

Characterization of phosphoric acid- and lime-stabilized tropical lateritic clay

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Abstract The effect of calcium-based stabilizers such as lime on the geotechnical properties of tropical soils has been reported by many researchers. However, the amount of literature available on the micro-structural, molecular, and leaching characteristics of lime and in particular phosphoric acid-stabilized lateritic clays has been limited. This research was carried out in an attempt to identify the time-dependent soil-chemical reactions. In addition, the possible mechanisms that contributed to the stabilization process were discussed in the light of various spectroscopic and microscopic techniques such as X-ray diffractometry (XRD), energy-dispersive X-ray spectrometry (EDAX), field emission scanning electron microscopy (FESEM), Fourier transform infrared spectroscopy (FTIR), and nuclear magnetic resonance spectroscopy (NMR) etc. Based on the results it was found that in lime-treated samples, the coating action of free iron oxides on clay particles imposed inhibitive effects on the dissolution of clay alumina. On the other hand, in phosphoric acid-stabilized soil, due to the increase in solubility of metal oxides and also the subsequent release of clay alumina the formation of cementitious compounds were more feasible. From engineering point of view, the phosphoric acid-stabilized lateritic soil showed the highest degree of improvement with an approximately threefold strength

increase in comparison with the natural soil over an 8-month curing period.

Keywords Laterite clay · Stabilization · Phosphoric acid · Lime · Molecular structure · Leaching · Coating

Introduction

Tropical soils have significant differences from the more common soils of temperate climates. In these regions, rock weathering is intensive and is characterized by the rapid breakdown of feldspars and ferromagnesian minerals, the removal of silica and bases (Na_2O , K_2O , MgO), and the concentration of iron and aluminum oxides (Mitchell and Soga 2005). This process is termed laterization (Gidigas 1972) and involves leaching of SiO_2 and deposition of Fe_2O_3 and Al_2O_3 . Laterite as a soil group, rather than a well-defined material, is found in these humid tropics where heavy rainfall and warm temperatures under good drainage conditions leads to the formation of thick horizons of reddish lateritic soil profiles rich in iron and aluminium, and kaolinite clays (Townsend 1985). These soils mostly occur at hillside and therefore provide excellent borrow areas for extensive use in various construction activities. However, their optimum use has been limited by a number of difficulties in construction related to their workability, field compaction, and strength.

As reported in previous studies, in comparison to lime treatment, phosphoric acid stabilization is a potentially attractive alternative for treating lateritic soils (Lyons and McEwan 1962; Sutton and McAlexander 1987; Medina and Guida 1995). This is because phosphoric acid reacts with free iron and aluminum oxides present in the soil environment, producing heat and rapid formation of

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insoluble hard cementitious products (Winterkorn 1962). Furthermore, kaolinite as an important aluminum source is one of the major mineral compositions of clay deposits found in tropical regions.

In general, two basic phosphate reactions are distinguished with Al and Fe oxides and hydroxides. These reactions differ in the time scale over which they occur and the stability of products formed. The initial reaction appears to be a fast ligand exchange of surface hydroxyl groups with phosphate and the formation of inner-sphere surface complexes (Laiti et al. 1996; Nanzyo 1984; Persson et al. 1996). On the other hand, for the longer term reactions, two possible mechanisms have been suggested. The first one involves the slow diffusion of phosphate ion into micropores (van Riemsdijk et al. 1984). Another proposed mechanism is the slow weathering of surface in the presence of phosphate and the formation of metal phosphate precipitate (Laiti et al. 1996; Lookman et al. 1997; Li and Stanforth 2000).

Throughout these years considerable research has been carried out in studying the effect of lime stabilization on engineering properties of soils (Balasubramaniam et al. 1989; Locat et al. 1990; Bell 1996; Narasimha Rao and Rajasekaran 1996). However, the use of phosphoric acid as a stabilizer has been limited. This is due to the lack of standard laboratory work on acid stabilization which makes it very difficult to predict their long-term performance.

In the present paper an attempt was made to identify the mechanisms responsible for the improvement of phosphoric acid- and lime-stabilized lateritic soils. Hence, the changes in the micro-structural, molecular, and leaching characteristics of treated soil were monitored at different time intervals. In addition to evaluating the relative success of acid treatment in comparison with alkaline stabilizers, unconfined compressive strength test were performed on various mix designs.

Materials and experimental programme

Materials

The residual lateritic soil used in this investigation was a reddish color clayey soil with high amounts of iron oxides commonly found in tropical regions. The chemical and physical properties of the natural soil are presented in Table 1. The lime used in this investigation was a laboratory-grade hydrated lime. In addition, the phosphoric acid was a Merck analysed, 85% H_3PO_4 , of specific gravity 1.71.

Preparation of specimens

Prior investigations conducted in the laboratory on the lateritic soils showed that oven drying significantly changes its plasticity and compaction properties (Eisazadeh 2010). Therefore, all specimens were prepared from air-dried soil. The air-dried soil was then broken into smaller sizes and sieved through a 2-mm sieve. The sieving was done to ensure that the soil was of uniform grade throughout the experiment. Also, based on the literatures published on acid- and lime-stabilized soils (Medina and Guida 1995; Bell 1996), two percentages of acid and lime content (i.e. 3 and 7%) by weight of the dry soil was selected for the treatment.

In this research, a standard and rational step by step protocol was used for preparing various mix designs. Also to ensure that any observed changes and in particular the strength developments were attributable solely to the action of the stabilizers on the soil particles, the possible contributions caused by the variations in water content and compactive effort needed to be eliminated. Hence, first of all, the required amount of water known as optimum water content (OWC) was determined for the natural soil in

Table 1 Characteristics of the natural laterite clay

Engineering and physical properties	Values	Chemical composition (oxides)	Values (%)
pH (L/S = 2.5)	4.86	SiO ₂	21.55
Specific gravity	2.75	Al ₂ O ₃	24.31
External surface area (m ² g ⁻¹)	57.89	Fe ₂ O ₃	29.40
Liquid limit, LL (%)	75.8	Na ₂ O	0.07
Plastic limit, PL (%)	39.60	K ₂ O	0.11
Plasticity index, PI (%)	36.20	P ₂ O ₅	16.71
BS classification	MH	SO ₃	3.98
ICL (%)	5.00	CO ₂	3.65
Maximum dry density (mg m ⁻³)	1.33	Soluble phosphorus (P)	0.40 ppm
Optimum moisture content (%)	34.00	Soluble aluminum (Al)	0.05 ppm
Unconfined compressive strength (kPa)	288.10	Soluble silica (SiO ₂)	0.10 ppm
Loss of ignition (%)	6.32	Soluble calcium (Ca)	0.10 ppm

accordance with clause 3.3.4.2 of BS 1377: Part 4: 1990a. Second, in the case of acid stabilization, the air-dried soil was directly mixed with the required amount of water and phosphoric acid until a uniform mixture was achieved, whereas in lime mix designs, prior to addition of water, the soil and lime were first mixed thoroughly in the dry state. Also based on laboratory trials an additional quantity of water (2% wet of the optimum water content) was added to all mix designs to take into account the loss due to evaporation. Finally, after compacting the samples into thin wall PVC tubes under constant compactive effort as specified in clause 4.1.5 of BS 1924: Part 2: 1990b, they were wrapped with thin plastic film and sealed to the atmosphere with rubber tight lids. The samples were then stored in a controlled temperature room ($27 \pm 2^\circ\text{C}$) until required for testing at each of the three curing periods, 1, 4, and 8 months. In addition, for more convenience in the presentation of the results, a specimen designation scheme was used. Letters in the specimen designation indicated soil name and type of treatment, respectively (i.e., LC: laterite clay, AT: acid treated, LT: lime treated, M: months).

Testing programme

In general, the characterization studies carried out in this investigation can be divided into three main categories, i.e., micro-structural, molecular, and leaching analyses which are comprehensively discussed in the following paragraphs.

The main goal of extraction test was to monitor relative changes in the concentration of elements released from soil-stabilizer matrix at different time intervals. The test was performed based on a procedure slightly modified from the Environmental Society of Canada (EC 1990) known as equilibrium extraction (EE) test, which allowed the determination of soluble elements using distilled water as the leachant. The leaching was conducted at a liquid to solid ratio of 8. For each test, 20 g of solid particles was mixed with distilled water using a magnetic stirrer for 24 h. Then, after filtration, pH was measured, and the supernatant were preserved for ICP analysis of the four main elements, i.e., Si, Al, P, and Ca.

In this paper, powder X-ray diffraction technique was employed to assess the mineralogical changes induced by stabilizers on the soil structure and also to detect new crystalline compounds that were formed during the stabilization process. The XRD analysis of cured samples was carried out by using a Bruker D8 advance diffractometer. The scans were performed using $\text{CuK}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$) at an angle scan (2θ) between 6° and 90° with a 0.02° step size and dwelling time of 1 s at each step. The patterns obtained were compared with standard dataset

of the Joint Committee for Powder Diffraction Standards (JCPDS 1995).

A JSM-6701F JEOL field emission scanning electron microscope (FESEM) equipped with energy-dispersive X-ray spectrometer (EDAX) was used to obtain high-resolution images of soil fabric prior and after treatment. Each sample was sputtered with platinum for 120 s at 30 mA under high vacuum until they were completely covered and ready to be used for the microscopic analysis. The EDAX analysis was also carried out to detect major elemental changes on the surface of treated particles. The results were presented based on Al:Si, Ca:Si, and P:Si ratios at different time intervals.

Fourier transform infrared spectroscopy (FTIR) analysis was performed in an attempt to study the changes in the molecular structure of treated samples. Hence, 2 mg of dried soil was ground in an agate mortar and mixed with 200 mg KBr. The transparent pressed KBr disc (pellet) was then placed in the path of the infrared spectra and scanned using a Perkin Elmer Spectrum 2000 instrument. Finally the adsorption bands were recorded at characteristics wave numbers between 400 and $4,000 \text{ cm}^{-1}$.

The ^{27}Al and ^{29}Si MAS NMR spectra of acid and lime-treated samples were measured in order to better elucidate the stabilization process, by determining the local structure around the Al and Si atoms. All measurements were performed on a Bruker AVANCE 400 MHz solid-state NMR instrument, equipped with a MAS probe. The samples were confined in a zirconium rotor with 7 mm diameter.

Unconfined compressive strength (UCS) test was used as an index of soil enhancement, with at least three specimens being tested for each mix design. It was also used to determine the UCS of the natural soil compacted at the optimum moisture content. The test was carried out in accordance with BS 1924: Part 2: 1990. Hence, after reaching the specified curing time, samples were taken from the thin wall PVC tubes and directly tested at an axial strain rate of 1% per min. During the test, the applied load and changes in the axial deformation were recorded automatically by the data acquisition unit (ADU) with failure being defined as the peak axial stress. At the end of each test, the failed soil specimen was dried and weighed to determine its moisture content.

Results and discussion

In recent years, several investigations on the leaching characteristics of soil–lime mixtures have been reported (Boardman et al. 2001; Goswami and Mahanta 2007). However, very few attempts to understand the leachability of phosphoric acid-stabilized lateritic soils have been made. The concentrations of soluble Si, Al, P, and Ca

released from treated samples into the aqueous solution are shown in Fig. 1. As can be seen, the 7% phosphoric acid-stabilized soil released approximately 66 ppm soluble P at the early stages of curing while reaching an optimum of approximately 171 ppm after 4 months' curing. The former was probably due to the reaction of free iron and aluminum oxides present in the soil environment with phosphoric acid which resulted in a relatively lower P concentration at 1-month curing period. On the other hand, considering the weathering action of the acid on the soil structure and bearing in mind the high phosphate content of the tropical soil, an increase in the level of soluble P with curing time was justifiable. With further curing to 8 months, a downward trend in the soluble P concentration was observed. On the other hand, in the 7% lime-stabilized soil, the soluble Ca concentration decreased sharply with curing time. The reduction in the level of stabilizer ions extracted from soil matrix was consistent with the formation of new reaction products which rendered them less soluble. Consideration of the soluble Al data in both graphs

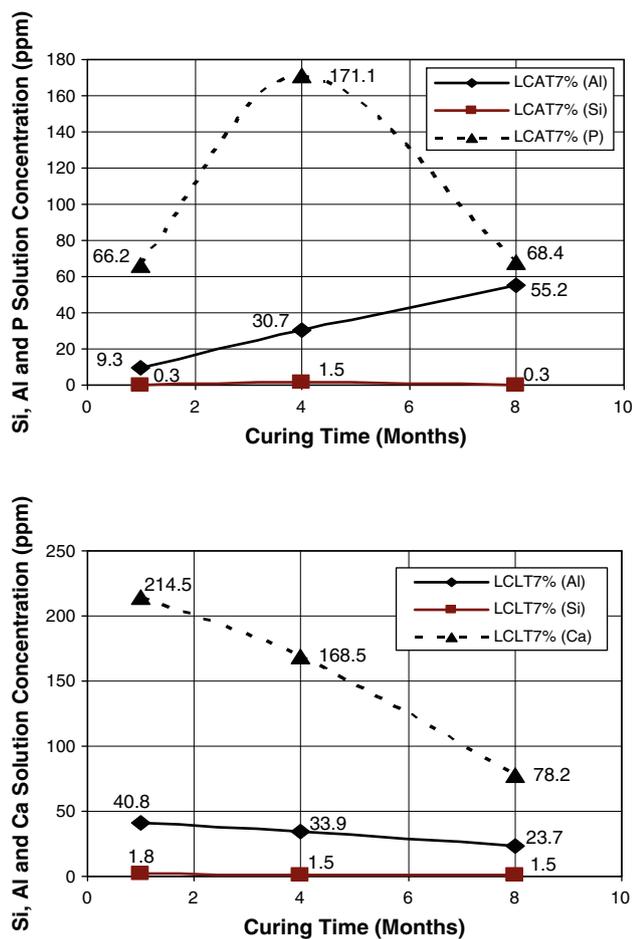


Fig. 1 Concentration of soluble Al, Si, P or Ca in the aqueous solution with respect to phosphoric acid and lime treatment following 1, 4, and 8 months' curing period

revealed some interesting points. As apparent, the Al release for acid-treated samples seemed to increase with curing period while in contrast, the Al concentration of solutions stabilized with lime showed a decrease. The former can be attributed to the highly acidic nature of pore water which resulted in the dissolution of clay alumina and the free aluminum oxides present in the soil environment. While the latter was likely to be caused by the coating action of free oxides and their lower dissolution in an alkaline environment in comparison with the acidic medium which prevented the attack of lime on the clay mineral structure.

X-ray diffraction (XRD) patterns for the natural and chemically treated lateritic soil are illustrated in Fig. 2. As can be seen, kaolinite ($2\theta \approx 12.5^\circ, 18.5^\circ, 20^\circ, 36^\circ, 63^\circ$), quartz ($2\theta \approx 21.5^\circ, 25^\circ, 38^\circ$), goethite ($2\theta \approx 18.2^\circ, 21.8^\circ$), and calcite ($2\theta \approx 29.4^\circ, 43.2^\circ$) were the major minerals present in the laterite clay samples (Brown 1961). On the other hand, with respect to the natural soil, no fundamental changes in the XRD patterns of acid- and lime-treated samples were observed. Nevertheless, there was a slight decrease in the intensity of peaks for kaolinite mineral. This was probably caused by the attack of stabilizers on mineral lattice. Furthermore, several new reflections regarding the formation of new products at various 2θ angles were evident. These peaks were observed at $27.2^\circ, 35^\circ$, and 66° 2θ for phosphoric acid and at $14.9^\circ, 28.8^\circ$, and 29.5° 2θ for lime-treated samples. The former peaks were tentatively assigned to aluminate and iron phosphate hydrates and the latter to calcium aluminate hydrate compounds (JCPDS 1995).

Energy-dispersive X-ray (EDAX) analysis was performed in order to obtain more information regarding the surface composition of treated particles. The time-dependent changes in the Al:Si, Ca:Si, and P:Si ratios of various mix designs are shown in Table 2. In addition, EDAX spectrums corresponding to the natural and 7% lime and phosphoric acid-treated lateritic soil are presented in Fig. 3a–c, respectively. As can be seen, high intensities of silicon (Si), aluminum (Al), phosphorus (P), and iron (Fe) peaks are clearly evident in both the untreated and treated samples. The concentration of these elements and in particular the iron element confirmed the lateritic nature of the soil and moreover the presence of free iron oxides on the surface of clay particles (Carroll and Starkey 1971). The lime-treated samples also contained calcium (Ca). The presence of calcium is consistent with the application of lime into the soil environment. It should be noted that the high intensities of platinum (Pt) peak was due to the thickness of coating which was taken into consideration when determining Al/Si, Ca/Si, and P/Si ratios. As shown in Table 2, for all design mixes, the Al:Si ratio varied <10% between the untreated and treated samples. These

Fig. 2 XRD patterns for the natural and 8 months' cured lime and phosphoric acid-treated samples

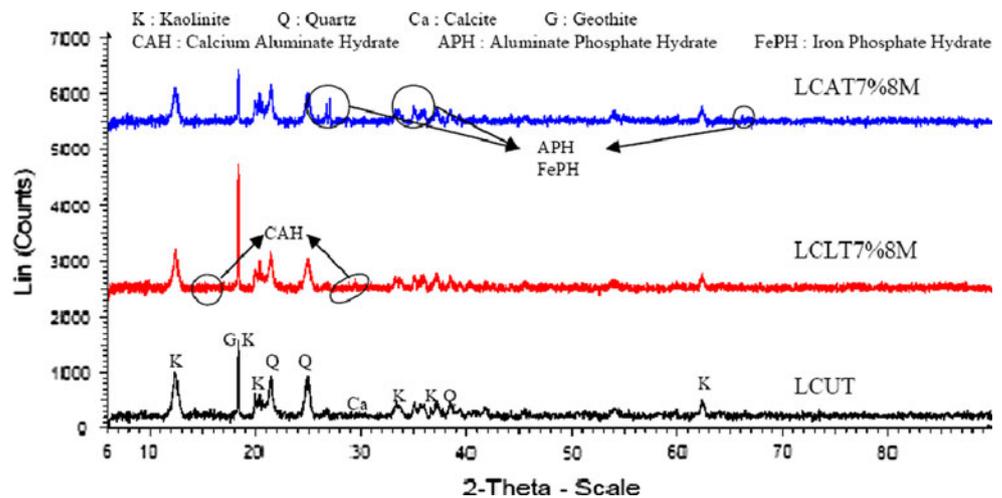


Table 2 Al:Si, Ca:Si, and P:Si ratios of various mix designs obtained from EDAX analysis

Sample description		Ratio (wt%)		
Mix design	Curing time (months)	Al:Si	Ca:Si	P:Si
LCUT	–	1.13	0.00	0.77
LCAT7%	1	1.22	0.00	0.55
LCLT7%	1	1.05	0.94	0.58
LCAT7%	4	1.10	0.00	0.46
LCLT7%	4	1.12	0.66	0.47
LCAT7%	8	1.12	0.00	0.72
LCLT7%	8	1.21	0.39	1.00

LC laterite clay, LT lime treated, AT acid treated, UT untreated

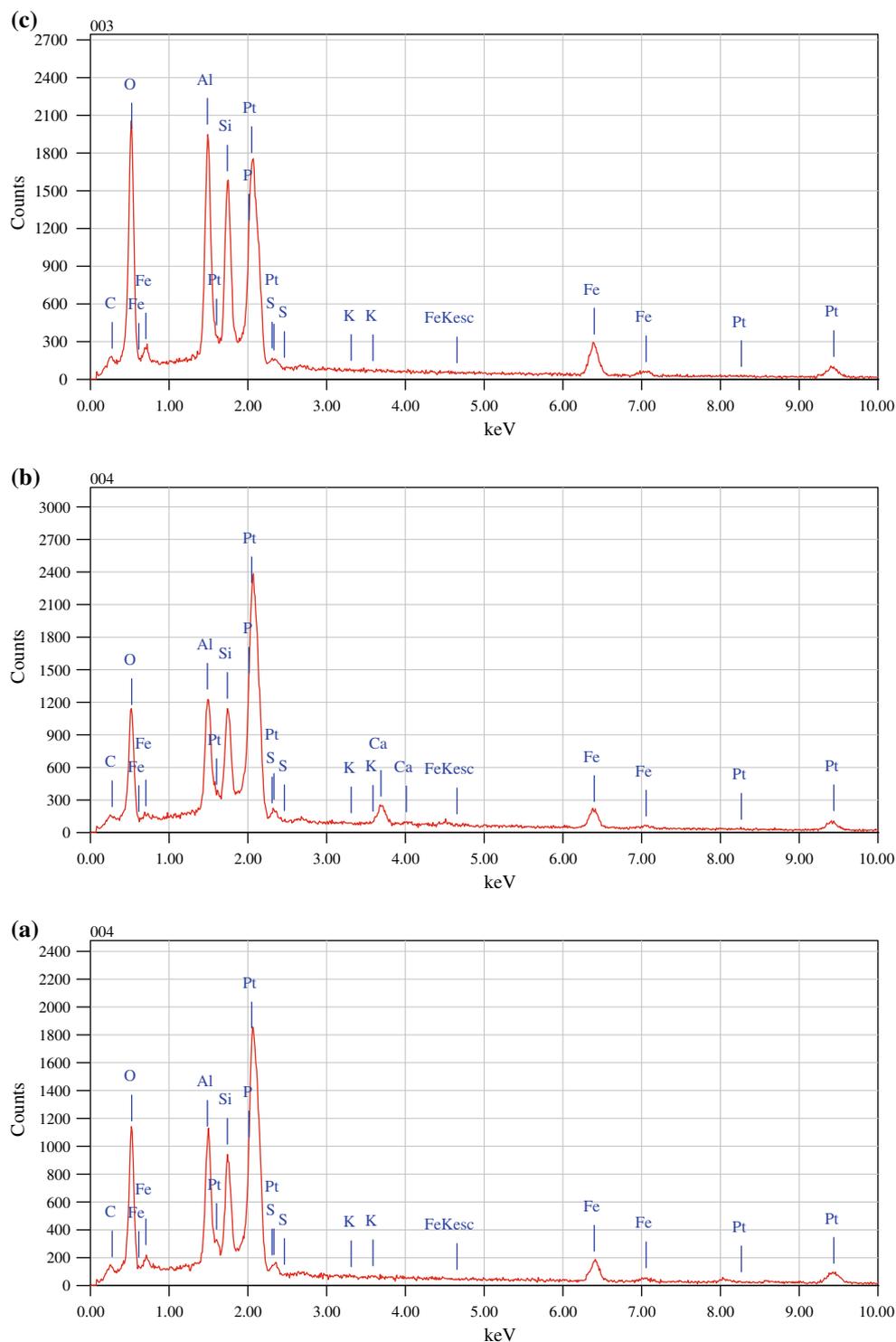
results suggested that the soil structure did not change significantly following the treatment with chemical stabilizers. However, as indicated by the P:Si and Ca:Si ratios, there was an appreciable change in the surface composition of clay particles. As can be seen for the acid-treated mixes the P:Si ratio decreased after 4 months of curing. This behavior suggested an increase in the sources of silica present at the surface of clay particles due to the dissolution of the coating. At 8 months' curing period, the P:Si ratio showed a noticeably higher value. This was tentatively caused by the formation of a gel-like phosphate hydrate compound on the surface of clay particles. On the other hand, a decrease in the amount of calcium present at the surface of clay particles was believed to be the main cause of reduction in the Ca:Si ratio of lime-treated samples from 0.94 to 0.39 on increasing the curing period from 1 to 8 months.

In this paper, FESEM was used to study the morphology of stabilized soil. As can be seen in Fig. 4, the free oxides present in the untreated soil (LCUT) environment have coated and bonded the clay particles together. On the other

hand, from the images of lime and phosphoric acid-treated samples after 8 months of curing, it was apparent that the vigorous action of acid on clay minerals and in particular the free oxides have transformed the soil structure into a more integrated composition consisting of sharper edged particles. Furthermore, in lime-treated samples, the soil fabric revealed a more particle-based microstructure. In addition, the formation of new compounds in the form of white lumps was evident in both mix designs (Willoughby et al. 1968; Rajasekaran and Narasimha Rao 1997).

Fourier transform infrared spectroscopy (FTIR) can be used to determine the functional groups of soil minerals by measuring the absorption bands at characteristic wavelengths of bonds that vibrate independently of one another. The common features of FTIR spectrums (Fig. 5) for natural and chemically stabilized lateritic soil were as follows: The bands at 1,105 and 1,032 cm^{-1} corresponded to the perpendicular and in-plane Si–O stretchings. Peaks at 3,620 and 3,696 cm^{-1} were attributed to the OH stretching vibrations of inner and inner-surface hydroxyl groups, respectively (Madejova and Komadel 2001). The latter band is characteristic of a kaolinite mineral, while the former is commonly found in many different phyllosilicate minerals. The band at 913 cm^{-1} suggested the presence of hematite (Gadsen 1975). There was also some quartz present as indicated by the band at 796 cm^{-1} (Marel and Beutelspacher 1976). Most of the other bands such as the Si–O vibrations observed at 698, 540, 470, and 430 cm^{-1} confirmed the presence of kaolinite mineral. The remaining bands were assigned to water vibrations. Band at 3,446 cm^{-1} was a stretching vibration, whereas the 1,638 cm^{-1} band was a H–O–H bending band of water with an overtone occurring at 3,378 cm^{-1} . As can be seen from the FTIR spectrum of 8 months' cured samples, the chemical treatment did not cause any major changes in the functional groups of soil particles. However, in the lime-

Fig. 3 EDAX spectra of **a** untreated, **b** lime-treated and **c** phosphoric acid-treated laterite clay



stabilized soil, a new absorption band at $1,385\text{ cm}^{-1}$ with a weak intensity attributed to the Ca–OH bond of lime was evident (Nacamoto 1970).

As can be seen, in the ^{27}Al NMR spectrum of the natural lateritic soil (Fig. 6), a broad peak at 70 ppm corresponding to the tetrahedral Al, and a relatively sharper peak at

approximately 0 ppm arising from the octahedral Al were observed (Okada et al. 2006). The intensity of octahedral peak supported the presence of kaolinite mineral with 1:1 silica:alumina structure in the soil environment. After 8 months of curing, the spectrum of phosphoric acid-treated samples revealed slightly different local structure to

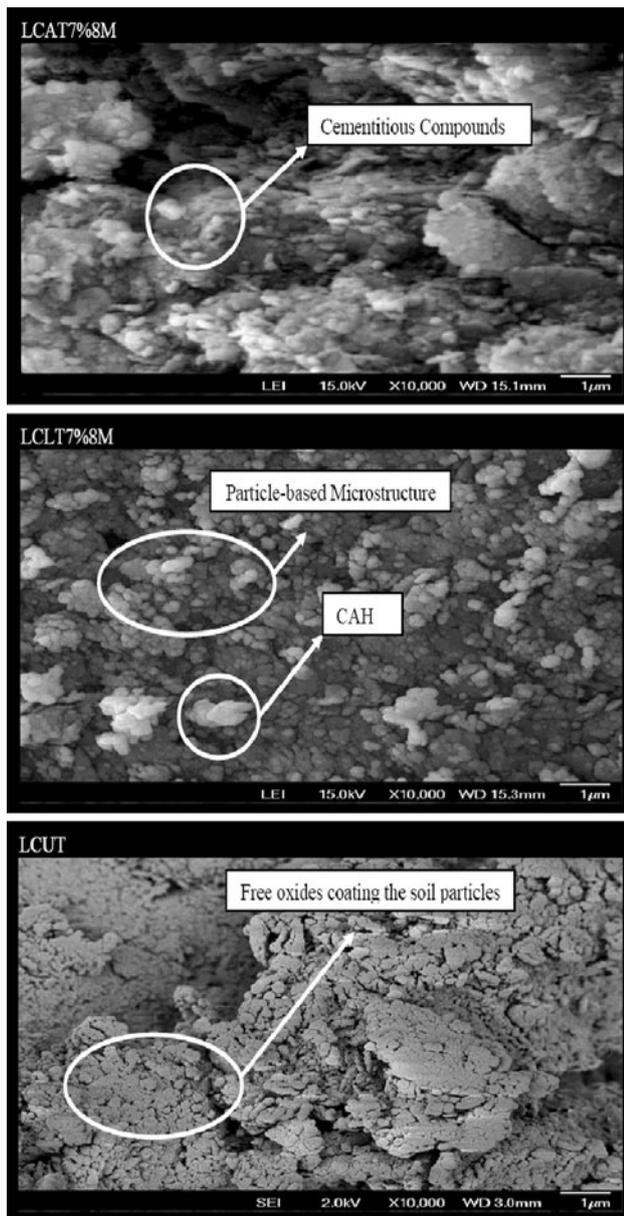


Fig. 4 FESEM images of untreated (LCUT), lime-treated (LCLT7%8M), and phosphoric acid-treated soil (LCAT7%8M) at 8 months' curing period

that of the natural soil. That is a tetrahedrally coordinated Al band at approximately 73.5 ppm and an octahedrally coordinated Al band at 5.5 ppm. However, for lime treatment the molecular structure was rather similar to that of the natural soil. The ²⁹Si NMR spectra of untreated and lime-treated laterite clay are shown in Fig. 7. As apparent, the deconvoluted spectrum of the natural soil revealed a major peak at -92 ppm attributed to the bi-dimensional (Q₃) clay structure. Although, after 8 months of curing, some marginal variation in the size of this peak was observed, however, no new peaks or chemical shifts in the spectrum of lime-treated samples were evident.

The unconfined compressive strength of acid- and lime-stabilized laterite clay at different time intervals are shown in Fig. 8. It was apparent that in overall the phosphoric acid treatment was more effective in improving the strength characteristics of the natural soil. As can be seen, the 7% phosphoric acid-treated samples gained a compressive strength of 843 kPa (approx. 3 times of the untreated soil strength) in comparison with the 633 kPa achieved for the same amount of lime over the 8 months' period. This was attributed to the reactions between the free phosphate ions present in the soil medium and the released clay alumina that resulted in the formation of aluminite phosphate hydrate compounds that bonded the soil particles together. Furthermore, at the last 4 months of curing, a marginal increase in the strength of 7% lime-treated samples was observed, whereas, for samples containing 7% phosphoric acid, the strength raised by approximately 40% in this period. The coating action of free oxides which impeded the progression of pozzolanic reactions was believed to be the main reason responsible for this type of behavior.

Conclusions

In this research, a variety of analytical techniques that were linked to the micro-structural, molecular, and leaching characteristics of the soil were performed on lime and phosphoric acid-treated lateritic soil. This was carried out in an attempt to identify the mechanisms that contributed to the stabilization process.

According to the data obtained for laterite clay mix designs, it was found that the stabilization process is highly sensitive to the impurities present on the surface of clay particles. For instance, the coating action of iron oxides was believed to have a detrimental effect on the progression of pozzolanic reactions which resulted in a marginal gain of strength at the last 4 months of curing. This was consistent with the results obtained from leaching test which indicated that the highly alkaline conditions caused by application of lime had little effect in dissolving the free oxides. The role of free oxides in providing an adsorbing surface complex was also confirmed by EDAX, FTIR, and NMR analysis which suggested that a surface modification phenomenon over a long curing period was mainly responsible for the strength development. On the other hand, in phosphoric acid-treated samples due to an increase in the solubility of metal oxides in a low pH environment and also the subsequent release of clay alumina the formation of cementitious compounds were feasible. This was in a good agreement with XRD and FESEM results which revealed the presence of new products in soil-stabilizer matrix. These products were tentatively assigned as aluminite and iron phosphate hydrate compounds.

Fig. 5 FTIR spectra for untreated, phosphoric acid and lime-treated lateritic soil at 8 months' time interval

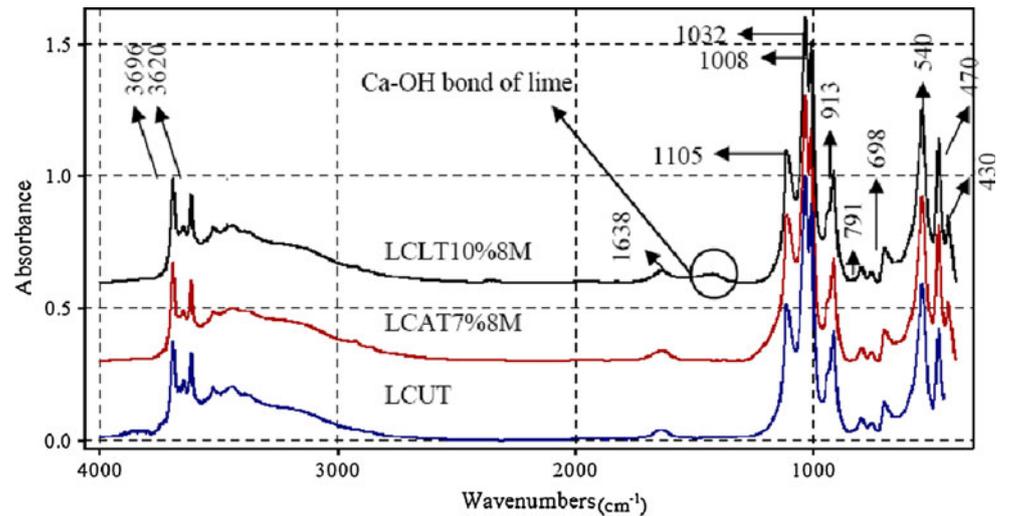


Fig. 6 ²⁷Al MAS NMR spectra of the natural, phosphoric acid and lime-treated lateritic soil

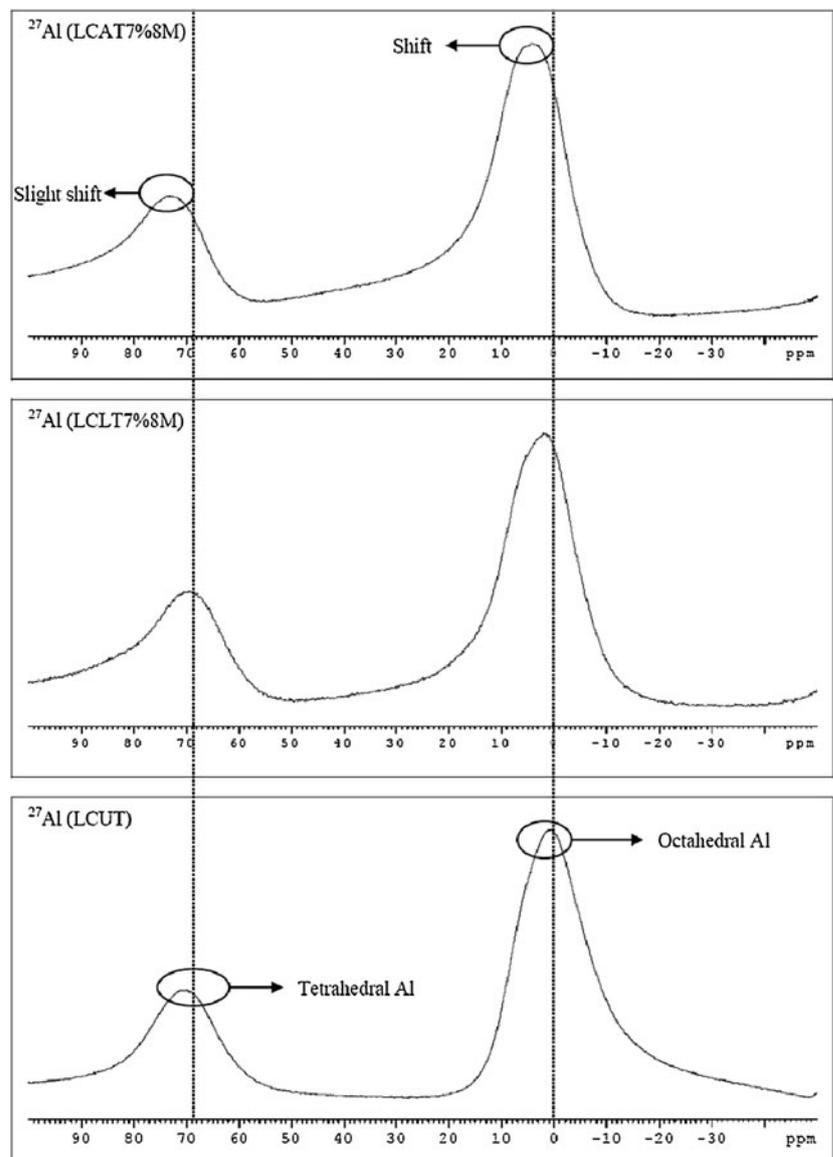


Fig. 7 ^{29}Si MAS NMR spectra of the natural and lime-treated lateritic soil

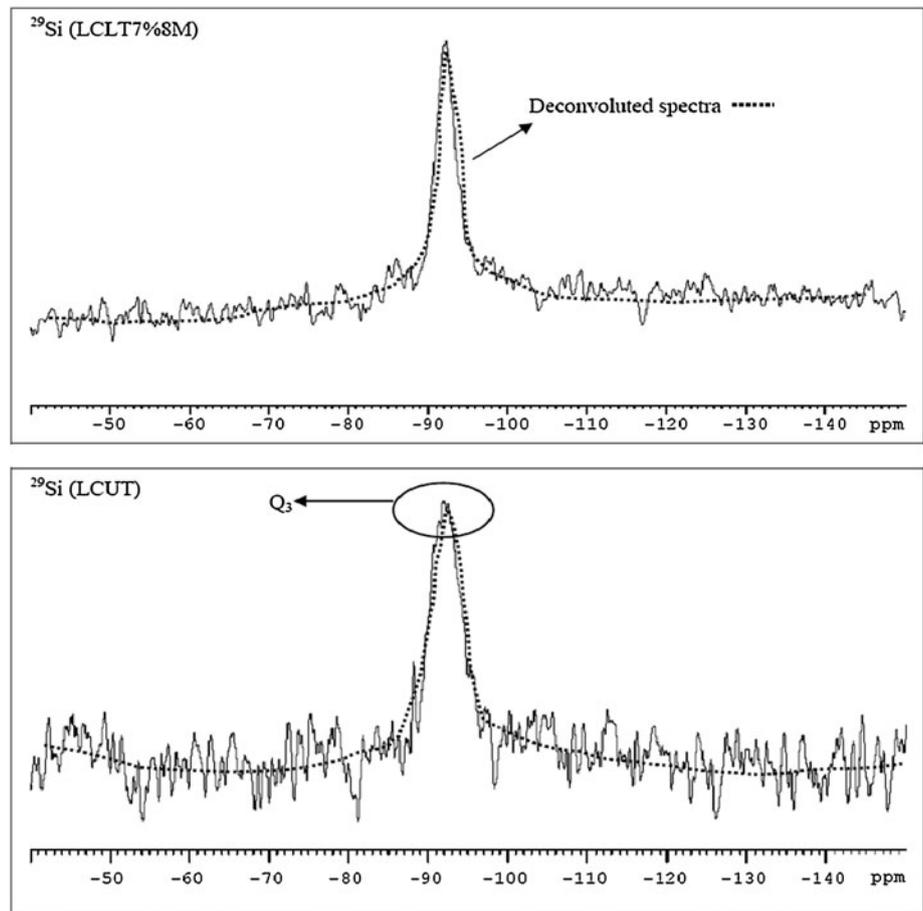
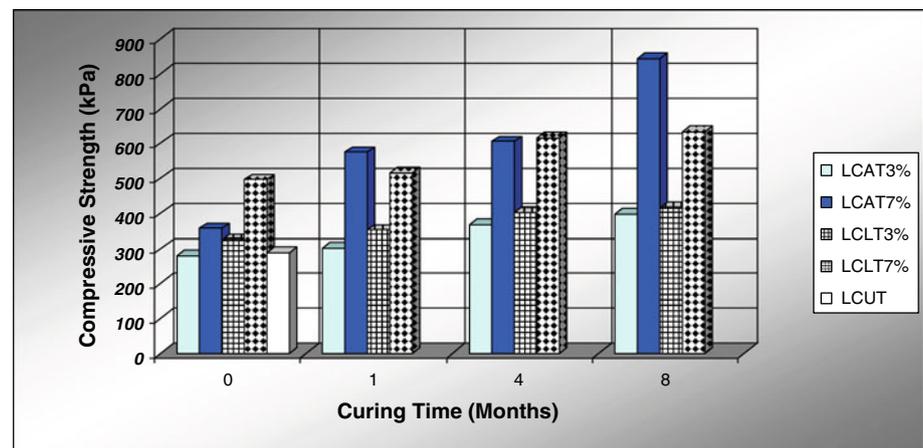


Fig. 8 Strength gain for lime and phosphoric acid-treated laterite clay with different stabilizer content and curing time



Finally, according to the unconfined compressive strength test, the 7% phosphoric acid-treated sample resulted in an almost threefold strength improvement in comparison with the natural soil. The latter indicated that phosphoric acid can be used in a wise manner as an alternative to alkaline stabilizers for treating acidic lateritic soils without having to remove the weak soil and replacing it with a superior material.

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