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Physico-Chemical Characterization Of Lime Stabilized Tropical Kaolin Clay

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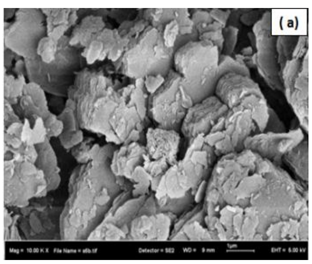
Graphical abstract



Abstract

Lime stabilization is one of the techniques used to improve the mechanical properties, particularly the strength of soft clay soil. However the effectiveness of lime at long term is still ambiguous. This paper aims to determine the suitability of lime for stabilizing tropical kaolin clay soils, and to assess typical changes in soil structure due to mineralogical influences at different during period and lime content in both short and long term. The microstructure characterizations have been investigated using x-ray diffraction (XRD), and Field Emission Scanning Electron Microscopy (FESEM). Furthermore, to illustrate the effect of lime on the strength, series of laboratory tests were carried out by unconfined compressive strength. The results indicated that the addition of lime resulted in an improvement in compaction properties. In addition, the unconfined compressive strength (UCS) of stabilized clay has increased with the addition of lime. The influence of the time factor on the development of strength lime treated samples was equally proportional with the lime content factor. The formation of calcium aluminate silicate hydrate (CASH) was observed from the XRD test after 200 days, and the presence of the cementious products were further verified in FESEM analysis. It is therefore, proved the effectiveness of lime to stabilize kaolin clay in long term duration.

Keywords: Lime, stabilisation, kaolin clay, pozzolanic reaction, mineralogy

Abstrak

Penstabilan kapur adalah salah satu teknik yang digunakan untuk meningkatkan sifat-sifat mekanikal, terutamanya kekuatan tanah liat lembut. Namun keberkesanan kapur pada jangka panjang masih samar-samar. Kertas kerja ini bertujuan untuk menentukan kesesuaian kapur terhadap tropika tanah liat kaolin, dan untuk menilai perubahan biasa dalam struktur tanah yang dipengaruhi bahan mineral di dalam tempoh yang berbeza dan kandungan kapur dalam kedua-dua jangka pendek dan panjang. Analisis struktur mikro telah dikaji dengan menggunakan 'x-ray diffraction' (XRD), dan 'Field Emission Scanning Electron Microscopy' (FESEM). Selain itu, untuk menggambarkan kesan kapur pada kekuatan, siri ujian makmal dilakukan dengan kekuatan mampatan terkurung. Hasil penelitian menunjukkan bahawa kehadiran kapur menyebabkan peningkatan sifat pemadatan. Selain itu, kekuatan mampatan terkurung (UCS) dari tanah liat stabil telah meningkat dengan penambahan kapur. Pengaruh faktor masa kepada pembangunan sampel dirawat kekuatan kapur adalah berkadar sama dengan faktor kandungan kapur. Pembentukan kalsium aluminat silikat hidrat (CASH) dapat diperhatikan pada ujian XRD selepas 200 hari, dan kehadiran produk bersimen telah disahkan lagi dalam analisis FESEM. Oleh itu, terbukti keberkesanan kapur untuk menstabilkan tanah liat kaolin dalam tempoh jangka panjang.

Kata Kunci: Kapur, penstabilan, tanah kaolin, reakso pozzolanic, moneralogi

1.0 INTRODUCTION

Lime stabilization is one of the oldest forms of stabilization in the world. It has a history of success in many types of land over 5000 years. It is 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. Limestone is widely used in Malaysia. Moreover, the supplies of lime in the form of hydrated lime are relatively high in Malaysia, making a lime economically viable option for the treatment of tropical soils.[1] 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.[2]

The reaction between lime and clay occurs in two phases: modification and stabilisation. [3,4] The key reactions occurring in lime-treated clay are soil hydration, ion exchange, flocculation, and pozzolanic reaction. The short-term reactions (modification or flocculation) include hydration, migration of lime, pH and cation exchange reactions, and carbonation. During this phase, the calcium ions (Ca^{++}) from hydrated lime migrate to the surface of the clay particles and displace water and other ions. The soil becomes friable and granular as a result of flocculation of soil aggregates, making it easier to work and compact. These reactions influence the physical properties (i.e. Atterberg limits and particle size distribution) of the soil. This is accompanied by an increase in strength caused by both the dehydration and fundamental changes in the clay particle chemistry. Whereas, the long-term reactions that take place after the modification phase are time dependent and continue for a long period. It is termed solidification/stabilization and is the results of pozzolanic reactions.[5-8] The formation of various cementing products will bind the aggregates, thus increase the strength of the clay soils.

While most research considered only the physical characteristics of the soil parameters, details study to determine the mechanisms of lime-clay reaction on microstructure area were limited.[9] To understand the mechanisms involved in lime-clay reactions in details, fundamental knowledge on physical and microstructure characteristics were equally important. Therefore, this paper aims to investigate the strength and the microstructure of lime treated clay using unconfined compressive strength test, XRD diffraction and Field Emission Scanning Electron Microscope (FESEM) analysis.

■2.0 MATERIALS & TESTING PROGRAMME

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 1 and Table 2). The lime used was hydrated lime Ca $(OH)_2$ at 5% and 10% content. It has been supplied as a powder state from a lime factory in Pasir Gudang, Johor.

Table 1 The Physical Properties and chemical composition for Kaolin clay

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_2O_3	30.31
Specific Gravity	2.65	Fe_2O_3	1.02
Liquid Limit, LL (%)	40.8	Na_2O	0.79
Plastic Limit, PL (%)	22.6	K_2O	8.78
Plasticity Index, PI	18.2	P_2O_5	4.03
(%)BS Classification	CI	SO_3	2.05
ICL (%)	2	CO_2	1.4
Maximum dry density (Mg/m3)	1.5		
Optimum moisture content (%)	24.3		
Unconfined compressive strength (kPa)	130		

Table 2 Chemical composition of the hydrated lime

Hydrated Lime	
CHEMICAL COMPOSITION	VALUES (%)
Calcium Oxide, CaO	74.23
Phosphorus Oxide, P2O5	0.08
Magnesium Oxide, MgO	0.74
Calcium Sulphate, CaSO4	0.12
Ferric Oxide, Fe2O3	0.17
Aluminium Oxide,	0.11
Al2O3 Silica, SiO2	0.14
Loss on Ignition, LOI	24.35

To study the effect of hydrated lime on physical properties and the microstructural characterization, both treated and untreated kaolin were tested by similar laboratory tests. Physical tests were carried out by compaction and unconfined compressive strength (UCS). The increase in strength is determined by analysing the effect of kaolin soil on curing period and lime content. Then the microstructure and mineralogical studies were further verified by XRD-diffraction and field emission scanning electronic microscope (FESEM). Prior to analysis, the suitability of lime for long term stabilisation of kaolin soil was investigated by carried out initial lime consumption (ICL) test

■3.0 SAMPLES PREPARATION

Clause 3.3.4.1 of BS 1377: Part 4: 1990 has been adopted to determine the compaction characteristic in this study, which it is achieved by compacting the soil in three equal layers into a 1 L Proctor mould by applying 27 blows of 2.5 Kg rammer dropping from the controlled height of 300 mm.[10] The moisture-density relationship curve has been obtained for each individual soil with the stabilizer with their different content to investigate the effect of stabilizer on the compaction parameters for clay soils. The clay samples were oven dried initially. For the preparation of mixtures of lime and clay, the required amounts of dry kaolin were mixed with 5% and 10% lime by weight of kaolin in the dry state until equal distribution of lime in the mixture was obtained. Dry mixtures of lime and clay are mixed with distilled water. These mixtures were used for compaction and unconfined compressive strength. Moisture contents selected for this study corresponded to the optimum moisture content (OMC) of treated and untreated kaolin; determined from compaction tests. These water contents were used for the preparation of samples for unconfined compressive strength.

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, the samples were removed using a hydraulic jack and wrapped with thin plastic film. The plastic containers were covered 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.

4.0 RESULTS AND DISCUSSIONS

4.1 Initial Consumption of Lime Test

Standard initial consumption of lime (ICL) test was performed at different solid to liquid ratio (S/L), to indicate the initial amount of lime needed for modification process. pH of 12.4 is required to sustain both modification and stabilisation process. Figure 1 shows the effect of pH when lime is added to kaolin clay. It is shown in Figure 1, lime content of 2% is necessary to provide pH value of 12.4. However, Eades and Grim (1960) suggested the addition of lime in the range of 4-6% for long term strength gained. Therefore, 5% and 10% by weight of hydrated lime were added clay to study the effect of lime content on stabilized kaolin clay.

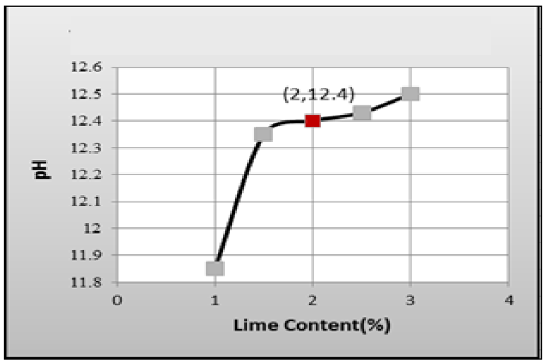
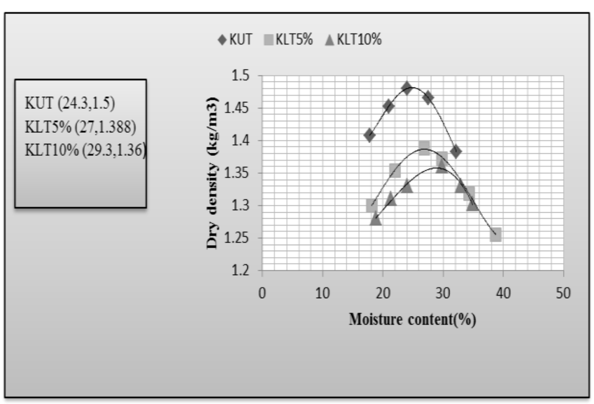


Figure 1 Results of the ICL test for Kaolin clay soil

4.2 Compaction Test

Standard compaction test was carried on lime stabilized kaolin clay prior to strength (UCS) test to determine the compaction characteristics. Figure 2 illustrates the dry density-water content relationships for kaolin clay with different lime contents.



** KUT = Untreated Kaolin, KLT = Lime-treated Kaolin*

Figure 2 The effect of compaction characteristics on lime stabilized kaolin clay

As shown in the chart, the effect of lime on the compaction properties of kaolin samples is obvious. This shows the effectiveness of lime to stabilize kaolin clay. The addition of lime increased the optimum moisture content (OMC) and decreased the maximum dry density (MDD) with increasing lime content. This finding was consistent with previous study by Umesha et al. (2009)[11] The reduction in the dry density occurs as a result of agglomerated particles due to flocculation. Furthermore, as lime requires more water for the pozzolanic reactions that leads to increase OMC with the content of the lime growing.

4.3 Unconfined Compressive Strength

Figures (3a) and (3b) present the development of UCS for lime-treated kaolin samples with various curing time and lime content respectively. As shown in Figure (3a), the lime-treated soil specimens show an increased in UCS at day 7 and day 14. While, significant increase in strength value at 28 days was noted for lime stabilized specimen compared to untreated specimen.

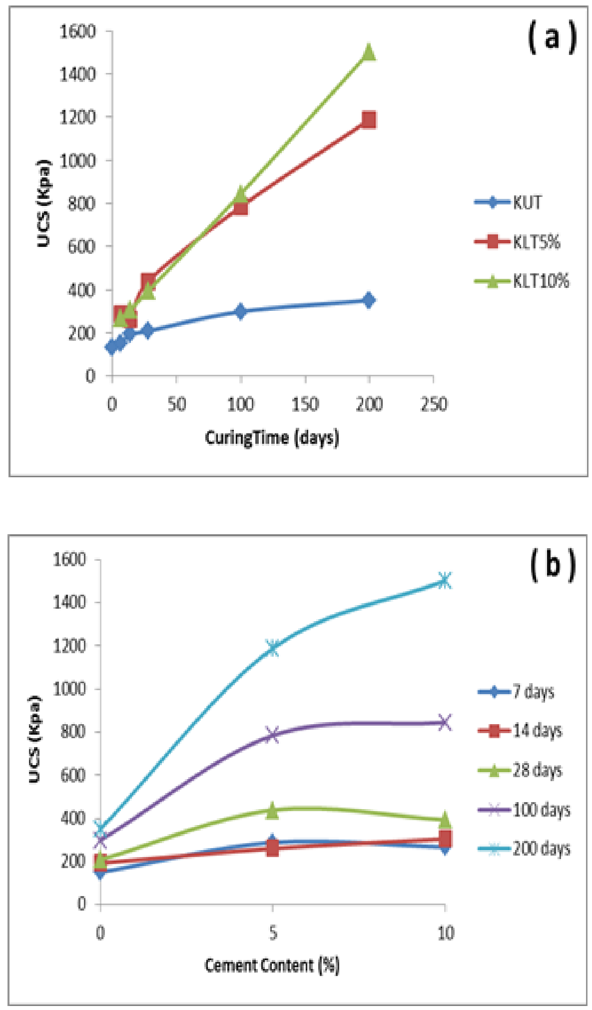


Figure 3 Variation of UCS of lime treated kaolin clay with (a) curing time and (b) lime content

The UCS increased about more than 4 times of the original untreated strength after 200 days curing. The consistent increase in strength of lime treated specimens may be associated with the increase in pH value, which promotes the alkaline condition. Consequently, this allowed pozzolanic reactions to take place for further strengthening of the soil.[4,1]

Figure (3b) demonstrates the influence of the time factor on the development of strength on lime treated samples. As shown in Figure 3b, the effect of curing time on lime treated specimens was relatively consistent as compared to the effect of lime content

(Figure 3a). The significant increases in strength was noted on specimens cured at longer curing time. This is corresponding to the development of cementitous products resulting from pozzolanic reactions as time prolonged.

4.4 Microstructure Analysis

As mentioned in section 2.0, soil strength development of lime treated kaolin clay samples was further verified using the X-ray diffraction (XRD), FTIR spectroscopy and field emission scanning electronic microscopic (FESEM). This section explained the microstructure analysis by XRD, FTIR spectroscopy and FESEM on lime treated kaolin clay samples.

4.4.1 X-Ray Diffraction (XRD)

In this study, powder XRD has been collected to determine the crystalline minerals present in the natural soil and to monitor the mineralogical changes caused by treatment. Samples for XRD test were prepared by grinding the soil using a pestle and a mortar to a fine homogeneous powder. Then it was placed in the elliptical opening of an aluminium holder and evenly distributed using a microscope slide until a smooth surface was achieved as seen in Figure 4.

Figure 4 Samples preparation and apparatus of XRD-Diffraction test

Figure 5 shows the XRD pattern of untreated 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. XRD investigation was conducted for the period of 200 days, to evaluate the processing of minerals at optimum lime content (OLC) of 10%, as shown in Figure 6. A new reflection is attributed to the formation of Gismondine (CASH), resulted from the reaction between the Portlandite and the mixture of soil.[12] The alkaline environment is responsible for the pozzolanic reaction to ensure silica and alumina dissolved from solution. As the time prolonged, pozzolanic reaction continues, thus produce more CASH to increase the strength of the treated 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 retards the bonding between the surface lime and clay particles. Hence, due to this, the partially crystallize CASH, is still not entirely converted to the crystallize compounds CSH and CAH. Furthermore, it can be noticed the presence of calcite, which is resulted from the carbonation of calcium, from the atmosphere. The details results of the untreated and lime treated kaolin clay samples at the 200 days curing time are tabulated in table 3. The mineral compositions are based on their categories as shown in the table 3.

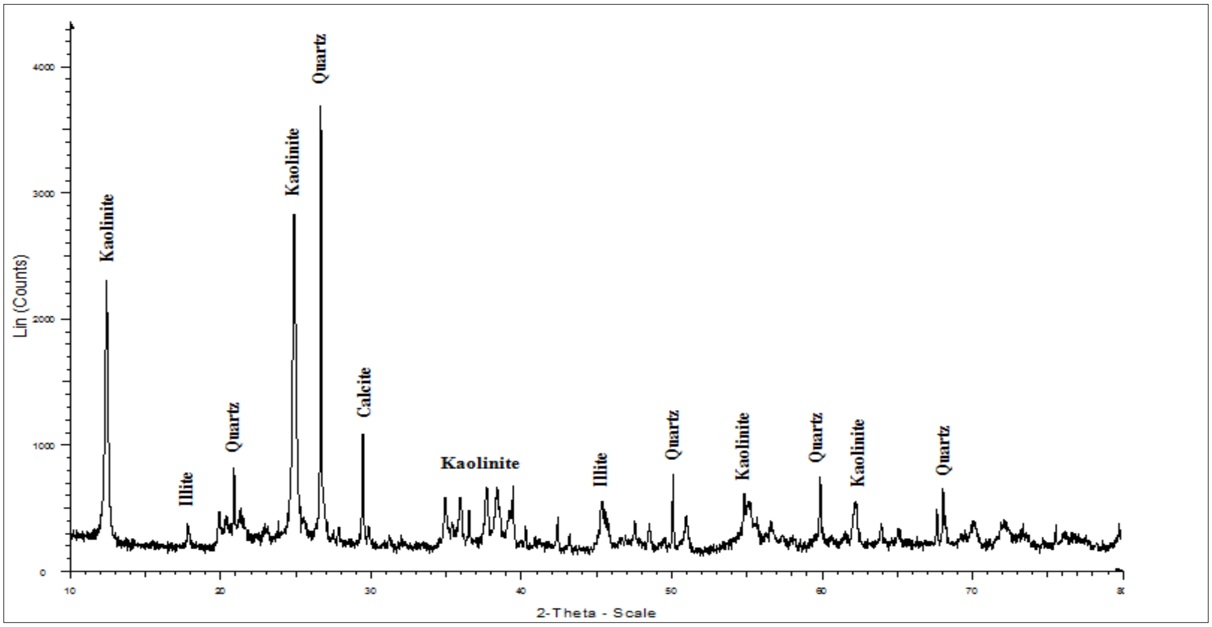


Figure 5 XRD of untreated Kaolin clay soil

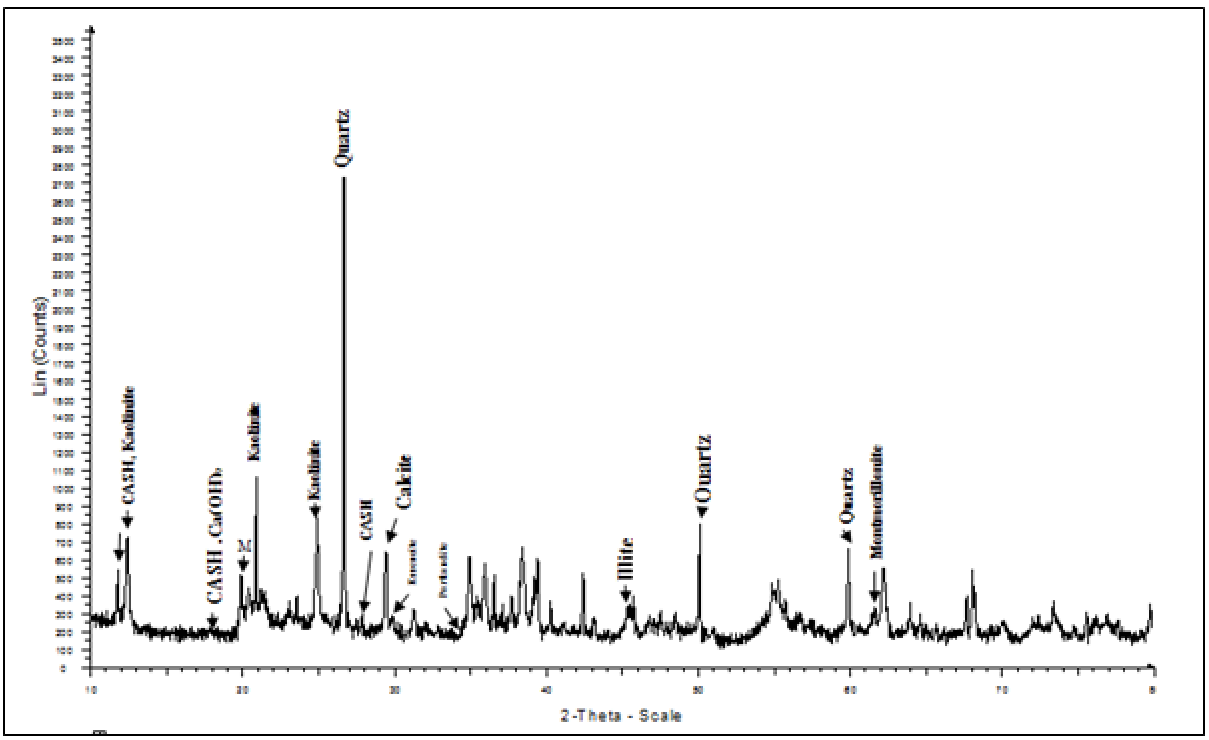


Figure 6 XRD for Kaolin clay samples treated by 10% cement at 200 days curing time

Table 3 XRD of untreated and lime treated Kaolin clay soils

Minerals		Compositions	Category
Un-treated Kaolin clay	**Lime treated Kaolin clay**		
Kaolinite	Kaolinite	$Al_2(Si_2O_5)(OH)_4$	Clay mineral
Calcite	Calcite	$CaCO_3$	Non-clay mineral
Illite	Illite	$(K,H_2O)Al_2Si_3AlO_{10}(OH)_2$	Clay mineral
Quartz	Quartz	SiO_2	Non-clay mineral
	Portlandite	$Ca(OH)_2$	Additives
	Gismondine	$CaAl_2Si_2O_8.4H_2O$	Cementitious products
	Muscovite	$KAl_2(Si_3Al)O_{10}(OH,F)_2$	Clay mineral
	Esseneite	$CaFe[AlSiO_6]$	Non-clay mineral
	Montmorillonite	$(Na,Ca)_{0.3}(Al,Mg)_2Si_4O_{10}(OH)_2$	Clay mineral

The micrograph of natural kaolin clay is presented in Figure (7a). It can be noticed that the neatly arranged book-like kaolinite particle is the predominate feature of the natural soil.[13] The kaolin generally has a to-face aggregate and edge -to-edge flocculated structure.[14,15] The images of 10% lime treated kaolin clay cured for 200 days is shown in figure (7b). It can be observed that clay particles have transformed from flaky form into flocculating structure. Furthermore, the morphology changes at the edges of clay particles and sufficient cementing compounds are due to pozzolanic reaction coated and they are attached to soil particles together, which make the voids in the soil less distinct. In addition, the formation of white lumps compounds on the surfaces of soil particles was apparent.

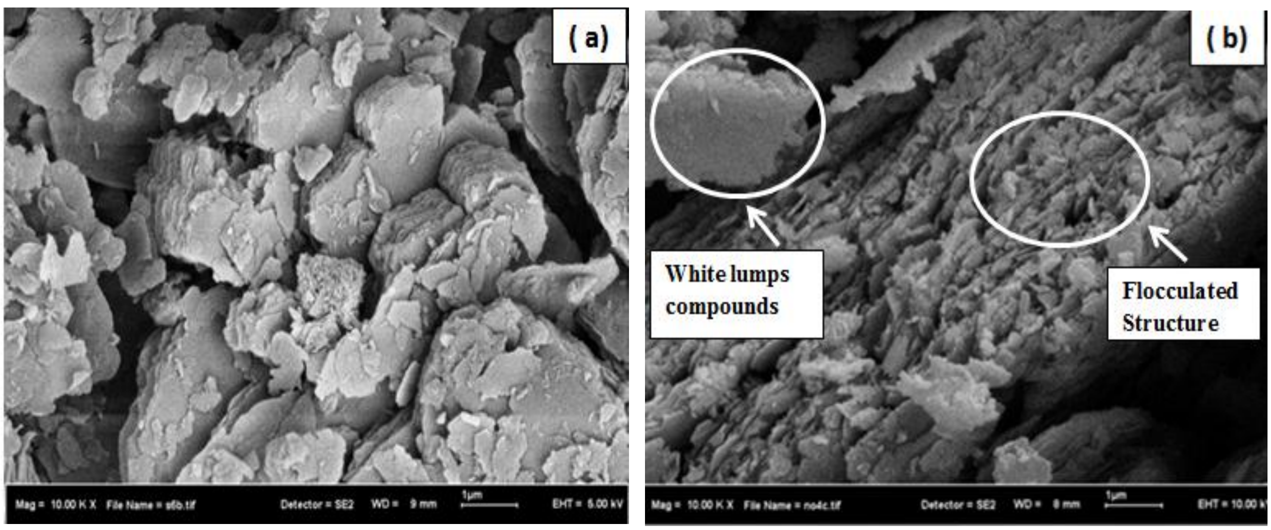


Figure 7 FESEM image of (a) untreated Kaolin clay soil and (b) 10% lime treated Kaolin clay after at 200 days curing time at magnification of X10.000.

4.4.2 FTIR Spectroscopy

Regarding to the Brown Kaolin clay, FTIR spectra from natural Kaolin soil samples are shown in Figure 8. FTIR analysis is carried out to identify the molecular changes in soil sturtures. Two strong bands at 3696 and 3620 cm-1 identified Kaolinite associated with octahedral stretch vibrations from OH. Sharp bands were also observed at 1115 cm-1, 1031 cm-1, and 1007 cm-1 and were attributed to Si-O stretching. The band at 912 cm-1 corresponds with OH deformation of hydroxyl groups. Most other bands, such as Si–O vibrations observed at 794 cm-1, 698 cm-1, 540 cm-1, and 470 cm-1 also confirmed the presence of kaolinite, and another small band at 1422 cm-1 marked the presence of calcite, while bands at 1638 cm-1 marked bonding vibrations of water molecules (H-O-H).

FTIR spectroscopy was also conducted on lime treated Kaolin clay samples as presented in Figure 9. No significant changes in FTIR spectra for lime were observed. Nevertheless, increased intensity of absorption at band 1425 cm-1 did correspond with increased sources of the calcium supplied by lime. Also, it was observed a slight increase in intensity from Si-O stretching due to ongoing degrees of polymerization over time.

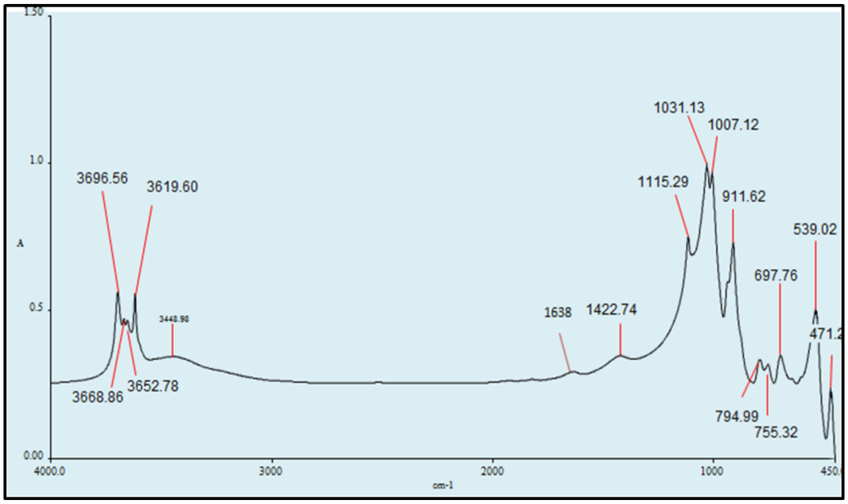


Figure 8 FTIR Spectra for natural Brown Kaolin clay soil

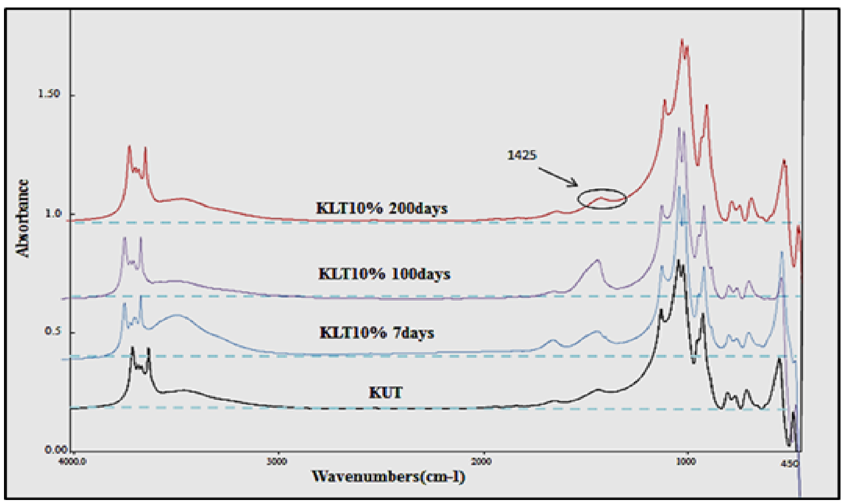


Figure 9 FTIR spectrums of natural and lime treated Kaolin Clay at different time intervals

■5.0 CONCLUSION

Based on the findings, it can be concluded that:

1) The addition of lime has improved the compaction properties of the kaolin clay samples through an increase in the optimum moisture content and a decrease in the dry intensity resulting from the lime content.
2) Two variables influencing the amount of development strength have been studied. These variables are curing time and lime content. Most of the samples show similar performance where the time factors and lime content were proportionally related to each other.
3) The strong alkaline conditions due to the addition of lime to the soil are assumed to be responsible in releasing silica and alumina from clay mineral. Consequently, a new cementitious product which is known as CASH is produced when lime reacts with clay. This is proven by the images obtained from FESEM micrographs, which shows a clear formation of cementitous material. It is believed that this product is able to increase in strength due to the pozzolanic reactions.

Inclusively, the factors that contribute to the effectiveness of lime to stabilize kaolin clay are the lime content, curing time, soil type and clay minerals.

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