Strength and Micro-Structure Evolution of Compacted Soils Modified by Admixtures of Cement and Metakaolin

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Abstract

Metakaolin, widely applied in concretes and cement-stabilized soft clays to improve their macro performance, was evaluated for use in compacted soils in highway and backfill engineering. The compactability and strength performance of cement-modified soils with metakaolin were evaluated in the terms of their applicability and effectiveness. The cement-based modified soil with metakaolin was more insensitive to water and more convenient in the field rolling compaction. The strengths, in addition to the unconfined compressive strength and splitting tension strength, were improved significantly. Up to a threshold ratio of metakaolin to cement ranging from 1/3 to 1/2 in this case, the strengths increased gradually, while they unexpectedly decreased thereafter. This phenomenon was different from the previously reported metakaolin applications in concretes and cement-stabilized soft clays, which was probably due to the water content and the ratio of metakaolin to hydrated calcium hydroxides of the cements. Microstructure analysis by X-ray diffraction, scanning electronic microscopy, thermo-gravimetric analysis and mercury intrusion porosimetry, demonstrated that the addition of metakaolin led to a higher quantity of hydration products and a denser micro-porosity distribution.

Keywords: Metakaolin; Cement-based modified soil; Compactability; Strength performance; Microstructure.

1. Introduction

Metakaolin (MK) is an amorphous aluminosilicate compound produced by the calcinations of pure kaolinite at a temperature of 500°C to 550°C. During the calcinations process, the chemical bound water in the interstices of kaolinite is driven off, and the crystalline structure is destroyed. Compared with industrial by-product materials, such as fly ash, rice husk ash and blast-furnace slag, commercial MK usually has higher purity, finer particle size and larger specific surface, leading to higher activity. Previous researchers reported that highly reactive MK was widely used as an effective mineral additive in the formulation of the high-performance concrete and cement paste. With the addition of MK, the unconfined compressive strength (UCS) and the splitting tensile strength (STS) of concrete increased significantly, while the free shrinkage strain, shrinkage and cracking were suppressed (Qian and Li, 2001; Ding and Li, 2002; Li and Ding, 2003; Batis *et al.*, 2005; Khatib and Hibbert, 2005; Poon *et al.*, 2006; Gleize *et al.*, 2007; Justice and Kurtis, 2007; Kim *et al.*, 2007; Gesoğlu and Mermerdas, 2008). Additionally, the resistances of concretes with MK to chloride ion penetration and diffusion, acid attack, and carbonation, as well as damage prevention under freezing and thawing conditions, were also obviously improved (Palomoa *et al.*, 1999; Boddy *et al.*, 2001; Gruber *et al.*, 2001; Courard *et al.*, 2003; Batis *et al.*, 2005; Akhras *et al.*, 2006; Poon *et al.*, 2006; Kim *et al.*, 2007). Recently, MK was also evaluated as an additive for the cement-based stabilized soft clays, and the results showed that the UCS increased two- to three times, the hydraulic conductivity was suppressed 10- to 100 times for the filling effect, and the hydration

1 and pozzolanic reaction with calcium hydroxide (CH) accelerated (Kolovos *et al.*,
2 2013; Zhang *et al.*, 2014; Deng *et al.*, 2015).

3 Because of the above mentioned good applicability for concrete, paste, and
4 stabilized soils, the MK ability to improve the performance of compacted soils
5 modified by cement became an interesting and attractive topic, where compactability
6 and mechanical performance were focused on highway and backfill engineering.

7 Compaction and strength tests were conducted to evaluate the use of MK in the
8 modification of backfill and embankment materials. Proctor compaction tests were
9 first conducted to evaluate the compactability of the modified soils. Thereafter,
10 unconfined compression tests (UCT) and splitting tension tests (STT) were performed
11 to investigate the strength performance at the maximum dry density. Finally, X-ray
12 diffraction (XRD), scanning electronic microscopy (SEM), thermo-gravimetric
13 analysis (TGA) and mercury intrusion porosimetry (MIP) were used to study the
14 internal micro mechanism.

15 **2. Materials and Methods**

16 **2.1 Materials**

17 The soils were obtained from a typical site that utilized the hydraulic filling
18 method at Lianyungang City in eastern China. Note that the original sand and clay
19 layers were both involved during cutter-suction dredging, hence, soils with both sand
20 and clay were widely formed in this site. The characterization of the studied soil is
21 shown in **Table 1**. The soil has 33% sand, 46.5% silt and 20.5% clay, and the liquid
22 limit (*LL*), plastic limit (*PL*) and plasticity index (*PI*) are 55.1%, 38.1% and 17.0,

1 respectively. This soil is both categorized as MHS according to the Unified Soil
2 Classification System (ASTM D2487-11) and (JTG E40-2007, China). The soil needs
3 to be improved when used as a material for highway and backfilling engineering (JTJ
4 034-278, Chinese standard).

5 Ordinary Portland cement (OPC 42.5 R/N) and metakaolin produced in China
6 and German, respectively, were adopted for this study. The chemical oxides of the
7 Portland cement and MK agent are listed in **Table 2**. Note that CaO and SiO₂ are the
8 two main oxides of OPC, and their total content was 84%; the ratio of CaO to SiO₂
9 exceeded 2.0, and the content of MgO was less than 2.0%, both of which meet the
10 specification requirements of European Cement Standard (EN197-1). The
11 predominant oxides of MK are SiO₂ and Al₂O₃, and their total content is
12 approximately 92%. In addition, the average particle size of MK is less than 2 μm,
13 and the specific surface area is approximately 10 m²/g.

14 **2.2 Compaction test**

15 The modified proctor compaction test with compaction energy of 2687 kJ/m³
16 (ASTM D1557-12) was adopted to evaluate the compactability and to determine the
17 maximum dry density (MDD) and the optimum moisture content (OMC) of
18 cement-based modified soils. During the compaction tests, the soils, cement (mass
19 ratio of cement to dry soil at 6% and 8%) and MK agent (mass ratio of MK to dry soil
20 at 0%, 2%, 4% and 6%) were first thoroughly mixed at the desired water contents;
21 later, these mixtures were divided into five parts and successively compacted into a
22 metal cylinder. The wet/dry density and water content were calculated, and the

compaction curve, OMC and MDD were obtained. Note that the common OPC content (6% and 8%) of the filling materials in highway engineering was selected, and the MK content (2%, 4% and 6%) was preset aiming to investigate the MK effect on compactability and strength performance.

2.3 Cylindrical sample preparation and curing method

To prepare the testing specimens (50 mm in diameter and 50 mm in height) for the unconfined compression tests (UCT) and the splitting tension tests (STT), distilled water was continuously supplied to the premixed soil-cement-MK powders until the water content was equal to the OMC. Thereafter, the mixtures were sufficiently agitated to maintain a uniformed water distribution and were placed into a cylindrical metal mould that was pre-lubricated to avoid damage in the removal procedure (Chen *et al.*, 2009). Finally, the samples were statically compacted to the maximum dry density. Note that three parallel samples were prepared to improve the reliability.

The weight and dimensions of the extracted samples were measured with a precision of 0.01 g and 0.1 mm, respectively. Additionally, the deviation (dry density, moisture content, diameter and height) among three parallel samples was controlled as below: within $\pm 0.5\%$ of MDD, $\pm 0.5\%$ of OMC, ± 0.5 mm of the diameter and ± 1 mm of the height. These qualified specimens were sealed in plastic bags to avoid variation of the water content and later cured at $20 \pm 2^\circ\text{C}$ and 95% relative humidity for 6, 13 and 27 days. Thereafter, all specimens were saturated in a water tank for 24 hours to minimize suction (JTJ 034-278, Chinese standard; Consoli *et al.*, 2011), arriving at total curing periods of 7, 14 and 28 days.

2.4 Unconfined compression test and splitting tension test

UCTs were first conducted to evaluate the strength of the compacted soils. Upon arriving at the preset curing periods, these samples (superficially dried with an absorbent textile) were placed on an automatic loading machine with maximum capacity of 50 kN, and compressed at a rate of 1 mm/min to obtain the unconfined compression strength (UCS). STT was performed to obtain the splitting tension strength (STS) according to the Brazilian method (NBR 7222, Brazilian Standard Association, 1983). Note that during data processing and analysis, the deviation of the UCS and STS was within 10% of the mean value.

2.5 Hydration product and microporosity test

To investigate the micro mechanism of the MK effects on the strength of the compacted cement-based modified soils, XRD, SEM and TGA methods were adopted to identify the hydration products, while the MIP test was applied to clarify the microporosity distribution. Note that for the complex system of soil-hydration product admixtures, the hydration product was first judged by XRD, and then, SEM and TGA were employed to further confirm the findings. In addition, to differentiate the hydration and clay minerals, the XRD of untreated soils was also performed. In this research, specimens with 6% cement and different MK contents at a 7-day curing period were analysed by the micro mechanism.

The XRD analysis was carried out using a diffractometer equipped with a copper anticathode. Lyophilized samples were first milled into fine powders, and then scanned with a 2θ value ranging from 10° to 60° . The testing data were

1 semi-qualitatively analysed using JADE 5.0 software. Note that the lyophilization
2 utilized the following procedure: immersion into liquid nitrogen (-196°C) for instant
3 freezing and transfer into a vacuum chamber for sublimation for approximately 24
4 hours (Penumadu and Dean, 2000).

5 To perform the SEM test, cube specimens remote from the shearing surface were
6 cut from the cylindrical samples and lyophilized according to the above method. The
7 lyophilized samples was coated with a gold layer with a thickness of 200 to 300 Å (1
8 Å=0.1 nm) to provide electrical conductivity and prevent an electric charge from
9 accumulating on the surface (Al-Rawas and McGown, 1999).

10 To perform the TGA analysis, lyophilized samples were first milled into a fine
11 powder and later placed in a ceramic crucible and heated from room temperature
12 (approximately 20°C) to 900°C at a constant heating rate of 10°C/min in a dynamic
13 nitrogen atmosphere (Chen *et al.*, 2011; Argane *et al.*, 2015).

14 MIP is a method to determine the pore size distribution of porous materials based
15 on the unique relationship between the intrusion pressure and equivalent pore
16 diameter (Washburn, 1921). The calculation expression is shown below:

$$D = -\frac{4\gamma \cos\theta}{p} \quad (1)$$

18 Where D is the pore diameter, γ is the surface tension of mercury, θ is the contact
19 angle, and p is the applied pressure. In this study, γ of 0.48 N/m and θ of 140° were
20 recommended based on previous research (Diamond, 1970; Penumadu and Dean,
21 2000). Note that the pressure p applied in this study ranged from 3.7 kPa to 241.1
22 MPa, resulting in the range of pore diameters of 0.005 to 340 μ m.

3. Results and discussions

3.1 Compactability of cement-modified soils

Fig. 1 presents the compaction curves of the cement-based treated soils. It is interesting that the addition of MK smoothes the compaction curve at a given cement content (6% or 8%), indicating that MK can suppress water sensitivity and resulting in more convenience for field compaction.

The changes in OMC and MDD are shown in **Fig. 2**. When the soil is only modified by the 6% and 8% cement content, the OMC slightly increases from 14.2% to 14.7%, which is consistent with the previous studies (Rahman, 1987; Basha *et al.*, 2005; Hossain *et al.*, 2007). The OMC increases with increasing cement content because more water is required for cement hydration and because additional water is held in the flocculent structure between the cement and soil (Basha *et al.*, 2005; Hossain *et al.*, 2007). It should be mentioned that that if soils were treated by other materials, such as volcanic ash, rice husk ash, lime, fly ash and cement kiln dust, the OMC also increases with increasing binder content due to similar mechanisms (Miller and Azad, 2000; Lim *et al.*, 2002; Koliass *et al.*, 2005; Guney *et al.*, 2007; Kavak and Akayarli, 2007; Kumar *et al.*, 2007).

Fig. 2 shows that the MK enhanced the OMC of cement-modified soils. For instance, the OMC values with the 6% cement content were 14.2%, 14.8%, 15.6%, and 16.5% when the MK content was 0%, 2%, 4% and 6%, respectively. This finding can probably be attributed to the fact that active metakaolin rapidly reacts with the calcium hydroxide (CH), accelerating the hydration work of cement within the first 24

h (Wild *et al.*, 1996).

The MDD of the soils modified only by cement increased slightly from 1.87 to 1.89 g/cm³ when the cement content increased from 6% to 8%, agreeing with previous studies (Rahman, 1987; Singh *et al.*, 2008; Horpibulsuk *et al.*, 2006 and 2010). On the contrary, in the case of 6% cement content, when MK was added from 0% to 6%, the MDD decreased from approximately 1.883 to 1.78 g/cm³.

3.2 Strength performance

The effects on the UCS are shown in **Fig. 3**. When the 6% and 8% cement was solely used as the modifying material, the UCS values at the 7-day curing were 1.53 MPa and 2.28 MPa, respectively, both of which meet the requirements of subbase for Second/Third Class Road (1.5-2.0 MPa) and for First Class Road/highway (1.5-2.5 MPa) (JTJ 034-278, Chinese standard). When the composite with 6% cement and 2% MK was applied, the UCS at the 7-day curing periods was 3.0 MPa, indicating a higher strength performance and with less usage of modifying materials. In addition, at the 14-day and 28-day curing periods, the UCS of the soil treated with 6% cement and 2% MK also surpassed that modified with only cement at 6% and 8% content.

It is interesting that the UCS was not proportional to the MK content and that a threshold value was observed. When the MK content was less than this threshold, the UCS increased with the MK content; while above the threshold, the strengths decreased. For example, the UCS with 6% cement content at the 14-day curing period increased significantly from 2.37 to 4.12 MPa when MK content increased from 0% to 2% and later decreased from 4.12 to 2.21 MPa when MK content increased from 2%

1 to 6%. **Table 3** illustrates the UCS growth efficiency, defined as the ratio of UCS with
2 MK to that without. This table confirms that the MK threshold contents were 2% and
3 4% when the cement contents were 6% and 8%, respectively.

4 The effects on STS are shown in **Fig. 4**, and its growth efficiency is supplied in
5 **Table 3**. It is sure that the STS tendency was absolutely the same as that of UCS
6 because the linear relationship between STS and UCS (**in Fig. 5**) exists, and the ratio
7 between them was 0.08. The ratio is almost same as that reported in previous
8 investigations (Porbaha *et al.*, 2000; Ismail and Ryden, 2014).

9 After detailed evaluation of the changes in the UCS and STS data, the threshold
10 ratios between MK and cement content ranged from 1/3 to 1/2 for the compacted soils
11 in this case. Additionally, the optimal mass ratio in view of the strength performance
12 and economy varied from 1/5 to 1/4 for cement-based stabilized soft clays and from
13 1/10 to 1/5 for concrete (Qian and Li, 2001; Ding and Li, 2002; Kim *et al.*, 2007;
14 Gesoğlu and Mermerdas, 2008). Hence, the influence of MK on the strength of
15 compacted soils was different from that of concrete and cement-based stabilized soft
16 clay, which was probably closely related to the free water and ratio of MK to CH
17 hydrated by the cement. Murat (1983) did important works on unconfined
18 compressive strength of pastes with MK/CH ratios after 7- and 28-day curing, as
19 shown in **Fig. 6**. There is a maximum strength value in the data followed by a decline
20 with an incremental increase of the MK/CH ratio because excessive MK addition may
21 cause a reaction with the hydration products, resulting in a change from C_4AH_{13} to
22 C_2ASH_8 . Note that the MK/CH values at the peak strengths after the 7- and 28-day

curing were approximately 4.0 and 2.5, respectively. It could be estimated that the ratio of MK to cement at the peak strength is located in the range of 0.65 to 0.40 because the mass of the hydrated CH gel is generally approximately 15%-20% of the hydration products under fully hydration conditions. However, the cement was difficult to hydrate fully in the case of the compacted soils because of less available water; hence, the threshold value of MK/cement (1/3-1/2) could be considered in agreement with the results by Murat (1983). Additionally, because the water existed fewer and fewer from the concrete, soft clay to the compacted soils, the ratios of MK to cement were growing, which was consistent with the former finding.

3.3 Micro mechanism

The XRD, SEM, TGA and MIP tests were performed to investigate the micro-mechanism of the MK effect on compacted soils. Among them, XRD, SEM and TGA methods were used to analyse the hydration products, whereas the MIP method was applied to depict the porosity distribution.

3.3.1 XRD, SEM and TGA analysis

XRD results (in **Fig. 7**) indicate that the hydration products are composed of portlandite (CH), Ettringite (Aft/Afm), Calcium silicate hydrates (CSH) and Calcium silicate aluminates hydrations (CASH). Note that the referred 2θ values of the above hydration products are listed in **Table 4**. By comparison, it was sure that the intensity of the hydration products after the MK addition changed despite being the same types of hydration products. Note that CASH was obviously located at 23 and 36 degrees in the 2θ values for the modified soils with MK, which was probably owing to the main

1 mineral composition (Al_2O_3) of MK reacting with CH and SiO_2 , and producing
2 additional CASH.

3 **Fig. 8** provides the SEM photos of the samples with and without MK. After
4 detailed examination of **Fig. 8a, c** and **e**, it is obvious that the microstructure of the
5 samples modified by 6% cement and 2% MK is the most homogeneous and dense. In
6 **Fig. 8b, d** and **f**, the hydration products, including CSH, CASH and Ettringite
7 (Aft/Afm), were obviously observed, which was consistent with the XRD results.
8 These generated hydration products covered the surface of the soil particles,
9 strengthening the bonding, while also filling the pores of the compacted soils.

10 In the traditional TGA analysis of the cement-based hydration products, it is
11 often considered that the first mass loss (located at approximately 100°C) is caused by
12 dehydration reactions of several hydrates (e.g., CSH, CAH, and Ettringite)
13 (Alarcon-Ruiz *et al.*, 2005; Lemonis *et al.*, 2015); the second mass loss at 250°C by
14 the dehydroxylation of the clay minerals (Kolias *et al.*, 2005); and the third main mass
15 loss at $450\text{--}500^\circ\text{C}$ by the dehydroxylation of portlandite (CH) (Alarcon-Ruiz *et al.*,
16 2005; Chen *et al.*, 2011; Argane *et al.*, 2015). The TGA results of this study are
17 presented in **Fig. 9**. Among these results, **Fig. 9a** shows that the mass loss of the soil
18 treated only by cement was 6.9%, while this value increased to 7.8% and 7.3% with 2%
19 and 6% MK content, respectively. This change was owing to the increased generation
20 of hydration products, leading to greater mass loss at the high-temperature
21 calcinations. It needs to be mentioned that the increased generation of hydration
22 products can also be indicated in the DTG curve (**Fig. 9b**). For example, the

1 relatively higher and wider peaks of several hydrates (e.g., CSH, CAH, and Ettringite)
2 for modified soils with the MK agent were observed and attributed to the increased
3 hydration of the cement accomplished by the MK and the increased reaction between
4 MK and hydrated CH.

5 After the combined analysis and comparison of **Fig. 9a** and **b**, it can be found
6 that the mass loss (7.8%) with 2% MK was more than that (7.3%) with 6% MK;
7 meanwhile, the peak of several hydrates (CSH, CAH, and Ettringite) was also higher
8 and wider, probably indicating more hydration products (CSH and Ettringite).

9 3.3.2 Pore size distribution

10 **Error! Reference source not found.****Fig. 10a** and **b** show the pore size distributions
11 that were determined by the MIP test. It clearly shows that the cumulative intrusion
12 without MK is 0.132 mL/g, but this value decreases to 0.119 and 0.126 mL/g with 2%
13 and 6% MK, respectively, indicating that the structure with 2% MK is the densest,
14 and this finding is consistent with the SEM analysis.

15 In the differential volume curve (**Fig. 10b**), there existed a tendency of main pores
16 (from 0.01 μm to 0.1 μm) without MK, with 2 and 6% MK, moved from larger to
17 less successively. To further quantitatively investigate this change, the pore
18 distribution was first classified as five groups ($< 0.01 \mu\text{m}$, $0.01 \mu\text{m} \sim 0.1 \mu\text{m}$, 0.1
19 $\mu\text{m} \sim 1 \mu\text{m}$, $1 \mu\text{m} \sim 10 \mu\text{m}$, $> 10 \mu\text{m}$) after Horpibulsuk *et al.* (2006), and the volume
20 of each group is depicted in **Fig. 10c**. It shows that for the soil treated only with
21 cement, the volumes of the above five groups were 0.01, 0.067 0.032, 0.0088 and
22 0.015 mL/g, respectively. Among the groups, the porosity was the most concentrated

1 in pore zone ranging from 0.01 to 1 μm . These volumes were redistributed with MK
2 incorporation, and the larger pores were suppressed and migrated to the smaller sizes.
3 When the MK content was 2%, the pores ranging from 0.01 μm ~0.1 μm was the
4 highest, while those in other groups were lower. The MIP results totally confirmed
5 that the MK incorporation suppressed the large pores; furthermore, the large pore was
6 the least at the optimal MK content.

7 **4. Conclusions**

8 Metakaolin, a fine powder material widely used in the fields of concrete
9 construction and ground improvement, is introduced to the field of compaction filling
10 of cement-based treated soils. The compactability and strength performance of
11 cement-stabilized sandy soil with MK were first assessed by the compaction tests,
12 unconfined compression tests and splitting tension tests, respectively. Thereafter,
13 XRD, SEM, TGA and MIP were also applied to further analyse the changes in the
14 microstructure and the internal mechanism. The main results are listed as follows:

15 (1) With the MK addition, the cement-modified soil shows water insensitivity,
16 resulting in the greater convenience for rolling compaction.

17 (2) The mechanical performance of cement-based modified soil with MK,
18 including UCS and STS, has also been improved significantly. UCS and STS are not
19 proportional to the mix proportion of MK at a constant cement content, and there
20 exists the threshold value where the strength arrives at the maximum. In this case, the
21 threshold ratio of the MK to cement lies in the range of 1/3 to 1/2.

22 (3) Microscopic results show that the main products are CSH, CASH and

Aft/Afm. Although the addition of MK obviously cannot change the types of hydration products, it can upgrade the amounts, improving the density of the microstructure and cementation bonding.

Acknowledgements

The first author is grateful to the National Science Foundation of China (Grant No. 51378117, 41572280, 41372281 and 41330641) and the six talent peaks project in Jiangsu Province, P. R. China (Grant No. 2014-XCL-003) for their supports.

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1 **Table 1 Physical Properties of Soil Samples**

Properties	Values
Liquid limit $LL(\%)$	55.1
Plastic limit $PL(\%)$	38.1
Plasticity index PI	17.0
Medium sand content ($0.425\text{ mm} < \text{diameter} < 2\text{ mm}$)	6.4
Fine sand content ($0.075\text{ mm} < \text{diameter} < 0.425\text{ mm}$)	26.6
Silt content ($0.002\text{ mm} < \text{diameter} < 0.075\text{ mm}$)	46.5
Clay fraction ($\text{diameter} < 0.002\text{ mm}$)	20.5
Soil classification—ASTM D2487-11	Elastic silt sand (MHS)
Soil classification—JTG E40 (China)	MHS

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1 **Table 2 Oxides compositions of ordinary Portland cement and metakaolin**

Oxides content (%)	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	SO ₃	MgO	Na ₂ O	K ₂ O	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		

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Table 3 Strength growth ratio of UCS and STS

Strength type	Cement content (%)	Curing times (d)	MK content (%)			
			0	2	4	6
UCS	6	7	1	1.95	1.39	1.26
		14	1	1.74	1.19	0.93
		28	1	1.43	0.96	0.79
	8	7	1	1.11	1.39	1.02
		14	1	1.19	1.68	1.37
		28	1	1.09	1.33	1.11
STS	6	7	1	1.7	1.2	1.07
		14	1	1.6	1.15	0.95
		28	1	1.52	1.2	1
	8	7	1	1.21	1.49	1.1
		14	1	1.28	1.61	1
		28	1	1.08	1.32	0.94

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Table 4 Typical values of 2θ

hydration products	CH	Aft/Afm	CSH	CASH
2θ values	17.5	16; 42	29; 32	22; 23; 36
	Li and Ding, (2003); Kolias <i>et al.</i> , (2005); Kim <i>et al.</i> , (2007); Yi <i>et al.</i> , (2015)	Kim <i>et al.</i> , (2007) Shen <i>et al.</i> , (2015) Yi <i>et al.</i> , (2015)	Kamruzzaman <i>et al.</i> , (2006) Buchwald <i>et al.</i> , (2007) Kim <i>et al.</i> , (2007) Yi <i>et al.</i> , (2015)	Kim <i>et al.</i> , (2007) Mazouzi et al., (2014)

Fig. 1 Compaction curves

Fig. 2 Relationship between MDD, OMC and MK content

Fig. 3 Relationship between UCS and MK content

Fig. 4 Relationship between STS and MK content

Fig. 5 Relationship between UCS and STS

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Fig. 6 Relationship between MK/CH ratio and UCS (After Murat, 1983)

Fig. 7 XRD diffraction grams

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Fig. 8 SEM photos after 7-day curing

(a) 6%C, 1: 500; (b) 6%C, 1: 5000; (c) 6%C 2%MK, 1: 500; (d) 6%C 2%MK, 1: 10000; (e) 6%C 6% MK, 1: 500; (f) 6%C 6%MK, 1: 10000.

Fig. 9 TGA curves after 7-day curing

Fig. 10 MIP results

(a) Cumulative volume vs. pore size diameter (μm); (b) Log Differential volume vs. pore size diameter (μm); (c) Pore volume vs. Ranges of pore size