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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		