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PORE SIZE DISTRIBUTION AND HYDRAULIC CONDUCTIVITY OF DIGESTED SLUDGE CAKE

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ABSTRACT : The investigation, reported in this paper uses pore size distribution data to estimate hydraulic conductivity of digested sludge cake. This also focuses on the assessment of drying techniques in the preparation of dry sludge cake for mercury porosimetry studies. For the prediction of hydraulic conductivity, different theoretical models for fine grained soils are critically reviewed. Analyses of the results show that the predictive models can not relate hydraulic conductivity to the measured value without introducing a matching factor. However, hydraulic conductivity (K) can be predicted using pore size distribution measurement from an empirical relationship of the form $K = C\ (PSP)^{b}$, where PSP is the pore size parameter and C and b are constants.

KEY WORDS : Pore size, hydraulic conductivity, digested sludge, fabric, mercury porosimetry, sludge cake.

INTRODUCTION

The measurement of pore size distribution is a technique of inferring the relative arrangement of particles and pores (i.e. the fabric) in a porous material. Usually information available in the case of digested sludge cake focuses on porosity. However the porosity measurement of a digested sludge cake may not provide sufficient information concerning the nature and distribution of void space within the sludge cake. The size and the extent of connceted pores within the sludge cake greatly influence the flow of fluid through it. Attempts have been made by different investigators to study the pore structure of different materials (Bhasin, 1975, Diamond, 1970, Green-Kelly, 1973, Lawrence, 1977 and Ritter and Drake, 1945). Garcia-Bengochea et al (1979) mentioned that the pore system is extremely complex in geometry; the pore network in terms of pore size and diameter is an abstract concept. A possible development is to include the pore size distribution as a means of interpreting the characteristics of the porous medium in terms of its permeability. This analysis focuses on the pore size distribution of digested sludge cake and its effect on water retention and water transfer.

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In soils, several methods (Garcia-Bengochea et al, 1979) have been used for measuring the pore size distribution measurement. Bhasin (1975) also presented a critical review of the various techniques available to measure the pore size distribution of silty-clayey soils. He concluded that only the mercury intrusion method is capable of measuring the entire range of pore sizes upto five order of magnitude of fine grained soils. Therefore this investigation is concerned with mercury porosimetry or mercury intrusion method and application of pore size distribution measurement in calculation of hydraulic conductivity of digested sludge cake.

LITERATURE REVIEW

Mercury intrusion or mercury porosimetry

Washburn (1921) first proposed the measurement of pore size distribution by mercury penetration. Ritter and Drake (1945) published the experimental data. The concept was introduced into soil engineering by Diamond (1970). The pore size distribution of material is calculated from the well know physical relationship describing the penetration of a non-wetting fluid into a small pore of solids of diameter d, under pressure p, know as Washburn equation

$$P = \frac{4\ \sigma \mathrm{Cos}\phi}{d} \qquad (1)$$

here σ = surface tension of mercury (= 4.554×10^{-4} kN/m at 20° C, Rahman, 1989)

and ϕ = the air mercury contact angle, on dried sample of digested sludge cake (= 147.2°, Rahman, 1989)

In applying the above method the body is modeled as a bundle of capillary tubes and every intrusion of mercury at a particular pressure is assumed to fill the cylindrical tubes that correspond to the diameter specified by equation (1).

Hydraulic conductivity and fabric

Lambe (1954) was one of the first investigators to examine hydraulic conductivity of compacted fine grained materials. Subsequent studies of the hydraulic conductivity of compacted clays by Mitchell et al (1965), giving results similar to those obtained by Lambe. The Hydraulic conductivity of porous media has normally been predicted by the Kozeny-Carman equation (Carman, 1937). However, Lambe (1954) and Micheals and Lin (1954) have shown that the Kozeny-Carman equation is not suitable for predicting the hydraulic conductivity of fine grained materials. In general, factors that control the hydraulic conductivity of fine grained materials as concluded by different authors (Lambe 1954, Garcia-Bengochea et al, 1979) are (1) the geometry of the porous network (i.e. fabric) (2) the characteristics of permeability and (3) the surface interaction between the permeating fluid and the porous media.

Different hydraulic conductivity models

Childs and Collis-George (1950) presented an equation for hydraulic conductivity based on the probability of occurrence of all pore size sequence in the flow channel. In a similar way Marshall (1958) also tried to develop a similar relationship between hydraulic conductivity and pore size distribution considering pore groups of equal porosity. Theoretical background and development of both models (Childs and Collis-George's and Marchall's) are given in the respective reference, so only summary is presented here. If the pore size distribution function is divided into pore groups each having a smallest mean diameter of c,d,e .... etc arranged in increasing order that correspond to contributing porosity elements α, β, r ..... then according to Childs & Collins-George, equation for hydraulic conductivity is

$$K = M\ (c^2\alpha^2 + d^2\beta^2 + e^2 r^2 + \dots\dots\dots\dots + 2c^2\ \alpha\beta + 2c^2\,\alpha\, r \dots\dots\dots\dots\dots 2d^2\beta r\dots\dots) \qquad (2)$$

where, M is called matching factor and the value of M is derived on the basis of Poiseuille's law (Child and Collis-George, 1950).

Marshall's equation for hydraulic conductivity

$$K = \frac{\P\,\omega g\,\varepsilon^2}{32n^2}\ (d_1^2 + 3d_2^2\ +\ \dots\dots\dots\dots\dots\ (2n\text{-}1)\,d_n^2) \qquad (3)$$

ε = porosity

$\P\,\omega$ = density of water

n = no. of division of equal porosity

d_1, d_2, d_n = mean pore diameter in each division.

Millington and Quirk (1961) considered effective pore area available for flow per unit area of section to be $\varepsilon^{2/3}$ instead of ε and suggested modification of Marshall's equation by a porosity component of $\varepsilon^{4/3}$ instead of ε^2 in equation (3).

$$K = \frac{\varepsilon^{4/3}\P\,\omega g}{32n^2\mu}\ (d_1^2 + 3d_2^2\ +\ \dots\dots\dots\dots\dots\ \times\ (2n\text{-}1)\,d_n^2) \qquad (4)$$

The model derived by Childs and Collis-George, Marshall and Millington and Quirk shall be abbreviated as CCG-model, MS-model and MMQ-model respectively.

Garcia-Bengochea et al (1979), following the Child & Collis-George and Marshall methods, tried to define a pore size parameter (PSP) as

$$PSP = \sum_{i=1}^{n} \sum_{j=1}^{n} d^2 f(d_i) f(d_j \qquad (5)$$

Here d is the smaller of d_i and d_j and f (d) is the volumetric frequency of occurrence of pores with diameter d ......> d + Δd.

Then an empirical relation between hydraulic conductivity (K) and PSP was being attempted as follows :

$$K = C\,(PSP)^b \qquad (6)$$

where, C is a constant termed 'shape factor' and b is a statistical regression parameter.

EXPERIMENTAL PROGRAM

Preparation of sludge cake

Using a well stabilized digested sludge, the cakes were prepared by compressing the sludge in a pressure plate apparatus (Rahman, 1989) at different equilibrium pressures. This is indicated by the measurement of moisture content (ø) approaching a constant value which is independent of time.

Drying of sludge cake

Pore size distribution measurement processes require dry sample. The samples used for this study was dried following the procedure of air drying, oven drying and supercritical drying methods (Rahman, 1989). The objective is to select a suitable drying technique that is able to remove water from sludge cake with least swelling or shrinkage and (hope fully) causing minimal disruption to the cake structure. Samples of about 0.03x0.01x0.01 m^3 were cut from the prepared digested sludge cake and subjected to air, oven and supercritical drying methods.

Measurement of liner shrinkage

The lengths (L_0) of the wet sludge cakes were measured using slide calipers (accuracy 2.0 × 10^{-5}m). The samples were then dried by three selected (air, oven and supercritical) drying methods. The lengths (L_f) of the dry samples were then measured. The linear shrinkage was calculated as follows

$$\text{Linear shrinkage} = \frac{L_0 - L_f}{L_0} \times 100\ (\%)$$

Mercury porosimeter

The analysis was carried out using an "Erba Science Model Mercury porosimeter, series 2000". At the beginning of a test, dried samples (10^{-4} to 2.5×10^{-4} kg) were placed in a dilatometer (container for holding the specimen) and de-aired under a vacuum of 10^{-3}m Hg (< 19 kPa). The dilatomer was filled with mercury and was subsequently placed in a presser assembly (for applying pressure on mercury within the dilatiometer) of the porosimeter in order to apply pressure on the mercury surrounding the specimen. Measurement of the applied pressure (range ambient, 101 to 2.0×10^4 kPa) and the corresponding mercury intrusion (i.e. penetration of a needle into dilatometer) were recorded automatically in an Apple II $^+$ computer which was linked to the porosimeter. The amount of mercury penetrated was calculated by multiplying the area (6.36×10^{-9} m^2) of the needle by the penetration reading. From this the pore diameter corresponding to applied pressure was calculated by equation (1) .

MATERIAL EXAMINED

Measurement of linear shrinkage

Sludge cakes were collected at equilibrium pressures of 20 to 2.64×10^3 kPa and dried by three (air, oven and superritical) drying methods.

Pore size distribution measurement

(a) Untreated digested sludge

 (i) Sludge cakes were collected at selected pressures (50 and 10^3 kPa) and dried by the three drying methods.

 (ii) Sludge cakes collected at equilibrium pressures of 20 to 2.64 $\times 10^3$kPa were prepared using the supercritical drying technique.

(b) Digested sludge after freezing and thawing (samples were held in a freezer overnight at - 10°C and then left to thaw at room temperature). Sludge cakes were collected at equilibrium pressures of 20, 30, 50 and 75 kPa and dried by the supercritical drying method.

(c) Digested sludge at isoelectric point (the p^H of the samples were adjusted to 2.48 : this corresponds to the isoelectric point). Sludge cakes were collected at equilibrium pressures of 20, 30, 50 and 75 kPa and dried by the supercritical drying method.

Determination of hydraulic conductivity

The hydraulic conductivity of untreated and treated (i.e. after freezing and thawing and at isoelectric point) digested sludge samples were determined using constant head permeameter (Rahman, 1989). The moisture content of these sludge samples varied from 92 to 97%. The initial depth of the samples were 0.02 to 0.03 m. This led to final depths of about 0.004 to 0.01 , after stabilization for 7 to 8 days at different equilibrium pressures. Stabilization was indicated by the measurement of hydraulic conductivity approaching a constant value which is independent of time.

RESULT AND DISCUSSION

Studies on drying technique

Table 1 summarizes the results of linear shrinkage measurement of untreated digested cake subjected to different equilibrium pressures. This shows that maximum shrinkage at different pressures occurs in air drying, whereas there is minimum shrinkage with supercritical drying. The oven drying method also show higher shrinkage, but slightly less than that for air dried samples. One of the important forces that brings about such shrinkage (i.e. particle re-orientation and disturbance) during the air and oven drying methods, is the surface tension force associated with pore water. The difference in oven and air drying may also be due to rate of drying. According to Tovey and Yan (1973), where the air drying of wet samples take a relatively longer time than does oven drying, a greater re-arrangement of the fabric takes place in air dried samples than in oven dried samples. A critical region is delineated by the phase diagram of a material as that condition where condensation and vaporisation does not occur. Therefore, it is possible to enter this region with fluid in the liquid phase and leave the region with liquid in the vapour phase, avoiding the viscous drag or the surface tension effects that occur in normal drying.

Table 1 shows a range of linear shrinkage of about 0.3 to 1.7%, 4.3 to 31% and 6 to 35.6% (the values are at equilibrium pressures of 20 and 2.64 X 10^3 kPa respectively) with the supercritical, the oven and the air drying methods respectively. Measurements by Tovey and Yan (1973) indicated that in freeze and critical point drying, with a sample of kaolin (Ø = 160 to 270%) or montmorillonite (Ø = 1000 to 1300%), the linear shrinkage was less than 0.5%. Oven and air dried sample of these substances resulted in shrinkage of 24 and 29% respectively. Such determinations have not been report in the published literature for digested sludge cake.

Figure 1 summarizes the results of assessment of pore size distribution of untreated digested sludge cake. The cakes were collected at equilibrium pressures of 50 and 10^3 kPa and the samples were prepared by the three different drying methods. The figure shows that the total volume of mercury intrusion is maximum with supercritical drying technique (9.81×10^{-4} and 4.27×10^{-4} m^3 kg^{-1}at 50 and 10^3 kPa respectively). It is minimum (7.2×10^{-5} and 6.7×10^{-5} m^3 kg^{-1}) with the air drying technique. This shows that the sample prepared by supercritcal drying had a more open structure. The oven dried samples also show a lower mercury intrusion (1.08×10^{-4} and 1.01×10^{-4} m^3 kg^{-1}) than do supercritical dried samples but higher than that with the air drying

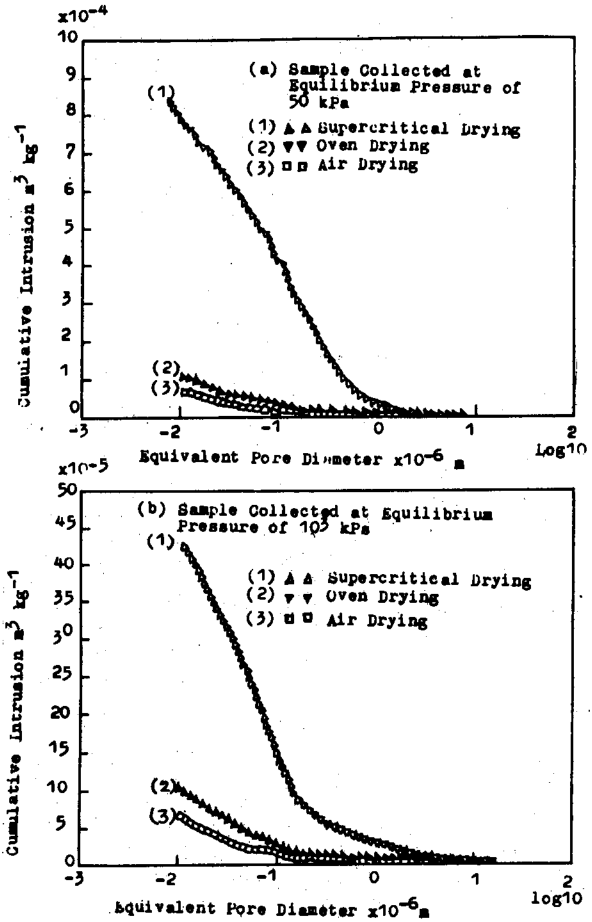

Fig 1 . Effect of Drying at Different Drying Techniques.

technique. These results are consistent with the data shown in the Table 1. Therefore, the measurement of sample shrinkage gives some indication of the changing state of the fabric. Overall it appears that the supercritical drying method is the most suitable means of preparing samples in order to minimize shrinkage and fabric re-orientation.

Figures 2 and 3 show the cumulative mercury intrusion (volume of voids intruded divided by weight of dry sample) versus pore diameter. The pressure values (in kPa) in Figure 2 indicate the equilibrium pressures at

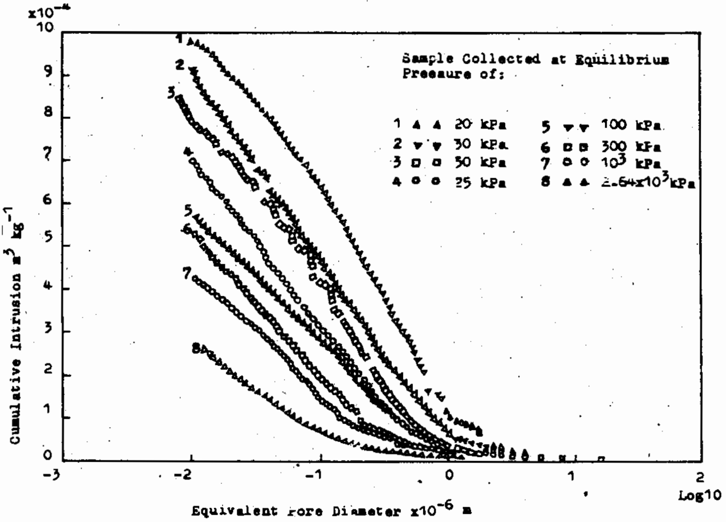

Fig 2(a). Pore Size Distribution of Untreated Digested Sludge Cake.

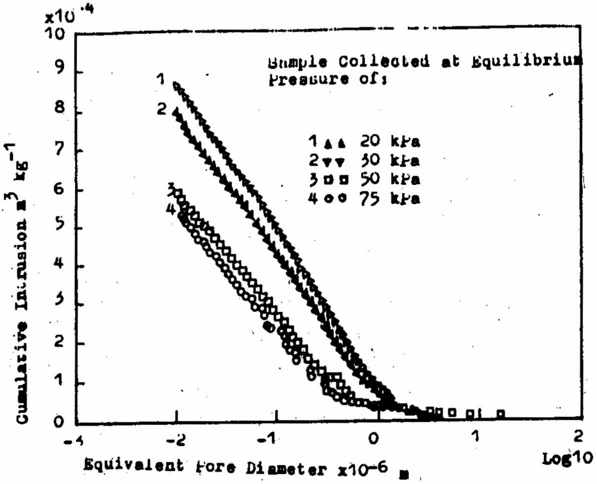

Fig 2 (b). Pore Size Distribution of Digested Sludge After Freezing & Thawing.

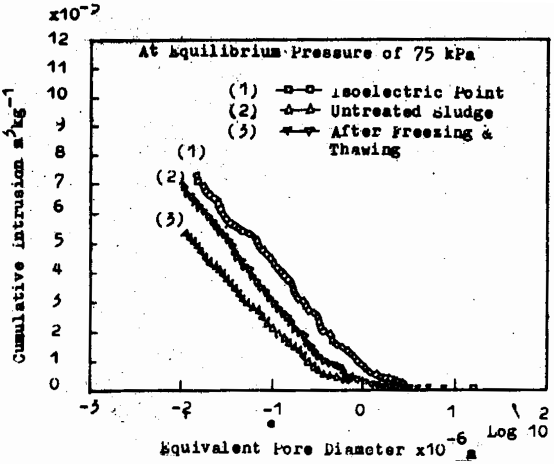

Fig 2 (c) : Pore Size Distribution of Digested Sludge at Isoelectric Point.

which samples were collected for the pore size distribution test. Figure 2 shows that as equilibrium pressure increases, the overall scale of the pore size distribution of digested sludge cake decreases. Thus there is a significant variation of cake structure at different equilibrium pressures. Pore structure of the digested sludge cake collected at lower pressures is more open than that of the cake collected at higher pressures. Figure 3 demonstrates that at the same equilibrium pressure, sludge, after freezing and thawing shows less mercury intrusion than does untreated sludge and also sludge at isoelectric point. Sludge at the isoelectric point shows more mercury intrusion than does untreated sludge.

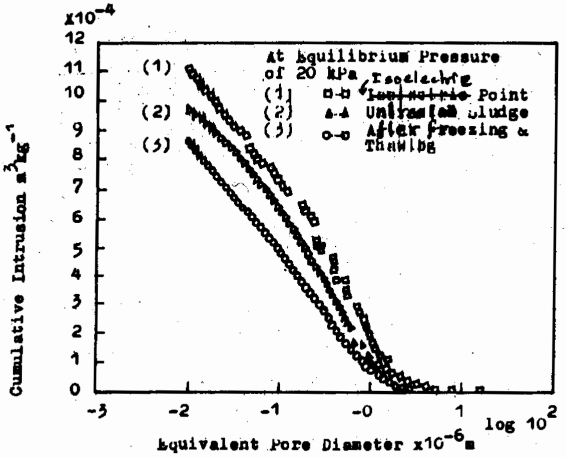

Fig 3 (a): Effect of Treatment of Pore Size Distribution of Digested Sludge at

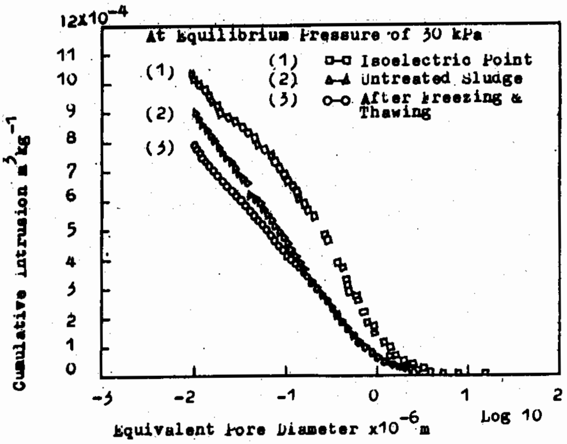

Fig 3(b). Effect of Treatment on Pore Size Distribution of Digested Sludge.

Hydraulic conductivity

Figure 4 shows the dependence of hydraulic conductivity (K) on the moisture content (Ø) and also the comparison of calculated values of hydraulic conductivity (using CCG, MS and MQ methods) with the observed values. Hillel (1980) and Gardner & Mayhugh (1958) proposed the following empirical relationship between K and Ø

$$K = A\,(10)^{B\varnothing} \qquad (6)$$

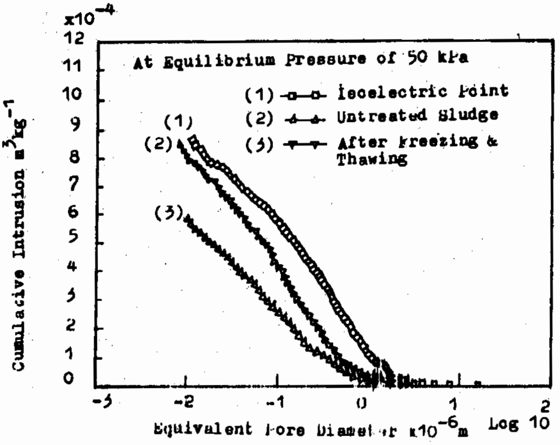

Fig 3(c) . Effect of Treatment on Pore Size Distribution of Digested Sludge

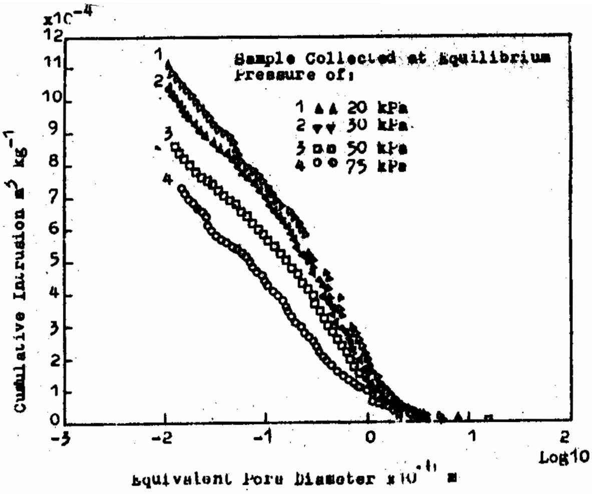

Fig 3(d) : Effect of Treatment on Pore Size Distribution of Digested Sludge

Table 1. Shrinkage in Air, Oven and Supercritical dried digested sludge cake

Sample collected at equilibrium pressure (kPa) of	Linear shrinkage, %		
	air drying	oven drying	supercritical drying
20	35.61	30.95	1.65
30	35.02	30.41	1.57
50	34.41	29.48	1.49
75	33.78	28.83	1.32
100	32.36	27.81	1.24
150	30.28	26.04	1.05
300	25.96	21.88	0.92
600	18.61	15.22	0.84
900	12.84	10.05	0.81
1200	9.42	7.28	0.65
1500	7.74	5.60	0.41
2640	5.96	4.27	0.31

Fitting equation (7) to the data shown in figure 4 by linear regression between K and Ø leads to the evaluation shown in Table 2. Clearly the satisfactory fit achieved in all cases supports the use of equation (7). Also it shows that K is sensitive to Ø. Table 2 also indicates that the values of B's are more or less the same but the values of A's are sensitive to the type of treatment.

Prediction of hydraulic conductivity

It is apparent from figure 4 that all the predicted K values are about 5 - 22 times higher than the measured values of K at corresponding moisture content but have a similar slope to the measured values. Hillel

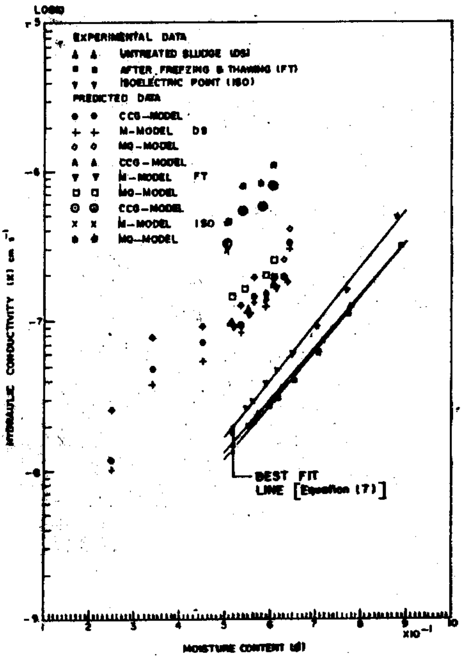

Fig 4. Hydraulic Conductivity of Treated and Untreated Digested Sludge.

(1980) emphasized that although the predictive models should in theory yield K values close to the experimental values, in practice a matching factor (ratio of experimental to prdicted value at a given Ø) is needed to adjust the values. The data obtained from different predictive models represented in Figure 4 follow a similar trend to experimental results. Thus a log-linear regression analysis was also made for predicted K and Ø. This allowed comparison with the experimental relationship obtained by fitting with the equation (7). Table 2 also shows the different values of coefficients A and B in equation (7) for the predicted hydraulic conductivity of untreated and treated sludge. It is apparent from the table, that the data from the MS model provides a better result than the other two (CCG and MQ) models. However, the regression coefficients of the other two models are also satisfactory. The slope of equation (7) varies from 2.5 to 3.8 in the different predictive models. Therefore, it is possible to predict hydraulic conductivity using these models, by introducing matching coefficients as shown in Table 2.

The three (CCG, MS and MQ) models were then examined in terms of the following equation

$$\log K = \log C + b \log (PSP) \tag{7}$$

This approach was suggested by Garcia-Bengochea, et al (1979). The linear regression by least square method was used to estimate the value of C and b using PSP values from mercury porosimetry and the experimental K values at their corresponding moisture content. The coefficient of determination (R^2) is also calculated. All these values are presented in Table 3. The coefficient of determination are in all cases very good (vary from 69 to 97%) and also better than those determined by Garcia-Bengochea, et al (vary from 64 to 91%). From the three models tested, the M model provides better coeffcients of determination (77 to 97%). Overall it appears that statistcal methods based on pore size distribution of digested sludge can not relate predicted hydraulic conductivity to measured value effectively with out being matched with an empirical matching factor (Table2). Thus a more sophisticated theoretical model is needed to improve prediction of K from pore size distribution data. However, as an alternative, K can be predicted using pore size distribution measurement via equation (6).

CONCLUSIONS

On a critical examination of the test result and the analysis made in this study, the following observations and conclusions can be made :

(1) The supercritical drying method appears to be most promising technique for preparing digested sludge cake for pore size distribution measurement.

(2) Hydraulic conductivity of untreated and treated sludge is directly related to moisture content ($0.55 < \varnothing < 0.89$). The Hydraulic conductivity can be represented by an equation of the form

$K = A(10)^{B\varnothing}$, where A and B are constants.

Table 2 . Parameters of Equation $K = A(10)^{B\varnothing}$

Sludge Type or Predictive Model	A × 10^{10}	SE(A) ,%	B	SE(B), %	R, %	M	Comment
DS	2.09	3.7	3.59	5.5	99.9	--	Experimental Data
FT	1.98	7.4	3.57	10.9	99.7	--	
ISO	2.26	5.9	3.75	8.9	99.8	--	
CCG	20.8	17.2	3.17	33.2	97.4	0.21	DS
MS	29.1	15.6	3.00	30.0	96.5	0.20	
MQ	73.0	15.1	2.51	29.3	96.2	0.18	
CCG	26.4	24.7	2.98	43.8	98.5	0.21	FT
MS	25.7	20.9	3.03	45.7	98.0	0.20	
MQ	72.3	22.8	2.50	40.5	97.5	0.18	
CCG	37.7	49.9	3.82	89.3	94.4	0.05	ISO
MS	50.3	53.04	3.61	94.9	94.4	0.06	
MQ	109.0	52.8	3.30	94.4	92.7	0.04	

Note :

Unit of A & B are consistent with K × 10^{-2} m s^{-1} and Ø expressed as decimal fraction.

DS = Untreated Digested Sludge.
FT = Digested Sludge after Freezing and Thawing.
ISO = Digested Sludge at Isoelectric Point.
SE(A) = Standard Error of A.
SE(B) = Standard Error of B.
R = Pearson Product Moment Correlation between ® and log K.
M = Ratio of experiental to predicated hydraulic conductivity at a given Ø.

(3) It is apparent that all predicted hydraulic conductivity values (by MS, CCG and MQ models) provide estimates which are 5 to 22 times higher than the measured values for unterated and treated sludge at a given moisture content. In view of this, it is apparent that statistical method based on pore size distributon measurement of digested sludge can not relate the predicated hydraulic conductivity to measured values without introducing an empirical matching factor. Thus a more sophisticated theoretical model is required in order to improve the accuracy of prediction of hydraulic conductivity from pore size distribution data. Again the hydraulic conductivity can also be predicted using pore size distribution measurement, using an empirical relation of the form $K = C\,(PSP)^b$ where C and b are constants.

Table 3. Regression Parameters of Equation $K = C\ (PSP)^b$

Sludge Type	C ×10^7	SE(C)	b	SE(b)	R,%	R^2,%
			CCG-Model			
DS	73.1	30.7	1.09	12.0	96.5	93.2
FT	96.9	38.7	1.13	16.4	98.0	96.0
ISO	11.9	40.4	0.92	22.8	94.4	89.1
All samples	8.6	25.9	0.74	11.1	87.0	75.8
			MS-Model			
DS	67.6	26.0	1.05	9.9	97.4	94.9
FT	118.4	34.4	1.16	14.4	98.5	97.0
ISO	9.9	38.7	0.87	21.7	94.4	89.0
All samples	7.8	24.5	0.70	10.3	87.7	76.9
			MQ-Model			
DS	149.1	36.0	1.29	15.0	96.1	92.5
FT	207.9	48.7	1.36	22.0	97.5	95.0
ISO	10.3	45.0	0.98	27.9	92.7	86.0
All samples	7.2	29.6	0.75	13.6	82.8	68.6

Note :

Unit of C and b are consistent with K ×10^{-2} m s^{-1}

DS = Untreated Digested Sludge.

FT = Digested Sludge after Freezing and Thawing.

ISO = Digested Sludge at Isoelectric Point.

SE(C) = Standard error of C.

SE(b) = Standard error of b.

R = Pearson Product Moment Correlation between log (PSP) and K.

R^2 = Coefficient of Determination.

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NOTATION

A, B = constants,
b = statistical regression parameter,
C = constant termed 'shape factor'.
CCG = Childs and Collis-George,
c, d, e = pore diameters,
d_1, d_2, d_n = mean pore diameter in each division,
DS = untreated digested sludge,
FT = digested sludge after freezing and thawing,
ISO = digested sludge at isoelectric point,
K = hydraulic conductivity,
L_o = length of wet sludge cake,
L_f = length of dry sample,
M = matching factor,
MS = Marshall,
MQ = Millington and Quirk,
n = no. of division of equal porosity,
p = pressure,
PSP = pore size parameter,
R = Pearson product moment correlation,
R^2 = coefficient of determination,
SE(A) = standard error of A,
SE(B) = standard error of B,
SE(b) = standard error of b,
SE(C) = standard error of C,
α, β, r, ε = porosity,
¢ = contact angle,
σ = surface tension of mercury,
Ø = moisture content,
¶ ω = density of water.