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Gaseous diffusion in porous media. Part 3—Wet granular materials

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Abstract

The diffusion of hydrogen through granular materials partly saturated with water was measured by a non-steady state technique previously described. The two types of sample used consisted of solid particles (unimodal pore-size distribution) and porous particles (bimodal pore-size distribution), and all measurements were made on samples being dried from saturation. Coefficients of diffusion D were calculated, and for the range over which the larger pores were emptying the empirical equation $D = D_v (\epsilon/\epsilon_v)^\sigma$ fitted all materials, where ϵ is the fractional air-filled volume, ϵ_v is the volume occupied by the larger-pore phase, and where D_v is the diffusion coefficient when only this phase is air-filled. For all materials $\sigma \simeq 4$ whether the samples were uniform or of mixed sizes. No such relationship existed over the subsequent range in which the smaller pores were drained. Over this range D must be a function of at least five independent variables— the total porosity ϵ_T, the crumb porosity ϵ_c, the shape factor for the crumbs or inter-crumb pores k, the shape factor for the particles forming the crumbs or crumb pores k_c, and the moisture content of the sample. The spatial distribution of pores within a porous medium can be as important as the sizes of the pores. The factors k and m, previously introduced as particle-shape factors, now have a greater significance as measures of the geometrical complexity of a porous system. Adding water can either increase or decrease the complexity, depending on the amount added and the nature of the system. The agricultural significance of diffusion between the crumbs D_v and within the crumbs D_c is discussed, and it is suggested that D_c, or its associated complexity factor k_c, might be used as an index of soil structure.

Introduction

DECREASING the air-filled pore space in a porous medium decreases the rate at which gases will diffuse through it. Though adding water is a convenient way of obtaining a continuously variable pore space, there is little evidence to show whether it acts differently from other ways of decreasing pore-space, such as, for example, repacking, or mixing particles of different sizes. Nor is there much evidence to show whether diffusion through different types of porous media is influenced in the same way by wetting. Hannen (1892) used a non-steady state method to measure carbon dioxide diffusion through dry and partly saturated packings of soils and sands. His data, re-calculated to allow for the exponential rate of decay of carbon dioxide concentration in the non-steady state (from the data available this can only be approximate), show for dry materials a relationship of the form $D/D_0 \simeq 0 \cdot 6 \epsilon$. All the points for wetted

materials lie below this line when graphed, suggesting that, in his experiment, moisture was more effective than packing in decreasing diffusion. Buckingham (1904) used a steady state technique on both wet and dry sands and soils. From his data for carbon dioxide, the relationship $D/D_0 = \epsilon^2$ emerged, but his range of moisture contents was too small to establish the role of moisture, other than as a pore filler. Soils were the only materials to which Penman (1940) added moisture, wetting them before they were packed into the diffusion cylinder, rather than adding water to already packed soils. His values of D/D_0 for moist soils do not differ from those for other (dry) materials packed to the same porosity. Hagan (1941) measured diffusion rates through soils wetted with up to 18% moisture, and concluded that moisture content affected the permeability to carbon bisulphide more than any other variable studied. By extrapolating regression lines for their diffusion data, Blake and Page (1948) showed that, where diffusion through one soil ceased at a porosity of $0 \cdot 1$, in another it reached zero only when the porosity was zero. They suggested that this may have resulted from the blocking of some of the air-filled pore-space by water films, but they did not give moisture contents. Taylor (1949) measured diffusion through a loam soil, a quartz sand and through powdered glass, interpreting his data in terms of 'an equivalent diffusion distance' (actually this is equivalent to $(D/D_0)^{-1/2}$). This parameter bore no simple relationship to moisture content, to moisture potential or to air content, and the effect of moisture on diffusion was obscured. Measurements on soils *in situ* by Raney (1949) have too few supplementary data for the effects of cultivation to be separated from the effects of moisture. Bruce and Webber (1953) used Raney's method on a loam soil and suggested that the relationship between diffusion and moisture content was closely related to pore-size distribution; they also stressed the inadvisability of extrapolating for diffusion rates at moisture contents outside the experimental range. Call (1957) measured the diffusion of ethylene dibromide through soils packed into tubes to simulate field conditions and his results supported $D/D_0 = 0 \cdot 66$ ($\epsilon - 0 \cdot 1$). He too stressed the importance of 'blocked pores' but, with no data for moisture contents, the effect of moisture is difficult to separate from effects of dry porosity, soil condition, or possibly anisotropy of structure induced by compacting the soils. Rust *et al.* (1957) wetted non-soil particles, and found $D/D_0 = 0 \cdot 68 \epsilon - 0 \cdot 01$, whereas for the dry materials $D/D_0 = 0 \cdot 60 \epsilon + 0 \cdot 04$. Their results suggest that moisture-blocked pores decrease diffusion more than pores blocked in other ways, and the values for wet soils lie close to the theoretical curve for cylindrical particles with blocked pores (De Vries 1950) and in no case above the curve for spherical particles. In an analogous study of the electrical conductivity of electrolyte solutions saturating various granular

materials, Wyllie and Gregory (1953) investigated the effect of depositing a non-conducting compound between the particles. This they put into position in the dry packing in solution. The deposit of silica, which remained when the solvent was removed by evaporation, occupied the same sites as water would be expected to occupy when added to the same type of packing. If the analogy is exact, their results suggest that the relation $D/D_0 \propto \epsilon^{4.2}$ might be expected.

Theoretical investigations of the type used for dry materials are few. The equation $D/D_0 = \epsilon^{3/2}$ of Marshall (1959) makes no distinction between wet and dry materials. Millington (1959) modifies his relationship for dry soils ($D/D_0 = \epsilon^{4/3}$) to read $D/D_0 = n^2(\epsilon_1^{4/3}/m^2)$, where m equal-volume pore-size groups make up the total porosity ϵ, and when n of these are drained, the air-filled pore-space is given by $\epsilon_1 = n(\epsilon/m)$. The effect is to predict a lower rate of diffusion through moist materials than through dry materials of equal air-filled porosity. De Vries (1950) has suggested a 'blocked-pore' factor for use with his equation, but no existing method distinguishes at a given moisture content between pore-space which is merely water-filled and pore-space which might be termed 'blocked'.

Qualitatively the presence of water seems more effective in decreasing gaseous diffusion than are other agents that decrease air-filled pore-space to the same extent, but quantitatively it is impossible at present to predict the decrease in diffusion rate for a given type of material.

Measurement of diffusion

The apparatus described in Part I (Currie 1960a) was used to measure the diffusion of hydrogen through the porous samples. To use the technique of Part II (Currie 1960b), it was first necessary to overcome difficulties not encountered with dry materials.

The supporting gauze, as previously, had to be rigid, able to retain the granules, and offer the minimum resistance to diffusion. In addition, with moist samples, the apertures had to remain open in diffusion experiments, but had to be able to transmit water as the sample was brought to moisture equilibrium on a suction plate. Three attempts to avoid the use of permeable supports failed.

In the first, granular packings were stabilized by adding adhesive in solution and then allowing it to dry, but the large quantity needed to produce stability raised doubts about the nature of the resultant pore system (Wyllie and Gregory (1953) showed the effect of adding a cementing phase to various granular materials). Similarly, sintered-glass blocks were thought to be atypical of unconsolidated packings.

In the second attempt, a U-shaped tube of square section was used—copying van Bavel (1952)—with one end fitted into the sample tube recess and the other open to the atmosphere. Though this supported the porous material without in any way impeding 'longitudinal' gas flow, it had two disadvantages. In the second limb, the upward diffusion of hydrogen was assisted by convection to an unacceptable extent; and with some wet materials, a moisture gradient was set up within the sample, which was capable of sealing the bottom of the U-tube while leaving the sample as a whole unsaturated.

In the third attempt, the lower end of the straight cylindrical tube was sealed and hydrogen from the gas tube diffused to a concentration in equilibrium with the air in the porous sample. Though a simple working equation was developed for this system, in practice the interval over which the changing concentration could be followed was too small to allow D to be reliably estimated.

Because these attempts failed, it was necessary to resort to the permeable support. By careful choice of suitable open-meshed nylon fabrics, supports fulfilling all requirements for specific materials could be provided.

The second new difficulty was the variability of moisture distribution throughout the samples. When samples packed in 3 in. tubes were nearly saturated, some downward drainage occurred. At best, a measured diffusion coefficient could then be related to a mean value of saturation but diffusion through the sample often ceased when only the lower end of the sample was blocked by moisture. Shorter tubes (1 in.) were satisfactory, and when made of Perspex allowed the presence of moisture gradients to be observed. Occasionally, the visual checks were supplemented by destructive testing. To maintain the convenient time scale of experimental operations, the cross-sectional area of the tubes was altered from approximately 22 cm² to about 8 cm². These tubes were fitted with flanges at the upper ends to enable them to fit snugly into the apparatus in place of the standard brass tubes.

The wet samples required careful preparation to give reproducible diffusion coefficients. All the measurements were made on samples being dried from saturation. After an initial diffusion measurement when dry, the sample was evacuated in a desiccator; air-free distilled water was then admitted and allowed to saturate and almost submerge the sample before the whole was restored to atmospheric pressure. The total pore-space calculated from the weight of liquid held at saturation agreed well with that derived from the weight and density of the solid phase, indicating that saturation was complete. The sample was then placed on a suction plate (sintered glass or other porous material), brought into equilibrium with a chosen water potential, a set of diffusion measurements made, and the moisture content determined by weighing. A series of such operations at increasing moisture tensions was continued until the suction plate technique became inadequate. When further increases in tension failed to remove enough water, the remainder was removed slowly, either by passing a stream of air through the sample, or by bringing the sample to equilibrium over saturated solutions of selected salts. A final diffusion measurement was made after the sample had been completely dried over phosphorus pentoxide. Comparison of initial and final diffusion coefficients for the dry sample showed whether the particles had been obviously re-orientated in the sample during handling. In practice, agreement between the coefficients was always well within the 1% tolerance permitted between duplicate determinations on the same sample.

In samples prepared by desaturation, the continuity of air-filled pore-space with the outside atmosphere was ensured. Saturating dry samples by small increments of water often entrapped air, isolating pockets that could not contribute towards gaseous diffusion through the sample. For this reason, which must not be regarded as a hysteresis effect, reproducible results were obtainable only on the drying cycle.

Materials used. The materials used were in two main groups—uniform solid particles and uniform porous particles. The first group comprised glass spheres, sand and carborundum and, for convenience, the sintered-glass blocks (Porosity 2); the second included pumice and soil crumbs. Three soils were used, chosen as having different crumb porosities, ϵ_c (i.e. the fractional volume of the crumb free from solid matter), and expected to have differing internal structure. Two of these soils were unstable in water and all three swelled on wetting. Though this swelling (and shrinking) doubtless has an important influence on the diffusion

of gases and ions, particularly in agricultural soils, it was considered desirable to exclude it from the present work. The soils were therefore heated to 800° C, which stabilized the crumbs, increased crumb porosities by from 6 to 9% of the crumb volume, and probably modified the original structure slightly.

Experimental results

With all the materials tested, adding water caused a decrease in the diffusion coefficient (Fig. 1), but only in packings of
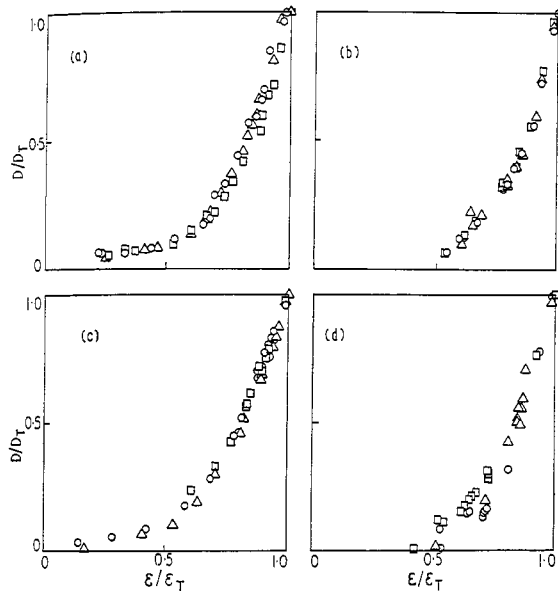

Fig. 1. Relative diffusion/porosity relationships for uniform solid particle systems on drying from saturation (left to right): (a) sintered-glass blocks; (b) glass beads; (c) sand; (d) carborundum.

Sample 1, □; Sample 2, ○; Sample 3, △.

solid particles was this consistently greater than that caused by equal decreases in porosity achieved by repacking or particle mixing. When plotted on a log–log basis (Figs 2 and 6) the data for all materials may be fitted over a substantial (lower) range of porosity by $D/D_0 = \eta\epsilon^\sigma$ where η is a function of the total porosity ϵ_T and of the corresponding diffusion ratio D_T/D_0 for solid particles, or of the inter-crumb porosity ϵ_v (Fig. 9) and corresponding diffusion ratio D_v/D_0 for porous particles. For both types of particle $\sigma \simeq 4$. When there is any deviation from this equation over the lower range, it occurs as $\epsilon \to 0$ in the sense that $\sigma < 4$, whereas theories involving 'blocked pores' and 'cut-off' values at low porosities lead to $\sigma > 4$. Particle shape influences the rate of diffusion through dry materials (Currie 1960b), but from Figs 1 and 2 it is apparent that in wet materials particle shape has much less effect on diffusion, for fairly obvious reasons. For the former, particle shape is the main determinant of pore shape, but in wet materials the pore shape is very much modified by the presence of water. Whatever the shape of the empty pore, the water menisci between the particles 'round off' the boundaries of the air-filled volume, so that at a given degree of saturation, most solid-grain packings will exhibit the same pore shape. Exceptions to this are inevitable, and for particles of irregular shape a higher degree of saturation is

expected to be needed before the pore shape ceases to be characteristic of the particles. Packings of porous particles are in this category, for they must be completely saturated
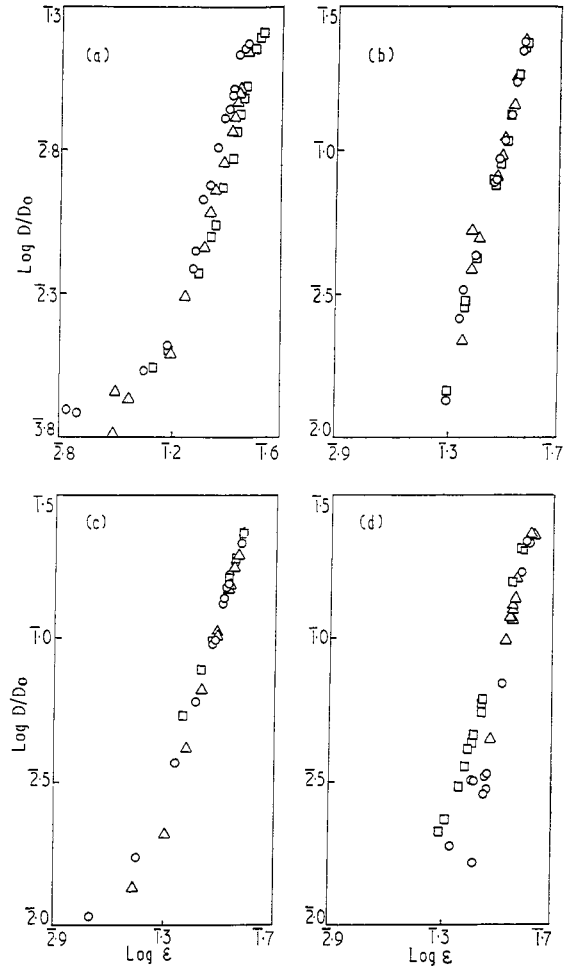

Fig. 2. Logarithmic diffusion/porosity relationships for uniform solid particle systems: (a) sintered-glass blocks; (b) glass beads; (c) sand; (d) carborundum.

Sample 1, □; Sample 2, ○; Sample 3, △.

before they become representative of the solid particle packing. Figs 5 and 6 show this and will be discussed later.

Figures 1 and 2 refer to materials of uniform particle size having sigmoid moisture retention curves (Fig. 3, curves (a–d)) characteristic of a normal pore-size distribution. To show that it is the normal distribution of pore size and not uniformity of particle size which governs this type of moisture/diffusion relationship, the measurements were repeated on a mixture of 5–6 and 0·75–0·8 mm diameter spheres, and on mixtures of 0·5–1·0 and 1–2 mm crumbs for one of the soils. Moisture/diffusion curves (Figs 4 and 8) and moisture retention curves (Fig. 3 and Fig. 7, curve (e)) for these mixtures are similar to those obtained for particles of uniform size.

Porous particles give packings with at least two distinct kinds of pores, namely those between the particles, the *inter-crumb* pores, and those within the particles, the crumb pores

(Fig. 9). (Orr and Dallavalle (1959) use the terms 'voids' and 'pores' respectively for these two groups.) Such a system has discrete zones of smaller pores separated by a continuous
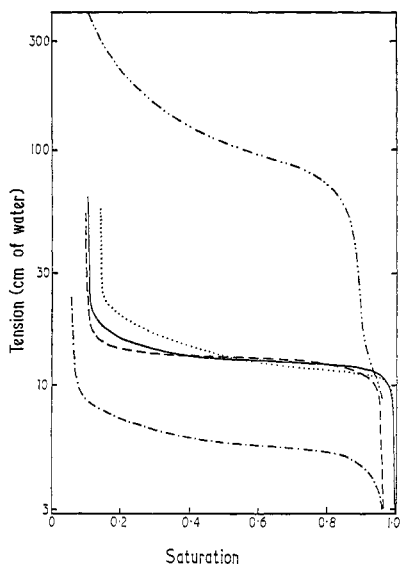

Fig. 3. Moisture retention curves for solid particle systems.

(a) Sintered glass   — ·· — ·· — ·· —
(b) Glass beads   — — — — —
(c) Sand   —————
(d) Carborundum   · · · · · · · · · · · · · ·
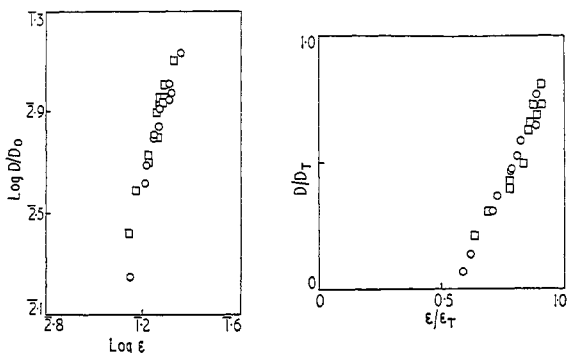(e) Mixed glass beads — · — · — · — · —

Fig. 4. Diffusion/porosity relationships for mixed solid particles.

Sample 1, □; Sample 2, ○.

system of larger pores, and is characterized by a 'double sigmoid' type of moisture retention curve (Fig. 7—only the lower sigmoid is shown). Desaturation/diffusion curves for such samples (Figs 5 and 6) reveal two distinct phases corresponding to desaturation, first the inter-crumb pores, and second the crumb pores. In the first phase, the process is identical with the desaturation of the simple pore system, the saturated granules behaving exactly as solid particles of the same shape and volume. (Four systems were fully investigated, and limited experiments with ten similar materials produced no evidence contrary to this finding.) In the second phase, there is no consistent relationship between the volume of the crumb pores and the increase in diffusion brought about when they are drained. This will be better
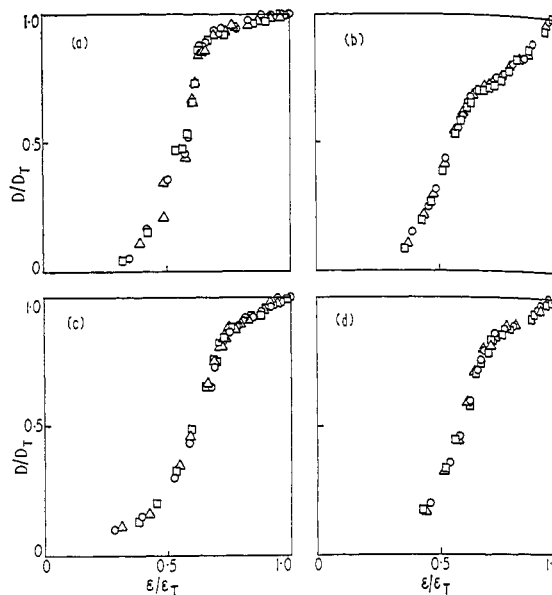

Fig. 5. Relative diffusion/porosity relationships for uniform porous particle systems. (a) Pumice; (b) HPP soil crumbs; (c) Ba80 soil crumbs; (d) TBL soil crumbs.

Sample 1, □; Sample 2, ○; Sample 3, △.

understood with reference to the crumb material. The equation of De Vries (1950) for a dry packing of porous crumbs may be written as

$$\frac{D}{D_0} = \frac{1 - (1 - kD_c/D_0)(1 - \epsilon_v)}{1 + (k - 1)(1 - \epsilon_v)}$$

where k is a shape factor for the crumb, and where ϵ_v is the inter-crumb porosity. D_c/D_0 represents the diffusion ratio for a continuous expanse of crumb material and will itself be a function of the crumb porosity ϵ_c, and the shape of the particles forming the crumb (shape factor k_c). As the crumb material is a consolidated medium, k_c will have a wider range of values than for unconsolidated media and will confer on D_c/D_0 an extended lower range of values. At the beginning of phase two, when the inter-crumb pore-space is completely empty, and the crumb pore-space is completely full $D_c = 0$ and at this stage

$$\frac{D}{D_0} = \frac{\epsilon_v}{1 + (k - 1)(1 - \epsilon_v)}$$

i.e. the saturated crumbs act as solid particles of the same shape and volume. As phase two continues, water is withdrawn from the crumbs, and D_c becomes positive as the total air-filled pore-space increases to include a crumb component. Search for a simple power law relating D_c and crumb pore-space has so far been unsuccessful. The value of D_c must depend on the porosity of the crumb, the fraction of this occupied by water, and the shape of the particles making up the crumb, so that the measured diffusion ratio D/D_0 must be dependent on at least five independent variables, for to these three have to be added the inter-crumb pore-space, and the shape factor for the crumbs as units. Not much is known about the internal geometry of the crumbs, but the crumb porosities ϵ_c are given in the Table. The ease with which the different crumbs were saturated suggests that the
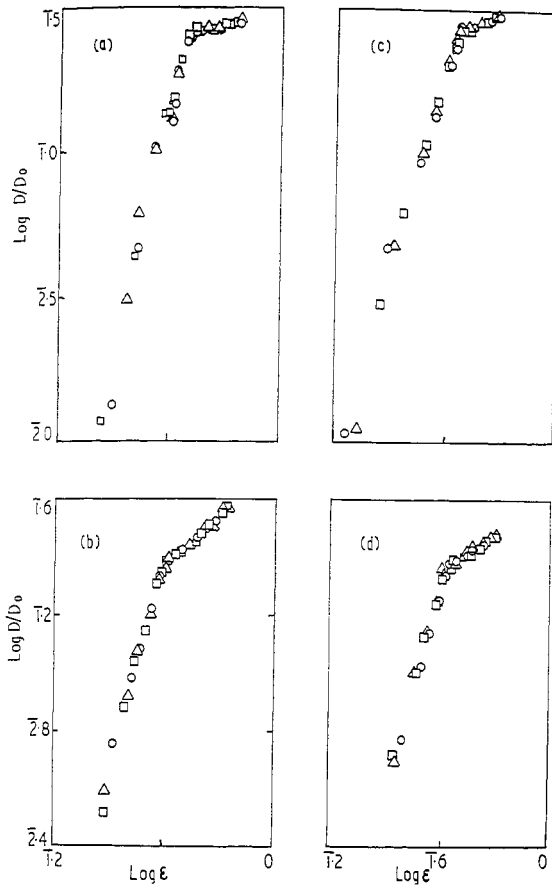
Fig. 6. Logarithmic diffusion/porosity relationships for uniform porous particle systems. (a) Pumice; (b) HPP soil crumbs; (c) Ba80 soil crumbs; (d) TBL soil crumbs.

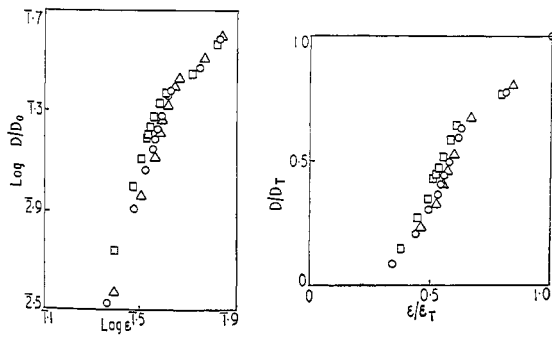Sample 1, □; Sample 2, ○; Sample 3, △.

Fig. 8. Diffusion/porosity relationship for mixed porous particles.

$$0 \cdot 5 - 1 \cdot 0 : 1 \cdot 0 - 2 \cdot 0$$

Sample	1	:	2	□
	2	:	1	○
	3	:	0	△
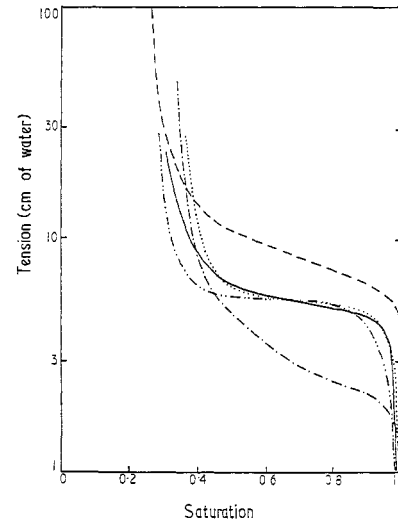
Fig. 7. Moisture retention curves for porous particle systems.
(a) Pumice ————————
(b) HPP soil crumbs ············
(c) Ba80 soil crumbs ————————
(d) TBL soil crumbs ————————
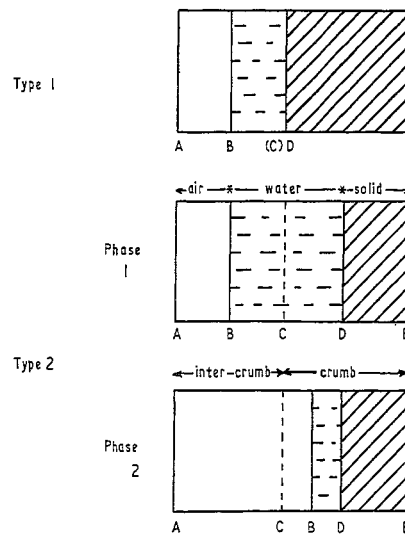(e) Mixed HPP soil crumbs ————————

Fig. 9. Diagrammatic pore distribution for solid particle systems (type 1) and porous particle systems (type 2).

1. Expressed as a fraction of total volume

		Corresponding diffusion coefficient
Total pore-space	$\epsilon_T = AD/AE$	D_T
Inter-crumb pore-space	$\epsilon_v = AC/AE$	D_v
Crumb pore-space	$\epsilon_p = CD/AE$	—
Air porosity	$\epsilon = AB/AE$	D

2. Expressed as a fraction of crumb volume

| Crumb porosity | $\epsilon_c = CD/CE$ | D_c |

internal geometry of the HPP crumbs was least complicated and that of the pumice most complicated. The agricultural history of the soils suggest that the HPP crumbs have a better

Table. *Data for dry samples*

	ϵ_T	D_T/D_0	ϵ_c
Sintered glass	0·303	0·137	
	0·301	0·148	—
	0·342	0·162	
Glass beads	0·378	0·236	
	0·373	0·237	—
	0·376	0·239	
Sand	0·387	0·230	
	0·379	0·214	—
	0·381	0·224	
Carborundum	0·386	0·204	
	0·408	0·216	—
	0·419	0·228	
Pumice	0·701	0·285	
	0·703	0·276	0·517
	0·700	0·280	
HPP	0·673	0·378	
	0·676	0·375	0·418
	0·677	0·374	
Ba8O	0·612	0·307	
	0·617	0·306	0·350
	0·612	0·306	
TBL	0·633	0·300	
	0·638	0·298	0·295
	0·634	0·301	
Bead mixtures	0·213	0·128	
	0·226	0·136	—
Soil mix 1 : 2	0·656	0·363	
2 : 1	0·674	0·381	0·418
3 : 0	0·686	0·393	

internal structure than either the Ba8O or TBL crumbs, the first of which are from a continuously cultivated arable soil, and the second from a boulder-clay sub-soil in which a high degree of particle orientation might be expected.

Only two of an almost infinite number of types of porous system have been considered here, but they are those most commonly encountered in practice. Pore-size distribution is of apparent importance, but other materials may have similar pore-size distributions without necessarily exhibiting the same moisture/diffusion characteristics, or the same moisture retention curves, for example one in which the larger pores are isolated in a continuous matrix of smaller pored material. It is therefore necessary to specify also the spatial distributions of these pore-size groups and the continuity of the pores. Moisture retention curves seem the necessary compromise between these three requisites.

In Part II, shape factors, k and m were calculated for all the dry materials used, recognizing that the values for the porous particles were 'effective' shape factors. Values for k and m were calculated for one sample from each of the four different materials of this type (Fig. 10). Both pass through a sharp minimum at the water content when only the crumbs are saturated, showing a very clear transition from phase 1 to phase 2 and these values represent the factors for the true shape of the crumb. The values for the four materials differ in a way consistent with the observed differences in particle shape. The factors k and m, originally introduced as particle shape factors, can now be given greater significance as measures of the complexity of the pore system, a concept equally applicable to consolidated and unconsolidated materials. Starting from dry materials and adding water (moving from right to left in Fig. 10), there is a steady decrease in complexity as the crumb pores are filled, reaching the minimum at crumb saturation. Further additions begin to fill spaces between crumbs, increasing the tortuosity of the remaining air path and so increasing the complexity again.

Discussion

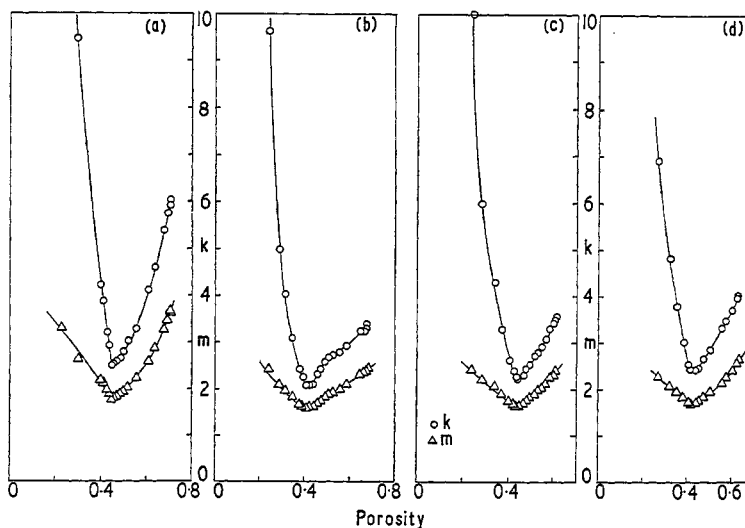It is apparent for a given porous material that, unless certain geometrical properties are known, the effective

Fig. 10. The effect of moisture on 'shape factors'.
(*a*) Pumice; (*b*) HPP soil crumbs; (*c*) Ba80 soil crumbs; (*d*) TBL soil crumbs.

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diffusion coefficient cannot be interpreted in terms of moisture content. Further detailed examination of published data reviewed earlier must be restricted to those materials for which these specifications appear complete. Taylor's data for quartz sand can be fitted with a fourth power relationship but not those for powdered glass. In assessing the other somewhat contradictory data, it must be remembered that adding water can simplify or complicate the system for diffusion according to type of medium. Values for k and m calculated from Penman's data are lower for wet than for dry soils, consistent with what is known of the method of packing. Call's data are not inconsistent with a fourth-power relationship between diffusion and porosity, bearing in mind that the packings were not of the same total porosity or all of the same crumb porosity, factors ignored in fitting a linear relationship. Other results interpreted by their authors in terms of 'cut-off' values, may be satisfactorily explained by $D/D_0 \propto \epsilon^4$, for it is not always possible to distinguish between first and higher power relationships, especially over limited range of porosity and with a high experimental error. The relation $D/D_0 \propto \epsilon^{4 \cdot 2}$, suggested from the work of Wyllie and Gregory, indicates that the analogy between cement and water is correct. Millington's equation by comparison gives $D/D_0 \propto \epsilon^{10/3}$ for wet materials.

Most agricultural soils can be placed in the two categories studied here. Soils where sand predominates are of the first type. Most soils, however, contain some clay and because their primary particles aggregate into discrete units separated by larger pores, they are of the second type. These units are formed either by natural processes (wetting and drying, freezing and thawing) or by artificial processes (cultivation); they are the blocks, prisms, plates, etc., of the undisturbed sub-soil, and the clods, crumbs and aggregates of the cultivated layer. The larger inter-crumb pores permit surplus water to be removed easily and allow gases to diffuse between the 'crumb' surface and the soil surface; the smaller crumb pores hold the reserve of water for plant growth and are the site of much of the biological activity in the soil. For this activity to proceed satisfactorily, i.e. in a direction beneficial to crop growth, oxygen must reach these smaller pores and carbon dioxide must be removed, both gases diffusing through partly saturated crumb pores. The interchange of gases between respiring organisms and roots in the soil involves two of the factors studied here, firstly D_v/D_0 for the diffusion in the inter-crumb pores, and secondly D_c/D_0 for the diffusion between the crumb surface and the

respiring surface. In addition to gaseous diffusion within the crumbs, there is also ionic diffusion induced by nutrient uptake by the organisms. By analogy, D_c/D_0 will be the same for ions in the saturated crumb as it is for gases in the dry crumb, but the same power law will not necessarily govern both at intermediate saturations. The value of D_c/D_0 or its associated complexity k_c is likely therefore to be an important parameter for soil structure, applicable both to individual crumbs and to larger units in an isotropic soil. Though the full relationship has yet to be determined, the necessary parameter can probably be calculated from the values of ϵ_c, D_v and D_T obtained from the methods described.

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