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Research paper

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 concrete 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.

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; Al-Akhras, 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 and pozzolanic reaction with calcium hydroxide (CH) accelerated (Kolovos et al., 2013; Zhang et al., 2014; Deng et al., 2015).

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

Compaction and strength tests were conducted to evaluate the use of MK in the modification of backfill and embankment materials. Proctor compaction tests were first conducted to evaluate the compactability of the modified soils. Thereafter, unconfined compression tests (UCT) and splitting tension tests (STT) were performed to investigate the strength performance at the maximum dry density. Finally, X-ray diffraction

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(XRD), scanning electronic microscopy (SEM), thermo-gravimetric analysis (TGA) and mercury intrusion porosimetry (MIP) were used to study the internal micro mechanism.

2. Materials and methods

2.1. Materials

The soils were obtained from a typical site that utilized the hydraulic filling method at Lianyungang City in eastern China. Note that the original sand and clay layers were both involved during cutter-suction dredging, hence, soils with both sand and clay were widely formed in this site. The characterization of the studied soil is shown in Table 1. The soil has 33% sand, 46.5% silt and 20.5% clay, and the liquid limit (*LL*), plastic limit (*PL*) and plasticity index (*PI*) are 55.1%, 38.1% and 17.0, respectively. This soil is both categorized as MHS according to the Unified Soil Classification System (ASTM D2487-11) and (JTG E40-2007, China). The soil needs to be improved when used as a material for highway and backfilling engineering (JTJ 034-278, Chinese standard).

Ordinary Portland cement (OPC 42.5 R/N) and metakaolin produced in China and German, respectively, were adopted for this study. The chemical oxides of the Portland cement and MK agent are listed in Table 2. Note that CaO and SiO_2 are the two main oxides of OPC, and their total content was 84%; the ratio of CaO to SiO_2 exceeded 2.0, and the content of MgO was less than 2.0%, both of which meet the specification requirements of European Cement Standard (EN197-1). The predominant oxides of MK are SiO_2 and Al_2O_3, and their total content is approximately 92%. In addition, the average particle size of MK is less than 2 μm, and the specific surface area is approximately 10 m^2/g.

2.2. Compaction test

The modified proctor compaction test with compaction energy of 2687 kJ/m^3 (ASTM D1557-12) was adopted to evaluate the compactability and to determine the maximum dry density (MDD) and the optimum moisture content (OMC) of cement-based modified soils. During the compaction tests, the soils, cement (mass ratio of cement to dry soil at 6% and 8%) and MK agent (mass ratio of MK to dry soil at 0%, 2%, 4% and 6%) were first thoroughly mixed at the desired water contents; later, these mixtures were divided into five parts and successively compacted into a 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.

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 mm < diameter < 2 mm)	6.4
Fine sand content (0.075 mm < diameter < 0.425 mm)	26.6
Silt content (0.002 mm < diameter < 0.075 mm)	46.5
Clay fraction (diameter < 0.002 mm)	20.5
Soil classification—ASTM D2487-11	Elastic silt sand (MHS)
Soil classification—JTG E40 (China)	MHS

Table 2
Oxides compositions of ordinary Portland cement and metakaolin.

Oxides 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		

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 ±0.5% of MDD, ±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 ± 2 °Cand 95% relative humidity for 6, 13 and 27 days. Thereafter, all specimens were saturated in a water tank for 24 h 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 semi-qualitatively analysed using JADE 5.0 software. Note that the lyophilization utilized the following procedure: immersion into liquid nitrogen (−196 °C) for instant freezing and transfer into a vacuum chamber for sublimation for approximately 24 h (Penumadu and Dean, 2000).

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