

Characterization of Hydrated Lime-Stabilized Brown Kaolin Clay

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Abstract

The effect of the calcium-based stabilizers like lime on the clay soil characterizations has been studied in this research. This study was carried out in an attempt to identify the time-dependent soil-chemical reactions for lime chemically stabilized Brown Kaolin clay samples. The data for the study of characteristics of treated samples were obtained from X-ray diffractometer (XRD), field emission scanning electron microscopy (FESEM), and Fourier transform infrared spectroscopy (FTIR). Furthermore, to illustrate the effect of lime on the strength, a series of laboratory tests were carried out by unconfined compressive strength. Based on the micro-structural analyses, it was observed that kaolinite is rapidly exhausted by pozzolanic reactions to produce the Gismondine (CASH), which is responsible on gain the strength for the lime treated samples with progressing time. On the other hand, it was noticed Illite mineral did not show any reacted with advancing time. In addition, based on the morphology of the treated samples, the presence of the cementious products were observed. These outcomes proved the effectiveness of lime to stabilize kaolin clay.

1. Introduction

The clay mineralogy has significant effect on the soil characterization. Kaolinite is geochemically and industrially extremely versatile. These minerals form in tropical climatic conditions of the region where rainfall is comparatively high, and there is good drainage to confirm cation leaching and iron acids granitic rocks [1]. On the other hand, Kaolin soil is soft clay deposits. Furthermore, it has predominantly poor engineering characterizations, which are lead to occur high compressibility, high plasticity, poor workability, and low shear strength. Therefore, using these soft deposits for construction structures, lead to many geotechnical problems. For that, to encounter these problems, chemical stabilization should be applied.

The calcium-based stabilizer confirmed its efficiency in improving the soil properties as reported by several researchers [2-4]. In Malaysia, limestone is widely used. Furthermore, the presence of lime in the form of hydrated lime is

comparatively high in Malaysia, making a lime economically applicable selection for the treatment of tropical soils [5]. Lime used to improve the bearing capacity of the layers in highway, railway, and airport buildings. It is also used as the basis for lightweight structures, such as fill material for retaining walls, and as a side support in excavations and trenches. Therefore, due to the proven versatility of lime stabilization, the method has been gained wider acceptance in different countries of the world, and most recently in Southeast Asia [6].

2. Mechanism of Lime Reactions

When a soil is mixed with lime, two mechanisms reactions occur. These reactions can be classified into short-term reactions (modification or flocculation) and long-term (solidification/stabilization) as stated by [7]. During the short-term phase, the calcium ions (Ca^{++}) from hydrated lime migrate to the surface of the clay particles and displace water and other ions. Thus, as a result to this reaction, increasing in the pH of the pore water was occurred. Furthermore, the soil becomes friable and granular due to the flocculation of soil aggregates, making it easier to work and compact include. Cation exchange and carbonation were included also within this term of reactions. These reactions influence the physical properties (i.e. Atterberg limits and particle size distribution) of the soil. Moreover, an increase in strength caused by both the dehydration and fundamental changes in the clay particle chemistry is accompanied in this reaction.

While, the reactions that take place after the modification phase, which are attributed to the long term reactions, are time dependent and continue for a long period. These reactions lead to produce the pozzolanic reactions [8]. The formation of various cementing products will bind the aggregates, thus increase the strength of the clay soils.

It should be noted that the most of previous research considered only the physical characteristics of the soil parameters [9,10]. Thus, the details studies to determine the mechanisms of lime clay reactions based on the microstructure area were limited. Therefore, this paper aims to

investigate the characteristics of hydrated lime treated Brown Kaolin samples using XRD diffraction, Field Emission Scanning Electron Microscope (FESEM), Fourier Transform Infrared Spectroscopy (FTIR) analysis, and to evaluate the strength development of lime treated samples, UCS test was carried out on the cured samples.

3. Materials

Slightly acidic brown pure kaolin was used as a material in this study. The bulk soil is purchased from Tapah, where it is located in the west of Peninsular Malaysia. The Atterberg limits, particle size distribution, density and pH of kaolin were determined in the laboratory. The physical and chemical composition of the soil properties are listed in Table 2. The lime used was hydrated lime Ca (OH)₂.

Table 2. The Physical Properties and chemical composition for Kaolin clay

4. Samples Preparation

Two different amounts of lime, i.e., 5 and 10% by weight of the dry soil, were chosen for this research to prepare the treated samples. The optimum moisture content and the max dry density were determined for the natural and the treated samples for both the percentages of lime. Clause 3 .3. 4. 1 of BS 1377: Part 4: 1990 was adopted to accomplish the compaction test. Dry Mixtures of lime and clay are mixed with distilled water. UCS samples were prepared by placing a mixture in a mold (38 mm diameter x 76 mm length) under constant stress of compaction, as specified in BS1924: Part2: 1990 (clause 4.1.5). Then, sample was removed using a hydraulic jack and wrapped with thin plastic film. They were covered plastic container with a tight lid to prevent the moisture lost during the curing time. The samples were stored in a temperature-controlled room (27 ± 2 °C) and tested at different curing periods of 7 days, 14 days, 28 days, 100 days and 200 days.

5. Testing Programme

ENGINEERING & Physical Properties	VALUES	Chemical Composition Oxides	VALUES (%)
CEC (meq/100 g)	19.2	SiO ₂	49.5
pH (L/S = 2.5)	4.34	Al ₂ O ₃	30.31
Specific Gravity	2.65	Fe ₂ O ₃	1.02
Liquid Limit, LL (%)	40.8	Na ₂ O	0.79
Plastic Limit, PL (%)	22.6	K ₂ O	8.78
Plasticity Index, PI	18.2	P ₂ O ₅	4.03
(%)BS Classification	CI	SO ₃	2.05
ICL (%)	2	CO ₂	1.4
Maximum dry density (Mg/m ³)	1.5		
Optimum moisture content (%)	24.3		
Unconfined compressive strength (kPa)	130		

To study the effect of hydrated lime on soil characteristics, the treated soil was characterized prior and after the treatment. The methods and equipment used in each of these analyses are specified as follow.

Powder XRD was collected to define the crystalline minerals existed in the natural soil and also to monitor the mineralogical changes caused by lime treatment. Samples for XRD test were prepared by grinding the soil using a pestle and a mortar to obtain a homogeneous fine powder. A Bruker D8 advance diffractometer was used to accomplish scans in an angle scan (2θ) ranging between 10 to 80° with a 0.02° step size and dwelling time of one second at each step. Then, the mineralogy analysis was carried out based on the characteristic Bragg data available in the standard powder diffraction file (JCPDS, 1995).

A JSM-6701F JEOL Field Emission Scanning Electron Microscope (FESEM) was used also to study the morphological changes and the topographic features associated with clay particles before and after treatment. Furthermore, FTIR was conducted to examine the molecular alterations in the structure of treated samples. Hence, 2 mg of dried soil was ground in an agate mortar and blended with 200 mg potassium bromide (KBr) to produce a homogeneous powder, which was then pressed into a solid pellet. The transparent KBr disc (pellet) was then placed in a sample holder where it was scanned using a Perkin Elmer Spectrum 2000 instrument to yield a pattern of the beam transmitted through the sample from 400 to 4000 cm⁻¹.

Unconfined compressive strength (UCS) test was performed on cured specimens at an axial strain rate of one percent per minute in accordance with BS 1924: Part 2: 1990. The results were used as an index of improvement.

6. Test Results and Discussion

6.1. Compaction Test

Standard compaction test was carried on lime stabilized Brown Kaolin clay prior to strength (UCS) test to determine the compaction characteristics. Figure 1 reveals the dry density-water content relationships for kaolin clay with 5% & 10% lime contents. As shown in the figure, the addition of lime increased the optimum moisture content (OMC) and decreased the maximum dry density (MDD) with increasing lime content. This was consistent with the study conducted by [11]. The reduction in the dry density occurs as a result of agglomerated particles due to flocculation. Furthermore, because the lime requires more water for the pozzolanic reactions that leads to increase OMC with the content of the lime growing.

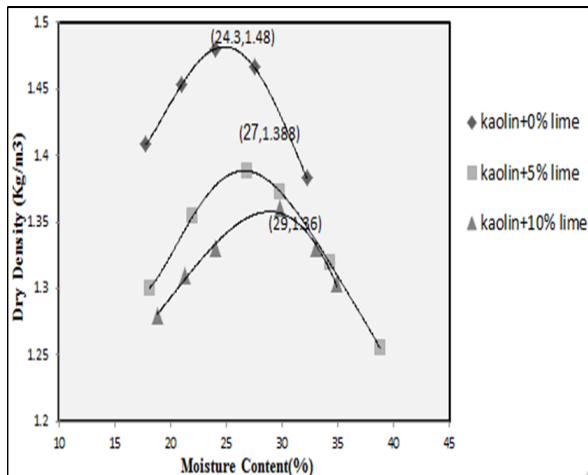


Figure 1: The compaction characteristics of lime stabilized Brown Kaolin clay

7. Microstructure Analysis

7.1. X-ray diffraction (XRD)

Figure 2. shows the XRD pattern of untreated Brown Kaolin clay. The main mineralogical constituents of untreated kaolin clay are kaolinite $Al_2(Si_2O)(OH)_4$, quartz SiO_2 , calcite $CaCO_3$, and illite $(K,H_3O)(Al_2Si_3AlO_{10}(OH)_2)$. However, kaolinite is the predominant clay mineral. Furthermore, XRD-ray patterns of untreated and 10% lime treated Brown Kaolin clay samples after 7, 100, and 200 days were shown in Figure (3). As shown in this figure, it can noticed new compounds at 7 days curing, which are observed at 2θ of (19.8, 35.3, 40.4, 50, 54, 56), (17.7, 20, 29.8, 34.5, 55), and (29.2, 34.2, 47.4, 64.7) for Montmorillonite, Muscovite, and Portlandite respectively. For the long period at 100 and 200 days, a new reflection is attributed to the formation of Gismondine (CASH), which is resulted from the reaction between the Portlandite and the mixture of soil. This finding was in agreement with [12] study. As the time prolonged, the alkaline environment which is responsible for the pozzolanic reaction to ensure silica and alumina dissolved from solution will increase. Thus, the pozzolanic reaction continues to produce more CASH, which lead to increase the strength of the lime treated Kaolin soil. Furthermore, it should be noted that the high percentage of quartz does not assist in increasing the strength of lime treated Kaolin clay. Instead, it retarded the bonding between the surface lime and clay particles. For this reason, the partially crystallize CASH, still not entirely converted to the crystallize compounds CSH and CAH. Furthermore, it can be noticed the presence of calcite, which is result from the carbonation of calcium. Thus, as stated previously it can form

comparatively weak cementing agents such as calcium carbonate, and this reaction is undesirable because it exhausts some lime that would normally be used to form more resistant cementitious products [13].

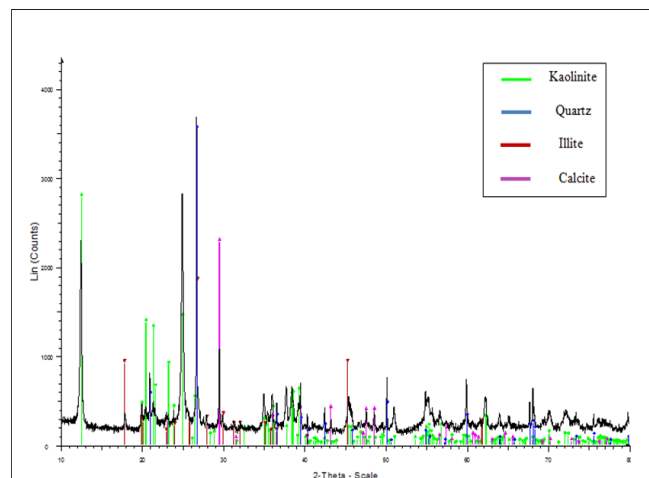


Figure 2: XRD of untreated Kaolin clay soil

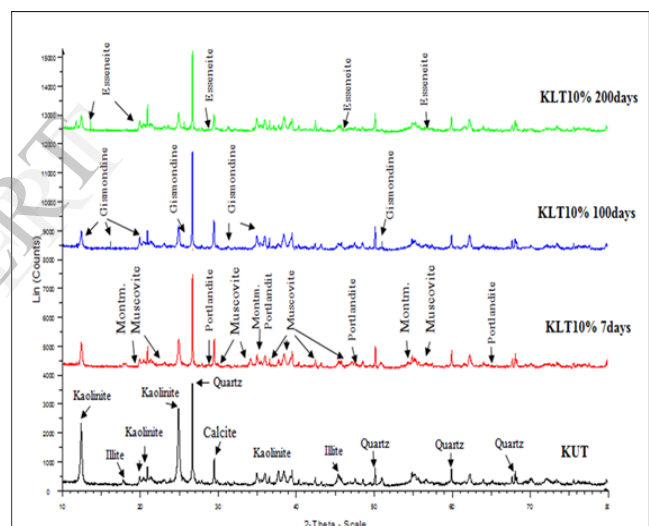
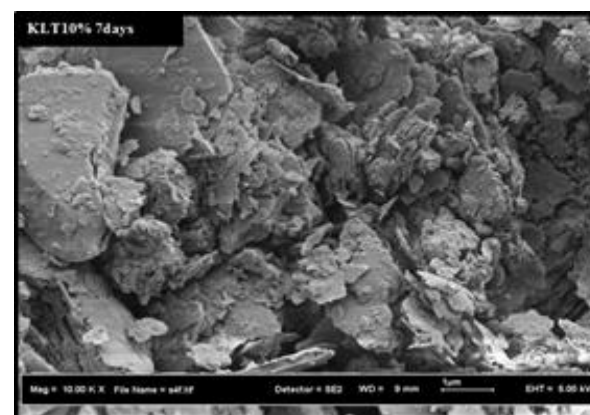


Figure 3: X-ray diffraction patterns for untreated and lime treated Brown Kaolin clay soil after 7, 100, and 200 days curing time

7.2. Scanning Electron



Microscope Analysis

The micrograph of natural kaolin clay is presented in Figure 4. It can be noticed, the neatly arranged book-like kaolinite particle were the predominate feature of the natural soil [14]. The kaolin generally has a to-face aggregate and edge -to-edge flocculated structure [15]. Figure 5. shows the micrograph of lime stabilized Brown Kaolin clay samples at different intervals time. It was evident there is insignificant modification in the texture of the lime-kaolin matrix at 7 days curing time. In addition it can detect little flocculated parts with small signs of $\text{Ca}(\text{OH})_2$ as a result to starting lime dissolution. At 100 and 200 days curing, it was apparent that the degree of crystal formation and the fabric texture of lime-stabilized matrix are developed with time. It can recognize parts of reticulation phases and the evident of white lumps. These changes are attributed to the progressing in the process of lime reaction and producing calcium alumina silicate hydrated (gismondine), which is responsible for the long-term strength performance of stabilized specimens.

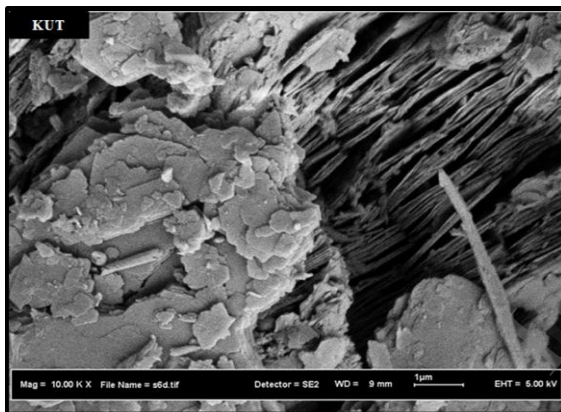


Figure 4: FESEM image of untreated Kaolin clay soil

8. Molecular Characteristics

8.1. FTIR

Infrared spectroscopy was carried out in this research, to examine the influences of hydrated lime on the molecular structure of treated Kaolin soils.

The FTIR spectrum of natural Brown Kaolin soil is shown in Figure (6). As can see, kaolinite was identified by two strong bands at 3696 and 3620 cm^{-1} . These bands were related to the octahedral OH stretching vibrations. Furthermore, it can be detected sharp bands at 1115 cm^{-1} , 1031 cm^{-1} , and 1007 cm^{-1} that are attributed to the Si-O stretching.

The band at 912 cm^{-1} corresponding to the OH deformation of hydroxyl groups. Most of the other

Figure 5: FESEM images of 10% Lime treated Kaolin Clay after 7, 100, and 200 days

bands such as the Si-O vibrations observed at 794 cm^{-1} , 698 cm^{-1} , 540 cm^{-1} , and 470 cm^{-1} , also

confirmed the presence of kaolinite mineral.



Furthermore, the small band at 1422 cm^{-1} relates to the presence of calcite. While, the bonding mode vibration of water molecules H-O-H is observed at 1638 cm^{-1} .

FTIR spectroscopy was also conducted on lime treated Brown Kaolin clay samples as presented in Figure (7). It can detect increasing the intensity of absorbance band at 1425 cm^{-1} due to increasing the sources of calcium, which are supplied from the lime addition. It was also observed a slight increasing in the intensity of Si-O stretching due to development the degree of polymerization with time

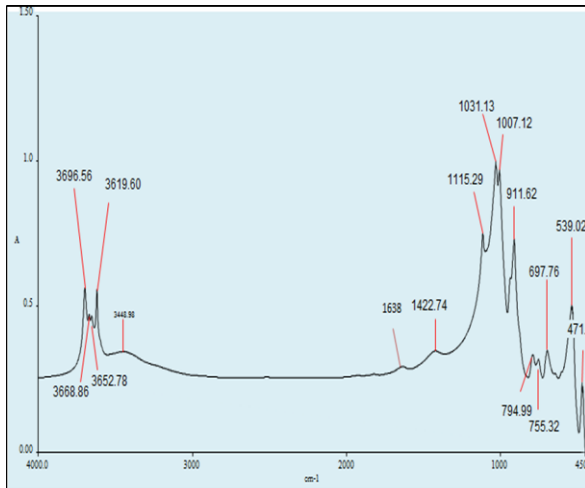


Figure 6: FTIR Spectra for natural Brown Kaolin clay soil

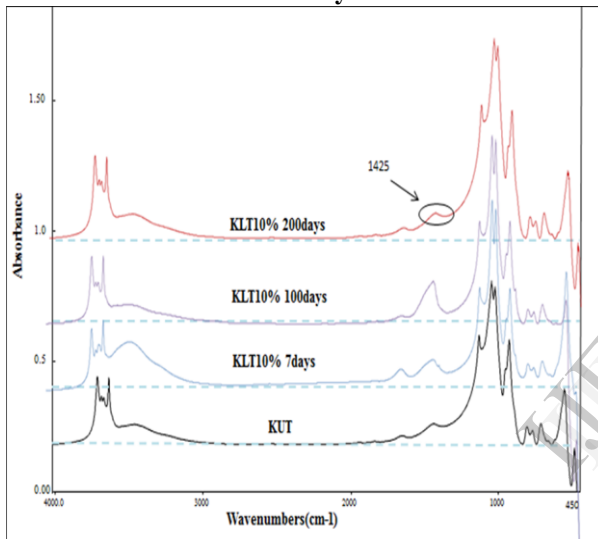
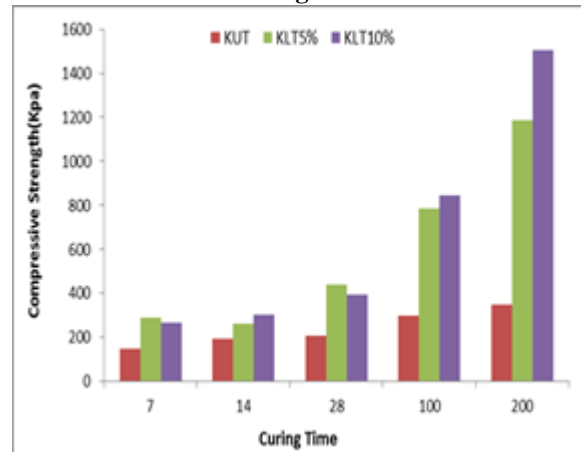


Figure 7: FTIR spectrums of natural and lime treated Brown Kaolin Clay at different time intervals

9. Strength Analyses (UCS)

In order to assess the degree of improvement in the Brown Kaolin soil, UCS test was performed on lime-treated samples. Analysis of the data presented in Figure 8. As can be seen from the Figure, in comparison to the maximum value of lime 10%, it can notice that 5% lime-treated soil specimens shows an increased in UCS at the early stages of curing time 7, 14 and 28 days. However, the UCS of the 10% lime treated cured samples showed increased about more than 9 times of the original untreated strength after 200 days curing. The promising increased in strength of lime treated specimens is may associate with the increase in pH value, promoting alkaline condition. Consequently, allowed pozzolanic reactions to take place for further strength [12].

Figure 8: Unconfined compressive strength (UCS) of lime-treated Brown Kaolin soil at curing time



10. Conclusion

Based on the results of the microstructural characteristics of lime-kaolin, which were obtained from this study, it can conclude regarding the strength gain for the lime treatment. The high alkalinity obtained from the lime treatment, leads to dissolve the lime to react with soil silica and alumina, which are released from the quartz and kaolinite minerals to produce the calcium silicate and calcium aluminium silicate hydrated (CASH) as indicated by the X-ray diffraction of lime treated Brown Kaolin samples. These compounds form matrix that participates to flocculation the soil particles by coating and bonding them together, and with time, it strengthens the soil. This pozzolanic compound is responsible for the long-term strength performance of the stabilized clay soil. This was proved by the images obtained from FESEM micrographs, showed a clear formation of cementitious materials. Furthermore, the increasing in the intensity of Si-O stretching due to development the degree of polymerization with time was also indication to the effectiveness the lime in stabilized the kaolin sample.

Overall, the factors contribute to the effectiveness of lime to stabilize kaolin clay was associated with lime content, curing time, soil type and clay minerals.

10. References

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