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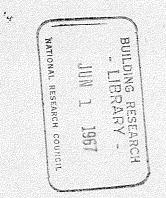
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REPLACEMENT OF WATER BY AIR IN SOIL PORES

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ΒY

P. J. WILLIAMS





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April 1967

DEPLACEMENT PAR L'AIR DE L'EAU DES PORES DU SOL

SOMMAIRE

Les sols composés de minéraux ayant le même indice de réfraction que l'eau sont translucides après saturation en eau. Toute présence d'air dans les interstices est parfaitement visible. On a utilisé ces sols pour étudier le processus de remplacement par l'air de l'eau des interstices.

Quand les pressions externes de l'air et de l'eau sont réglées de façon à produire un égouttement rapide de l'eau, l'air se répand brusquement au travers du sol poreux lorsqu'on établit une certaine différence entre les pressions de l'air et de l'eau. Si l'égouttement se produit plus lentement, l'air en solution se dégage progressivement, agrandissant les bulles, et produisant une rupture progressive des ménisques d'une façon difficilement explicable par la théorie de la capillarité.



Replacement of Water by Air in Soil Pores

By P. J. WILLIAMS*

Soils composed of minerals having the same refractive index as water are translucent when saturated. Any air in the pores is fully visible. Such soils have been used to observe the manner in which air may replace water in soil pores. Where the external air and water pressures are established such as to cause rapid drainage of water, the air suddenly spreads through the pore structure at a certain air/water pressure difference. Where drainage occurs more slowly, a progressive diffusion of air in solution occurs, giving enlargement of bubbles, as well as a progressive breakdown of menisci not easily explainable from capillary theory.

WHEN a soil is not completely saturated, the interfaces between the air and water in the soil are confined by the soil pores. Because of capillarity, a pressure difference then exists between the water and the air; this is represented by the capillary equation:

$$p_a - p_w = \frac{2\sigma}{r} \cos \theta \qquad . \qquad . \qquad (1)$$

where

 $\sigma = interfacial energy$

r = the radius of curvature of the interface.

In the case of water and soil minerals the contact angle θ is generally assumed to be 0° , and the radius of curvature *r* is equivalent to the radius of the pore in which the interface lies. The lower the moisture content of the soil the greater is the difference $p_a - p_w$, because the interfaces then lie in smaller pores and constrictions of pores. This constitutes the well-known suction/moisture content relationship.

This relationship can be determined in pressure membrane apparatus consisting of a cell with a drainage outlet covered by a membrane. The soil sample is placed on the membrane and the air pressure, p_a , in the cell raised. The membrane has pores so small that they remain water-filled at all values of p_a in question and the air cannot pass through it. Water, however, drains out of the soil until the water pressure, p_w , in the soil is in equilibrium with the atmospheric pressure. The moisture content of the sample is then determined. The test is repeated for a number of different values of p_a .

Several days are required for the moisture

content of even small samples to attain equilibrium. The low permeability of the membrane and of the soil itself (as the moisture content is reduced) is generally thought to be the main cause. In recent years membranes have become available which have substantially greater permeability. When using these membranes with quite permeable soils it would be expected that drainage should be completed in a few minutes or hours, i.e., that the water would assume a uniform pressure throughout the sample within that time.

In tests using such membranes it was found that the drainage occurring after the application of an air pressure increment rapidly decreased, becoming imperceptible after a few minutes. A suction/moisture content relationship could be obtained on the basis of a series of pressure increments applied over, for example, one hour. The relationship so obtained differed, however, from that obtained by conventional procedures (Fig. 1); in particular there was a marked inflection, due to the release of a considerable quantity of water at a certain value of $p_a - p_w$, (Williams, 1966 (a)). For a range of values of $p_a - p_w$, the water content was lower in the conventional test, implying that some drainage continued over a long period of time. The presence of the inflection in the rapid test can be more easily explained than can its absence in the slow test.

The pore system of a soil consists of interconnected openings of various sizes, giving a network of channels with innumerable variations of diameter. Consider a soil homogeneous in a microscopic sense and initially saturated. For relatively low values of $p_a - p_w$,

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only a few large pores, either opening directly to the surface or in direct connection with the external air channels nowhere narrower than of radius r, will be emptied of water. As $p_a - p_w$ is increased somewhat, the radius r is decreased, Strings of interconnected air-filled pores extend a little deeper into the sample, but their penetration is limited by occurrence of constrictions smaller than r. When the value of $p_a - p_w$ is such, however, that these strings extend through perhaps ten to 100 pores (representing a layer perhaps 0.5 mm thick) it becomes likely to the point of certainty that there will always be additional openings of appropriate radius, through which the air may advance further. There is, therefore, a critical pressure difference, $p_a - p_w$, at which air-filled channels, instead of being confined to a thin surface layer, can spread through the sample. This value of $p_a - p_w$ is referred to as the air-intrusion value and is associated with a substantial drainage of water. The corresponding value of requals r_c , which is the radius of the largest continuous opening through a soil sample big enough to contain a statistically representative assemblage of particles.

The term air-entry value has been used (Bishop and Henkel, 1962) to describe the air-water pressure difference at which air can be forced through initially-saturated fine-pored filters used in soil strength testing. Brooks and Corey (1964), in a theoretical treatment, refer to a similar quantity, "bubbling pressure . . . a measure of the maximum pore size forming a continuous network of flow channels within the medium". Topp, Klute and Peters (1966) have made observations on the influence of duration of test and other factors on the form of suction/ moisture content curves.

The explanation of the air-intrusion value already given leaves open the question of why this kind of suction/moisture content curve differs from that obtained by the slower conventional test procedure. Several authors believe that the form of the curve obtained by the latter procedure is a measure of poresize distribution (Donat, 1938; Schofield, 1938) and that for any value of $p_a - p_w$ the remaining water content is situated only in pores of equal or smaller radius than that given by equation (1). This would require nucleation and enlargement of air bubbles in pores of this size but which are totally isolated by smaller pores. Powers (1962) believes a process of this type to occur in cement.

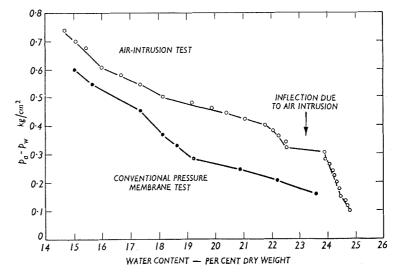
Experiments are now reported that enabled direct observation of the manner of water replacement by air under various test conditions.

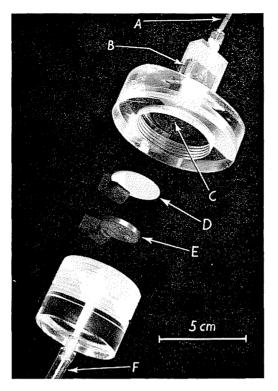
EXPERIMENTAL WORK

Soils were prepared by crushing the minerals chiolite (5 NaF.3A1F₃) or cryolite (Na₃A1F₆). These translucent, smoky-white minerals have a refractive index similar to that of water (in which they are substantially insoluble). The crushed material is white and opaque, but when mixed with water it is transparent in thin layers. Air within the pores is then fully visible.

Small pressure membrane cells were constructed of Perspex, such that the sample in the cell could be viewed through a microscope. Depending on the magnification desired and the grain-size composition of the sample, the microscope could be focused on a plane up to about 2 mm within the sample. One type of cell is shown in Fig. 2 and another in Fig. 3. The cells were so designed that there is a sufficient ratio of membrane area to sample height and volume for drainage from the sample to lead to hydrostatic equilibrium

Fig. 1—Suction/moisture content relationship obtained for a silt containing some clay, by a conventional pressure membrane test, and by an air-intrusionvalue test in which each air pressure increment was applied as soon as perceptible drainage had ceased





throughout the water in the sample in a short time. In addition, the dimensions of the cell are determined by the working distances of the microscope at the magnifications desired.

The following are examples of the four kinds of test carried out:

Observations of Drainage from Pores During Rapid Test.—A sample of chiolite consisting of a 74μ -250 μ particle diameter fraction was prepared as a slurry, boiled, cooled, and placed in the apparatus (Fig. 2). A preliminary test showed that the permeability of the sample with the membrane in the cell was such that a 20-cm head between the top and bottom of the sample gave a flow of $1.8 \text{ cm}^3/\text{min}$.

The drainage was measured in a pipette and is shown as a function of time in Fig. 4 (it could also be shown on a similar plot to that in Fig. 1). Photomicrographs taken at the times shown by the arrows in Fig. 4 are reproduced in Fig. 5. Incident lighting was used and the saturated sample appears dark. In the first two photographs air is visible only in a thin surface layer, while slight compression of the sample and membrane assembly gives a small amount of drainage. In the third photograph the air has spread substantially, and in the next taken only ten seconds later, when the air pressure was 0.055 kg/cm^2 , the air has spread throughout the sample. This pressure, therefore, represents the air-intrusion value.

A second test of the same type is illustrated in Figs. 6 and 7. The sample consisted of equal parts of $250 \,\mu$ -149 μ , $149 \,\mu$ -74 μ ,

Fig. 2—(Left) Pressuremembrane apparatus used for tests with low microscope magnification *A*—Port for compressed air. *B*—Part of sample viewed under microscope. *C*—Base of sample. *D*—Membrane. *E*—Bronze filter. *F*—Drainage (to burette).

 74μ - 44μ , and $<44 \mu$ fractions. The sample was mixed to a slurry and not boiled. Entrapped air bubbles are visible and the presence of the finer grain-size fractions also reduces the transparency of the material. The intrusion of air gave a somewhat different pattern of air-filled regions in this test.

In some experiments with fine-grained and relatively compressible material, the paths taken preferentially through the soil by the air were in fact observed to be fully-developed cracks. As the air entered down these paths the soil separated, the crack finally having a width perhaps ten times the radius of the air-water interfaces. Associated with such cracking is an increment of drainage, which is

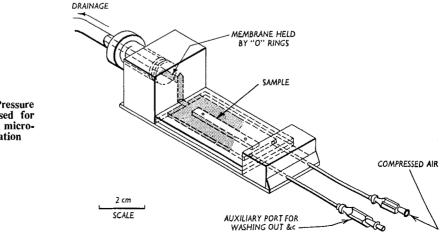


Fig. 3—(Right) Pressure membrane cell used for tests under high microscope magnification distinct from that due to intrusion of air into pores.

Intrusion of air apparently took place only by continuous extension of the air-filled regions: in the boiled samples no new isolated air bubbles arose and in the unboiled samples no significant enlargement of isolated bubbles already present was observed. In Fig. 10 a string of air-filled pores is seen (in transmitted light) after air-intrusion in a sample consisting of equal parts of $<44 \mu$, 44μ – 74μ , 74μ – 149μ fractions, and 1/2 part of 149μ – 250 μ . The dark regions are shadows of airfilled pores. Air-intrusion occurred in jumps, in which strings of perhaps twenty or even fifty or more pores empty apparently simultaneously. At the height of the air-intrusion process these jumps occur at short intervals of time or simultaneously, in various parts of the sample.

Observations of Slow Enlargement of Entrapped Bubbles.—In these tests the value of $p_a - p_w$ was raised to just below the airintrusion value, which had been determined previously. Bubbles trapped some millimetres in from the sample surface, following its preparation as a slurry, were then observed. In Fig. 8(a) bubbles are shown about twenty minutes after the application of the air pressure, in this case 0.22 kg/cm^2 (the airintrusion value was 0.25 kg/cm^2 for this material which was similar to that described in the previous paragraph). By this time

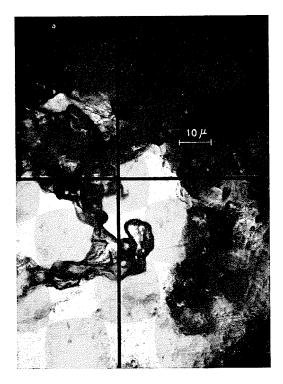


Fig. 10—Air intrusion into string of pores. The dark areas are air-filled

volume changes of the bubble associated with transient pressure changes in the water were complete. The bubbles enlarged slowly during the next two or three days, and in Fig. 8(b) the same bubbles are seen after twenty-six hours. Because of poor control the air pressure varied slowly by up to $\pm 5\%$ in this case. Other tests showed that this has no significance for the observation.

Attempts to Observe Nucleation of Air Bubbles.—A third series of tests also involved samples where $p_a - p_w$ was raised to a value just below the air-intrusion value. The highest value of $p_a - p_w$ used was 0.3 kg/cm^2 using a $< 44 \mu$ fraction with more than 80% by weight medium and coarse silt, and with an air-intrusion value of 0.35 kg/cm^2 . $p_a - p_w$ was established both by raising the air pressure, and in other tests by lowering of the water pressure. The sample was repeatedly examined by traversing with the microscope to ascertain whether new air bubbles arose during a period of days. In some cases photographs were taken repeatedly. In no cases were bubbles observed to have formed.

Changes at Air-water Interfaces During Tests of Long Duration.-It was noticed that although no new bubbles were formed when the value of $p_a - p_w$ was maintained for long periods somewhat below the air-intrusion value, there was a progressive extension in the amount of air-filled regions of the sample. Observations were therefore made under high magnification, over periods of days, of interfaces between the soil water and external air. The progressive advance of such interfaces into the soil is seen in Fig. 9. In this example $p_a - p_w$ was established at 0.30 kg/cm², using the material described above having an air-intrusion value of 0.35 kg/cm^2 .

DISCUSSION

The experiments show that there are at least three distinct processes by which air may replace the water in the soil pores. The simplest process is demonstrated by experiments of the first type and is sufficiently described by equation (1). Air-water interfaces retreat inwards from the soil surface until they are confined by pores such that their radius is that given by equation (1), for the appropriate pressure conditions $p_a - p_w$. When $p_a - p_w$ is equal to or greater than a certain critical value-the air-intrusion value-there are interconnected air-filled pores through the sample. Fig. 10 is a projection, but it is possible to make an estimate of the radius of the interfaces as $5 \mu - 15 \mu$. In this case the value of $p_a - p_w$ was 0.24 kg/cm^2 , and the value of 6μ is, therefore, predicted by equation (1).

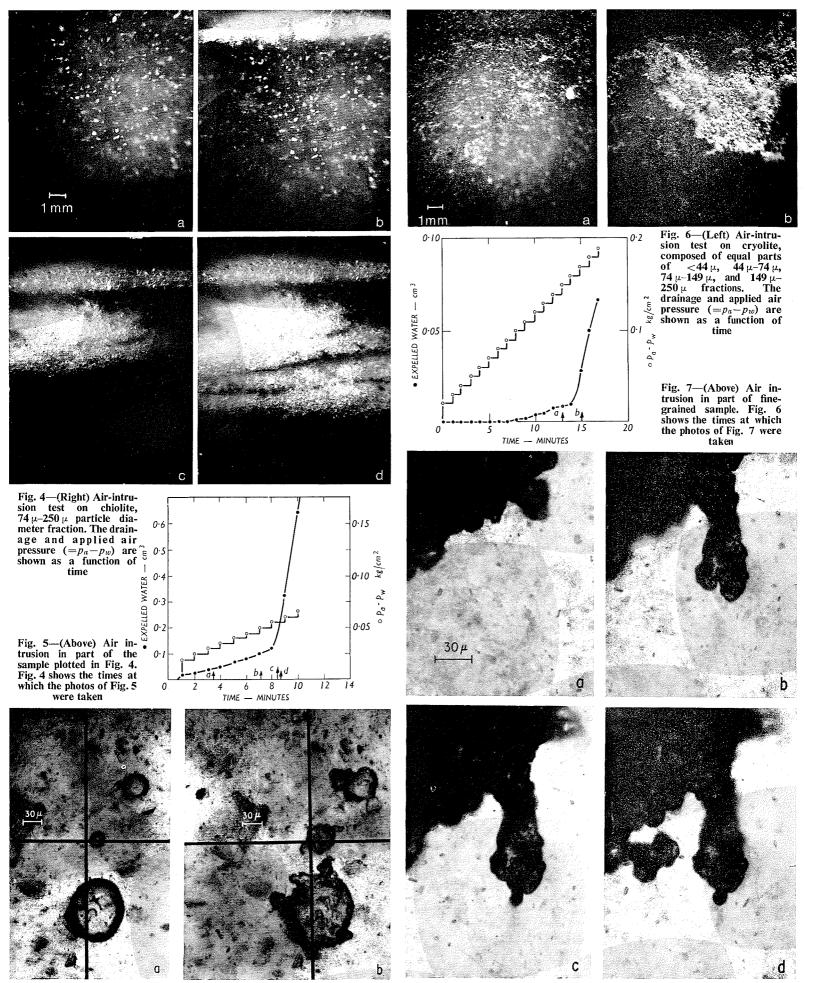


Fig. 8--Entrapped air bubbles (a) bubbles 20 min after establishment of $p_a-p_w=0.22 \text{ kg/cm}^2$; (b) 26 h later

Fig. 9—Progressive advance of air into soil by break-down of menisci under constant $p_a - p_{iv}$ of 0.30 kg/cm²; (a) 24 h after establishment of $p_a - p_{iv}$; (b) after 12 days; (c) after 14 days; (d) after 19 days

Fig. 5 shows that in a soil composed of material in a narrow grain size range, and which is not susceptible to significant compression, the spreading of air occurs rather uniformly at the air-intrusion value. The somewhat banded appearance is due to some sorting (sedimentation) that appears to be inevitable when samples are placed. The slurry has to be inserted in small quantities through the air port or base of the cell; tapping the cell is necessary to ensure filling of the space above the membrane.

The second material, illustrated in Fig. 7, was somewhat compressible in bulk and tended to decrease slightly in volume under the raising of the air pressure. This compressibility is largely responsible for the tendency for air-intrusion to occur preferentially along certain paths (incipient cracks) or along the sides of the pressure membrane cell. The air-intrusion value is also well-defined, however, for this material.

The formation of true cracks, observed in some tests with compressible materials, is not to be regarded as a replacement of water in pores by air. It is a consolidation due to the effective stress set up in the soil skeleton by the establishment of the air-water pressure difference. This cracking commonly occurs for values of

$$p_a - p_w < \frac{2}{r_c},$$

i.e., before the air-intrusion value is reached. The drainage associated with this volume change must not be confused with that occurring as air replaces water in the pores.

A second process involving replacement of water in pores by air, demonstrated by experiments of the second type, occurs much more slowly. It depends on the rate of diffusion of air through the water to bubbles. This occurs if the interface radius of entrapped bubbles is greater than that of the interfaces to the external air. The solubility of air in water depends on the pressure of the air (according to Henry's Law the mass of dissolved gas in a definite mass of the liquid at a given temperature is very nearly proportional to the (partial) pressure of the gas). If the pressure of the water is uniform, then the air pressure will be lower where the interface radius is greater, according to equation (1). There is thus a concentration gradient for air in solution from the vicinity of small interfaces towards larger interfaces. Diffusion occurring along this gradient results in air coming out of solution, when the saturation concentration for the air pressure at the larger interface is exceeded. Entrapped bubbles can thus increase in size. As bubbles grow they will tend to penetrate smaller pores until the interfaces have a radius equal to that of the interfaces to the external air.

The general equation for diffusion between two locations is:

$$\frac{dQ}{dt} = K \frac{dc}{dx} dy dz \qquad . \qquad . \qquad . \qquad (2)$$

where

 $\frac{dQ}{dt}$ = rate of diffusion, cm³/s^{*}

 $K = diffusion \ coefficient, \ cm^2/s.$

- dc = difference in concentration between the two locations, cm³/cm³
- dx = distance between the locations, cm.
- dy dz=cross-sectional area for diffusion, cm², and the volumes of air, cm³, are reduced to S.T.P.*.

The diffusion coefficient of air in water is $2 \cdot 10^{-5}$ cm²/s (Liebermann, 1958). The geometry of the system under consideration is complicated but insertion of appropriate values into equation (2) indicates that the observed rate of growth of the bubbles is compatible with that to be expected.

The third process of drainage observed in the experiments of the fourth type described above, occurs very slowly. There is no immediate explanation for the gradual advance of interfaces through the soil. This occurred in the test illustrated (Fig. 9) with $p_a - p_w$ about 14% less than the airintrusion value for the material in question. It implies that the right-hand term in equation (1) is lower by this amount than in the case of the "rapid" procedure under which the air-intrusion value is measured.

It is possible that the radius of the pore openings is changed by local particle rearrangement, especially in the neighbourhood of the air-water interfaces. Because of the irregular shapes of particles the local stresses arising from interfacial energy effects are complex (such stresses due to ice-water interfaces are discussed by Everett and Haynes, 1965), and might result in such particle movements. The consolidation process described above also presumably involves particle reorientation.

The contact angle of the air-water interface to the particle surface has been assumed to be 0 and has been ignored in equation (1). In fact there may be a small contact angle 0 such that the right-hand term must be increased by a factor $\cos \theta$. The contact angle might vary with time. Topp (1966) investigated the effect on the interfacial energy air-water of the proximity of various materials. In the present apparatus the Perspex could be responsible for a decrease of perhaps 3%. This appears insufficient to account for the breakdown of the menisci.

An alternative possibility is that there

^{*} For dimensional conformity, the mass of air dissolved in a given mass of water is here converted to the volume at S.T.P. dissolved in unit volume.

must be a certain minimum hydraulic gradient ("threshold" gradient) in the pore water for drainage to continue steadily to completion. The last, very small increments of drainage to establish equilibrium might only occur sporadically at long intervals because the gradient is then less than the threshold gradient.

The capillary equation, equation (1), may be insufficient to explain the process by which a slow breakdown of menisci occurs. The equation does not take into account, for example, the presence of properties of the adsorbed water layers surrounding the particles. Slow movement of water may take place in these layers and there may be changes in contact angle or air-water interface radius in connection with this.

The formation of new bubbles apparently did not occur in the pressure range covered by the experiments. If a new bubble were to arise it would have to have a certain minimum size to persist. The radius would have to be such that the pressure of the air in the bubble was in equilibrium with the concentration of dissolved air in the vicinity of the bubble. For a sample in which interfaces already present have assumed a uniform radius r as given by equation (1), then a new bubble must have the same or greater radius. An exception to this would be bubbles occurring in association with hydrophobic sites (discussed below), although such bubbles were not observed in the present experiments. It is probable that the concentration of dissolved air was insufficient to allow the necessarily nearly instantaneous formation of such a bubble.

The formation of vapour bubbles would not be expected in the present experiments. Vapour in such bubbles would have to have a pressure as low as the vapour pressure of water at the temperature in question, about 0.024 kg/cm^2 absolute (i.e., -0.976 kg/cm² when atmospheric pressure is taken as 0). From equation (1), substituting p_v , the pressure of the vapour, for p_a , it is apparent that the pore water pressure, p_w , would then have to be lower than this by an amount depending on the bubble radius r.

Although the formation of new air bubbles was not observed in the cryolite soil, it is possible this occurs in soils of other minerals (especially quartz). Liebermann (1957) has shown that bubble nuclei of extremely small size occur on chemically-cleaned glass. These nuclei are believed to be air attached to a hydrophobic site; the air-water interface at such a site would be convex towards the air and the pressure of the air therefore sufficiently low that there would be no tendency for the minute bubble to dissolve. They would increase in size when the appropriate concentration of dissolved air occurs.

DETERMINATION OF AIR-INTRUSION VALUE

The air-intrusion value, the difference in pressure between soil air and water at which the air becomes continuous through the soil, is a single-valued characteristic of a soil's pore structure. It is of significance in the study of partially-saturated soils, for both geotechnical and agronomic purposes. Knowledge of the air-intrusion value of a soil permits evaluation of susceptibility to frost heave (Williams, 1966 (a)). The intrusion of ice into a soil on freezing is in many respects analogous to the intrusion of air.

An apparatus (now available commercially*) has been designed and constructed, and a procedure developed to facilitate determination of the air-intrusion value of natural soils. More than fifty different soils, other than more or less pure clays, have been tested and in all cases it has been possible to measure an air-intrusion value. Because of their extremely low permeability, clays require special consideration and have not been investigated in detail. In developing a procedure for the test which would have general application, regard was paid to various factors varying from soil to soil, and which might influence the results obtained.

The procedure for the test has been described in detail (Geonor, 1966). The factors considered and the design of the instrument are outlined here. The apparatus consists basically of a pressure membrane cell which is rapidly assembled. The sample completely covers the membrane which is of the permeable type, and is contained in a Perspex ring. The sample is 1 cm to 2 cm high and 20 cm² in cross-sectional area. The proportion of the surface area of the sample in contact with the Perspex containing-ring is small. The small height of the sample in relation to its volume results in short flow paths for the water draining from the sample, and equalization of pore-water pressure occurs relatively quickly after each increment of air pressure. Drainage is observed in a burette.

In the case of substantially compressible samples, it is desirable to consolidate the sample after placing it in the Perspex containing-ring, before carrying out the airintrusion test. Drainage due to consolidation during the course of the test may otherwise be so substantial as to obscure drainage due to air intrusion. To allow such preliminary consolidation, there is provision for placing a rubber membrane directly over the sample. The consolidating load is applied by raising the air pressure, causing the

^{*} Geonor A/S, Oslo, Norway.

rubber membrane to bear on the sample with the same pressure. After drainage has ceased, the cell is opened and the rubber membrane removed. The test is then carried out in the usual way.

In carrying out the test, the sample is generally prepared as a slurry. Tests can also be carried out, with some modification, on undisturbed samples. After the slurry is placed, a small quantity of water is added on top of the sample and a measurement of the permeability of the sample and membrane made by lowering the drainage burette and observing the flow.

The permeability measurement can be used to determine the appropriate rate of application and size of air pressure increments. For the apparatus in question the relationship between observed permeability, and appropriate manner of application of air pressure, was determined by analysis of a large number of trials on different soils. In plotting the results of a test (drainage as a function of time), it is necessary to choose scales for the axes which reveal clearly the acceleration of drainage associated with intrusion of air. The amount and rate of drainage vary substantially with soil type. The permeability observation is also used as a general guide to the choice of axis scales.

Drainage must necessarily involve a slight difference in water pressure p_w from the top to the base of the sample. Any correction necessary on this account can also be determined from comparison of the permeability measurement and the rate of drainage at the moment of air intrusion.

In relatively coarse-grained soils the point of air intrusion is usually very clearly defined. For finer-grained soils, it is often less conspicuous and more care is necessary in selecting the rate of increase of air pressure and appropriate scales for plotting of results. Fig. 11 (a to d) shows typical examples of tests carried out on various soils.

As in the case of the experiments described earlier, cracking of the sample may occur giving a smaller preliminary period of accelerated drainage. This has been investigated by tests in which the sample was visible through a window in the cell. If cracking occurs it is prior to, or possibly simultaneous with, air intrusion. The accelerated drainage as a result of cracking is always less than that occurring in association with air intrusion.

CONCLUSIONS

There are three distinct processes by which air may replace water in the pores of a soil when a difference in pressure $p_a - p_w$ is established between the air and water:

(a) A process explained by the normal "capillary" equation, equation (1):

$$p_a - p_w = \frac{2\sigma}{r} \cos \theta$$

and the radius r of the air-water interfaces. If this process alone occurs it gives a curve of moisture content versus $p_a - p_w$ in which there is a conspicuous air-intrusion value of $p_a - p_w$, corresponding to an interface radius r_c equal to that of the largest continuous openings through the soil.

(b) A process which occurs more slowly, involving diffusion of air (from air external to the sample) to entrapped air bubbles, with a resulting enlargement of these.

(c) A process of slow advance of interfaces into the soil, possibly due to a change with time, of interfacial energy, contact angle or interface radius, or to a process not explained by the capillary equation, equation (1). This occurs under constant $p_a - p_w$.

Additional drainage may occur because of consolidation due to the stresses on the soil skeleton, arising from the air-water pressure difference. This drainage may occur suddenly with the development of cracks in the soil.

Under appropriate experimental conditions the air-intrusion value may be measured for all soils in a homogeneous state, with the possible exception of those largely composed of clay particles.

ACKNOWLEDGMENTS

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