Research Article

Strength and Pore Structure Development of High-Plasticity Clay Treated with MK-Blended Cement

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This paper focuses on the strength development and pore structure evolution of high-plasticity clay mixtures treated with metakaolin- (MK-) blended ordinary Portland cement (OPC). The unconfined compressive strength (f_{cu}) of treated soil mixtures is measured to study the effect of MK replacement. The microstructural study is carried out by mercury intrusion porosimetry (MIP), scanning electron microscopy (SEM), and X-ray diffraction (XRD). The results showed that the MK replacement led to the decrease of unconfined compressive strength of OPC-treated soil mixture cured for 28 days, but the influence on 7 days strength was negligible. However, the MK addition also enhanced the unconfined compressive strength significantly. The MK addition provided more cementitious products by secondary hydration and pozzolanic reaction, which would give rise to a notable filling effect by turning the large pores (1 to 10 μm) into smaller ones (0.1 to 1 μm). However, the MK replacement led to a decrease of cementitious products due to the lack of calcium hydroxide (CH) in soil mixture, and thus the effect on pore structure refinement was reduced. Nevertheless, the presence of MK enhanced the unconfined compressive strength cured for 7 days, due to the high reactivity of MK with CH.

1. Introduction

There was a general increase in engineering projects constructed under the soft ground in China. Deep mixing is an efficient technique for soft ground treatment. The soil was mixed in situ with the binder, and then the soft ground was transferred into composite ground by the treated soil-mixture columns [1, 2]. Cement is the most typical binder used in the deep mixing method. The cement content typically used in Japan and the USA is 20–30% and 10–50%, respectively [2]. When dealing with the soft ground, it often associates with a large amount of cement for strength purpose. Moreover, the production of cement is energy exhausted and generates vast CO_2 emission of approximately 900–935 kg CO_2/t in China [3]. Therefore, it is urgent to decrease the usage of cement for the global environmental

issue and increase the strength of cement-treated soil mixture for engineering needs.

Metakaolin (MK) is an aluminosilicate material, obtained by calcining kaolin clay at the temperature of 500–800°C [4, 5]. It contains high-reactivity silicon and alumina compounds, and the typical mass content of SiO_2 and Al_2O_3 is about 50–55% and 40–45%, respectively [6]. The MK has an acceleration effect on cement hydration and pozzolanic reaction [7, 8], which results in a significant increase of compressive strength by approximately 5–20% MK replacement (mass content), and therefore it is utilized for high-performance concretes, mortars, and cement pastes [5–11].

Recently, MK-blended cement has been utilized for soil stabilization [12–14]. The cement-treated soil mixture shows a significant enhancement on unconfined compressive

strength (f_{cu}) with MK addition due to the notable refinement on pore structure by the filler effect [13, 14], in particular for the compacted soil mixture. However, the MK replacement would result in the reduction of strength at a high clay-water to binder (w/b) ratio [12], which differs with the effect on concretes or cement pastes as mentioned before. Therefore, the strength performance and the pore structure evolution of the cement-treated soil mixtures with MK replacement were not well understood, in particular when treating the high-plasticity clay.

This paper focuses on the strength development and pore structure evolution of the cement-treated soil with MK replacement. The strength performance was conducted by unconfined strength tests (UCTs), and the microstructural investigations were performed by mercury intrusion porosimetry (MIP), scanning electronic microscope (SEM), and X-ray diffraction (XRD). In addition, the study in comparison with the cement-treated soil mixture with MK addition was performed.

2. Materials and Testing Methods

2.1. Materials. The untreated soil for the experiment was collected from a construction site located in Jiaxing city, in the east coastal region of China. The soil (JX clay) has the liquid limits (LL) of 54.1% and plastic limits (PL) of 26.0%, and it was classified as high-plasticity clay (CH) according to ASTM D2487 (2011). The physical properties of the soil are shown in Table 1. MK was bought from BASF German, with mean particle size <4 μm and specific surface area of about 10 m^2/g. The SiO$_2$ and Al$_2$O$_3$ mass contents were 52% and 40%, respectively. Note that, it was exactly the same material used in lianyungang clay (LYG clay) stabilization for comparison [14]. Ordinary Portland cement (OPC) type 42.5 N/R under the brand name of Hailou China was used. The oxides compositions of the MK and OPC are shown in Table 2.

2.2. Sample Preparation. As shown in Table 3, the water content of untreated soil was set to 65% (approximately 1.2 LL). The mass content of binder (OPC + MK) to wet soil was 18.2% (30% for binder to dry soil ratio), and the MK replacement was 0%, 10%, 20%, and 30%. The sample was designated as C*i*MK*j*, where *i* was the OPC mass content (%) and *j* was the MK mass content (%), respectively.

The soil was firstly oven dried at a temperature of 60°C for 24 h, then crushed, and sieved to pass a 2.0 mm sieve. The sample preparation started with mixing the dry soils, OPC, and MK powders in a mechanical mixer for 5 min to achieve a homogeneous powder mixture. Soon, water was added into the mixture and continued mixing for 5 min. After that, the mixtures were cast in PVC cylinder molds (46 mm in inner diameter; 100 mm in height) by hand-vibrating and then covered to minimize water evaporation and cured in the curing room at 20 ± 2°C and 95% humidity. After 48 h of curing, the samples were removed from the molds, transferred to sealed bags, and continued curing to 7 and 28 days.

2.3. Testing Methods. The unconfined compression tests (UCTs) were performed on the treated soils at 7 and 28 days of curing, and the unconfined compressive strength of the sample was designed as $f_{cu,curing\ age}$ (i.e., $f_{cu,7d}$ is the unconfined compressive strength of sample cured for 7 days). The UCT apparatus has the capacity of maximum loading to 50 kN. The axial strain rate was fixed at 1%/min, as per ASTM D4219 (2008). Before testing, thin layers of Vaseline were applied to the up and bottom of the sample. After testing, small pieces (about $1 \times 1 \times 1$ cm^3 cube) taken from the broken samples were frozen by liquid nitrogen (–195°C in boiling point) immediately and then dry-freezed for 24 h.

The pH measurement for the samples after 28 days curing was conducted as per ASTM D4972 (2013). The dry-freezed fragments of the samples were crushed to pass through a 2.0 mm sieve, and 10 g of the solid powders was added into 10 mL distilled water by a liquid-solid ratio of 1 : 1. The mixture was mixed to achieve homogeneity, and the pH values were measured using a pxsj-216f pH meter.

The MIP test was used to quantitatively evaluate the refinement of MK replacement on pore structures. It was conducted on the samples cured at 28 days with 0%, 10%, and 20% MK replacement, respectively. The pore diameter can be calculated using the capillary pressure equation based on Laplace's relation between pressure and corresponding pore diameter with a spherical air-water interface:

$$d = -\frac{4\tau \cos \alpha}{p},\tag{1}$$

where t is the mercury surface tension, α is the contact angle between mercury-air interface and soil particle, p is the pressure, and d is the pore diameter corresponding to the pressure applied. In this study, the MIP apparatus is AutoPore IV 9500 V1.09, with a pressure applied ranging from 10.5 kPa to 206.8 MPa. The contact angle was 130 degrees, and the mercury surface tension was 485 dynes/cm, respectively. Thus, the pore diameter estimated ranges from 0.006 μm to 119 μm.

SEM and XRD were used to investigate the micromorphology and identify hydration products of the broken sample from MK-blended cement (20% MK replacement) treated clay mixture. The SEM apparatus used was SU3500, Hitachi. It was conducted in low-vacuum mode. Tungsten filament electron source was used, and the accelerating voltage was 15 kV. The XRD analysis was performed with Cu-Kα (λ = 1.540562 Å) radiation on an X'TRA from ARL company, Switzerland. The instrument was operated at 40 kV and 40 mA. The scans covered the range 2–70° 2θ, with a nominal step size of 0.02° 2θ and time per step of 5 s. In addition, phase identification was carried out using the software program Jade 6 and the ICDD database, sets 1–54 (2004) combined with ICSD.

3. Results and Discussion

3.1. Unconfined Compressive Strength. Figure 1 shows the strength development of the cement-treated soils varies with the MK replacement. It can be seen that the $f_{cu,7d}$ of C18.2MK0 sample without MK is 1.05 MPa and it increases to

TABLE 1: Physical properties of soil for the experiment.

Natural water content (%)	Specific gravity	Liquid limits (%)	Plastic limits (%)	Particle size distribution (%)		
				Sand	Silt	Clay
51.3	2.7	54.1	26.0	3	44	53

Note. Sand (0.074 mm < $d \leq$ 2 mm); silt (0.005 mm < $d \leq$ 0.074 mm); clay ($d <$ 0.005 mm).

TABLE 2: Oxide composition of materials.

Oxide content (%)	SiO_2	Al_2O_3	CaO	Fe_2O_3	SO_3	MgO	Na_2O	K_2O	Loss on ignition
MK	52	40	1	2.5		0.8	0.5		
OPC	19	6.5	65	3.2	2.5	0.8	0.5	0.4	2.1

TABLE 3: Samples definition.

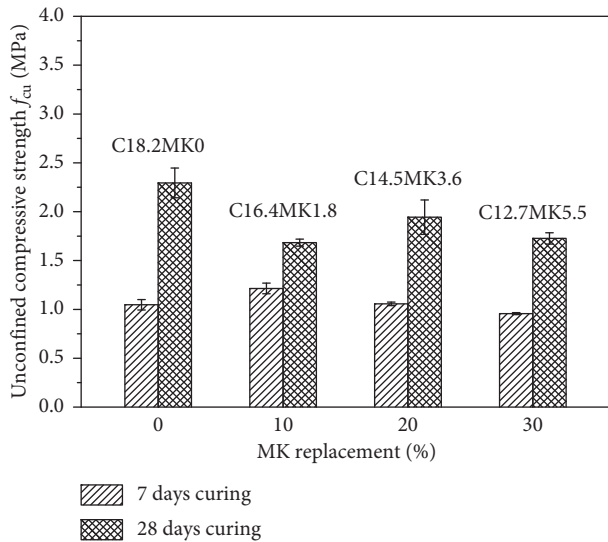| Label	MK replacement (%)	OPC (%)	MK (%)	Water content (%)
C18.2MK0	0	18.2	0	65
C16.4MK1.8	10	16.4	1.8	65
C14.5MK3.6	20	14.5	3.6	65
C12.7MK5.5	30	12.7	5.5	65

FIGURE 1: Unconfined compressive strength of the MK-blended cement-treated soil.

1.21 MPa with 10% MK replacement. After that, the strength decreases to 1.06 MPa and 0.96 MPa with 20% and 30% MK replacement, respectively. Therefore, the optimal MK replacement for early age (7 days) strength development is 10%. On the other hand, the sample without MK shows the highest strength of 2.29 MPa at 28 days curing, while it decreases to 1.68, 1.94, and 1.73 MPa with 10%, 20%, and 30% MK replacement, respectively. It can be seen that, the strength performance of MK-blended cement used in high-plasticity clay treatment differs, or opposite to that in concretes or cement pastes. For instance, the optimal MK replacement is 20% for concrete [8] and 10% for cement pastes [7], respectively. Note that, the w/b ratio used above is 0.45 and 0.30, respectively. Therefore, the variation in strength behavior may attribute to the high w/b ratio in treated high-plasticity clay mixtures (approximately 2.2) and the large amounts of fines fractions, i.e., soil particles.

Figure 2 shows the strength increment ratio of the high-plasticity clay treated with MK-blended cement. Note that the JX clay was treated in terms of MK replacement, whereas the LYG clay (water content of the untreated soil was set to 70%, approximately 1.2 LL) was treated in terms of MK addition [14]. It can be seen that the 10% MK replacement enhanced the $f_{cu,7d}$ by 16.0%, the 20% MK replacement kept the $f_{cu,7d}$ almost unchanged, while the 30% MK replacement caused a decrease of $f_{cu,7d}$ by 8.7%. Moreover, the MK replacement results in the decreasing of the $f_{cu,28d}$ of the cement-treated soils, though the C14.5MK3.6 sample with 20% MK replacement exhibits a second higher strength, which was still decreased by 15.3%. However, the strength development of treated LYG clay with MK addition is to the contrary. It shows that the strength increases with MK addition for both 7 and 28 days curing. For instance, the additive 6.7%, 20%, and 33.3% MK mass content resulted in the growing of the $f_{cu,28d}$ about 56.9%, 171.3%, and 201.6%, respectively [14].

In addition, it confirmed that the MK accelerates the hydration and enhances the compressive strength at early age [7]. The 7 days strength increment was higher than that of 28 days for both OPC-treated soil mixtures with MK replacement or MK addition.

3.2. Dry Density.
Figure 3 shows the dry density of the MK-blended cement-treated soil mixtures. The dry density of treated JX clay mixture was 1.254 g/cm³, and it decreased to 1.251, 1.242, and 1.213 g/cm³ with 10%, 20%, and 30% MK replacement. However, the dry density of treated LYG clay mixture was 1.649 g/cm³, it increased to 1.664 and 1.69 g/cm³ with 6.7% and 20% MK addition, and then it decreased to 1.669 g/cm³ with 33.3% MK addition [14]. On the whole, it can be seen that the higher dry density (or unit weight) of
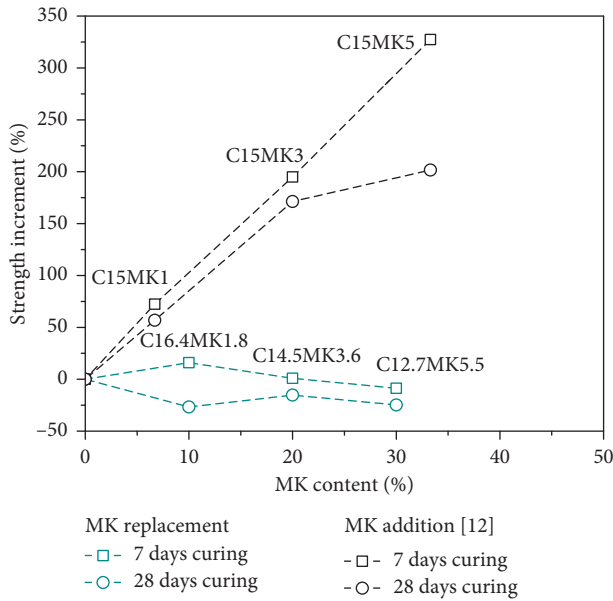
Figure 2: Strength increment ratio of the cement-treated soil with MK replacement and addition.
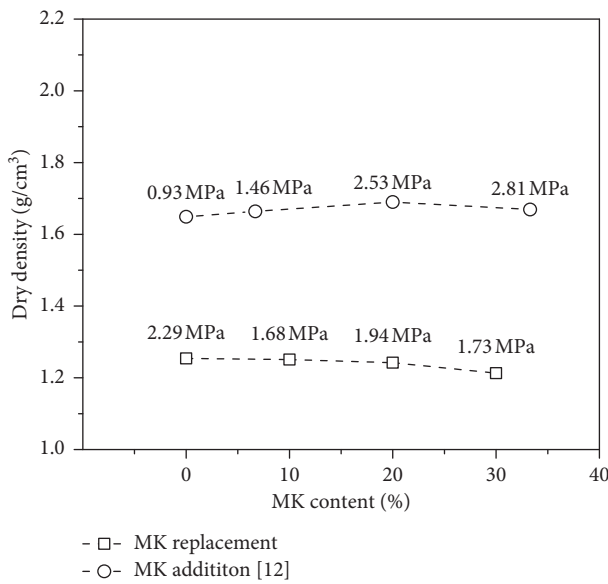

Figure 3: Dry density of MK-blended cement-treated soil mixtures cured for 28 days.

the pozzolan (e.g., MK, FA) blended cement-treated soil mixture, the higher compressive strength it gains [15]. While the differences in strength and dry density between treated JX clay and LYG-clay depend on the soil properties, clay-water to cement ratio, cement content, and MK content [2].

3.3. pH. Figure 4 shows the pH of cement-treated soil mixtures with MK replacement cured at 28 days. The pH of the soil mixture treated with pure OPC was 11.671, and it decreased to 11.511, 11.033, and 10.878 with 10%, 20%, and 30% MK replacement, respectively. Though the decreasing
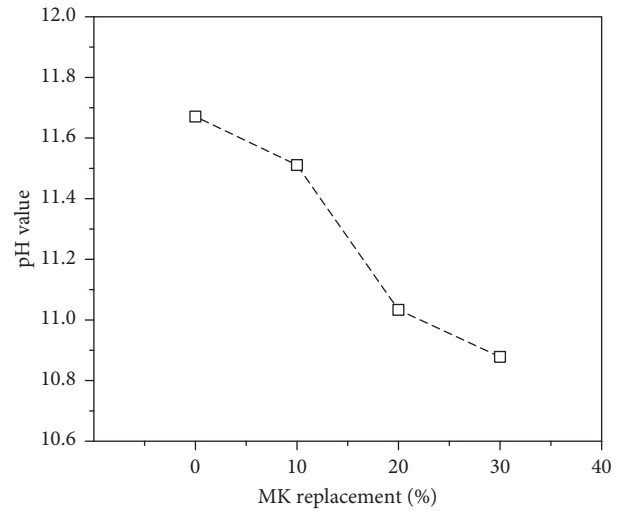

Figure 4: pH values of treated JX clay mixtures cured for 28 days.

in alkalinity of the pore solution slows the cement hydration to some extent [16], the cement hydration was eventually accelerated due to the high reactivity of MK with calcium hydroxide (CH) [8]. However, unlike the concrete or pastes, the strength of treated soil mixture additionally gains from the cementitious products by the reaction of reactive silica/alumina leaching from the soil particle with CH liberated from cement hydration [17]. Note that the pH at 10 to 11 is sufficient for the reactive SiO_2 and Al_2O_3 leaching into the pore solution [18, 19]. Therefore, the high early strength of cement-treated soil mixture was due to the quick removal of MK with CH, whereas the decrease of 28 days strength was due to the decreasing of pH value and CH amount.

3.4. Pore-Structure Evolution. As is shown in Figure 5, the pore volume of the cement-treated soil mixture without MK is 0.339 mL/g, and it was increased to 0.349 and 0.358 mL/g with the MK replacement of 10 and 20%, respectively. However, the cement-treated soil mixture with addition MK showed a tendency in decreasing the porosity within the threshold value [13, 14]. For instance, the pore volume decreases from 0.412 to 0.369 mL/g with MK addition increases from 0% to 20%, and then it decreases to 0.432 mL/g with 33.3% MK addition. Note that, the pore sizes were measured ranging from 0.006 to 119 μm for MK-blended cement-treated JX clay mixtures, while it ranged from 0.005 to 340 μm for LYG clay mixtures [14]. Therefore, the conflict of the smaller pore volume with smaller strength and dry density can be explained.

The pore volumes were classified into five groups (i.e., group 1 was $d > 10\,\mu$m, group 2 was $1\,\mu$m < d < $10\,\mu$m, group 3 was $0.1\,\mu$m < d < $1\,\mu$m, group 4 was $0.001\,\mu$m < d < $0.1\,\mu$m, and group 5 was $d < 0.1\,\mu$m) according to Horpibulsuk et al. [20], and the results are plotted in Figure 6. It can be seen that the dominant pore volume was group 3 and group 4 with the pore diameter size ranging from 0.01 to 1 μm, which is an agreement with the literature [14, 20, 21]. For instance,
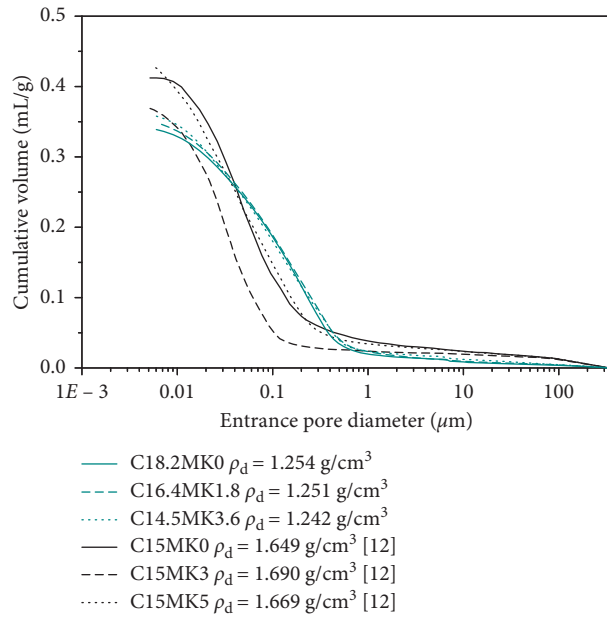
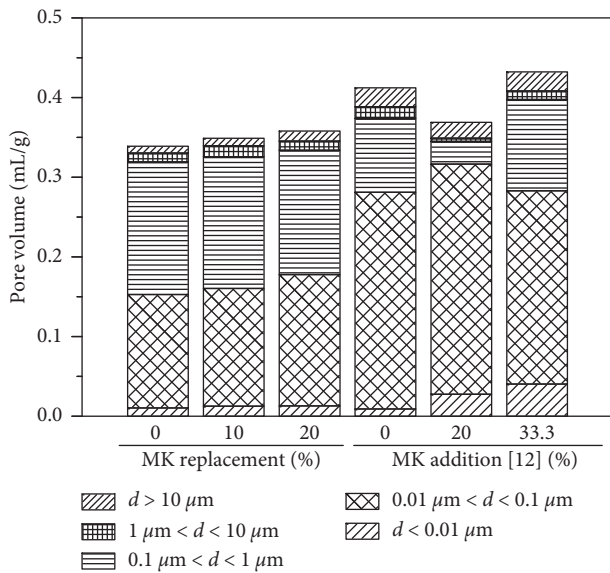FIGURE 5: Cumulative volume of the samples treated by MK-blended cement.

FIGURE 6: Pore volume of the samples treated by MK-blended cement.

the pore volume of the treated JX clay mixture ranging from 0.01 to 1 μm was about 91.2%, 89.7%, and 89.6% of the total volumes, with 0%, 10%, and 20% MK replacement, respectively. However, the effect of MK replacement on pore structure refinement was limited. It shows each group of pore volumes increases with the MK replacement, though a decrease was observed for pore volume ranging from 0.1 to 1 μm. For instance, the pore volume of the treated JX clay mixture without MK was approximately 0.009, 0.011, 0.167, 0.142, and 0.010 mL/g for group 1 to group 5, respectively. When the MK replacement increases to 10% and 20%, the cumulative pore volume increases by 3.0% and 5.6%, respectively, though the pore volume ranging from 0.1 to 1 μm decreased by 0.7% and 6.3%, respectively.

However, the effect of MK addition on pore structure refinement was significant [14]. The 20% MK addition resulted in the cumulative pore volume decreased by 10.5% and modified the porous structures by reducing the pores to a smaller size more efficiently. For instance, the pore volume ranging from 0.1 to 1 μm and from 1 to 10 μm decreased by 82.9% and 62.4%, respectively, whereas the smaller pore volume ranging from 0.01 to 0.1 μm and 0.005 to 0.01 μm increased by 103.0% and 204.4%, respectively.

Therefore, it can be concluded that the filling effect on pore structure refinement was limited for cement-treated high-plasticity clay with MK replacement. This could be attributed to the fact that the hydration products were insufficient to fill the pore structure and bond the soil particles.

3.5. SEM Analyses.

The SEM analyses are presented in Figure 7. The main hydration products were supposed to be the amorphous calcium silicate hydrate (CSH)/calcium aluminum silicate hydrate (CASH)/calcium aluminate hydrate (CAH) gels and ettringite (AFt) [13, 22–24]. It can be observed that the hydration products were similar to those formed in the cement-treated clay mixture [13, 23]. However, the amounts of these products were supposed to be less, in view of the lower strength and higher porosity mentioned in Sections 3.1 and 3.4.

3.6. XRD Analyses.

Figure 8 shows the XRD patterns of the untreated clay and MK-blended cement-treated clay mixture with 20% MK replacement after 28 days of curing. The major phases of the untreated clay were quartz, tosudite, and albite low, with the minor phases such as the halloysite, clinochlore, and muscovite. After treatment, the main minerals remained unchanged. Figure 9 shows the main hydration products of treated soil mixture were crystal ettringite (AFt)
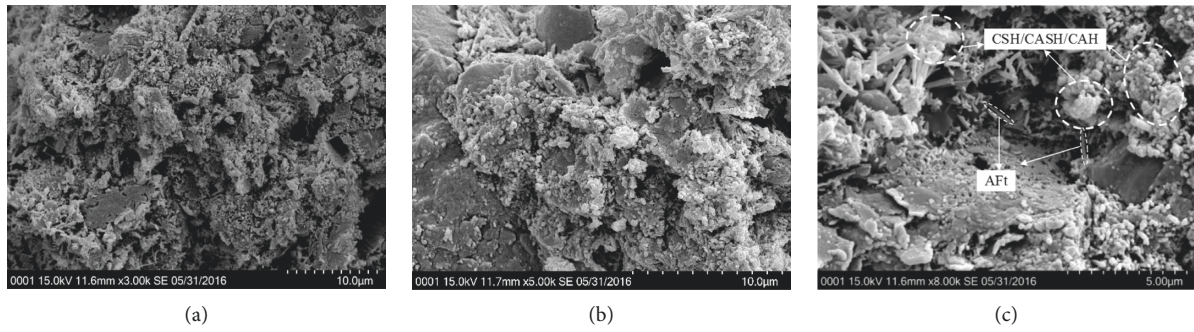
(a)

(b)

(c)

FIGURE 7: SEM photo of cement-treated JX clay mixture with 20% MK replacement. (a) 3000x; (b) 5000x; (c) 8000x.
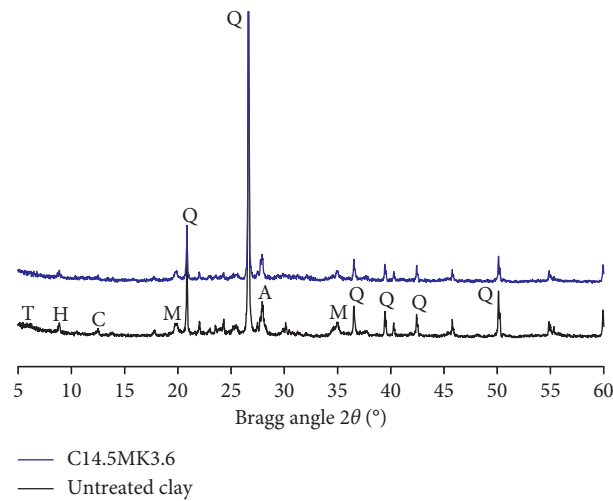

FIGURE 8: XRD patterns of cement-treated JX clay mixture with 20% MK replacement and untreated soil (Q, quartz PDF#86-2237; A, albite low PDF#72-1245; T, tosudite PDF#22-0956; H, halloysite PDF#29-1489; C, clinochlore PDF#83-1365; M, muscovite PDF#87-0691).
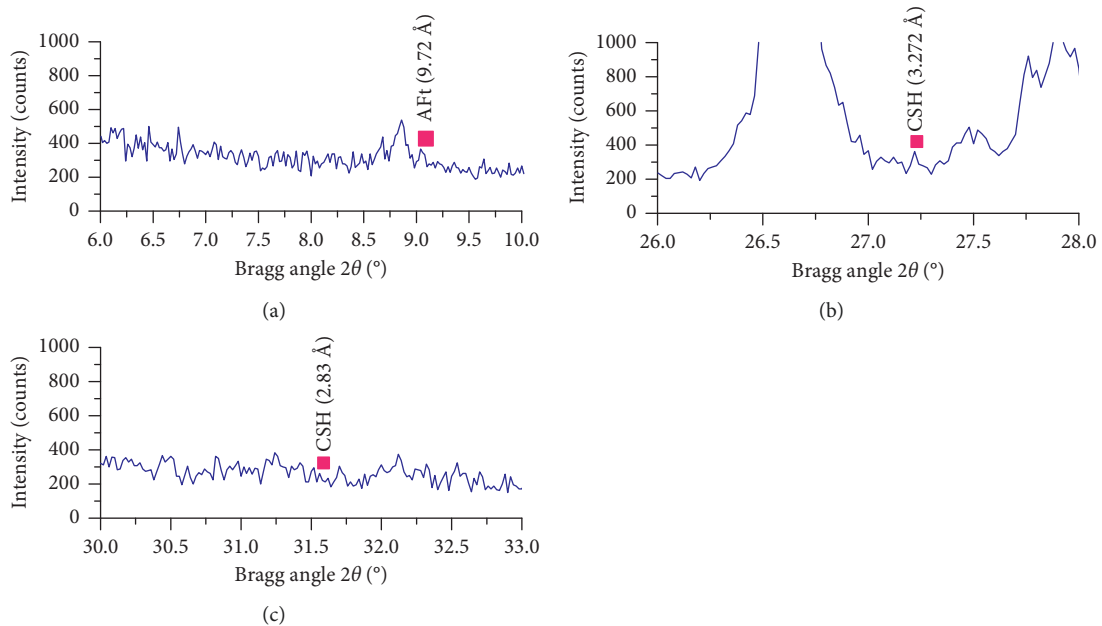

(a)

(b)

(c)

FIGURE 9: Hydration products identification (AFt PDF#41-1451/PDF#72-0646; CSH PDF#12-0475/PDF#29-0373). (a) Ettringite (AFt); (b) calcium silicate hydrate (CSH).

and calcium silicate hydrate (CSH) gel, which is an agreement with Du et al. [23]. However, the CH was not identified in treated high-plasticity clay mixture, attributing to the high clay-water to cement ratio and the quick removal of CH with MK [8]. Therefore, the amounts of CH were insufficient for further hydration or pozzolanic reaction, as well as the bonding and filling effects by cementitious gels [24, 25].

4. Conclusions

This paper investigates the strength performance and pore structure evolution of the high-plasticity clay treated by MK-blended cement. A comparison for the cement-treated soil with MK replacement and MK addition was performed. The unconfined compressive strength of the treated soil mixture was performed by UCTs, while the microstructure characteristics were performed by MIP, SEM, and XRD. Based on the results, the following conclusions can be drawn:

(1) The MK-blended cement can be used to treat high-plasticity clay. However, to enhance the unconfined compressive strength of the OPC-treated high-plasticity clay mixture, the MK must be used as addition. The 10%–30% MK replacement results in the unconfined compressive strength of OPC-treated JX clay mixture decreased by about 15.3% to 26.7% cured for 28 days. Nevertheless, the presence of MK enhanced the unconfined compressive strength cured for 7 days, due to the high-reactivity MK with CH.

(2) The filling effect on pore structure refinement of the OPC-treated JX clay mixture was reduced with MK replacement. The MK replacement results in an increase of the cumulative pore volume and pore structure slightly changes by decreasing the pore volume ranging from 0.1 to 1 μm. The MK addition results in a notable decrease of pore volume ranging from 0.1 to 10 μm simultaneously with an increase of smaller pore volume ranging from 0.005 to 0.1 μm.

(3) The MK replacement decreases the CH amount in cement-treated JX clay mixture and its pH value at 28 days curing. Though the MK accelerates the hydration and pozzolan reaction which provides high early strength for cement-treated soil mixtures, eventually the 28 days strength decreases due to the insufficient cementitious products to fill the pore structure and bond the soil particles.

Data Availability

The data used to support the findings of this study are available from the first author (e-mail: 230149190@seu.edu.cn) upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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