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Mechanical behaviour and micro-structure of cement-stabilised marine clay with a metakaolin agent

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HIGHLIGHTS

- Metakaolinite is an effective agent for cemented soils.
- Unconfined compression strength with 3% and 5% MK are about 2.0–3.0 times that of soils lacking MK.
- Cemented soils with MK more feasibly generate early strength.
- The addition of MK mainly changes the pore volume distribution and produces more CSH/Aft/CASH bonding and fissures.

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ABSTRACT

Metakaolin, a fine powder material, has been widely used as an effective additive to produce the high-performance concrete since 1990s, for its high efficiency and relatively low price. However, metakaolin has rarely been attempted in admixtures of cement-stabilised soft clays until presently. This paper focuses on the macro-strength and micro-structure development of cement-stabilized Lianyungang marine clay mixed with metakaolin. The results show that the unconfined compression strengths of cemented soils containing 3% and 5% MK are approximately 2.0–3.0 times of that of materials without MK, that is MK can effectively improve the quality of cemented soils. Additionally, the strength with MK after 7 day's curing periods is approximately 0.87 times of that after 28 day's, while this ratio is 0.58 for the soils lacking MK, which indicates that the cemented soils containing MK show sufficient earlier strength than those lacking MK. Finally, the microstructure analysis reveals that MK mainly changes the pore volume distribution, which ranges between 0.01 μm and 1 μm, and produces more CSH/Aft/CASH bonding and fissures due to the secondary hydration and pozzolanic reactions.

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 [21] and is usually used as a mineral additive in the cement and concrete [31,36,30,27]. The chemical equation of the calcinations is shown below:

$$2Al_2Si_2O_2(OH)_4 \overset{600-900\,^{\circ}C}{\rightarrow} 2Al_2Si_2O_7 + 4H_2O \quad (1)$$

MK will react with calcium hydroxide (CH) in the presence of water to produce a CSH-like cementing compound and a phase that contains hydrates of alumina [26]. Furthermore, previous studies have emphasized that MK has a higher pozzolanic activity (between 610 and 1150 mg CaO/g) than silica fume and fly ash (approximately 400–450 mg CaO/g) [21,8,15,16,18,19]. This activity was attributed to its chemical components, fine grain size and proportion of particles with sizes in the range of 1–10 μm [10,4]. It needs to be mentioned that the high-performance concrete mixture or cement paste was usually produced by replacing Portland cement with 8–20% metakaolin in engineering practices to take advantage of the positive effects of MK, such as the filler effect, accelerated hydration, and pozzolanic reaction with calcium hydroxide (CH). Among these effects, the filler effect occurs instantly, the accelerated hydration works within the first 24 h, and the pozzolanic reaction is maximised between 7 and 14 days [38].

The deep mixing method is one of the most popular 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 [11,39]. When the ground is improved, the deep

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mixing columns and soils among them form the composite foundation to support the upper embankments or structures. Therefore, the strength of the cemented soil, a key parameter, is usually designed to range from 0.8 MPa to 1.2 MPa, and ordinary Portland cement (OPC) is usually adopted as the cementation agent in the engineering practices [29,23]. Because MK effectively improves the strength and durability of concrete mixtures and cement pastes when used as an additive, the capability of MK to improve the macro-behaviour of cemented soil when used as an additive becomes an interesting subject of study. Furthermore, the micro-mechanism of MK in the cemented soils also requires investigation.

This study aims to investigate the strength and micro-structure evolution of cemented Lianyungang marine clays mixed with MK for the application of the deep mixing method. The strength and secant modulus E_{50} of cemented soils after 7 days' to 28 days' curing were compared to clarify the MK's efficiency. To achieve the above objectives, ordinary Portland cement and MK were first prepared and mixed at various mass ratios. This mixture was then remixed with Lianyungang marine clay (a type of marine clay typically located in eastern China) to obtain the cemented soil samples. The samples were then cured at standard conditions according to the standard [1]. Unconfined compression tests, scanning electronic microscopy (SEM), and mercury intrusion porosimetry (MIP) were performed for pre-defined curing periods to clarify the evolution of the macro-strength and micro-structure.

2. Materials and testing methods

2.1. Materials

Lianyungang clay, a type of quaternary marine sedimentation, is widely deposited in the eastern coastal areas of China, which is of the high water content, high sensitivity, and high compression while of low strength and low permeability. The basic properties of the selected soil samples are listed in Table 1 and it is characterized as a high plasticity (CH) clay by the USCS (Unified Soils Classification System [2], ASTM D2487-11).

The mineral component of the Lianyungang marine clay is listed in Table 2, where the samples were pre-treated by ethylene glycol and the semi-quantitative analysis was carried out by the Jade software. Note that the clay mineral is the main component and the content of the interstratified illite/smectite is over 40%.

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 was approximately 92%, the average particle size was less than 4 μm and the specific surface area was approximately 10 m^2/g.

2.2. Sample preparation and unconfined compression test

To prepare the testing samples, the selected marine clay was first dried at 30 °C, and distilled water was then added until the water content arrived at 70% (approximately 1.2 w_L, between the sample's natural water content, *i.e.* 61.5% and the maximum natural water content of the site, *i.e.* 75%). After one day of curing, the OPC (mass 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. Note that the selected cement content was less than that adopted in most ground improvement projects in Chinese highway engineering (usually ranging from 15% to 20%) [24] considering the effectiveness and economy of MK agent, while the MK content covered the ranges applied in concrete engineering (usually the ratio of cement mass to the total mass of the cement and MK ranges from 8% to 20% [31]).

The mixed clay-cement-MK paste was then agitated for 5–10 min and transferred into a plastic mould with detachable covers at both ends, which was of 100 mm in length and 50 mm in diameter. To improve the uniformity and reliability, the soil paste was artificially compacted by vibration, and more than three parallel samples were fabricated for each group to avoid discrete results. The density and moisture content of samples of the soil-cement-MK paste after compaction were measured instantly and listed in Table 4, where the water content decreased with cement and MK content because these above two materials increased the solid mass. After the first 24 h of curing, the cemented soil samples were removed from the moulds, wrapped in polythene bags and stored in a standard chamber at 95% humidity and 20 ± 2 °C. Note that this sample preparation procedure is widely recommended for the cemented soft clay [25,13,14,19].

After 7 and 28 days of curing, the samples were removed to perform the unconfined compression test (*i.e.,* UCT) to determine their strength (f_{cu}) and secant modulus (E_{50}). The shearing rate was defined at 1 mm/min in this study.

2.3. Mercury intrusion porosimetry (MIP)

MIP is a method used 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 [37]:

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

where D is the pore diameter, γ is the surface tension of mercury, θ is the contact angle, and P is the applied pressure. Note that the recommended contact angle of 140° and the mercury surface tension of 0.480 N/m were employed [7,28,32]. 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, six samples (i.e., 15% cement without MK, 15% cement with 3% MK and 15% cement with 5% MK after 7 and 28 days of curing) were tested.

Table 1
Physical properties of soft clay.

Natural water content (%)	Wet density (kN/m^3)	Void ratio e	Particle size distribution (%)			Liquid limits W_L (%)	Plastic limits W_p (%)
			Sand	Silt	Clay		
61.5	16.8	1.68	2.6	43.9	53.5	58.8	27.2

Table 2
Mineral composition of Lianyungang marine clay.

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

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		

Table 4
Density and water content of samples.

Cement content (%)	MK content (%)	Density (g/cm^3)	Water content (%)
12	0	1.655	55
12	1	1.638	49
12	3	1.643	47
12	5	1.646	46
15	0	1.649	52
15	1	1.664	46
15	3	1.690	44
15	5	1.669	43

The samples were lyophilised to minimise the shrinkage of cemented soils. 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 transferred to the vacuum chamber of a freeze dryer for sublimation, which was sustained for approximately 24 h [28].

2.4. Scanning electronic microscopy (SEM)

Scanning electron microscopy was used to further investigate the microstructure changes of the cemented soils containing MK and mechanism of action of MK. Two specimens (*i.e.,* 15% cement without MK and 15% cement with 3% MK cured for 28 days) were scanned in this study.

To perform this test, the cube specimens with 1 cm in length were pre-treated and lyophilized same as MIP test. After that, the dried specimens were trimmed flat and vacuum coated with a layer of gold in the thickness of 200–300 Å (1 Å = 0.1 nm) to make it electrically conductive to prevent electric charge build up on the specimens [5].

3. Results and discussions

3.1. Mechanical behaviours of cement-stabilized samples containing MK

Fig. 1 shows the typical stress–strain curves of the specimens mentioned above and preliminary results show the significant strength increases with the MK content. The stress–strain curves have a plastic ductile yielding appearance when the MK content was below 1% (f_{cu} < 1 MPa) and gradually tended towards brittle failure as the MK content was increased. A comparison of the strength of cemented soils with and without MK shows that cemented soils containing more than 3% MK were approximately 2–3 times higher than those lacking MK, which demonstrates the strong contribution of MK irrespective of the curing period and cement content.

The typical relationship between the unconfined compression strength and MK content is shown in Fig. 2. Interestingly, the strength of cemented soils nonlinearly increases with the MK content, but this growth slows when the MK content reaches 3% (with the exception of the sample containing 15% cement that was cured for 7 days). Uddin [35] and Bergado et al. [6] studied the influence of the cement ratio, a_w, on the strength of cemented soils and proposed three zones according to the strength growth: the inactive zone, the active zone and the quasi-inert zone. Thereafter, MK effect in this study can be classified into two zones, *i.e.,* the active zone and the inert zone, and the threshold was approximately 3% if imitating cement ratio effect. Note that the 3% MK (MK to wet soil) content, the optimal MK content with respect to increases in strength, corresponds to a 15–20% MK replacement ratio (*i.e.,* MK mass to cement and MK mass combined), which falls into the rational replacement ratio range from 8% to 20% proposed by Qian and Li [31] for the concrete tests.

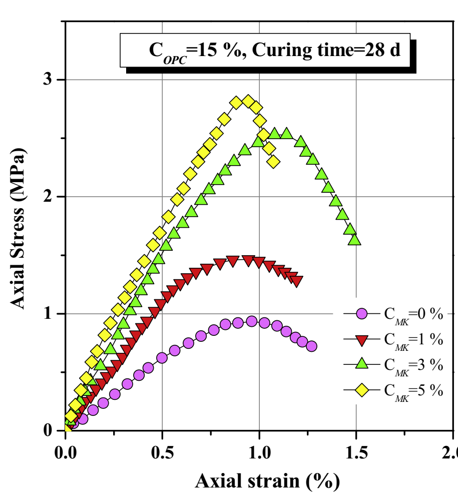

Fig. 1. UCT results with 15% cement after 28 days of curing.

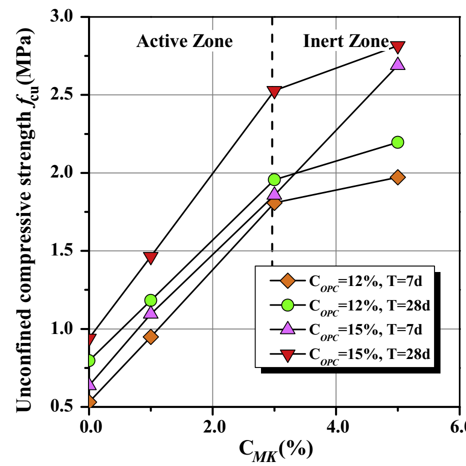

Fig. 2. Relationship between f_{cu} (MPa) and MK contents (%).

It is mentioned that the cement hydration process produces the calcium silicate hydrate (CSH) and calcium hydroxide (CH), and the secondary hydration reaction occurs to form additional cementitious aluminium containing CSH gel, together with crystalline products which include calcium aluminate hydrates (C_3AH_6 and C_4AH_{13}) and alumino-silicate hydrates (C_2ASH_8) if there exists the active $Al_2O_3{\cdot}SiO_2$ (the main component of MK). Since the optimal mass ratio of MK to CH ranges from 0.5 to 1 during the secondary hydration process, and the mass of CH accounting for 20% of cement can be produced during the cement hydration [24], MK replacement ratio should be from 10% to 20% under ideal conditions, which is consistent with this study. Additionally, when the mass ratio of MK to CH is 0.5, the product is towards C_4AH_{13} and CSH; when the mass ratio is 0.6, the product is towards C_3AH_6 and CSH; and when the mass ratio arrives at 1.0, the product is towards C_2ASH_8 and CSH respectively [24]. In other words, the strength evolution in Fig. 2 with cement content, MK content and curing periods is caused by the complex formations of the hydration and secondary hydration.

Considering that with MK is not only a type of special cemented soil, the empirical relationship of common cemented soils must be verified for this material. Fig. 3 showed the f_{cu} of cement-treated Lianyungang soft clay containing MK after 7 and 28 days of curing compared to that of Bangkok, Ariake and Shanghai clay using just ordinary Portland cement or slag-cement [12,22,18,19]. The linear fitting results are expressed as follows:

$$f_{cu,7} = 0.87 f_{cu,28} \quad \text{(cement treated clay with MK)} \tag{3}$$

$$f_{cu,7} = 0.58 f_{cu,28} \quad \text{(just cement treated clay)} \tag{4}$$

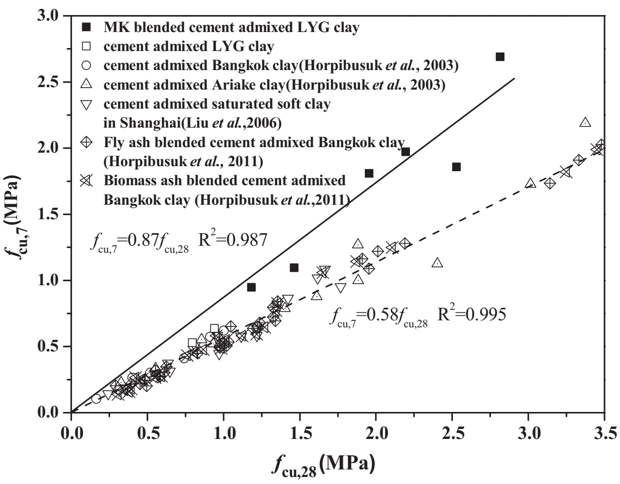

Fig. 3. $f_{cu,7}$ vs. $f_{cu,28}$ plot for different cement-treated clays.

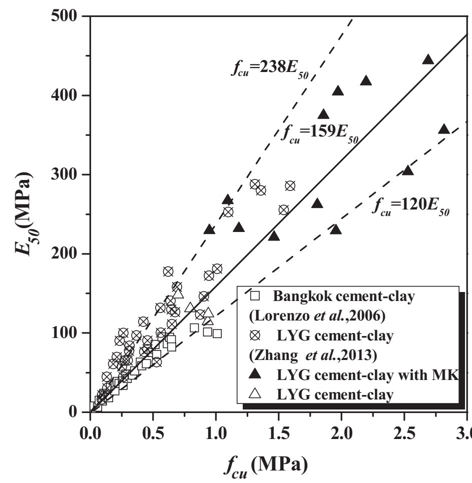

Fig. 5. Correlation of modulus of elasticity with compressive strength for the different cement–clay mixes considered.

Eqs. (3) and (4) indicate that the unconfined compression strength of the common cemented soils after a curing period of 7 days is only approximately 0.58 times that measured after 28 days of curing, while this ratio is 0.87 for soils containing MK. This finding demonstrates that MK can obviously enhance the strength of these materials at an early stage, which is similar to the finding of the application in concretes [38,34]. Noted that Horpibulsuk et al. [12] proposed a time-dependent strength prediction equation of cement stabilized high water content clays (Liquid limit index from 1.0 to 2.5) based on Abrams' law:

$$\frac{q_t}{q_{28}} = 0.038 + 0.281 \ln t \tag{5}$$

where q_t is the strength after t days of curing, t is the curing time (days). If the curing period t is preset as 7 days, the Eq. (5) is simplified as $q_7/q_{28} = 0.585$ which verifies authors' statistics (Eq. (4)) in this study.

The secant modulus (E_{50}), obtained from unconfined compression tests, is expressed as the ratio of the stress to strain when the axial stress is 50% of the unconfined compressive strength [3,33,29,9,23,20]. The relationship between the secant modulus and MK content is shown in Fig. 4. The modulus positively correlates with the MK content for a given cement content and curing

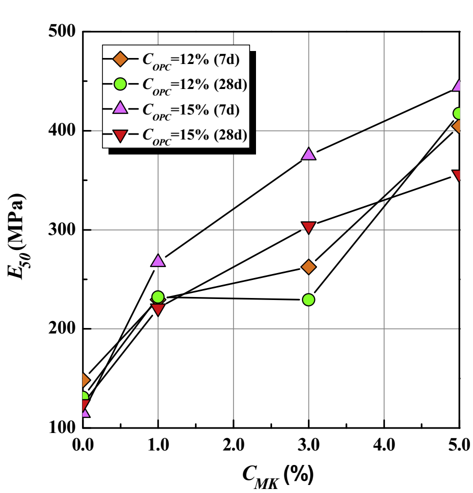

Fig. 4. Relationship between E_{50} (MPa) and MK contents (%).

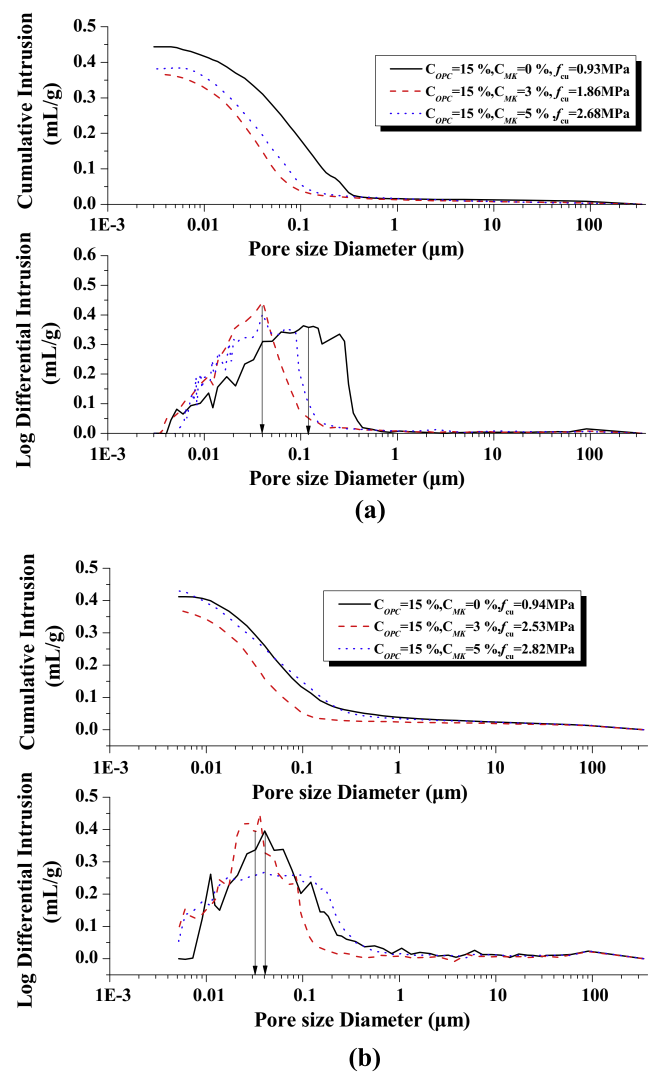

Fig. 6. Cumulative mercury intrusion and log differential intrusion vs. pore size diameter curve (a) after 7 days of curing, and (b) after 28 days of curing.

time, while the curve does not smooth when the MK content exceeds 3%, which is slightly different to that of the unconfined compression strength in Fig. 2.

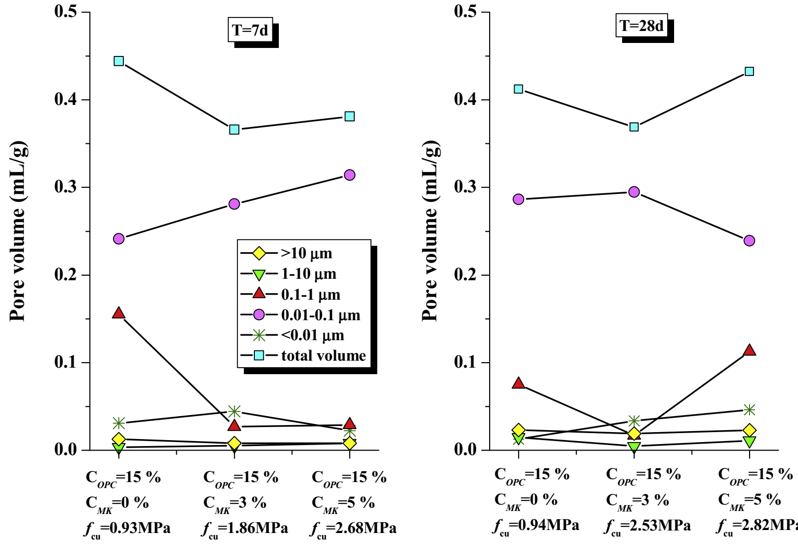

Fig. 7. Pore size distribution classified by an order of magnitude of different proportion of cement and MK.

Fig. 5 depicts the relationship between the secant modulus and unconfined compression strength of cement-treated clay containing MK for the ratios of the secant modulus to the unconfined compression ranging from 120 to 238. The ratio has also been discussed for common cemented soils. Lorenzo and Bergado [23] found that the ratio ranged from 147 to 175 for Bangkok clay mixed with cement contents ranging from 5% to 20%, and Zhang et al. [40] also reported that the ratio ranged from 150 to 275 for Lianyungang clay (the same clay examined in this study) for cement contents ranging from 10% to 20%. The data from the references and those obtained in this study are also gathered in Fig. 5. The results showed that the experimental relationship between the unconfined compression shearing strength and the secant modulus are close as almost all modulus values were approximately 120–238 times that of the unconfined compression strength.

3.2. Micro mechanism of cement-stabilised samples with MK

Fig. 6(a) and (b) shows the typical relationships between the mercury intrusion curves of samples containing 15% cement after 7 and 28 days of during the formation of cumulative intrusion volume (mL/g) and the log differential intrusion volume (mL/g). The cumulative pore volume decreased when the MK content varied from 0% to 5% and 3% irrespective of the curing time. The total pore

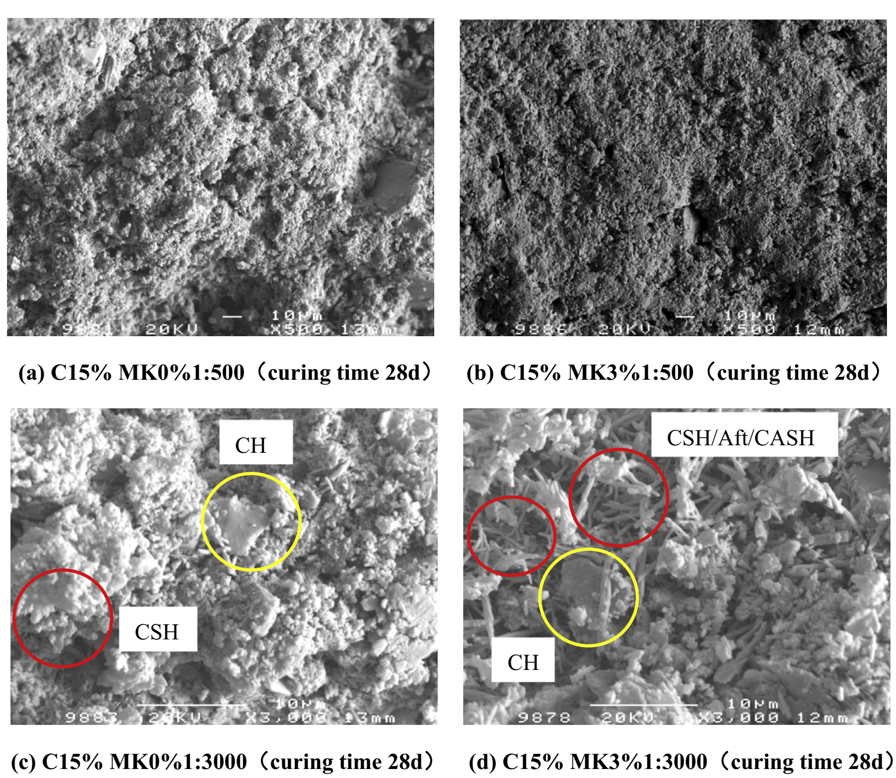

Fig. 8. SEM photos of MK-cement treated clay after 28 days of curing.

volume was minimised at a MK content of 3%, which is consistent with the zone analysis shown in Fig. 2, which is probably attributed to the reasonable MK proportion for secondary hydration. The effect of the MK content on the microstructure was further clarified in the log-differential intrusion volume analysis. The maximum pore diameter density at 0%, 3%, and 5% MK was 0.107 μm, 0.04 μm and 0.034 μm after 7 days of curing and 0.04 μm, 0.036 μm, and 0.04 μm after 28 days of curing, respectively. In addition, this density clearly changed when the pore distribution ranged from 0.01 μm to 1 μm. The micro-pore distribution was classified according to Horpibulsuk et al. [15] to further analyze the pore size distribution who counted the pore volume of pores that ranged among 0.01 μm, 0.1 μm, 1 μm, and 10 μm in diameter respectively. Fig. 7(a) and (b) shows the pore volume statistics calculated with the method proposed by Horpibulsuk et al. [17]. The addition of MK decreases the pore volume between 0.1 μm and 1 μm of cemented soils after 7 days' curing but those between 0.01 μm and 0.1 μm is on the contrary. The micro-volume between 0.01 μm and 0.1 μm after 28 days of curing is almost identical to that measured after 7 days of curing. Above all, volumes larger than 1 μm and less than 0.01 μm were almost identical irrespective of the curing period or MK content. Therefore, pore volume distribution between 0.01 μm and 1 μm affected the macro-strength but it is not the only main factor for cemented soils in this study. In other words, the cementation (or bonding) and fissures may constitute another key factor that affects the macro-strength.

The SEM results are shown in Fig. 8. Comparison of Fig. 8(a) and (b) at 500 times magnification preliminary showed that the frame structure of cemented soil containing MK was obviously denser. At 3000 times magnification, a large number of $Ca(OH)_2$ crystal (CH) and calcium silicate hydrate crystal (CSH) layers that enwrap the soil aggregate can be empirically observed. Fig. 8(c) and (d) shows that the reticulate and fibrous structure of calcium silicate hydrate crystals (CSH) and the hexagonal plate or prism hydrates of C_2ASH_8, C_4AH_{13}, and ettringite (Aft) interlace form the netted structure. In other words, the micro structure observation proves that the CH produced by cement hydration is consumed by MK to promote the secondary hydration and pozzolanic reaction, which generates the advantageous hydration products and reformed the microstructure. The new products and reformation of the microstructure effectively strengthen the bonding and fill the micro-pores of cement-stabilised soils, which improve the macro strength behaviour.

4. Conclusions

This paper proposed an effective agent, MK, to improve the behaviour of cemented soils in deep mixing projects and analysed the strength development and microstructure changes of cement-stabilised Lianyungang soft clay by using unconfined compression strength tests, mercury intrusion porosimetry and scanning electron microscopy. The following conclusions can be drawn from this study:

1. Metakaolin is a type of additive that effectively improves the strength of cemented soil. The unconfined compression strengths of cemented soil containing 3% and 5% MK were approximately 2.0–3.0 times that of soils lacking MK. The strength increased significantly when the MK content was less than 3%. This effect was attenuated at MK contents above 3%.
2. Comparing the unconfined compressive strength of cemented soils from Bangkok, Ariake and Shanghai without MK and that from Lianyungang containing MK cured for 7 or 28 days showed that the strength of materials containing MK after 7 days of curing was approximately 0.87 times that after 28 days of curing, while this ratio was only 0.58 for materials lacking MK. This finding shows that the addition of MK induces sufficient strength early during the curing process, which means that MK more feasibly generates early strength. Nevertheless, the relationship with MK between unconfined compression strength and secant modulus followed that without MK.
3. The addition of MK mainly changes the pore volume distribution between 0.01 μm and 1 μm, which demonstrates the filling effect and partly affects the macro-strength. The secondary hydration and pozzolanic reaction due to the addition of MK produces more CSH/Aft/CASH and strengthens the bonding and fissures, which constitutes another key factor.

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