

FORMATION OF CEMENTITIOUS COMPOUNDS IN LIME - RICE HUSK ASH - SOIL MIXTURES

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ABSTRACT

Even though some studies have been carried out on the reactions involved in lime-flyash mixtures, no information is available on the reaction products of lime-rice husk ash treated soil. This paper describes reaction products in the stabilization of soil by adding lime and rice husk ash to improve the engineering properties of the soil. X-ray diffraction and scanning electron microscopy techniques were used in the study. Samples were analysed at different stages of curing. The results are presented and discussed.

INTRODUCTION

Many procedures have been developed to improve physical properties of soils in which a wide range of stabilizing agents, additives and conditioners have been incorporated with the soils. Undoubtedly the most widely applied methods involve the use of inorganic cementive agents which rely for their effectiveness on the formation of cementive bonds between particles in the soil system. The two most common cementive stabilizing agents are normal Portland cement and hydrated lime. In the application of each of these materials, the soil components and the stabilizing agent are both involved in the chemical changes responsible for the soil alteration.

In recent years use of various waste products in civil engineering construction has gained considerable attention in view of the shortage and high costs of suitable conventional aggregates, increasing costs of waste disposal and environmental constraints.

Rice husk is a major agricultural by-product obtained from the foodcrop of paddy. For every four tonnes of rice produced, one tonne is rice husk. Rice husk has a chemical composition which typically corresponds to the following:- (1) cellulose (40-45%), lignin (25-30%), ash (15-20%) and moisture (8-15%). The ash is derived mainly from the opaline which is present in the cellular structure of husk and about 90% of which is silica. The silica content in the rice husk depends on the following: (a) variety of the rice: (b) soil and climate conditions: (c) prevailing temperature: and (d) agricultural practices ranging from application of fertilizers and insecticides and the like. The normal method of conversion from husk to ash is by incineration. Houstin (1972) reported that the properties of rice husk ash depend greatly on whether the husks have undergone complete destructive distillation or have only been partially burnt. He had classified rice husk ash into (1) high-carbon char, (2) low carbon (gray) ash and (3) carbon-free (pink or white) ash.

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The silica content of the husk can be enriched by converting the husks into ash. A number of researchers (Bartha and Huppertz, 1974, Singh 1977, Shah et al, 1979, Yeoh et al, 1979 and Ibrahim and Helmy 1981) have studied the physical and chemical properties. Chemically, rice husk ash consists of 82-87% of silica and exceeds that of fly-ash. The high percentage of siliceous material indicates that rice husk ash can be an excellent material for stabilization as previous research on fly-ash shows that the stabilized strength depends upon the percentage of silicon and aluminium oxides in the fly-ash (Goeker et al, 1956, Vincent et al, 1961 and Mateos and Davidson 1962).

Even though some studies have been carried out on the reactions involved in lime-flyash mixtures, no information is available on the reaction products of lime-rice husk ash treated soil.

MATERIALS

Rice Husk Ash (RHA)

Rice husk is generally considered a worthless by product of rice milling. At the mills, disposal of the husk is by burning in heaps near the mills. Before use the ash was oven-dried at 60°C. The properties of the ash are shown in Table 1.

Table 1 Composition and Properties of Open Field Burnt Ash

SiO ₂	90.73%
Ca (as an element)	0.022%
Fe (as an element)	0.226%
Cu (as an element)	0.002%
Mn (as an element)	0.042%
Moisture content	1.68%
Loss of ignition	2.70%
Spacific gravity	2.12%
Fineness, amount retained when wet sieved:	
BS No 30 (600 µm)	13%
BS No 200 (75 µm)	69%

Lime

The lime used is hydrated lime commercially named as "White Horse". The pertinent properties of the lime as supplied by the manufacturer conformed to the requirement of Type 1 Grade A lime as specified in the standard specification for lime for soil stabilisation (AASHTO M 216-68). The properties of the lime are given in Table 2.

Table 2 Composition and Properties of Hydrated Lime

Ca(OH) ₂	90 ± 1%
CaCO ₃	6%
Mg(OH) ₂	3%
Fe ₂ O ₃	0.15%
Al ₂ O ₃	0.5%
SiO ₂	0.9%
Moisture content	0.5%
Passing BS sieve:	
BS NO 100	99%
BS NO 200	95%

Soil

Residual granite soil, typical of the Malaysian residual soils, was used in the study. Table 3 shows the properties of the soil. The particle size distribution curve of the soil is shown in Fig. 1.

Table 3 Properties of Residual Granite Soil

Grain size distribution:	
Gravel	1%
Sand	52%
Silt	15%
Clay	32%
Physical properties:	
Natural moisture content	27%
Specific gravity	2.68%
Linear shrinkage	15%
Liquid limit	73%
Plastic limit	36%
Plasticity index	37%
Soil classification:	
Unified	SF
Textural	Sandy clay
AASHO	A-7-5
Engineering properties:	
Modified AASHO density,	1.71 Mg/m ³
Optimum moisture content	14.7%
Unsoaked CBR	32%
Soaked CBR	2%
Swell	1.97 mm

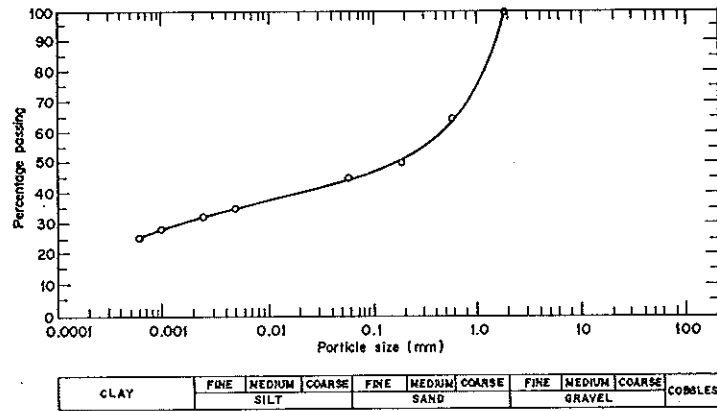


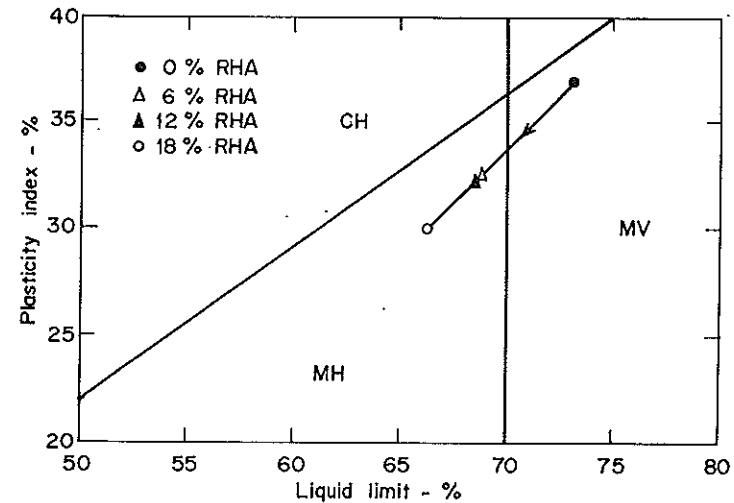
Fig. 1 Particle Size Distribution of the Soil.

GEOTECHNICAL PROPERTIES

Atterberg limits tests, modified AASHTO compaction and unconfined compression were carried out on the soil with different mix proportion of RHA and lime (by weight of dry soil). From Fig. 2 it can be seen that addition of RHA reduces both plasticity index and liquid limit. Fig. 3 shows that linear shrinkage decreases with increasing RHA content. In the compaction tests, for a given RHA content the maximum dry density is decreased but optimum moisture content is increased, by the addition of lime. By increasing the RHA content, maximum dry density is decreased further and at the same time the optimum moisture content increases. This is clearly illustrated in Fig. 4.

The unconfined compressive strength is the most popular method of evaluating the effectiveness of stabilization. As a general rule, for a given type of stabilization the higher the compressive strength, the better the quality of the stabilized material. Each specimen used in unconfined compression tests was compacted in the 50 mm by 100 mm mould at optimum moisture content and the modified AASHTO maximum dry density. Specimens were cured in groups of three, in a thermostatically controlled incubator set at 30° ± 2°C before being loaded in compression. Curing times adopted were 7, 28 and 56 days, and at least three specimen were tested for each case. In Fig. 5 a general pattern is observed in which the strength develops rapidly with addition of RHA until an optimum is reached, beyond which, the strength begins to decrease. The maximum strength significantly varies with curing time. However there is only a slight variation in optimum RHA content with curing time i.e. The optimum RHA content varies between 6% to 9% for the curing periods considered.

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CH : Clay of high plasticity
 MV : Silt of very high plasticity
 MH : Silt of high plasticity

Fig. 2 Plasticity Chart Showing Changes in Plasticity of Rice Husk Ash Treated Soil Samples.

The drop in strength due to further increase in RHA after the optimum amount may be attributed to the decrease in the maximum dry density as a result of the presence of RHA in excess of the amount required for reaction with the available lime.

Fig. 6 shows the variation of unconfined compressive strength with curing period. 6% RHA content gives an average increase in strength of 35% as the curing time is increased from 7 to 28 days at 30°C, while 9% RHA content gives an increase of 49% for the same increase in curing time.

MINERALOGY

X-Ray Diffraction Analysis

Hilt and Davidson (1961) studied trial mixtures of lime and soil at different moisture and lime contents in an attempt to produce recognizable reaction products for x-ray analysis. From this study, he concluded that it is advisable to mix the sample at high moisture content, and that the height of the new peak reflections grew successively larger as the percentage of lime was increased to about 20 percent, and

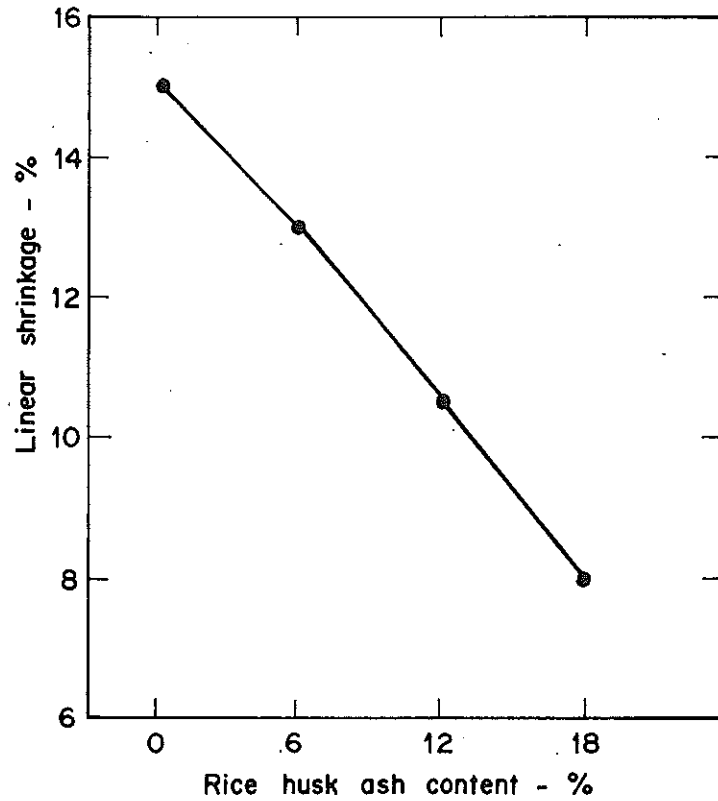


Fig. 3 Variation of Linear Shrinkage with Rice Husk Content.

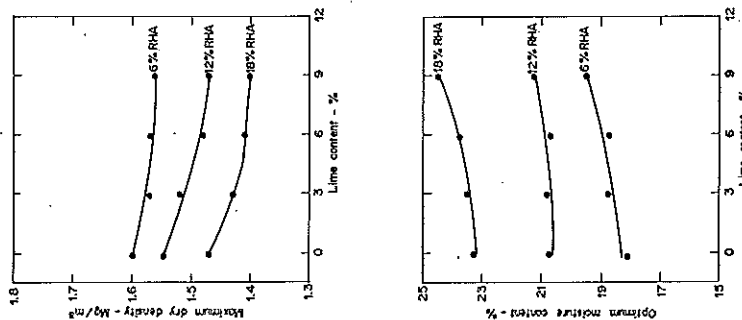


Fig. 4 Variation of Compaction Characteristics of the Residual Soil with Lime-RHA Content.

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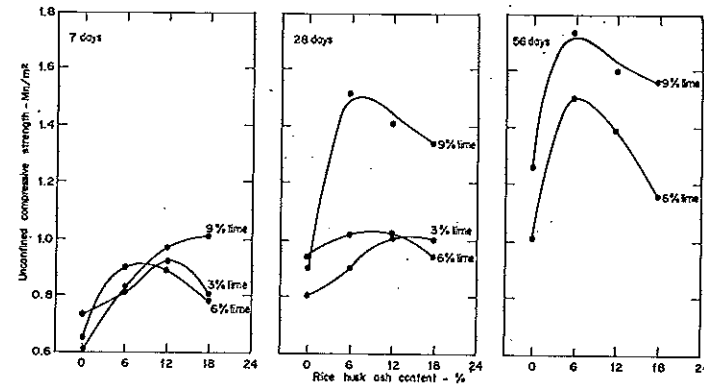


Fig. 5 Variation of Unconfined Compressive Strength of the Residual with Lime-RHA Content.

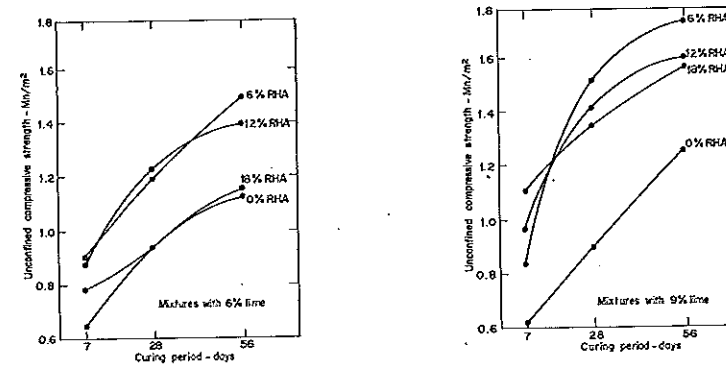


Fig. 6 Variation of Unconfined Compressive Strength of Lime-RHA-Soil Mixture with During Period.

beyond that lime content, the peak height remained relatively constant. Taking this into consideration, the compositions by weight of the mixtures chosen were as follows:

lime/RHA:	1 : 4
lime/residual soil:	1 : 4
lime/RHA/residual soil:	1 : 1 : 1

The minimum lime content of the mixtures was 25 percent by weight of mixture. Distilled water was added in the amount of 100 percent by weight of solid components.

After all the ingredients had been thoroughly mixed the pastes were placed in sealed plastic containers and cured in a thermostatically controlled chamber at 30°C.

Curing of these samples was extended to 90 days. X-ray diffraction studies were carried out after curing for 7, 28 and 90 days, while the scanning electron microscope was utilized for the samples cured up to 90 days only.

After the specified curing periods, the pastes were placed in small dishes and these were allowed to dry over concentrated sulphuric acid then ground to approximately 75 μm size before being mounted on the glass slide.

The x-ray diffractograph of the soil is shown in Fig. 7. Kaolinite clay mineral is identified by its strong diffraction lines at 7.19°A and 3.57°A , which correspond to the first and second order basal reflection respectively, which disappeared when heated to 550°C . The positions of the two peaks are unaffected by glycerol treatment. A minor amount of mica group minerals is also detected by their diffraction peaks at 10°A and 3.3°A . From the relative intensities of the (001) diffraction lines and particularly the missing 5°A peak, the mica present may be of the iron-rich type, such as biotite, glauconite, and lepidomelane.

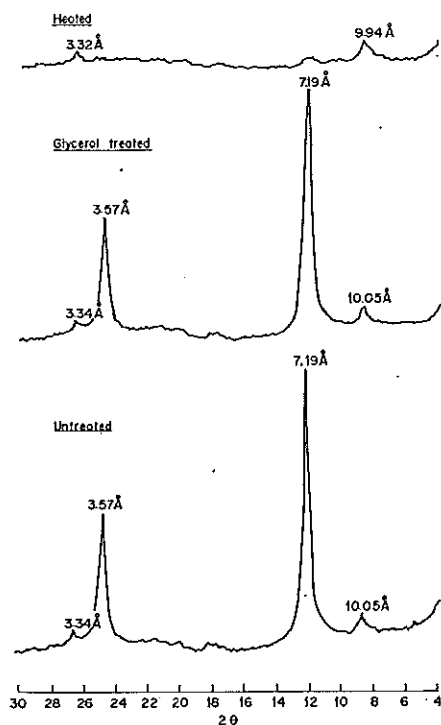


Fig. 7 X-ray Diffractograph of the Soil.

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Fig. 8 and Fig. 9 show the x-ray diffractographs of rice husk ash and the lime respectively.

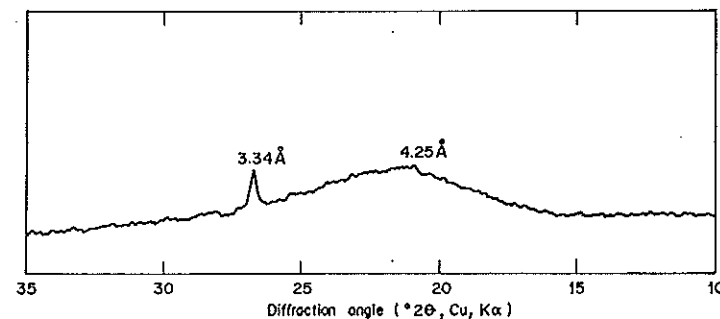


Fig. 8 X-ray Diffractograph of the Rice Husk Ash.

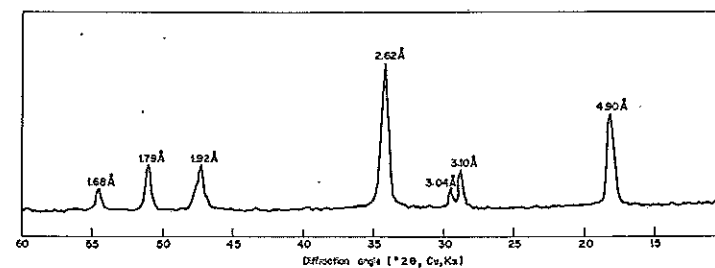


Fig. 9 X-Ray Diffractograph of the Lime.

Lime-RHA Mixtures:

The x-ray diffractograph for lime-RHA mixture cured at 30°C for 7, 28 and 90 days is shown in Fig. 10. It can be seen that after curing for 7 days the peaks observed previously in the x-ray diffractograph of lime (see Fig. 9) are no longer visible, indicating that the lime has been utilized in the lime-RHA reaction. The presence of CSH (Calcium Silicate Hydrate) gel is identified by a peak at 3.04°A and small peaks at 2.80°A and 1.80°A . As the curing period is extended to 90 days, the appearances of three peaks at 10.16°A , 3.04°A and 2.80°A indicate the presence of more crystallized calcium silicate hydrate 1 [CHS (1)].

A short term reaction product of lime-RHA mixture is the CSH gel which after prolonged curing transforms into a more crystallized CSH (1).

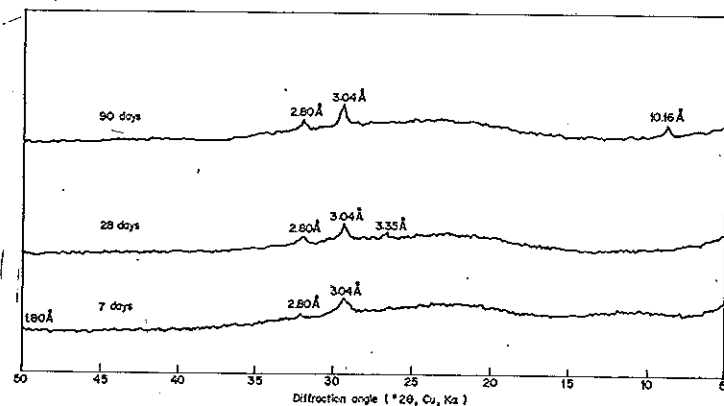


Fig. 10 X-ray Diffractograph of Lime-RHA Mixtures.

Lime-Soil Mixtures

Fig. 11 shows the result of the x-ray diffraction analysis on lime-soil mixtures after 7 and 90 days. After 7 days of curing peaks at 8.18, 3.04 Å, small peaks at 2.80, 2.49, 1.92 and 1.80 Å are observed. The peaks at 2.63 and 3.11 Å are for the unreacted lime. The intensities of the basal peaks for the kaolinite are significantly reduced.

After 90 day curing, the intensities of the x-ray peaks at 3.04, 2.49, 2.29 Å have increased and additional peaks appear at 7.59 and 3.83 Å. The peaks at 8.18 or 7.59 Å and the small peaks at 3.83, 2.49, 2.29 Å are indications of the presence of C_4AH_{13} and the rest of the peaks are related to the poorly crystallized CSH (1).

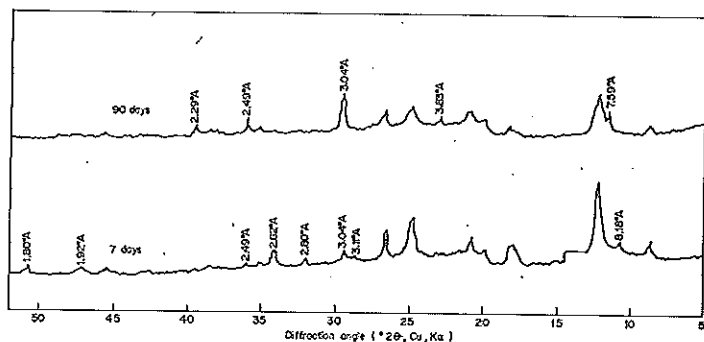


Fig. 11 X-ray Diffractograms of Lime-Soil Mixtures.

Lime-RHA-Soil Mixtures

X-ray diffractograms of the mixtures cured for 7, 28 and 90 days are presented in Fig. 12. After curing for 7 days new peaks appear at 12.63, 10.77, 8.18, 6.28, 4.19, 3.04, 2.88, 2.29 Å and small peaks at 5.40, 5.12, 2.80, 2.62, 2.49, 2.37, and 1.80 Å. The peaks for untreated residual soil as observed previously in Fig. 7 are considerably reduced in intensity.

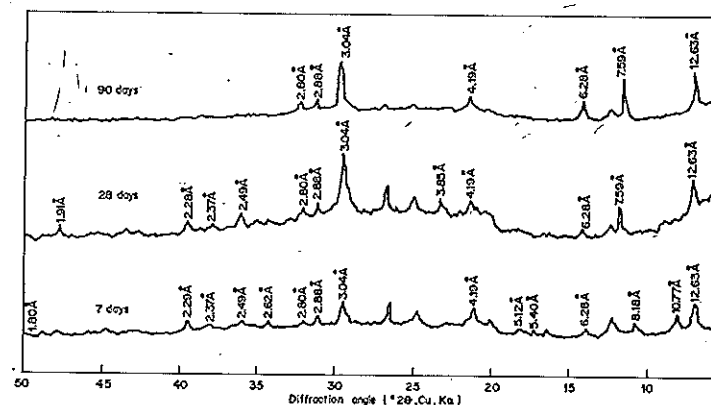


Fig. 12 X-ray Diffractograms of Lime-RHA-Soil Mixtures.

Extending the curing time to 28 days resulted in disappearance of the peaks at 2.62 Å (i.e. for lime). New peaks appear at 12.63, 7.59, 6.28, 4.19, 3.85, 3.04, 2.49, 2.28 and 1.91 Å with additional small peaks at 2.88, 2.80, 2.37 and 1.80 Å. By further extending curing time to 90 days, more peaks disappear in the diffractograph. Peaks observed at 12.36, 7.59, 6.28, 4.19, and 2.88 Å reveal the presence of dicalcium aluminate monosilicate-8-hydrate, C_2ASH_8 (Stratling's compound). In a study by Jambor (1963) the same compound was reported to result from reactions between lime and activated kaolin. Croft (1964) identified Stratling's compound as one of the products produced in the lime-kaolinite reactions at 40°C. The rest of the peaks are attributable to the calcium aluminate hydrate C_4AH_{13} and CSH (1).

Electron Microscopy

Fig. 13 shows a scanning electron microscope picture of rice husk ash, characterized by high surface area, and cellular structure. Fig. 14 shows the crystal form of calcium hydroxide. At the top are thin calcium hydroxide crystals of hexagonal shape. Fig. 15 illustrates the general appearance of untreated soil. The platy morphology of kaolinite mineral can be clearly seen.

The reaction products formed in the lime RHA mixture are illustrated in Figs. 16(a) to (c). The structure of RHA is completely vanished. Paste cured for 90 days is

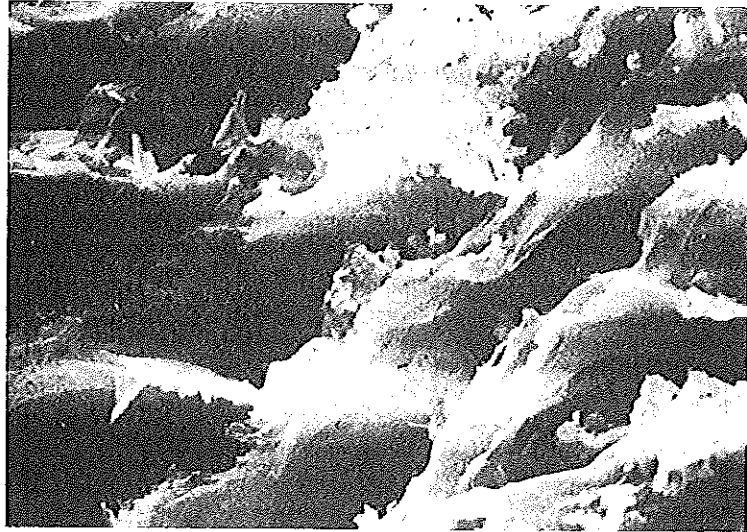


Fig. 13 Photograph of Rice Husk Ash ($\times 2,200$).



Fig. 14 Photograph of Lime ($\times 5,000$).

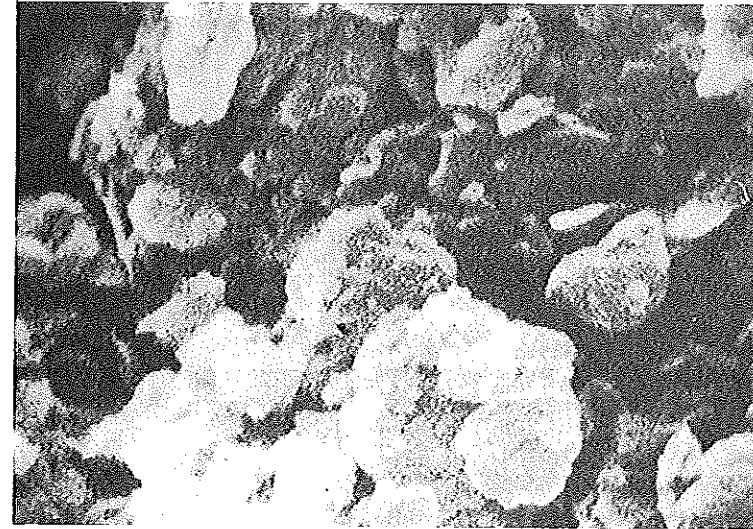


Fig. 15 Photograph of Untreated Soil ($\times 20,000$).

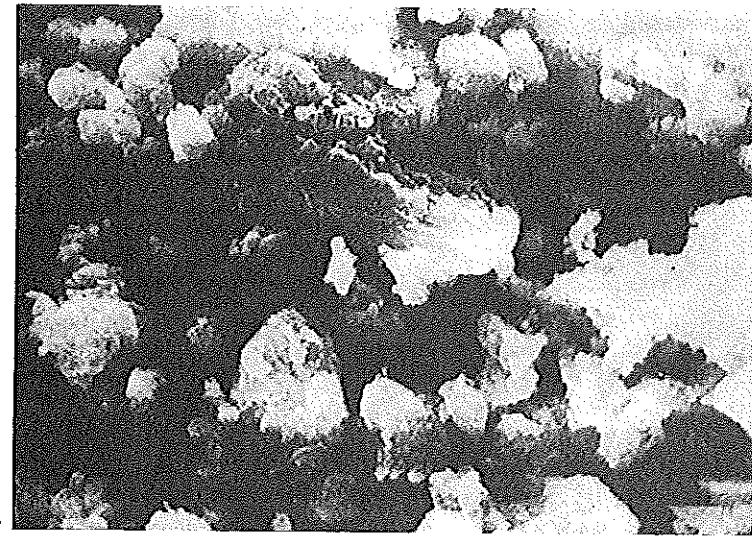


Fig. 16(a) Photograph of Lime-RHA Mixture ($\times 2,000$).

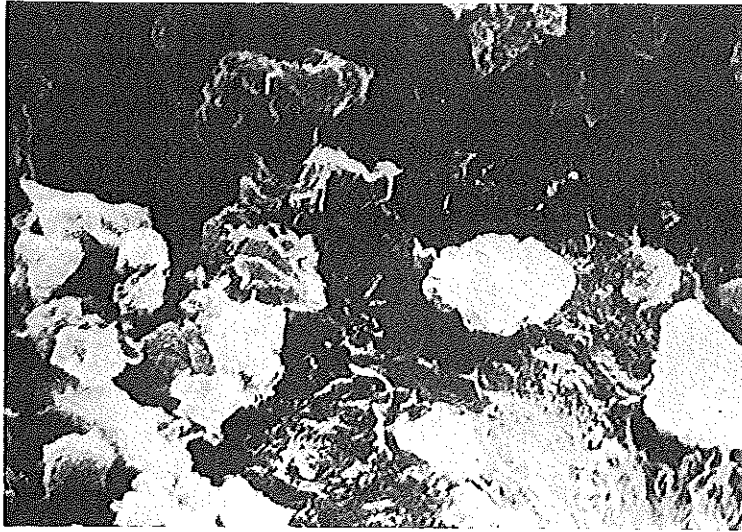


Fig. 16(b) Photograph of Lime-RHA Mixture ($\times 5,000$).

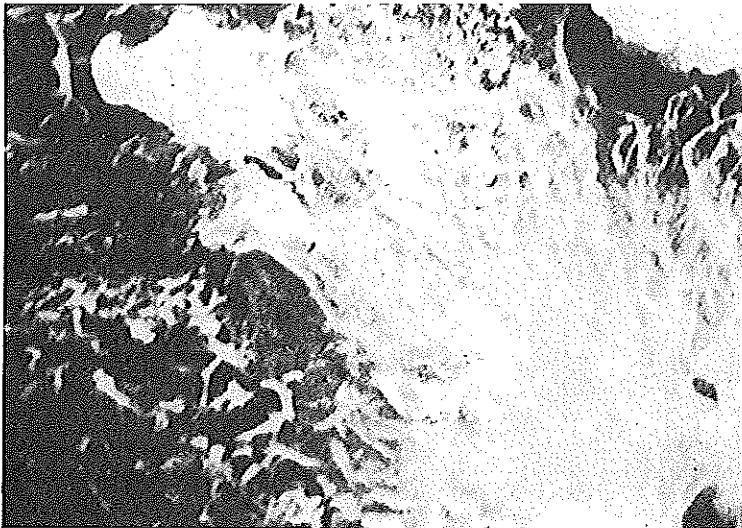


Fig. 16(c) Photograph of Lime-RHA Mixture ($\times 10,000$).

predominated by masses of flaky texture which is probably CSH (I). The formation of fine fibrous material (CSH gel) is clearly visible. Fig. 17 shows the reaction products of lime-RHA-soil mixture. It can be seen that new minerals are formed. The fibrous, flaky and hexagonal materials can be observed and probably these represent the CSH gel, CSH (I) and stratling's compound respectively, as indicated by the results of the x-ray diffraction analyses above.

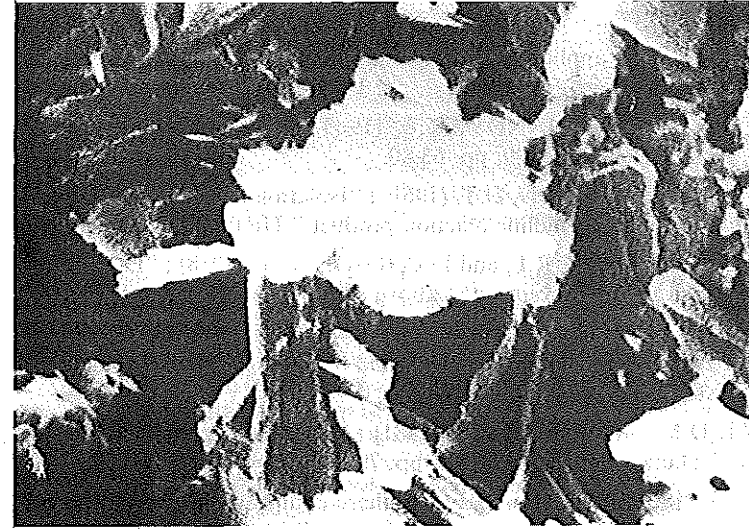


Fig. 17 Photograph of Lime-RHA-Soil Mixture ($\times 10,000$).

CONCLUSIONS

From the results of the study the following conclusions can be derived:

1. The important properties of the soil are enhanced by addition of lime and rice husk ash. This is due to the formation of various compounds when lime reacts with rice husk ash and the soil.
2. The initial increase in strength is attributed to the poorly ordered reaction products, calcium silicate hydrates (CSH) gel.
3. The development of long term strength appears to be due to the gradual crystallization of structurally-ordered new minerals, calcium silicate hydrate 1 [CSH (I)] and Stratling's compound (C_2ASH_8), from the initial disordered reaction products.

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