their sizes and functions. An important problem associated with characterization and classification of soil pores is lack of a standard terminology related to their distinct size ranges and functions and a second problem relates to the need for identifying pore size in terms of an equivalent cylindrical diameter (Danielson and Sutherland 1986). The second problem results from the complex and variable pore shapes and their interconnected nature.

The need for a standardized classification scheme has been identified by different researchers (Russell 1973; Greenland 1977; Bouma 1981; Cannell and Jackson 1981; Luxmoore 1981) they suggested various but almost similar classifications (Table 1). Russel (1973) divided the pores into four classes

according to their sizes i.e. coarse >200 μm; medium 20-200 μm; fine 2-20 μm; and very fine <2 μm. In the absence of a generally accepted terminology to relate pore size to function, Greenland (1977) proposed the terms fissure, transmission, storage and residual pores for four categories and he selected equivalent pore diameter of >500 μm, 50-500 μm, 0.5-50 μm and <0.5 μm respectively. Cannell and Jackson (1981) grouped the different pore sizes into three categories (1) large pores which are air filled at field capacity and range between 100 and 300 μm in diameter (2) medium pores, which are small enough to hold water against gravity and at that time water permit to be withdrawn by plant roots to potentials of about -1500 Kpa corresponding to diameters between about 60 and 0.2 μm and (3) small pores from which plants are unable to remove water. Luxmoore (1981) proposed a classification of micropores, mesopores and macropores in a letter to the editor of Soil Science Society of America Journal. The purpose of the letter and classification was to call attention to the need for some standard soil porosity terms. The suggested classification (microporosity, mesoporosity and macroporosity) corresponds to three dominant regimes of soil water behaviour and are associated with some convenient equivalent pore diameter ranges. Skopp (1981) presented his concern in a letter to editor about the soil pore classes suggested. He wrote that firstly pore sizes are based on soil water potential which is contingent with particular model (for instance capillary) which may not apply to cracks or slits; secondly the classification ignores the pore morphology (continuity and tortuosity) and thirdly it is difficult to put the arbitrary limits as they correspond to the soil water potential. In response to the Luxmoore (1981) proposal, Bouma (1981) suggested another already extant system (Brewer 1964) based on methods of measurement and replacing the term "void" by "pore" (Table 1). The system has five classes i.e. macropores (with four subclasses i.e. coarse, medium, fine, and very fine), mesopores, micropores, ultramicropores and cryptopores.

Although the pore size distribution of many porous materials have been satisfactorily measured by a variety of techniques, only a few have been adequately evaluated and commonly accepted for use with soils. The techniques used to measure pore size distribution on soils must be capable of measuring a very wide range of pore size with reasonable accuracy. At present no suitable single method is available to encompass the complete range of pore size present in soils and different methods in combination are used to measure the different pore sizes. The present paper attempts to review various techniques of measuring pore size in soils along with water desorption.

**Table 1**  
Defined size classifications of pores

Reference	Class name Subclass	Capillary potential (Kpa)	Equivalent diameter (μm)
Russel (1973)	Coarse		>200
	Medium		20-200
	Fine		2-20
	Very fine		<2
Greenland (1975) (1977)	Fissure		>500
	Transmission		50-500
	Storage		0.5-50
	Residual		<0.5
Cannell & Jackson (1981)	Large	> -10	100-300
	Medium	-10 to -1500	60-0.2
	Small	< -1500	<0.2
Luxmoore (1981)	Microporosity	< -30	<10
	Mesoporosity		-30 to -0.3
	Macroporosity	> -0.3	>1,000
Bouma (1981)	Macropores		
	Coarse		>5,000
	Medium		2,000-5,000
	Fine		1,000-2,000
	Very fine		75-1,000
	Mesopores		30-75
	Micropores		5-30
Ultramicropores		5-0.1	
Cryptopores		<0.1	

*Methods of pore-size measurement: Water desorption.* The pore size distribution of a soil has generally been obtained by observing the relationship between water content and suction. Water can be removed from a pore of a given radius when a suction is exerted on that water which is greater than the force acting to retain the water in the pore. The minimum suction required to empty a pore of given radius can be calculated from the relationship between suction and the radius of curvature of the meniscus at the air-water interface. The pressure difference (ΔP) across an air-water meniscus, as in a capillary tube, is expressed as

$$\Delta P = \frac{2\gamma}{r_c} = \frac{2\gamma \cos \theta}{r_p} \dots\dots\dots (1)$$

Where γ is the surface tension of liquid, r<sub>c</sub> is the radius of curvature of the meniscus, Cos θ is the contact angle of liquid to solid and r<sub>p</sub> is the radius of the tube. The pore will empty when the suction applied exerts sufficient force on the meniscus that its radius of curvature equals to the radius of the pore. The contact angle θ at this point is zero and value of

Cos zero is one (Cos 0 = 1). Thus the equation (2) can be rewritten as

$$\Delta P = \frac{2\gamma}{r_p} \dots\dots\dots (2)$$

Where the  $r_p$  is considered to be the equivalent cylindrical pore radius. As water is removed from the porous medium, the radius  $r_c$  of the curvature of air-water interface will decrease. When  $r_c$  decreases to the effective radius of a given pore that pore will drain as further water is removed. All pores with effective radii of a lesser value will still be water filled. When a given water content in a soil sample is attained by bringing the water matric potential to a desired value using a porous plate. The pore size dividing water-filled pores and drained pores can be calculated from Equ (2)

Pore size in soils of different textures has generally been obtained by the application of equ (2) to the moisture characteristic curve (Olson 1985; Marshall and Holmes 1988). This method is mainly used over the range covered by suction and pressure membrane procedures up to  $10^{-7}$  m radius.

However, in fine textured soils, the moisture content/suction desorption isotherm does not reflect the pore size distribution of the original soil. This is due to sample shrinkage and continuous changes in pore size distribution associated with changes in applied suction. Non-polar liquids have been used to avoid these problems instead of water for desorption but these require samples to be dried before saturation. When drying is done by direct evaporation of water, the decreases in porosity and changes in the distribution of pore sizes are inevitable. So, other drying methods like freeze-drying and soil water replacement by organic liquids are used.

*Comments.* Lawrence (1977) pointed out that in fine textured and swelling type soils, the calculation of pore size distribution by water desorption method is likely to be invalid for two reasons. Firstly, it is unlikely that the calculated pore size distribution represents the pore size distribution of the soil at all moisture contents. Because a stress on pore walls during an increase in suction causes a reduction in pore size and that the desorption of the water is delayed until a higher suction is reached; at that time the stresses on the pore wall have increased more and resulting in further decrease in pore size, consequently affecting the pore sizes as well as air entry point (Childs 1969). Secondly, during water desorption most of the fine-textured soils shrink and particle rearrangement takes place (Quirk and Panahokke 1962). Therefore, it is necessary that soil should be rigid and not change its volume with water content (Olson 1985). Moreover, a drying rather than a wetting soil is used because ( is more likely to be zero and the pores are not tubes of circular section so that an

effective size is measured (Marshall and Holmes 1988).

Regardless of the limitations of the water desorption method for measuring the pore size distribution, it serves as the most commonly used technique.

*Non-polar liquid desorption.* To avoid the problems which arise when a polar liquid such as water is desorbed from a fine textured soil, the removal of non-polar liquids for examples, benzene and tetrachloroethane has been used. Interaction between non-polar liquid and clay surface are weak and thus equ (2) may be applied with more confidence. The use of non-polar liquid desorption methods has been reported by Quirk and Panahokke (1962).

Desorption measurements are made on samples that have been dried and then saturated with a non-polar liquid. The apparatus and experimental procedure are very similar to those used for determining a moisture characteristics curve i.e. suction plate, pressure plate and vapour sorption methods. Pores between  $10^6$  nm and  $10^5$  nm equivalent diameter have been measured by using this methods. Equilibration time are often long (in the order of days) especially at higher suctions.

*Mercury intrusion technique.* Mercury intrusion porosimetry has been utilised as a reliable method for determining pore size distribution of a wide variety of porous solids. The technique has been further expanded for soils to measure the pore size and volume within soils in the diameter range between 200 and 0.001  $\mu\text{m}$  (Nagpal *et al* 1972; Sills *et al* 1973; Lawrence *et al* 1979; Newman and Thomasson, 1979; Olson 1985 and 1987; Kozak *et al* 1991).

The theoretical basis for using mercury intrusion technique to determine pore size distribution is identical to water desorption method, except that the pressure is required to force the mercury into the pores instead of suction to remove water from a saturated system. Because the mercury is a non-wetting liquid for most of materials, the contact angle  $\theta$  has a value greater than  $90^\circ$ . Thus it will enter the pore of a solid only if an external pressure is applied. The pressure required is dependent on the value of the contact angle, the surface tension, the size and geometry of the pore. The minimum pressure 'P' which must be applied to force mercury into a pore with radius 'r' is calculated from the equ (1).

As reported by Aylmore and Quirk (1967) that clay particles appear to be plate like, Sills *et al* (1973) when used the mercury intrusion porosimetry to measure pore size distribution in clay mineral mixtures, chose a slit-shape pore model instead of cylindrical pore model. For slit-shaped pores the parameter  $r_p$  in equation (1) is replaced by the plate separation 'd'.

The pore sample is dried, evacuated and inundated in mercury and pressure is applied hydraulically in discrete steps. The diminution of bathing mercury is measured and equated to the volume of pores invaded at each pressure step. Equ (1) is used to calculate the equivalent radii of the smallest pore filled with mercury, assuming a model of cylindrical tube of different sizes.

*Comments.* The mercury intrusion porosimetry method for determining pore size distribution in soil is convenient and fast. However, several sources of error have been identified.

Before soil can be intruded with mercury, the sample must be thoroughly dried. Direct drying by evaporating water may alter the size of natural pores, especially when soils are high in swelling type clay and the porosity determined by mercury porosimetry may not be representative of the original porosity of the sample in a moist condition (Thompson *et al* 1985; Olson 1987). But soil shrinkage with drying was not found to be a significant problem in mechanically strong soils high in sand or silt (Lawrence 1978; Olson 1985).

The value of mercury-solid contact angle is uncertain. Reported values range from 117 to 150° for different porous materials (Lawrence 1977). The value for clay-mercury systems has been reported to range from 139 for montmorillonite to 147° and for illite and kaolinite Sills *et al* (1973) used the value of 140° for kaolinite/illite mixture and goethite.

Uncertainty also exists concerning the surface tension of mercury. Values have been reported to range from 0.432 to 0.515 J<sub>m</sub><sup>-2</sup> and the most probable range is from 0.472 to 0.487; a value of 0.473 has commonly been used for soil porosity measurements (Danielson and Sutherland 1986). Lawrence (1978) pointed out that the error associated with surface tension variation is not very important when compared to those related to contact angles.

Apart from all the technical limitations (drying of sample, contact angle, mercury surface tension), there are also some practical limitations. For example, this technique can be used to measure the pore size ranged between 200 and 0.001 μm but in practice it measures only the soil matrix (intra-aggregate) porosity (which is relative uniform) and difficult to measure large pores inter-aggregated because of small sample size.

*Nitrogen sorption.* The nitrogen sorption method involves the analysis of either the adsorption or desorption branch of nitrogen isotherm usually obtained at -196°C (78° K). The nitrogen sorption technique has been used by Gregg & Sing (1991), Sills *et al* (1973) and Aylmore & Quirk (1967) to study the specific surface area and pore size distribution in the range below 20 nm in microporous systems. It is insensitive for pore sizes larger than 20 nm. Pore size distributions are often

obtained by assuming cylindrical pore and applying the Kelvin equation. This equation relates the pore radius *r*, to the relative pressure *p/p*<sup>0</sup> by the expression

$$\frac{RT}{V_m} \ln \frac{P}{P_o} = \frac{2\gamma \cos \theta}{rRT} \quad \dots\dots\dots (3)$$

Where *P*<sup>0</sup> is the saturated vapour pressure, *P* is the vapour pressure of the liquid  $\gamma$  is the surface tension, *R* is the gas constant, *T* is the absolute temp, *V<sub>m</sub>* is the molar volume of the liquid (nitrogen) and  $\theta$  is the solid-liquid contact angle.

Sills *et al* (1973) observed that slit-shape pores were present in fine textured soils rather than cylindrical. Therefore, they used a modified version of Kelvin equation, incorporating the thickness of the adsorbed film, to determine the plate separation for clay soils. The modified Kelvin equation is

$$d - 2t = - \frac{2V_m \gamma}{\ln P/P_o RT} \quad \dots\dots\dots (4)$$

Where *d* is the maximum distance of plate-separation at which capillary evaporation can occur for a given relative pressure, *p/p*<sup>0</sup>, and 't' is the thickness of the adsorbed layer.

*Comments.* Although nitrogen sorption has been used on large scale to determine specific surface area of both clay minerals and whole soil, the technique seems to have been relatively little used to measure pore size distribution. This technique can only be used to measure the pore <0.02 μm and becomes sensitive for pore >0.02μm. Whereas, in soil system, agriculturally important pores (which hold plant available water) generally ranged between 30-0.1μm. Techniques used to measure pore size distribution on soil should therefore be capable of measuring the agriculturally important pore range.

*Image analysis technique.* Image analysis techniques have been used by many researchers to measure voids in thin sections of the soil (Murphy *et al* 1977; Bouma *et al* 1979; Jongerius and Bisdom 1985; Haman *et al* 1995; Bodziony *et al* 1995; Ringrose-Voase 1996; Robertson and Campbell 1997). The introduction of image analysing computers, such as the Quantimet 720, makes it possible to measure and characterize fine pores (up to 20 μm) in the thin section, more rapidly and accurately than the conventional methods discussed before (Murphy *et al* 1977; Jongerius and Bisdom 1985). Jongerius and Bisdom (1985) reported that the combination of image analysing computer technique and back-scattered electron scanning images enables distributions of shapes of voids and their sizes up to 0.0075 μm to be measured.

*Principle of the image analysing computer.* In an image analysing computer, quantimet 720 (Fisher 1971) (Fig 1), an image is formed by a microscope (viewing thin sections

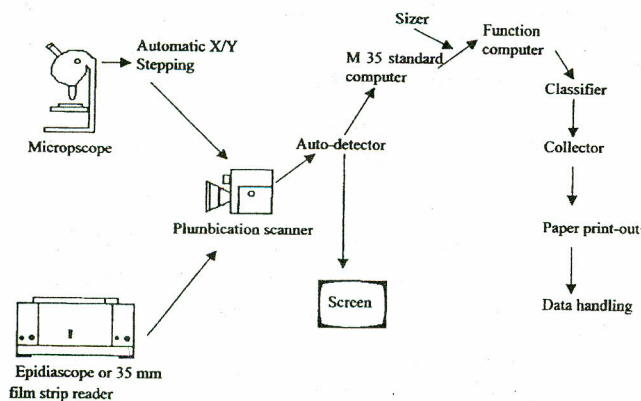


Fig 1. Schematic diagram of Quantimet 720 image analyser.

directly) an epidiascope or a 35 mm film strip reader (viewing photographs of thin sections) and is scanned by a plumbicon television camera and displayed on a monitor screen (Murphy *et al* 1977). The image is scanned in a raster of 720 line, each line consisting of 910 points known as picture points (pp). The full screen is thus composed of over 650,000 pp but measurements are made within a smaller variable live frame with a standard size of 625 lines of 800 points giving 500,000 pp. Each picture point is square and joins neighbouring points in horizontal and vertical directions and thus be considered as units of area or of linear measurement. The calibration of a pp is determined by the magnification; with a 32 x objective on a Lietz orthoplan microscope, the length of a pp is 0.41  $\mu\text{m}$  and with a 100 x objective 0.05  $\mu\text{m}$ . In the case of epidiascope which has three lenses 32 mm, 44 mm and 63 mm, the calibration of the pp depends on the degree of enlargement of the photograph.

The signal from the scanner is passed to a detector module and then to a monitor for display. In the detector, features are selected on the basis of their common grey level characteristics. The detector distinguishes in practice about six to eight different grey levels between or at white and black. The grey level of each picture point is separately assessed in the detector and the signal passed into a series of modules which make the areal measurement. A computed display is superimposed on the features being measured on the monitor that also shows numerically the results of a particular measurement.

Image analysis provides only one-dimensional information and the most important issue in the investigation of three-dimensional soil medium. Bodziony *et al* (1995) used the stereological analysis to investigate quantitative physical parameters (soil structure, aggregation, porosity) describing the three-dimensional structural changes of the soil. Computer Image Analysis method together with a scanning electron microscope even provide the better three-dimensional

analysis of the soil sample (Haman *et al* 1995). Forrer *et al* (2000) processed digitised image to quantify the concentration and flow paths of the mobile dye tracer (Brilliant Blue FCF) in soil profile. They analysed digitised slides/photographs on a workstation (sum APARC Ultra 1) using the software packages PCI, Version 6.01 (PCI, Ontario) and IDI, Version 4.01 (Interactive Data Language, Research System Inc., CO).

*Comments.* Impregnation and sectioning technique estimates the soil porosity and pore size distribution and also characterises the pores by shape and orientation. However, it is not necessary that the testimated total porosity will closely relate to the channelling porosity of the soil. Bullock and Thomasson (1979) show the total macroporosity measured from thin sections which is poorly correlated to equilibrium suction measurements. Moreover, it is not easy to make the measurements on pores <30  $\mu\text{m}$  diameter.

## Conclusion

Among the techniques mentioned above, water desorption and mercury intrusion porosimetry serve as the most common techniques. Water desorption is most convenient and suitable method for medium and light textured soils. Mercury intrusion porosimetry is the most satisfactory for the fine pores range  $10^5$  to 10 nm. An accurate characterisation of complete pore spectrum by a single method is quite difficult. Information concerning the geometry of the pores (shape, orientation and continuity) are as important as sizes of the pores. This information can be obtained by coupling computer image analysis with a scanning electron microscope and scanning reflected light microscope computer topography. The mercury intrusion, computer image analysis, stereoanalysis and computer image analysis plus scanning electron microscope techniques are costly and has limited use. The water desorption technique with all its limitation is widely used in most of the laboratory and is also a relative low-cost.

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