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Hydraulic conductivity of cement-stabilized marine clay with metakaolin and its correlation with pore size distribution

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ABSTRACT

Metakaolin (MK), widely used for high-performance concrete admixtures, is introduced into improving the percolation behaviors of cement-stabilized soft clays, which is a key performance when analyzing the permeability and consolidation in the grounds modified by deep mixing methods etc., the underground water seepage and the migration of pollutants in waterproof curtains by grouting methods etc. To investigate MK effect on the hydraulic conductivity of cement stabilized soils, the flexible wall permeameter was developed and mercury intrusion porosimetry (MIP) tests were performed. The results show that the addition can reduce the hydraulic conductivity 10–100 times when MK content arrives at 3% to 5%, which attributes to less total porosity and pore diameter. As the cement stabilized soils not only belong to a kind of special soils in geotechnical engineering, but also belong to cement-based reinforced materials, it is further discussed whether the unique expression of the hydraulic conductivity of these porous materials (clay, cement stabilized soils, cement pastes and concretes) existed. The results present that there is a good correlation between the hydraulic conductivity, the void ratio and median throat pore diameter for each kind of materials from clays to concretes respectively, and the hydraulic conductivity qualitatively increases with the void ratio and median throat pore diameter whatever clays, cemented soils and pastes or concretes. After several attempts, the combined variable nD_{50}^2 was adopted to normalize the macro and micro characterization of porosity, and the relatively unique relation depicting the hydraulic conductivity of these materials was proposed.

1. Introduction

Calcinations of pure kaolinite at temperatures from 550 °C to 900 °C produce an amorphous silica compound (metakaolin, i.e., MK), which is a very reactive aluminosilicate pozzolan (Janotka et al., 2010) and is usually used as a mineral additive in the cement and concrete (Qian and Li, 2001; Vejmelková et al., 2010). Caldarone et al. (1994), Wild and Khaitib (1996), Curcio et al. (1998) and Poon et al. (2001) found that the MK agent could obviously improve the early strength and that at a preset period whatever concretes or mortars. Boddy et al. (2001) and Gruber et al. (2001) found that the permeability of the Cl^- ion of the concrete with the MK agent sharply decreases which means the better concrete durability under sea water environments. Cassagnabère et al. (2011) compared the strength and durability of mortars with pure cement and those with ternary binders (cement + slag + metakaolin) in the view of the water absorption and oxygen permeability, and confirmed the MK's effectiveness and environmental friendship. Above all, the former researches showed the applicability and effectiveness of MK agent in concretes and mortars. It should be mentioned that the MK was also used as an agent to cement-based stabilized clays, as Kolovos et al. (2013) and Zhang et al. (2014) reported the application in the grouting and deep mixing projects respectively. The deep mixing method is another kind of ground improvement techniques to treat the soft clays and has been widely applied in China, Japan and Europe, where the soft clay and cement (in the form of a powder or slurry) are mixed by machines in situ. In the design of the deep mixing method, the strength and hydraulic conductivity of cemented soils should be both considered. Even though the MK effectiveness in strength of the cemented soils was reported (Zhang et al. (2014)), the MK effect on hydraulic conductivity is still not clear and needs to be further investigated.

The former researches on the hydraulic conductivity of pure cement stabilized soils and that of clays may help us perform this study. Terashi and Tanaka (1983) found that it decreased with the cement content while Chew et al. (2004) proposed that the hydraulic conductivity should be associated with the void ratio. The experiments of Broderic and Daniel (1990), Locat et al. (1996) and Lorenzo and Bergado (2006) confirmed the same results as Chew et al. (2004). Note that despite the relationship between the hydraulic conductivity and void ratio respectively existed for a kind of cemented soils,

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the statistical relationship appeared to be much dispersed when all data gathered.

Additionally, there is also no relatively unique relationship between the permeability and void ratio or clay fraction even though Tavenas et al. (1983) discussed that of clays. To uniform the relationships of clays, micro pore size distribution was taken into account according to the Poiseuille's theory for laminar flow though porous media (Childs and Collis-George, 1950) and several models have been developed to this end (capillary models, hydraulic-radius model, probabilistic models, summarized by Lapierre et al. (1990)). Since the pore size distribution by the mercury intrusion porosimetry (MIP) explicates the characterization of the entrance throat pore size and the cumulative pore volume, representative parameters of the functions must be selected for further analyzing, where Garcia-Bengochea et al.(1979), Juang and Holtz (1986) and Tanaka et al. (2003) did important works. Among them, Tanaka et al. (2003) proposed a relative simple combined variable (nD_{50}^2, where n is soil porosity and D_{50} is the median diameter of entrance pores when 50% of the total cumulative pore is attained in MIP tests) and tried to uniform the hydraulic conductivity of clays.

As the cemented soils with or without MK can be both classified to special soils and cement-based reinforced materials, the applicability of the empirical expressions of the hydraulic conductivity from clays, cement stabilized soils and concretes needs to be further evaluated.

To understand the percolation behavior of cemented soils and MK effect on the hydraulic conductivity, ordinary Portland cement (OPC) and MK were first prepared and mixed at various mass ratios, and then this mixture was then remixed with Lianyungang marine clay (a type of marine clay typically deposited in the eastern China; Deng et al., 2014) to obtain the cement stabilized soils. After curing at standard conditions according to the standard ASTM D1632 (2007), the percolation and MIP tests were performed to investigate the MK and micropore effect on the hydraulic conductivity. Furthermore, the data of the hydraulic conductivity and pore size distribution of the clays, cement stabilized soils, cement pastes and concretes are gathered to discuss and statistically evaluate the empirical expressions, which can be used for predicting and revealing the mechanism of percolation behavior of these materials.

2. Materials and testing methods

2.1. Materials

Lianyungang clay, a type of quaternary marine sedimentation, is widely distributed in the eastern coastal areas of Jiangsu province in the eastern China. The water content, sensitivity, and compression of this material are high, while its strength and permeability are low (Deng et al., 2014). The basic properties of the selected soil samples are listed in Table 1. Note that the liquid limit, plastic limit and natural water content of the marine clay are 58.8%, 27.2% and 61.5%, respectively, and the soils is classified as a high liquid limit clay according to the plasticity chart (ASTM D2487-11) in Fig. 1.

A semi-quantitative mineral analysis by X-ray diffraction was conducted, and its results are listed in Table 2. The total mineral content consists of quartz (23.2%), plagioclase (15.6%), feldspar (4.1%), calcite (12.1%) and clay fraction (45.0%, particle size less than 2 μm). The clay fraction contained the kaolinite, illite, chlorite and illite/smect mixed-layer clay mineral at percentages of 13.0%, 29.0%, 14.0% and 44.0%, respectively.

Table 1
Physical properties of soft clay.

Water content (%)	Wet density (kN/m^3)	Specific density G_s	void ratio e	Particle size distribution (%)			Liquid limits W_L (%)	Plastic limits W_p (%)
				sand	silt	clay		
61.5	16.8	2.70	1.68	2.6	43.9	53.5	58.8	27.2

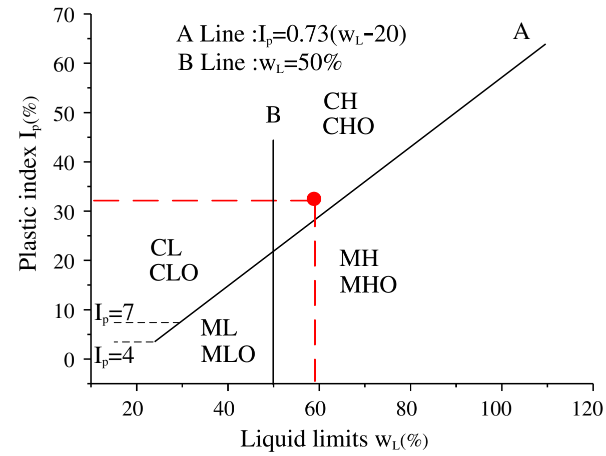

Fig. 1. Plasticity chart (ASTM:D2487-11).

Table 3 presents the oxides of the ordinary Portland cement (OPC 42.5 R/N) and metakaolin (Metamax from BASF German). Note that the Portland cement used in this study falls well within the guidelines of the European Cement Standard (EN 197-1), which specifies that the ratio of CaO to SiO_2 should exceed 2.0 and the MgO content should not exceed 2.0%. The total content of SiO_2 and Al_2O_3 in the MK is approximately 92%, the average particle size is less than 4 μm and the specific surface area is about 10 m^2/g.

2.2. Sample preparation

To prepare the testing samples, the selected marine clay was first dried at 30 °C, and tap water was then added until the water content reached 70% (approximately 1.2 w_L) to simulate the worst insite case. After one day of curing, the OPC (mass ratio content of cement to wet soil at 12% and 15%) and MK (mass content of MK to wet soil at 0%, 1%, 3% and 5%) were mixed with the prepared soils. The mixed clay–cement–MK paste was then injected into a plastic mold with detachable covers at both ends, that was 100 mm in length and 50 mm in diameter. After the first 24 h of curing, the cemented soil samples were removed from the molds, wrapped in polythene bags and stored in a standard chamber at 95% in humidity and 20 ± 2 °C in temperature. After 28 days of curing, the samples were removed to permeameter for determining hydraulic conductivities, and to freeze dryer and mercury intrusion porosimeter for determining the pore size distribution respectively. The properties of the stabilized soils after 28 days curing are listed in Table 4, including the water content, density, void ratio and porosity. Note that the specific gravity of cement stabilized soil, a main parameter used to calculate the void ratio and porosity, is considered same as that of the original soil, equal to 2.70 in this case because the former study shows that the cement adding does not substantially affect the specific gravity (Pakbaz and Alipour, 2012).

2.3. Hydraulic conductivity testing

As the hydraulic conductivity of cemented soils is relatively small, usually less than 10^{-8} cm/s, the common constant and variable head permeameters cannot be directly considered. In this study, the triaxial cell was redesigned as the permeameter shown in Fig. 2 referring ASTM standard (D5084-10). During this test, the cell pressure σ_3 was preset as 500 kPa and the seepage pressure σ_1 as 400 kPa. Note that there was almost no volume change that occurred when the cell pressure arrived at 500 kPa because the strength of the samples was almost

Table 2
Mineral composition of Lianyungang marine clay.

Total mineral (%)					Clay mineral (%)			
Quartz	Feldspar	Plagioclase	Calcite	Clay content	Illite	Kaolinite	Chlorite	Illite/smectite
23.2	4.1	15.6	12.1	45.0	29	13	14	44

higher than 1.0 MPa and estimated pre-yielding stress was more than 3.0–5.0 MPa. After the sample was installed and it commenced percolation, the leachate was collected and weighted per 2 h. It needs to be mentioned that the height of these samples was re-trimmed to 3 cm to reduce their seepage path because of the very low hydraulic conductivity of the stabilized soils with the 15% cement content. In other words, the hydraulic gradient of the samples with 12% cement content is 500, while that with 15% cement content is 1333. Note that in spite of the high hydraulic gradient of the cement stabilized soils with 15% cement content, the percolation test works well, and the linear flow is observed.

With Darcy law shown in the Eq. (1), the hydraulic conductivity can be calculated:

$$k = q/(Ai) \tag{1}$$

where q is leachate volume per time (m^3/s), A is sample area (m^2) and i is hydraulic gradient. The measured hydraulic conductivity is added in Table 4.

2.4. Mercury Intrusion Porosimetry (MIP)

MIP is a method to determine the pore size distribution of porous materials based on the unique relationship between the intrusion pressure and equivalent pore diameter proposed by Washburn (1921):

$$D = -\frac{4\gamma\cos\theta}{P} \tag{2}$$

where D is the pore diameter, γ is the surface tension of mercury, θ is the contact angle, and P is the applied pressure. In this study, a contact angle of 140° and a mercury surface tension of 0.480 N/m were recommended. Because the range of intrusion pressure was 3.7 kPa to 241.1 MPa for the PoreMaster-60 (by Quantachrome Corporation USA), the pore sizes measured ranged from 0.005 to 340 μm. In this study, eight samples (i.e., 12% and 15% cement with 0%, 1%, 3% and 5% MK after 28 curing days) were tested and the main parameters of the pore size distribution curves are also listed in Table 4.

Note that the samples were lyophilised to minimize the shrinkage of cemented soils before MIP tests. Small pieces of specimens were first trimmed to appropriate sizes and shapes and then immersed in liquid nitrogen (−196 °C) for instant freezing. The frozen specimen was then immediately transferred to the vacuum chamber of a freeze dryer for sublimation, which was sustained for approximately 24 h (Penumadu and Dean, 2000).

Table 3
Oxides composition of ordinary Portland cement and metakaolin.

Oxide content (%)	SiO_2	Al_2O_3	CaO	Fe_2O_3	SO_3	MgO	Na_2O	K_2O	Loss on ignition
OPC	19	6.5	65	3.2	2.5	0.8	0.5	0.4	2.1
MK	52	40	1.0	2.5		0.8	0.5		

3. Results

3.1. Hydraulic conductivity

Figs. 3 and 4 present the time-dependent leachate mass under a preset hydraulic gradient of the cemented soils respectively, where C12MK3 annotates the sample with 12% cement content and 3% MK in mass. Note that the seepage times of samples with 12% cement content last about 50 h while that with 15% cement content last about 120 h even though the hydraulic gradient with 15% cement content is about 2.6 times of that with 12% content, which means that lower permeability of soils with more cement content. The hydraulic conductivity can be calculated with Eq. (1) and q (leachate mass per time), which is measured by the slope of the curve.

Fig. 5 explicates the MK effect on the hydraulic conductivity of the cemented soils and shows that the addition of MK significantly reduces the hydraulic conductivity. It decreases from 10^{-8} cm/s to 10^{-9} cm/s at 12% cement content and decreases from 10^{-9} cm/s to 10^{-11} cm/s at 15% cement content when the MK content arrives at 5%. Note that when the MK content is more than 3%, the reduction of the hydraulic conductivity slows down. In other words, the optimal MK content may be 3% in the view of reduction efficiency in this study. It also should be mentioned that the increasing of the cement content also decreases the hydraulic conductivity which is consistent with the results of the cemented Bangkok clay by Lorenzo and Bergado (2006).

3.2. Pore size distribution

Since the MK addition effectively reduces the percolation behavior of the cemented soils, their micro pore size distributions are investigated by the mercury intrusion porosimetry. Figs. 6 and 7 present the curves of the accumulative mercury intrusion volume per gram soils (V_m) and the differential intrusion volume versus the entrance pore diameter of stabilized soils with 12% and 15% cement contents after 28 days' curing respectively. The results show that the accumulative intrusion volumes range from 0.473 mL/g to 0.425 mL/g of soils with 12% cement content and 0.442 to 0.402 mL/g with 15% cement content. It is interesting that the accumulative volume decreases with the cement and MK content, and the peak of the differential intrusion volume curve (d V_m/dlog D) moves towards the less pore diameter. This means that the MK content not only decreases the accumulative volume, but also changes the pore size distribution. Additionally, despite the ratio of the void ratio determined by the accumulative intrusion volume (e_M) and by the water content (e_w) ranges from 0.79 to 0.81, i.e., the pore volume determined by the water content is about 1.2 times more than by the mercury volume, it is consisted with the results of stiff Boom clay by Nguyen et al. (2013) who considered that the MIP technique can only cover a limited range of pore size, from 340 to 0.005 μm in apparent mercury entrance diameter D (corresponding to a pressure range from 3.7 kPa to 240 MPa). It is also mentioned that the unique peak of the log differential intrusion curve of the cement stabilized soils (in Figs. 6 and 7) is identical with the most observations of the common concretes and cement pastes (Roy et al., 1993; Cook and Hover, 1999).

Table 4
Properties of stabilized soils after 28 days curing.

Sample	Water content w (%)	Density (kN/m^3)	Void ratio[a] e_w (−)	Porosity[b] n (−)	Hydraulic conductivity (cm/s)	V_m (mL/g)	Void ratio[c] e_M (−)	e_M/e_w	D_{50} (nm)
C12MK0	57.9	16.6	1.57	0.611	9.97E−09	0.473	1.277	0.81	69.8
C12MK1	56.5	16.8	1.51	0.602	5.85E−09	0.455	1.229	0.81	65.9
C12MK3	51.3	16.9	1.42	0.587	1.09E−09	0.428	1.156	0.81	62.9
C12MK5	50.1	16.8	1.41	0.585	1.05E−09	0.425	1.148	0.81	56.5
C15MK0	53.6	16.5	1.51	0.602	5.83E−10	0.442	1.193	0.79	77.1
C15MK1	53.3	16.6	1.49	0.598	3.53E−11	0.437	1.180	0.79	47.2
C15MK3	48.2	16.8	1.38	0.580	1.39E−11	0.404	1.091	0.79	38.3
C15MK5	47.5	16.8	1.37	0.578	1.08E−11	0.402	1.085	0.79	40.1

[a] e_w determined by water content with the expression $e_w = \frac{G_s(1+w)\rho_w}{\rho} - 1$ where G_s is the specific gravity of soils, selected as 2.70 in this case, w is the water content, ρ_w and ρ are the densities of water and soils.
[b] The relationship between the void ratio and porosity is $n = e/(1+e)$.
[c] e_M defined as the ratio of mercury intrusion volume V_m to soil solid volume V_s and determined with the expression $e_M = V_m G_s$.

3.3. Micro pore effect on hydraulic conductivity of cemented soils with MK

Hydraulic conductivity and pore size distribution present the inherent relation if detailed examining the properties in Table 4. According to the existing researches, the void ratio e, D_{50} and nD_{50}^2 are selected to be on behalf of the characterization of micro pores, and the results are discussed in Fig. 8. It is obvious that the hydraulic conductivity k can be expressed as bellows (R is the correlation coefficient):

$$k = 10^{22.85*(e-1.868)} \quad (\text{cm/s}) \quad R^2 = 0.483 \tag{3}$$

$$k = 10^{95.64*(D_{50}-0.156)} \quad (\text{cm/s}) \quad R^2 = 0.727 \tag{4}$$

$$k = 10^{1438*(nD_{50}{}^2-0.00864)} \quad (\text{cm/s}) \quad R^2 = 0.663. \tag{5}$$

It can be found that the hydraulic conductivity is closely related with void ratio (or porosity, on behalf of total pore volume per gram of soils) and median entrance throat diameter of micro pores (D_{50}, on behalf of pore size distribution formation). Note that the combined variable $nD_{50}{}^2$ proposed by Tanaka et al. (2003) also showed a good correlation with hydraulic conductivity. Above all, the above equations confirm that hydraulic conductivity is the macro reflection of micro structures.

4. Discussions

The results of cement stabilized soils with MK present that there is a good relationship between the hydraulic conductivity and the variables of pore structures (the void ratio e or porosity n, median pore-throat diameter D_{50} and combined variable nD_{50}^2), and the pore structures mainly control the percolation behavior of these materials. It is necessary to clarify that compatibility of the relationships for the more materials since the stabilized soils in this study is not only a kind of special soils but also belongs to cement-based reinforced materials. To achieve this objective, about 130 data of the clays, rocks, concretes, cement stabilized pastes and soils with void ratio (or porosity), pore size distribution and hydraulic conductivity are collected from the literatures (concrete: Ahmad et al., 2005; cemented soils: Chew et al., 2004; concrete and rock: Gao and Hu, 2013; cement paste: Ma et al., 2013; Ye et al., 2006; Concrete: Roy et al., 1993; soft clay: Tanaka et al., 2003), and the relationships between the k and e, between k and D_{50} and between k and nD_{50}^2 were further statistically analyzed.

4.1. Relationship between k and e

The relationship between hydraulic conductivity k and void ratio e of the above databases is shown in Fig. 9. It is clear that there is a good linear correlation between log k and e for each kind of materials respectively, which is usually adopted to depict the percolation behavior for clays and cement stabilized soils. In other words, the percolation behavior of cement pastes and concretes also follow the same laws, same as clays and cement stabilized soils. On the other hand, there is not a unique function to depict the relationship for all the materials in these coordinates. Additionally, the slope between e and log k for the clays

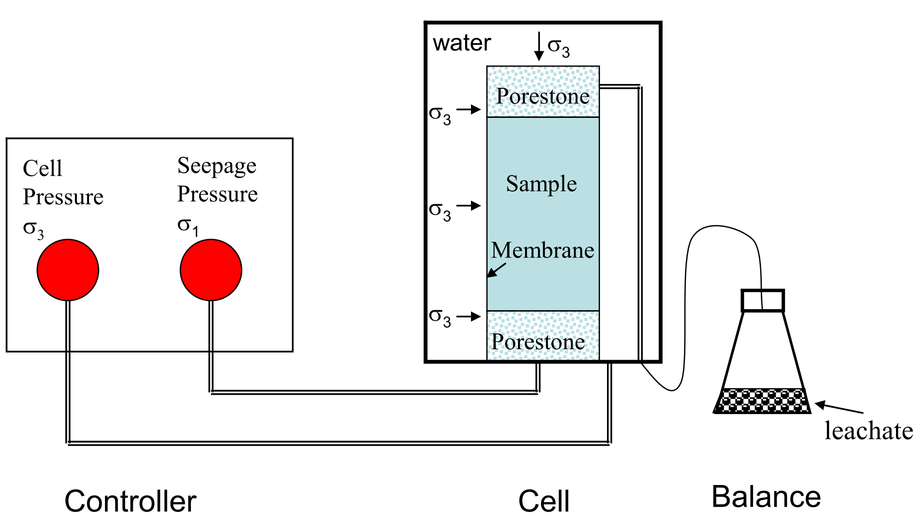

Fig. 2. Sketch of permeameter.

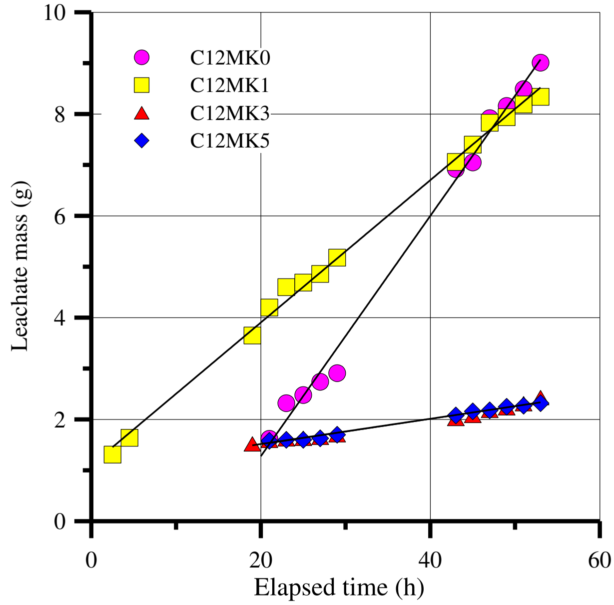

Fig. 3. Leachate mass of stabilized soils with 12% cement content.

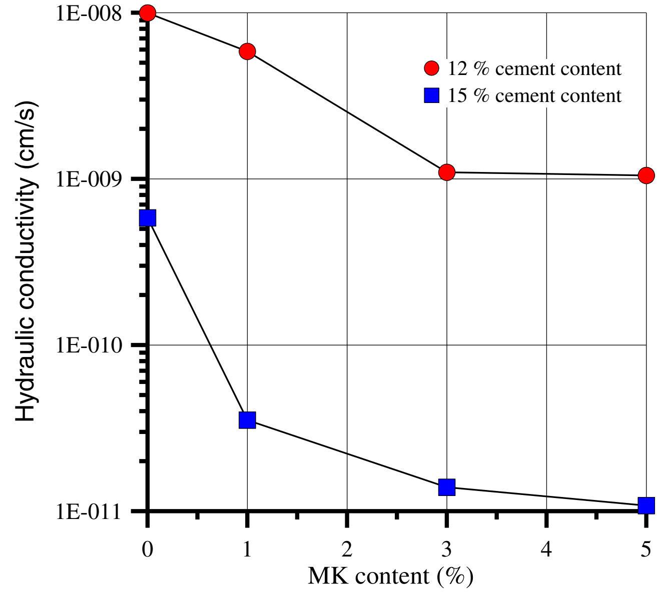

Fig. 5. MK effect on the hydraulic conductivity of stabilized soils.

and common cement soils (Tanaka et al., 2003; Chew et al., 2004) is very close, whist that of cemented soils with MK agent shows the similarity as that of the cement pastes and concretes, that implies the pore structures and engineering properties of the cemented soils with MK may be closer to those of the latter.

4.2. Relationship between k and median throat pore D_{50}

In the view of Poiseuille's theory for laminar flow though porous media (Childs and Collis-George, 1950), the water percolation behavior depends on the material pore sizes. The median throat pore diameter D_{50} is a representative variable to describe the pore size distribution. The relationship between hydraulic conductivity k and median throat pore diameter D_{50} of the above data is presented in Fig. 10. The hydraulic conductivity totally increases with the median throat pore diameter whatever clays, cemented soils and pastes, or concretes despite the data are slightly scattered. Note that some minor changes occur when the median pore diameter arrives at about 0.5 μm, i.e., the hydraulic conductivity changes significantly when the median pore size is less than 0.5 μm, while slightly when the pore size more than about 0.5 μm.

4.3. Relationship between k and nD_{50}^2

Since the analysis of cement stabilized soils with MK in Fig. 8 showed a good correlation between k and nD_{50}^2, this relation is recounted on the gathered data and shown in Fig. 11. The results show that the combined variable nD_{50}^2 can relatively simply uniform the hydraulic conductivity involving the materials from clays to concretes. The unique relation depicting the hydraulic conductivity is listed as Eq. (6). Note that its correlation coefficient R (0.82) shows the reasonable statistics.

$$k(\text{cm/s}) = 10^{-5.96}\left[nD_{50}^2\right]^{1.06} \qquad R^2 = 0.760 \tag{6}$$

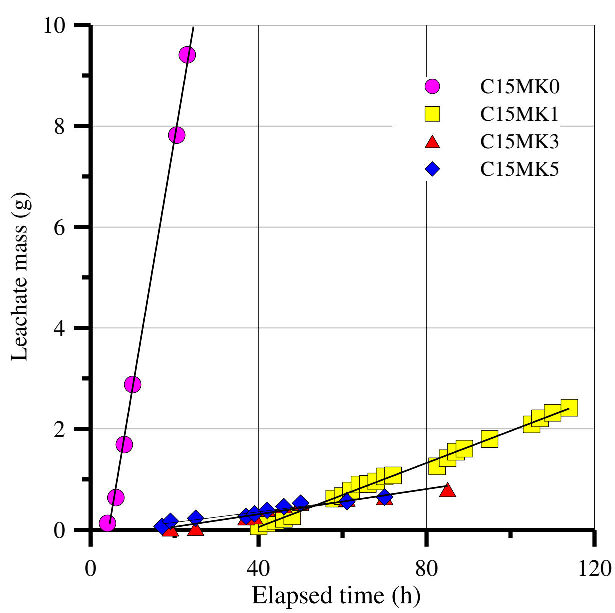

Fig. 4. Leachate mass of stabilized soils with 15% cement content.

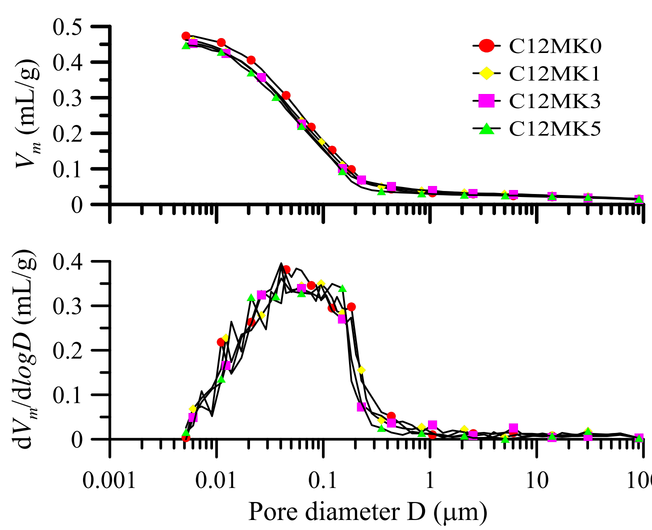

Fig. 6. MIP results of stabilized soils with 12% cement content.

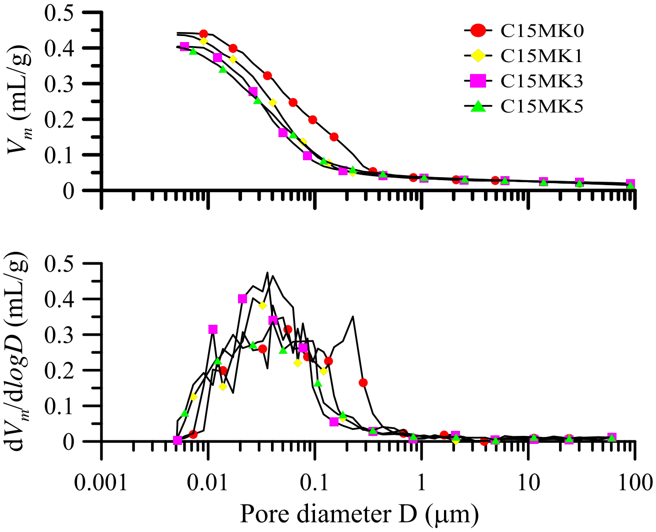

Fig. 7. MIP results of stabilized soils with 15% cement content.

Furthermore, if $\log \frac{k}{[nD_{50}^2]^{1.06}}$ is considered as the new general variable, the mean value is −5.96 and standard deviation is 0.70 respectively after the reanalysis. It can be deduced that 68.3% probability of data located in the range between the lines $k(\text{cm/s}) = 10^{-5.26}[nD_{50}^2]^{1.06}$ and $k(\text{cm/s}) = 10^{-6.66}[nD_{50}^2]^{1.06}$ shown in Fig. 11, according to the normal distribution theory of the statistics. It should be also mentioned that as this statistics is conducted based on the limit data, the parameters (e.g., −5.96 and 1.06) may be slightly changed if more data are supplied, but the combined variable nD_{50}^2 proposes the feasibility to uniform the hydraulic conductivity of materials from clays to concretes.

5. Conclusions

Metakaolin (MK), a new fine powder agent, widely used to improve the performance of concretes and pastes, is effectively introduced to cement stabilized soft clays. In this study, the percolation behavior (i.e., the hydraulic conductivity) of the cemented soils with and without MK was first discussed by the improved permeameter tests, and the significant association of the hydraulic conduction and the pore size distribution was found. To further analyze and clarify the intrinsic relationship between the hydraulic conductivity and pore size distribution of cement-based reinforced materials (concretes, cement pastes and cemented soils) and clays (or soils), the data of the above mentioned materials were collected, and the relationships between k and the void ratio e (porosity n), median throat pore diameter (D_{50}) and combined variable (nD_{50}^2) were discussed. The main results are listed as follows:

(1) Addition of MK effectively decreases the hydraulic conductivity. The hydraulic conductivity of cement stabilized soils with 3% to 5% MK is about 10 times less than that without MK at 12% cement content, and 100 times less than that without MK at 15% cement content. In the view of reduction efficiency of the hydraulic conductivity, the optimal MK content is 3%.
(2) The correlations of hydraulic conductivity and micropore parameters of cement stabilized soils with MK are established, which

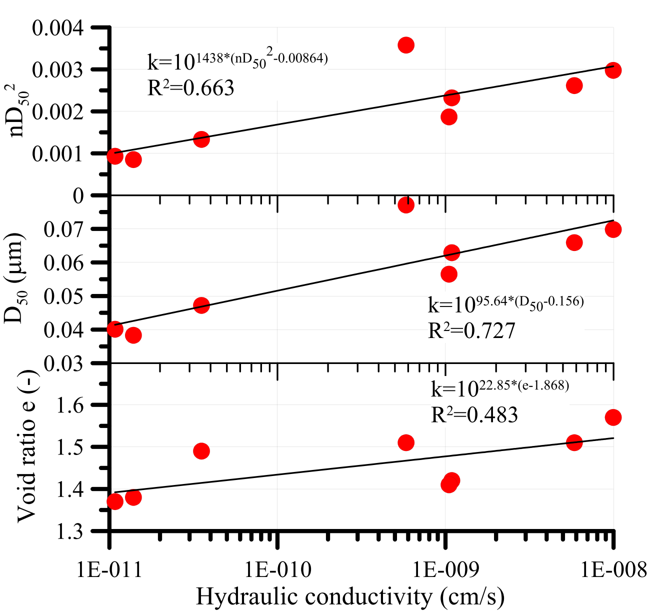

Fig. 8. Micro pore effect on hydraulic conductivity of cemented soils with MK.

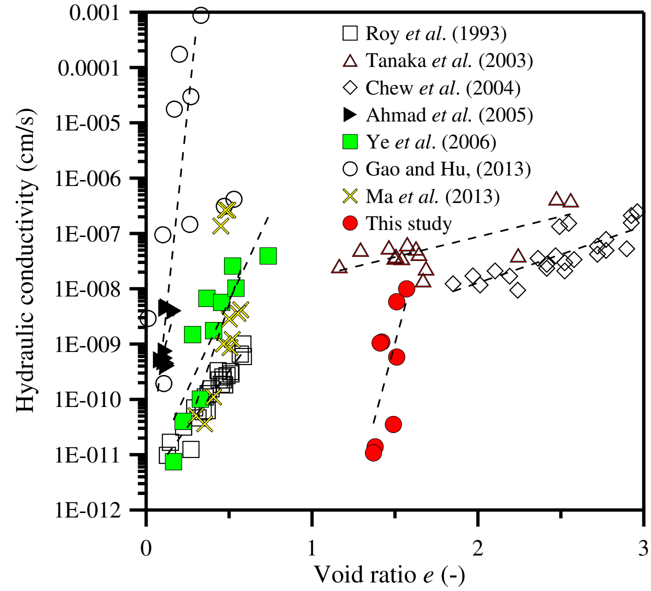

Fig. 9. Relationship between k and e.

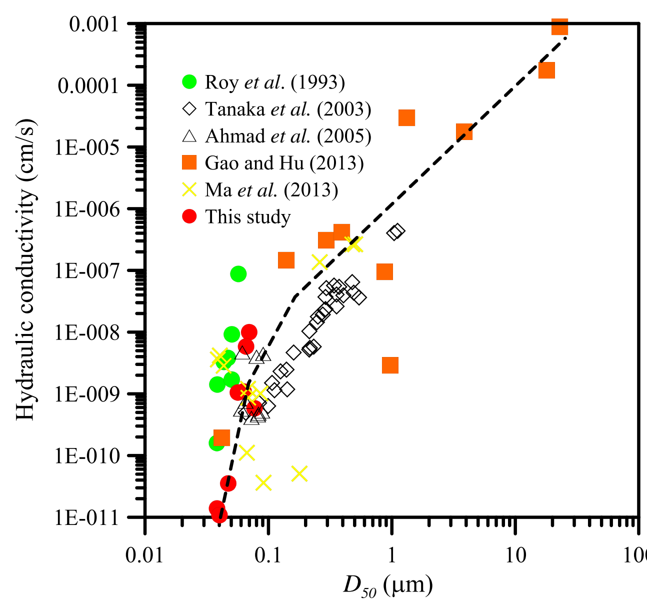

Fig. 10. Relationship between k and D_{50}.

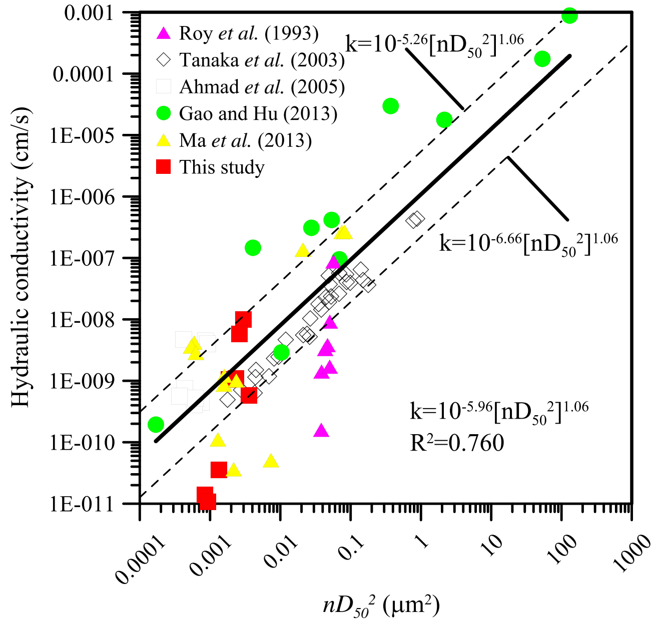

Fig. 11. Relationship between k and nD_{50}^2.

means that the effectiveness of MK to decrease the hydraulic conductivity is attributed to less total porosity and throat pore diameter.

(3) The statistical analysis of the hydraulic conductivity and void ratio of materials from clays to concretes shows that a good correlation for each kind of materials respectively. The slope between e and log k for the clays and common cement soils is very close, whist that of cemented soils with MK agent is closer to that of the cement pastes and concretes.

(4) The hydraulic conductivity totally increases with the median throat pore diameter whatever clays cemented soils and pastes or concretes. It changes significantly when the median pore size is less than 0.5 μm, while slightly when the pore size more than about 0.5 μm.

(5) The unique relation depicting the hydraulic conductivity of materials from clays to concretes can be expressed as $k(\text{cm/s}) = 10^{-5.96}[nD_{50}^2]^{1.06}$ and the 68.3% probability of data located in the range between the lines $k(\text{cm/s}) = 10^{-5.26}[nD_{50}^2]^{1.06}$ and $k(\text{cm/s}) = 10^{-6.66}[nD_{50}^2]^{1.06}$ according to the normal distribution theory of the statistics.

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