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Effects of Fluorogypsum and Quicklime on Unconfined Compressive Strength of Kaolinite

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ABSTRACT

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Coastal areas have environmentally and economically important roles but tend have weak soft ground, which is often vulnerable by waves and unsuitable for coastal construction, such as ports and waterfront areas. Hence, this soft ground, which usually contains large amounts of clays, needs to be ameliorated by using appropriate soil improvement techniques. A common approach to improve soft ground is soil–binder injection techniques to enhance its strength. When avaialbe, binders from industrial wastes can be used instead of commercial products, such as cement and lime, to reduce construction costs and minimize environmental disturbance. Reusing industrial wastes mitigates environmental pollution and reduces the costs of waste management. Construction materials, such as sand and cement, can be partially replaced with industrial wastes if the wastes are granular and induce cementation effects. Fluorogypsum (FG), a by-product obtained during the production of hydrofluoric acid, satisfies these conditions, as it is capable of binding granular materials. Approximately 894,000 metric tons are annually produced in the U.S. However, data on the mechanical strength of clay–FG mixtures are unavailable. In this study, we conducted unconfined compressive strength tests to investigate the mechanical behavior of kaolinite, which represented clay in soft ground, at different FG and quicklime contents. The effects of FG on the compressive strength of kaolinite–FG–quicklime mixtures depend on the curing time and weight ratios of the constituent materials. The composition of the mixture with the highest compressive strength was 30% FG, 5% lime, and 65% kaolinite. We infer that the stoichiometric ratios of mixtures control the chemical reactions for the maximum compressive strength at different quicklime contents based on a series of compressive tests.

ADDITIONAL INDEX WORDS: *Fluorogypsum, soft ground improvement, clay, cementation, unconfined compressive strength*.

INTRODUCTION

The population living in coastal areas is rapidly growing (Barragán and de Andrés, 2015). Coastal structures, such as ports and waterfront areas, are important facilities needed to accommodate economic and life quality demands for this growing population. However, coastal erosion due to other environmental actions can induce instability of shoreline, damage to coastal structures, and significant economic losses. For example, Pusan, a marine city in South Korea, has one of the largest ports in the world, and this port is continually expanding to accommodate an ever-increasing volume of shipments. However, the coastal region where this port is located presents soft clay soils that can cause significant consolidation settlements (Chung *et al.*, 2005). Similarly, the accelerating shoreline subsidence and erosion in Louisiana, USA, are producing significant economic and environmental losses, such as increased vulnerability to hurricane damage and oyster reef loss (Piazza, Banks, and Peyre, 2005). Thus, low-cost efficient engineering methods are needed to stabilize soft sediments and prevent shoreline erosion (Hillyer, Stakhiv, and Sudar, 1997).

The addition of ordinary Portland cement (OPC) to soils has been employed for soil stabilization as it can increase the soil strength due to pozzolanic reaction (Hillyer, Stakhiv, and Sudar, 1997). Industrial by-products, such as fly ash, phosphogypsum, lime sludges, and fluorogypsum (FG), may represent lower cost alternatives to OPC (Consoli *et al.*, 2007; Garg and Singh, 2006;

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Yan *et al.*, 1999). The addition of lime to vulnerable soils induces the formation of calcium silicate hydrate (CSH) and calcium alumina silicate hydrate (CASH), which can effectively produce soil stabilization (Alzubaidi and Lafta, 2013; Cong, Longzhu, and Bing, 2014). The hydrofluoric acid industry annually produces as a byproduct, has been approximately 894,000 metric tons of FG in the USA (Lofton *et al.*, 2018). FG can be used as a binding plaster in buildings and construction materials in aquatic conditions (Bigdeli *et al.*, 2018a; Bigdeli *et al.*, 2018b; Bigdeli *et al.*, 2020; Garg and Pundir, 2014; Lofton *et al*., 2018; Singh and Garg, 2009). The compressive strength of FG-granulated blast-furnace slag–OPC plaster increases during curing time (1–90 days), owing to the formation of gypsum, ettringite, and tobermorite (Garg and Pundir, 2014). In addition to being low-cost, FG is an ecofriendly by-product with lower CO_2 emission than OPC. FG-based soil stabilization and investigates the effects of composition and curing time on the unconfined compressive strength (UCS)) of FG-lime-kaolinite mixtures, in which the kaolinite is representative of soft soils. This research focuses on UCS because this mechanical property is widely used in geotechnical engineering and correlates well with the load-bearing capacity of cohesive soil.

METHODS

Materials

The materials used to prepare the test specimens were water-saturated kaolinite (Edgar Minerals, Inc.) pH-adjusted FG (Brown Industries, Inc.), and quicklime (Carmeuse). All the materials were used in powdered form. FG and quick lime were used as binders to improve the strength of the kaolinite. Kaolinite was selected to represent soft clay minerals with phyllosilicate structure. The kaolinite had a specific gravity of 2.65, specific surface of 28.52 m^2/g, median particle size of 1.36μm, and plastic limit of 26% water content. This material data was provided by the manufacturer, Edgar Minerals, Inc.

The chemical compositions and mineralogy of FG, quicklime, kaolinite and mixtures were identified through X-ray fluorescence (XRF) using PANalytical Epsilon 3^{XLE} X-ray fluorescence spectroscopy and X-ray diffraction (XRD), using a PANalytical Empyrean X-ray diffractometer. Also, scanning electron microscopy (SEM, using a JSM-6610 LV microscope) was applied to capture the microscopic images of the materials, and energy dispersive spectroscopy (EDS) with SEM was used to determine the chemical elements of selected areas in the images.

Unconfined Compressive Strength (UCS) Test

A total number of experimental cases is 12 experimental compositions was considered, with 4 different weight ratios of FG (*i.e.*, 15%, 30%, 50% 70%), and 3 weight ratios of quicklime (QL, *i.e.*, 2%, 5%, 10%) with kaolinite used as the remaining portion of the composition. The different specimens are identified as FGX-QLY, where X denotes the weight ratio of FG and Y denoted the weight ratio of quicklime. For each composition, a total of three cylindrical specimens were fabricated. After mixing the different dry components, deionized water was added to the powder mix to reach the optimum moisture content of 27% that corresponds to the maximum unit weight of the mixture, as obtained via a standard compaction test (ASTM D698). The mixture was blended for 8–10 min. Plastic mold of 152.4 mm height and 76.2 mm diameter were used to cast cylindrical samples. A circular metal disc of 38.1 mm thickness and 76.2 mm diameter was placed at the bottom of the mold, and the disc occupied a length of 25.4 mm in the mold. The mixture was poured into the mold in 3 layers and compacted with 25 rammer blows per layer. The mixture height was 5/6 of the plastic mold length. After the hammering process was completed, the specimen was dehydrated for at least 3 min to maintain its shape. Each specimen had a target unit weight of 15 kN/m^3. The cylindrical specimens were extruded (Figure 1(a)) and then cured for 7 or 28 days at 25 °C, 100% relative humidity. Next, each specimen was placed in a loading frame (VJT511-TriSCAN100), and the unconfined compressive strength of the specimen was determined according to ASTM D5102 (2004) (Figure 1(b)). The applied vertical strain rate was 1.27 mm/min. Unconfined compression test were performed after 7 days of curing and after 28 days.

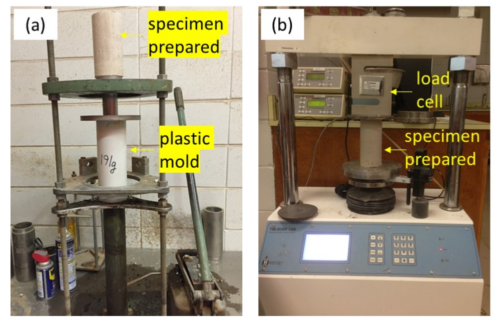

Figure 1. Experimental equipment and test configurations: (a) extruder and specimen extruded from the plastic mold, and (b) loading frame and specimen for the unconfined compression test.

RESULTS

SEM, XRD and XRF Results

The obtained SEM images of kaolinite, FG, and mixture FG70-QL5 after cementation are shown in Figure 2. The kaolinite, FG and quicklime cemented and bonded to each other (Figure 2). Table 1 shows the results of the chemical element analysis of the different FG-based mixtures considered in this study. Additionally, the EDS performed on cemented regions identified during the capturing of the SEM images provided elements of cementation. The cemented regions from six different FG-based specimens contained in average 6.54% C, 59.10% O, 12.48% Al, 12.15% Si, 3.82% S, 0.09% K, 5.13% Ca, 0.55% F, 0.58% Pt, and 0.08% Fe.

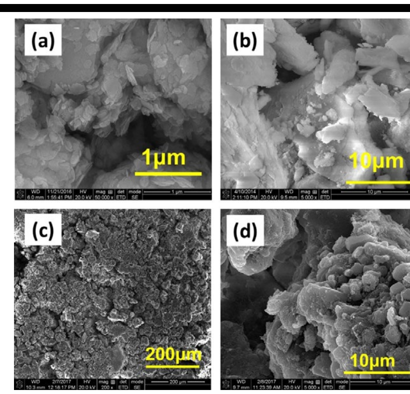

Figure 2. SEM images of materials: (a) kaolinite, (b) FG, (c) and (d) FG70-QL5 mixture (*i.e.*, 70% FG, 5% quicklime, and 25% kaolinite).

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Table 2 lists the chemical compositions of the following groups of specimens: FG, quicklime, kaolinite, and six FG-based mixtures. As expected, the chemical compositions of the specimens were different for different mixtures. The Al_2O_3 content decreased, and the CaO content increased for increasing FG content. Based on XRD patterns, FG specimens showed strong peaks of gypsum ($CaSO_4(H_2O)_2$) and calcium sulfate ($CaSO_4$), whereas the quicklime exhibited strong peaks of calcium carbonate ($CaCO_3$). Stronger peaks of gypsum appeared when the FG contents were higher, and stronger peaks of silica corresponded to lower FG contents. The XRD patterns of the FG-based specimens were related to gypsum and kaolinite rather than calcium carbonate and calcium sulfate.

Table 1. *Chemical element analysis of FG-based compositions through SEM/EDS.*

Element	FG 15% QL 2%	FG 30% QL 2%	FG 70% QL 2%	FG 15% QL 5%	FG 30% QL 5%	FG 70% QL 5%
C	5.70	6.18	8.24	6.59	6.56	5.96
O	60.22	55.43	59.09	60.30	57.59	62.00
Al	15.00	16.54	8.21	13.51	14.21	7.42
Si	14.78	16.84	7.38	13.03	14.09	6.80
S	2.42	2.95	4.86	1.82	3.19	7.67
K		0.09		0.07	0.13	
Ca	1.78	1.90	8.30	4.61	4.12	10.09
F	0.09		2.12			
Pt			1.74			

Table 2. *Compositions of mixtures and endmember minerals determined through XRF.*

Composition (%)	FG	Quicklime	Kaolinite	15% FG, 2% QL	30% FG, 2% QL	70% FG, 2% QL	15% FG, 5% QL	30% FG, 5% QL	70% FG, 5% QL
Al_2O_3	5.24	8.69	34.47	18.92	16.68	5.38	21.66	14	4.27
CaO	25.58	47.83		16.98	19.29	28.67	15.26	22.07	30.15
H_2O	16.9	8.18	10.95	20.73	16.64	18.26	16.13	17.38	18.72
SO_3	36.52			19.01	23.99	38.76	16.07	26.97	39.88
SiO_2	11.97	10.24	49.07	23.48	22.08	8.05	28.67	18.83	6.65
CO_2		25.06		0.88	1.32	0.88	2.2	1.76	1.32
K_2O	2.54		3.69						
MgO	1.25		1.82						
Ca/SiO				1.11	1.34	5.45	0.81	1.79	6.94
Ca/Al				0.90	1.16	5.33	0.70	1.58	9.53
Ca/(Si+Al)				0.58	0.72	3.10	0.44	0.97	4.01

UCS Test Results

The UCS of pure kaolinite at 33% optimum moisture content was approximately 300 kPa, and kaolinite exhibited ductile failure (Figure 3(a)). For specimens containing both FG and quicklime, brittle failure occurred, as shown in Figure 3(b) for composition FG30-QL5.

The UCS always increased with the curing time (Figure 3(c) and 3(d)). For example, the FG70-QL5 specimens experienced a 340% average increase from the 7-day UCS to the 28-day USC, whereas the FG70-QL10 specimens experienced a 540% average increase in USC. The additional curing time facilitated the chemical cementation and increased the UCS values for the different specimens.

The 7-day and 28-day UCS varied based on both the FG and quicklime contents. Increases in the FG content corresponded to increases in the 28-day UCS for mixtures containing 2% and 10% quicklime contents (Figure 3(d)). However, the 28-day UCS of mixture with 5% quicklime content increased for the FG contents up to 30% and then decreased the FG content. The 7-day USC increased for increasing FG content up to 30% FG, and then it remained stable for compositions containing 2% and 5% quicklime, whereas it decreased for increasing FG content for compositions with 10% quicklime. When considering the effects of quicklime content, it is observed that 7-day and 28-day UCS reached a peak at 5% quicklime content for 15 and 30% FG content, whereas the compositions with 50% and 70% FG content reached their maximum values of UCS for 2% quicklime content (Figure 4). The maximum 28-day UCS is reached by the FG30-OL5 composition, which can be considered the optimal composition from the point of view of UCS among those considered in this study.

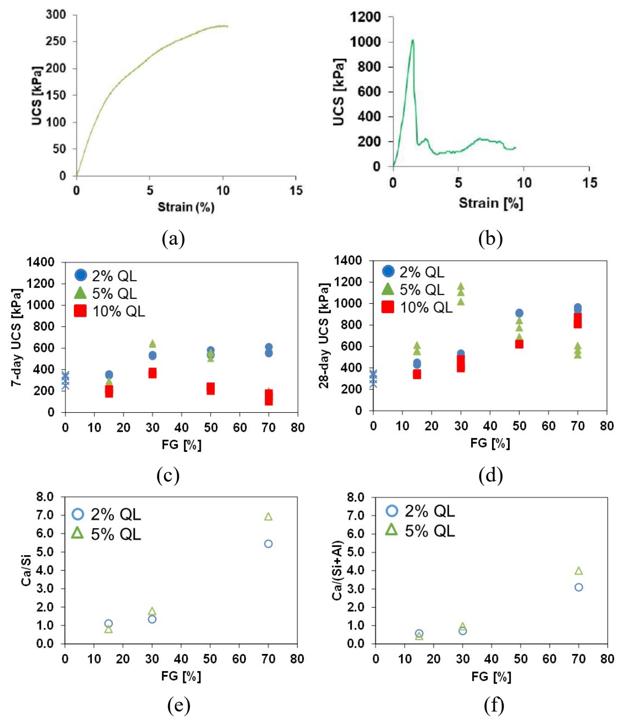

Figure 3. Experimental results: Stress–strain curve for variation in compressive strength with stress (a) 100% kaolinite and (b) 30% FG and 5% quicklime contents after 28 day curing time. UCS with varying amounts of FG with different quicklime contents and curing of (c) 7 days and (d) 28 days (remark– x shape: 100% kaolinite); (e) Ca/Si and (d) Ca/(Si+Al) ratios at different FG contents.

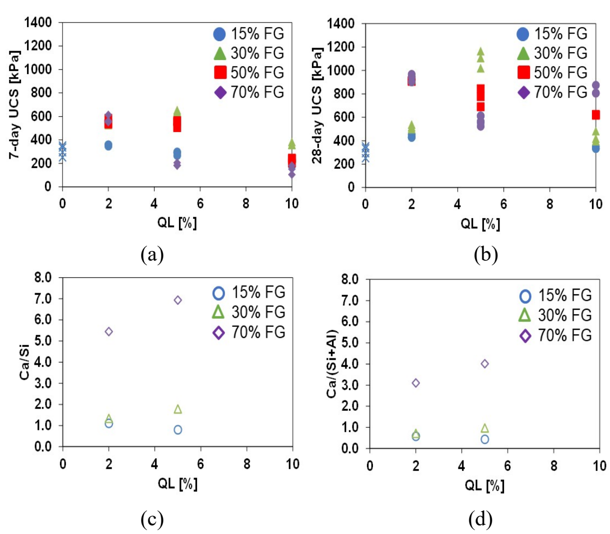

Figure 4. Experimental results for varying amounts of quicklime with different FG contents and curing periods: (a) UCS at 7 days of curing, (b) UCS at 28 days of curing (remark – x shape: 100% kaolinite).

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DISCUSSION

The optimal mixture ratio should be stoichiometrically analyzed. CSH consists of CaO, SiO_2, and H_2O (Raki *et al.*, 2010) and is divided into two types: tobermorite-like (CSH (I)) and jennite-like (CSH (II)) (Gougar, Scheetz, and Roy, 1996). The main reactions are expressed as follows:

$$CaO + H_2O \rightarrow Ca(OH)_2 \quad (1)$$
$$Ca(OH)_2 \rightarrow Ca^{2+} + 2OH^- \quad (2)$$
$$Ca^{2+} + 2OH^- + SiO_2 \rightarrow CSH \quad (3)$$

CASH was formed by CSH with aluminum oxide (Al_2O_3) (Tastan *et al.*, 2011), and the reaction can be expressed as follows:

$$Ca^{2+} + 2OH^- + SiO_2 + Al_2O_3 \rightarrow CASH \quad (4)$$

CSH and CASH expressed in the above equations are compounds of siliceous granular materials and binders that enhance the compressive strength of siliceous materials, such as kaolinite mixed with additives (Al-Mukhtar, Lasledj, and Alcover, 2010; Locat, Bérubé, and Choquette, 1990; Raki *et al.*, 2010; Tastan *et al.*, 2011). The effectiveness of cementation depends on the weight ratio of binders, such as CaO and $Ca(OH)_2$, and siliceous materials, such as SiO_2. When the CaO/SiO_2 ratio (C/S) is high, the binders can produce CSH or CASH rapidly. However, the mixture should contain sufficient SiO_2 to build the main fabric of the materials and assist the binders to form cementitious compounds between the main materials. Thus, the Ca/Si ratio is a critical parameter used for analyzing the strength of mixtures (Figure 5(c) and 5(d)).

CSH is the principal product of cement hydration. For example, the CSH produced after a 2-day pozzolanic reaction is the main and the heaviest component, compared to ettringite and monosulfate, which are also hydrate compounds (Gougar, Scheetz, and Roy, 1996).

FG mainly consists of CaO and SO_3 (Escalante-Garcia *et al.*, 2008). The XRD pattern of FG indicates anhydrite ($CaSO_4$) with high intensity, and gypsum ($CaSO_4.2H_2O$) with low intensity (Garg and Pundir, 2014). Most of the CaO present in FG reacts with water, which generates slaked lime ($Ca(OH)_2$) in Equation 1 (Chiaia, Fantilli, and Ventura, 2012; Tastan *et al.*, 2011; *Zrei et al.*, 2014).

The main components of kaolinite are SiO_2 and Al_2O_3 (> 82%), and the hydrate of SiO_2 induces silicic acid (H_4SiO_4), which can generate CSH with calcium hydroxide ($Ca(OH)_2$):

$$SiO_2 + 2H_2O \rightarrow H_4SiO_4 \quad (5)$$
$$Ca(OH)_2 + H_4SiO_4 \rightarrow CaH_2SiO_4 \cdot 2H_2O = CH + SH \rightarrow CSH \quad (6)$$

The CSH pozzolanic reaction of CaO from quicklime and SiO_2 occurs, as described in Equations (1) to (4). The CSH content is influenced by the water content based on the stoichiometry in Equation (1). Furthermore, Al_2O_3 present in kaolinite can form CASH, as expressed in Equation (4). Both reactions enhance compressive strength. For instance, when the samples are cured in water, CSH and CASH bind the grains in the main fabric and fill up the pores of the fabric (Aldaood *et al.*, 2014). Suitable Ca/Si and Ca/(Si+Al) values are 1.2–2.3 and 0.7–2.4, respectively, at the solution-to-solid ratio should be 0.4 (Richardson, 1999). Figures 3 and 4 show the Ca/Si and Ca/(Si+Al) ratios, respectively, and the maximum shear strength at 30% FG and 5% quicklime correspond to Ca/Si of 1.75 and Ca/(Si+Al) of 0.79 at a 33% water content, which is lower than the regular water ratio. Other water contents and water-to-solid ratios may result in higher strength.

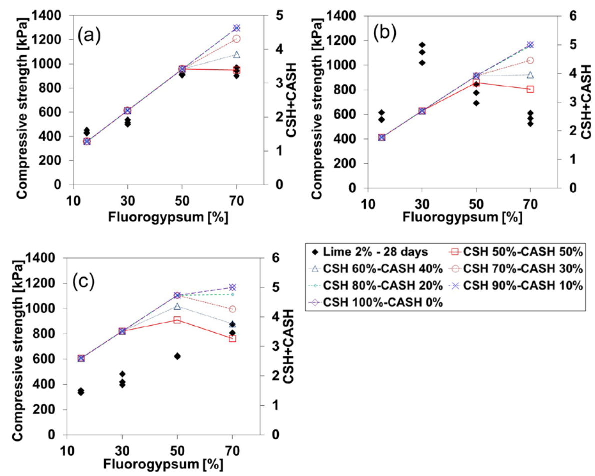

Figure 5. Correlation between compressive strength and CSH + CASH for various FG contents and curing period of 28 days: (a) 2% quicklime, (b) 5% quicklime, and (c) 10% quicklime.

Figure 5 shows the experimental results of the unconfined compressive strength tests and the CSH and CASH reactions based on the stoichiometry of Equations (1) to (4). We assume that the calcium ions would be consumed to both CSH and CASH evenly until the FG content reached 50%, as the SiO_2 and Al_2O_3 in the kaolinite are sufficient to form CSH and CASH. When the FG content reached 70%, the CSH and CASH contents could vary, owing to the lack of Al_2O_3 in kaolinite (Figure 5). The CSH and CASH trends may explain why the trends of the unconfined compressive strength in Figure 4 changes based on the material ratios of the mixture. As shown in Figure 5(a), the mixtures with 2% quicklime yield 80% CSH and 20% CASH reaction; 5% quicklime yield 50% CSH and 50% CASH reaction (Figure 5(b)), and 10% quicklime, 100% CSH and 0% CASH reaction (Figure 5(c)).

CONCLUSIONS

Coastal areas can be affected by erosion and subsidence and are often characterized by soft soils that are not appropriate for construction of structures and infrastructure systems. These effects can cause critical environmental issues and negatively impact the economy at a local or even global level. To mitigate these issues, the soil along the shoreline can be improved using different techniques, including the use of additives with binding properties. Among the different available binding materials, fluorogypsum (FG) is a low-cost industrial by-product that can react with soft soils and produce calcium silicate hydrate (CSH) and calcium alumina silicate hydrate (CASH). reactions with soils and improve soil properties of weak and soft soils typically located near coastal regions.

Unconfined compressive strength (UCS) tests were performed on different FG-quicklime-kaolinite mixtures to investigate their mechanical behavior. The experimental results show that these mixtures generally present an enhanced UCS when compared to kaolinite alone. The specific values of the UCS depend on the specific compositions and on the curing time, which affect the chemical reactions that govern the cementation effect. The observed UCS correlate well with ratios of Ca/Si and Ca/(Si+Al). The maximum 28-day UCS of the FG-quicklime-kaolinite mixtures was obtained for the mixture with 30% FG, 5% quicklime, and 65% kaolinite, and corresponded to optimal ratios of

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Ca/Si equal to 1.75 and Ca/(Si+Al) equal to 0.79 with 27% water content. It is also observed that the UCS could be influenced by the types of cementation reactions of CSH and CASH when available ions for the CASH and CSH reaction are limited in the pore fluid.

ACKNOWLEDGMENTS

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