

## Morphology and mineralogy of rice husk ash treated soil for green and sustainable landfill liner construction



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### ABSTRACT

The morphology and mineralogy of the soil treated with rice husk ash (RHA) under different molding moisture conditions. Leachate condition in landfills built with compacted clay soil is damaging the underground water flow with the hazards released from disposed and decomposing waste materials. This makes landfills dangerous infrastructure. The leakage can be dealt with through the deployment of green materials developed from agricultural waste. One of such wastes is rice husk combusted to derive ash. The test soil used in this exercise has been classified as highly plastic and poorly graded. The treated soil was examined by scanning electron microscopy and x-ray diffractometer methods. From the test results, the presence of goethite alongside quartz and kaolinite were observed in the XRD (X-ray Diffraction) spectra of 6% and 10 % RHA treated soil. The Goethite possessing an inner needle-like structure with a closed packed striated structure makes the composite a promising material for constructing landfill liners. This is because the closed packed striated structure of the goethite present in the composite will slow down the vertical seepage of leachate to allow its collection and removal by the leachate collection system. The composite will form a barrier between groundwater, soil, and substrata, and waste. From the SEM (Scanning Electron Microscopy), the uniformly distributed grain boundaries and smaller grain size of the composite (lateritic soil and RHA) will serve as a barrier to the movement of contaminants and other leachates to the groundwater and thus, making the composite a viable material for landfill liner system.

### Introduction

#### Background

Landfills are highly engineered containment systems, designed to minimize the impact of solid waste (refuse, trash, and garbage) on the environment and human health (Emmanuel et al. 2019). The primary purpose of the liner systems is to isolate the landfill contents from the environment and therefore, to protect the soil and groundwater from pollution originating from the landfill. The greatest threat to groundwater, which is posed by landfills is leachate (Rowe et al., 2001). Leachate consists of water and water-soluble compounds in the refuse that accumulates as water moves through the landfill (Eberemu et al. 2012). This water may be from direct rainfall, runoff or from the waste itself. Leachate may migrate from the landfill and

contaminate soil and groundwater, thus presenting a risk to human and environmental health. The overall use of the liner system the objective is to stop or limit the discharge of hazardous contaminants to groundwater (Osinubi et al. 2012). It is already understood that the purpose of a landfill is to isolate solid waste from the environment. This means that no harmful substances from the waste body could reach the environment in unacceptable quantities. The isolation of the waste material from the environment is achieved by providing an impermeable barrier all around the waste body called the liner (Moses et al. 2013; Onyelowe et al. 2020a). Geotechnical engineers are trying to include some of the advanced technologies in nondestructive testing methods for evaluating the performance of geomaterials utilized as liner materials. X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) are two of them. XRD is used for the mineralogical analysis of the materials while SEM is used to study the

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morphological surface analysis. A mineralogical analysis is the study of materials to determine the mineral composition and mineral structure. This analysis can be used to identify mineral species and understand their characteristics and properties as well as their stoichiometric contributions (Onyelowe et al. 2020b; Smith et al. 2001).

In rural areas, the environmental impacts of landfilling, affecting sustainable land management in the region, may be related mainly to the contamination of surface water and groundwater through leachate, and to the pollution of soil by direct contact with waste or leachate percolation, long after the closure of the landfill (Zurbrüg et al. 2012). Thus, a sustainable landfill, from the ecological and environmental point of view, should pose a negligible, or even zero, a risk to the environment both during the performance stage and long after the closure, hence the isolation of landfills should be long-term and self-sustainable (Laner et al. 2012; Gebhardt et al. 2012). This research work seeks to determine the mineralogical analysis and morphology of treated soil utilized as a liner material using X-ray diffraction and scanning electron microscopy methods. The microstructure of the stabilized soil is determined to assess how they perform under a given application (Widomski et al. 2015). The ash will be obtained through a controlled incineration system to take care of the carbon oxide gases released during the combustion

### General review

#### Overview

Nigeria is the largest in Africa in terms of population and population growth index. Different dumpsites are located around rural and urban communities (designed and non-designed). These dumpsites (majorly non-designed and some designed ones) pose serious environmental risks. These risks arise mainly from improper site selection, namely, serious slope stability problems caused by situating the landfill in the proximity of a steep slope. This is to say that the accumulated waste pile occasionally reaches a height of 10 m or above along a relatively steep slope, which poses slope stability problems. An additional risk arises from the lack of a proper containment system where the leachate water is being channeled to a nearby river or valley, creating serious environmental risks for the adjacent residential areas. A third risk arises due to operational problems, namely, improper information on the amount and type of waste, deposited, problems in compacting the old wastes that lie below the recently deposited wastes, and uncontrolled biodegradation that increases the possibility of methane explosions (Oyetola and Abdullahi, 2006). To meet the municipal waste discharge needs of the city, alternative landfills are cited for community and town usage (Onyelowe et al. 2019). However, this landfill site not only has a limited capacity but also possesses both operational and infrastructural problems. The primary problem of landfill sites is that after the construction of the landfill liner system, the site will be left idle for about 4 years due to the proximity to the users. This usually leads to a reduction in the moisture content of the compacted clay liner (CCL), which in turn leads to the formation of secondary fissures and cracks that cause the loss of the integrity of the landfill and hence, the relatively high permeability function of the CCL. Since municipal and hospital wastes have been periodically deposited on the landfill sites following the idle period, any lining system remediation process would require that all previously deposited waste be relocated elsewhere during the period of remediation, which would not be deemed environmentally or economically feasible (Bui Van and Onyelowe, 2018). Another problem is that most landfill site lacks the daily spread of soil cover material and proper sterile deposition of hospital wastes. Prior to the regular usage of the landfill, technical and administrative discrepancies that include the construction of interim waste deposition sites, transfer stations, and a trailer system should be resolved. From a city planning point of view, the early planning of the landfill site location is crucial to prevent residential and commercial development at those sites that are suitable for landfill siting since many factors need

to be taken into consideration in finding a suitable site (Osinubi et al. 2012). Lateritic soils have been extensively used in the last decades in dam and road construction; nevertheless, very little is known about the migration of pollutants through the compacted layers of such soils. The main mechanisms of solute transport through a porous medium are advection, mechanical dispersion, diffusion, and chemical reactions of the solute in the solution, such as radioactivity, and chemical reactions among solutes and solids, such as adsorption (Eberemu et al. 2012). Advection, i.e., the movement of a solute as it is carried by the water seeping through the soil, and mechanical dispersion, i.e., the mixing that occurs together with advection depend on the hydraulic conductivity of the soil. Diffusion, i.e., the movement of ionic and molecular constituents by their thermal kinetic energy in the opposite direction of the concentration gradient, is controlled by the diffusion coefficient of the chemical species of the soil. Adsorption, which is a physico-chemical process by which a solute is accumulated in a solid-liquid interface, is essentially due to clay minerals, which are negatively charged because of the isomorphous substitution of cations in the silicate or aluminate layers of the crystalline reticulate. Other retention mechanisms may occur, such as precipitation, ion complexation, and specific adsorption. The capacity of soils for attenuating pollution has been intensively studied in the last thirty years; an important application is the utilization of compacted clay liners in waste disposal sites or waste treatment plants to control subsoil contamination (Onyelowe et al. 2019). Although some soils exhibit some degree of deficiency, deficient soils are regarded as soils that do not meet some or all criteria required for their satisfactory performance as geotechnical structures. These could either be for base courses for roads, embankments for dams or roads, subsoil bases for foundations, clay liners for containment of leachates, and backfill for retaining walls (Laner et al. 2012). Improvement of deficient soils could either be by modification or stabilization or both. While the modification is the improvement of soil by addition of a modifier (cement, lime, etc.) to change (improve) its index properties, soil stabilization is the treatment of soils to enable the improvement of their strength and durability such that they become suitable for construction beyond their original classification. Over time, cement and lime have been the two main materials used for stabilizing soils. According to Neville (2000), the cost of these materials has rapidly increased due to the sharp increase in the cost of energy since the 1970 s. The cost of construction of roads with stabilized layers and other geotechnical structures has remained high due to the dependent on the utilization of industrially manufactured soil improvement additives (cement, lime, etc.). This has consequently continued to deter the underdeveloped and poor nations of the world from providing accessible infrastructure to their rural dwellers, who constitute a higher percentage of their population and are mostly agriculturally dependent (Alhassan and Mustapha, 2007). Thus, the use of agricultural waste (such as Rice Husk Ash) will considerably reduce the cost of construction. The environmental hazards these wastes cause as well as economically, add to the value chain of rice farmers. Information on world rice production (Oyetola and Abdullahi, 2006), shows that the global rice production by the 2015/2016 season was 472.09 million tons, while for 2016/2017, it is estimated to be 483.26 million tons, representing an increase of 11.17 million tons (2.37%) rise in the production. This translates to about 157.2 and 160.9 million tons of rice husk for 2015/2016 and 2016/2017, respectively, with a corresponding increase of 2.37% over these two seasons (Oyetola and Abdullahi, 2006). About 2.0 million tons of rice is produced annually in Nigeria (Oyetola and Abdullahi, 2006). Oyetola and Abdullahi projected rice production in Nigeria for the 2016/2017 season to stand at 3.120 million metric tons. This explained why mountain heaps of husk have become common sites at milling points in Nigeria and other developing countries. The search for the potential use of this waste, especially in geotechnical engineering, has prompted researchers over the last few years to study how it can be used in the improvement of soils.

Burning of rice husk generates up to 25 % of its weight as ash (K. C. Onyelowe et al. 2019). This will eventually help in the disposal of this waste material and reduce its hazardous effects on the environment. According to Oyetola and Abdullahi (2006), ash has been categorized as pozzolana, with about 67–70% silica and about 4.9 and 0.95% aluminium and iron oxides, respectively. Rowe and Van Gulck (2001), also reported 67.27 % silica in rice husk ash, while Oyetola and Abdullahi (2012), reported silica content of up to 84.55 %. The silica is substantially contained in an amorphous form, which can react with the  $\text{Ca}(\text{OH})_2$  liberated during the hardening of cement to further produce cementitious compounds. It was reported by Moses et al. (2013), that Portland cement, by the nature of its chemistry, produces large quantities of  $\text{CO}_2$  for every ton of its final product (K. C. Onyelowe et al. 2019). Therefore, replacing the proportions of the Portland cement in soil stabilization with a secondary cementitious material like rice husk ash will reduce the overall environmental impact of the stabilization process. Researches have recently been focused on the utilization of rice husk ash for the improvement of geotechnical characteristics of deficient soils in Nigeria for various other uses.

#### *Leachate generation and characteristics*

Numerous physiochemical and biological processes control the production and composition of leachate in landfills. In general, the composition of leachate will be a function of the type/age of waste deposited, the prevailing physicochemical conditions, and the microbiology and water balance of the landfill. Microbial action commences and triggers the decomposition of the putrescible waste and it occurs in three stages. In the first stage, degradable waste is attacked by aerobic organisms, resulting in the production of organic compounds, carbon dioxide, water, and heat. The second stage commences when all oxygen is consumed or displaced by carbon dioxide. Aerobic organisms, which thrive when oxygen was available, die off and the degradation process is then taken over by facultative organisms that can thrive in either the presence or absence of oxygen. These organisms can break down the large organic molecules present in food, paper, and similar wastes into more simple compounds such as hydrogen, ammonia, water, carbon dioxide, and organic acids. In the third and final stage (anaerobic, or methanogenic phase) methane-forming organisms multiply and break down organic acids to form methane gas and other products. The water-soluble degradation products from these biological processes, together with other soluble components in the waste, are present in the leachate. In addition, pH changes and acid formation may mobilize metals and increase their content in the leachate.

#### *Leachate retention and liner systems*

Both new landfills and lateral extensions of existing landfills need to provide an appropriate level of retention to protect the environment from the adverse effects of leachate entering the groundwater and even surface waters. This would generally comprise a leachate retention or liner system and a leachate collection system. Leachate is the liquid that has percolated through a solid waste, which has previously decomposed. The source of the liquid is primarily the water already present in the waste and any water induced from an external source such as rain water and ground water. To prevent the movement of leachate beyond the landfill site, an effective impermeable liner collection system becomes critical. Leachate collection pipes are entrenched near the bottom of the liner layer and are connected to the main pipe that leads to a leachate holding tank. At some sites, significant retention and attenuation can potentially be provided by the underlying geology of the site, which may be able to act as a component of the liner system. For others, it will be necessary to rely on a well-engineered liner system over the entire base area of the landfill for both retention and reduction.

#### *Landfill liner systems*

The landfills have been designed and constructed as a secure containment facility incorporating multilayer composite liner systems covering the entire surface area of the site (see Fig. 1). As the sites are lined, landfill gas and leachate can be collected and treated to ensure that there will be no untreated discharge from the landfill to the environment. The primary function of a landfill liner system is to protect groundwater from the impacts of leachate. This is achieved by the landfill liner slowing the vertical seepage of leachate to allow its collection and removal by the leachate collection system. The liner may also attenuate contaminants in leachate seeping through the liner to the point where the leachate that makes contact with the aquifer beneath the landfill has a minimal detrimental impact on groundwater. A further function of the liner is to retard the lateral movement of landfill gas from the landfill and the infiltration of groundwater. The landfill liner system should be designed to contain leachate over the period that the waste poses a potential environmental risk. The liner system should be designed and installed as presented in Fig. 1, following the quality requirements specified in an approved Construction Quality Assurance Program. The benchmark technique for a liner system for new landfills and lateral expansion of operating landfills is a system that forms a barrier between groundwater, soil, and substrata, and waste.

#### *Compacted clay liner system*

The ability of clay to retard water movement and absorb exchangeable cations makes it a suitable green construction material for a low-permeability liner. When assessing the suitability of a soil as a low-permeability liner, soil properties such as particle size distribution, morphology and plasticity (described by the soil plasticity index), and cation exchange capacity (CEC) should be determined. The potential for desiccation and subsequent cracking must also be considered. Clay used for the liner construction should have the following properties; no rock or soil clumps > 50 mm in any dimension, 70 per cent passing through a 75 mm sieve, 30 per cent passing through a 19 mm sieve, 15 per cent passing through a 2 mm sieve, soil plasticity index > 10, CEC > 10 mEq/100 g and minimal long-term degradation with exposure to leachate.

The clay liner must comprise a thickness of 900 mm and a hydraulic conductivity of less than  $1 \times 10^{-9}$  m/s using both fresh water and 50,000 ppm NaCl solution according to Australian Standard AS 1289.6.7.1–1999: Soil strength and consolidation tests – Determination of permeability of a soil – Constant head method for a remolded specimen. The liner must undergo construction by uniform moisture conditioning and compaction using a sheep-foot roller in layers with a maximum compacted thickness of 200 mm. There must be effective bonding between successive layers that include kneading between layers, scarification, and moisture conditioning. Successive layers must have a minimum horizontal overlap of 1 m to ensure that a preferential pathway for leachate flow is not created. Clay liners must have a smooth final surface that is graded at a minimum of 2% towards drainage lines and 1% along drainage lines. A Construction Quality Assurance (CQA) plan must be developed and implemented as a means of managing quality during construction and reporting so that the materials used, construction methods, and complete works comply with the landfill design.

#### *Microstructure/Morphology*

Microstructures determine the mechanical, physical, and chemical properties of materials. For example, the strength and hardness of materials are determined by the number of phases and their grain sizes. The electrical and magnetic properties and the chemical behavior (corrosion) are determined by the grain size and defects (vacancies, dislocations, grain boundaries, etc.) present in the material (Onyelowe et al. 2019). A complete description of microstructures involves describing the size, shape, and distribution of grains and second-

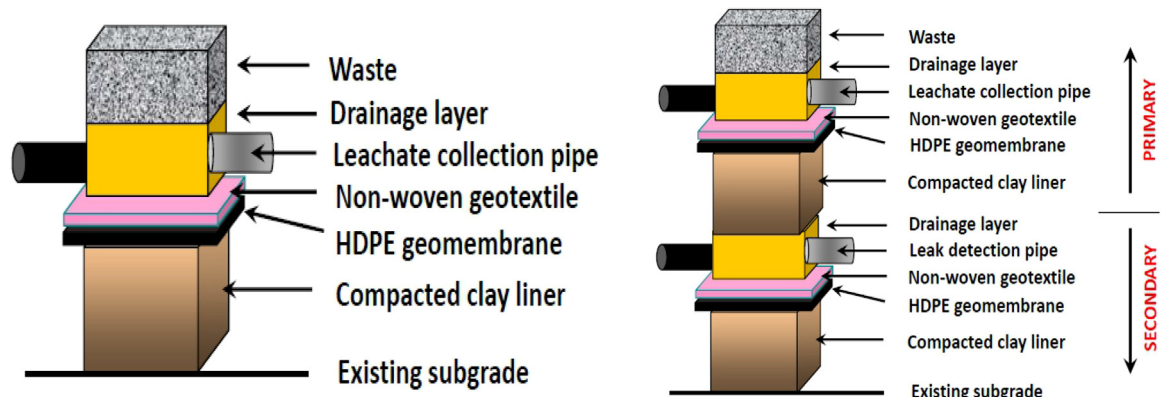


Fig. 1. Single and Double/Composite Liner Systems.

phase particles and their composition and, the defect structures, although these are often omitted (Onyelowe et al. 2019). Microstructural features of interest are the size, shape, and distribution of grains in the case of a single-phase material. In a two-phase or multi-phase material, the size, shape, and distribution of the secondary phases

are important in addition to the microstructural parameters of the matrix (the major component). The majority of the microstructural features and parameters of interest in engineering alloys are on a scale of a few tens of micrometers ( $1 \mu\text{m} = 10^{-6} \text{m}$ ) or less. Additionally, in recent times, there has been much interest in characterizing nanocryst-



Fig. 2. Rice Husk.

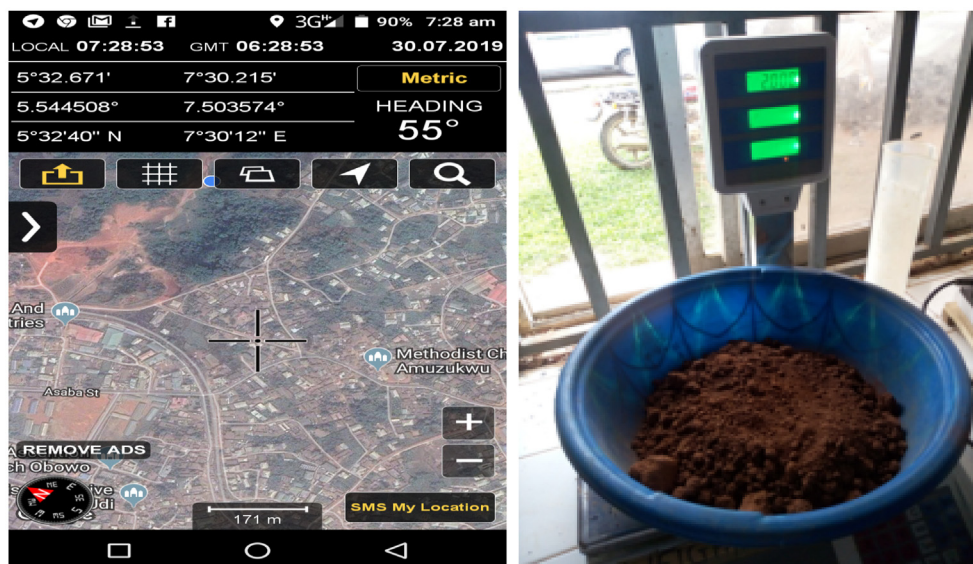


Fig. 3. GIS location of the borrow pit and the test soil sample.

talline materials with nanoscale grain sizes of “ 100 nm (1 nm =  $10^{-9}$  m). To observe these small and very small details, it is necessary to use several types of microscopes, covering a wide range of magnifications and resolutions. Microstructural features in the size range of a few tens of micrometers, i.e., submillimeter scale, may be observed using optical microscopy and scanning electron microscopy, which, however, can be used up to higher magnifications. Transmission electron microscopy is especially useful at very high magnifications, allowing observations on the nanoscale. Resolutions on an atomic scale are provided by field ion microscopy, scanning tunnelling microscopy, and atomic force microscopy. The latter two techniques have become very popular because they can provide additional information, namely, mapping magnetic and electrostatic forces (scanning tunnelling microscopy) and chemical interactions at surfaces (atomic force microscopy). Microstructural information may be viewed directly, recorded using photographic methods, stored digitally, or received by a television camera for input into an analysis system.

## Materials and methods

### Materials preparation

#### Rice husk ash (RHA) for soil stabilization

The Rice Husk used for this research was collected from Abakaliki rice mills, Ebonyi State, Nigeria. The husk was stacked in heaps near the mill and disposed of indiscriminately in farm areas. Samples were collected from different spots at the heap site and nearby farms (see

Fig. 2). The samples were burnt in a controlled incinerator. The ash was left to burn at a high temperature for twenty-four hours to obtain a certain degree of fineness. After the incineration, the RHA was left inside the furnace to cool and then collected the following day. The ash was then sieved using British Standard (BS) sieve size 425  $\mu$ m.

Rice Husk, as presented in Fig. 2, is the major byproduct in the processing of rice. The management of this waste has become a big challenge to some of the rice producers and the Ministry of Environment. Some of these wastes are left in open dumps while some are burnt in the open space and these two actions have been contributing to environmental pollution. Rice husk is a by-product from agriculture produce when it is harvested, the outermost layer of the paddy grain is the rice husk, also called rice hull. It is separated from the brown rice in rice milling. Burning rice husk produces rice husk ash (RHA), so for every 1000 kg of paddy milled, about 220 kg (22 %) of RHA is produced and when this husk is burnt in the boilers, about 55 kg (25%) of RHA is generated, if the burning process is incomplete, carbonized rice husk (CRH) is produced. The husk surrounding the kernel of rice account for approximately 20% by weight of the harvested grain. The exterior of rice husks is composed of dentate rectangular elements, which themselves are composed mostly of silica-coated with a thick cuticle and surface hairs. The mid-region and inner epidermis contain little silica. The use and disposal of rice husks have frequently proved difficult because of the tough, woody, abrasive nature of the husk, their low nutritive properties, and resistance to weather, great bulk and ash content. In fact, in South East Asia, the accumulating heaps of rice husk have become significant problems; The RH was burnt in



Fig. 4. X-Ray Diffractometer Experimental Set-up.



Fig. 5. Scanning Electron Microscopy Experimental Set-up; Phenom ProX, by phenom world Eindhoven, the Netherlands.

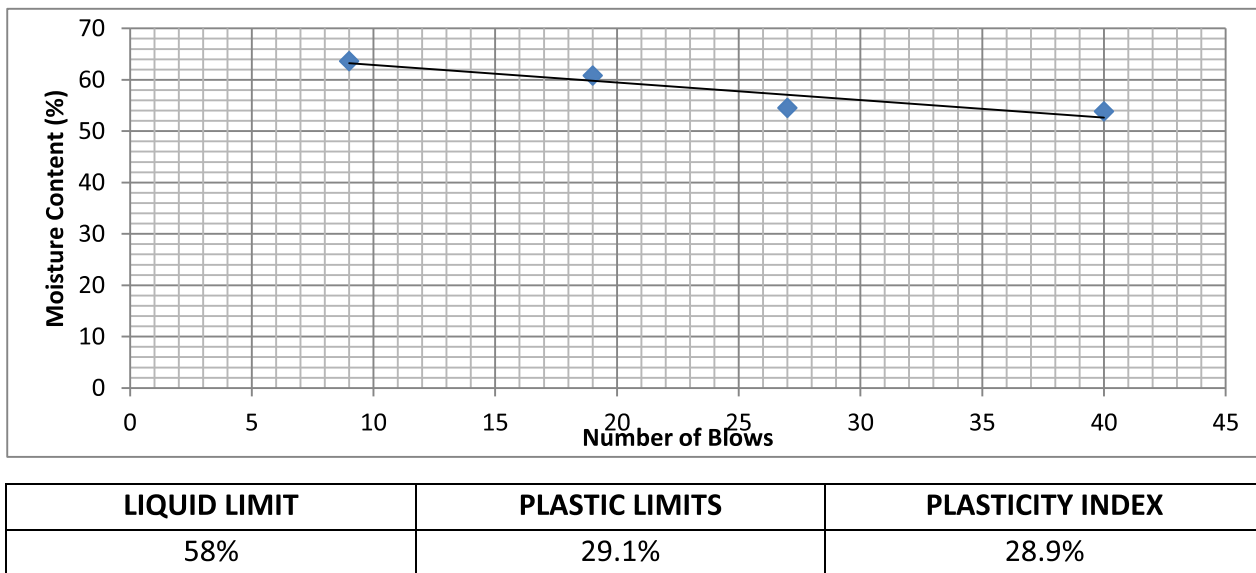


Fig. 6. Atterberg limits the behavior of natural soil.

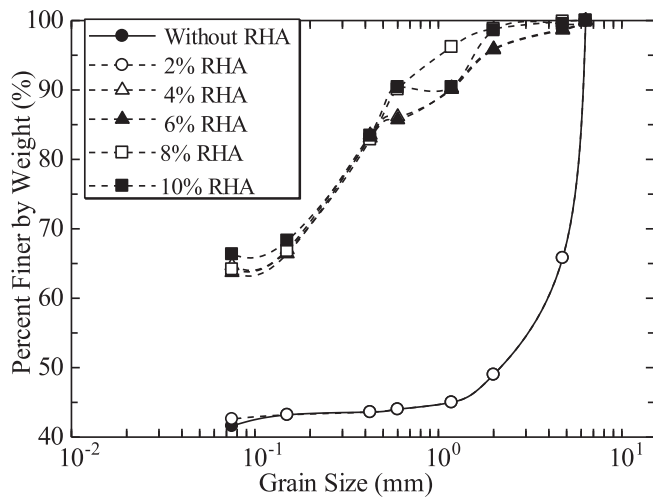


Fig. 7. Particle size distribution of the untreated and treated soils.

a controlled environment to form Rice Husk Ash (RHA). Rice husk is an agricultural waste obtained from the milling of rice. About 108 tons of rice husks are generated annually in the world. Meanwhile, the ash has been categorized under pozzolana, with about 67–70% silica and about 4.9% and 0.95% Alumina and iron oxides, respectively (Oyetola and Abdullahi, 2006). The silica is substantially contained in an amorphous form, which can react with the  $Ca(OH)_2$  liberated during the hardening of cement to further form cementations compounds. Soil stabilization aims at improving soil strength and increas-

Table 1  
Consistency and specific gravity of RHA treated soil.

RHA treated soil (%)	PL	LL	PI	SG
0	28.9	58.0	29.1	2.67
2	30.4	60.0	27.6	2.30
4	29.7	72.0	42.3	2.30
6	29.5	67.0	37.4	2.30
8	35.4	70.0	34.6	1.08
10	38.4	69.0	30.6	0.98

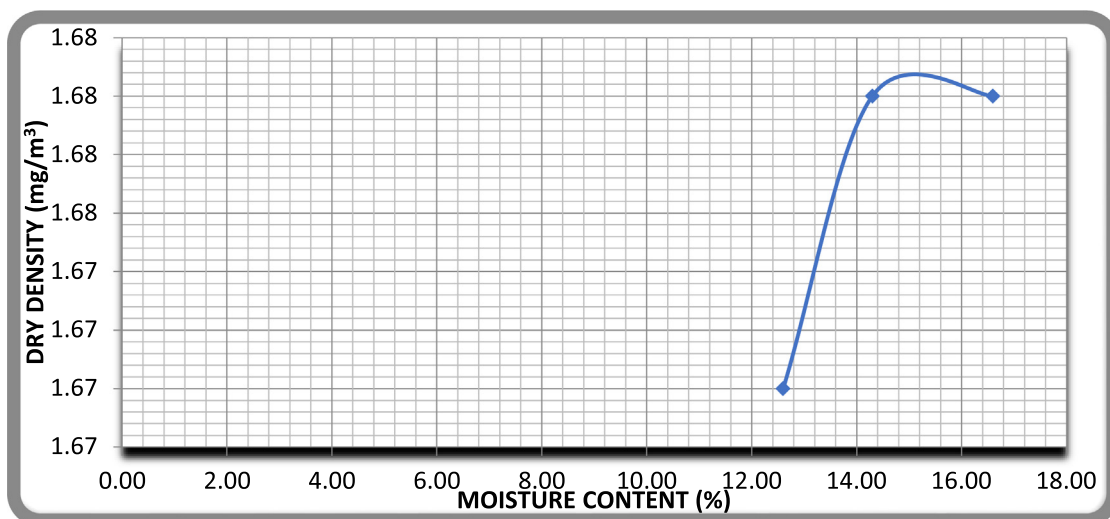


Fig. 8. Compaction of the natural soil.

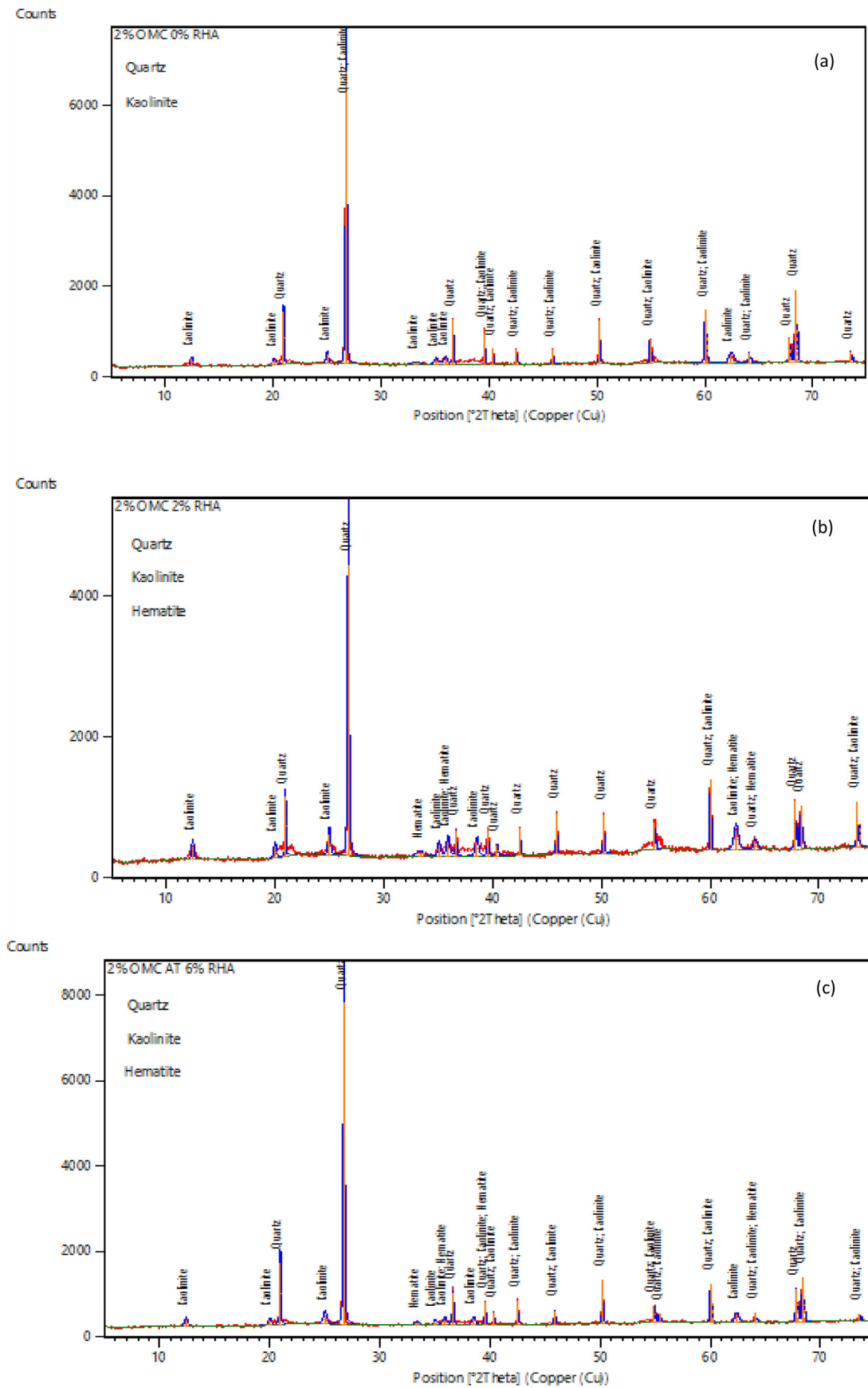


Fig. 9. XRD Spectra of treated soil (a) + 2% of OMC at 0% RHA, (b) + 2% of OMC at 2% RHA, (c) + 2% of OMC at 6% RHA (d) + 2% of OMC at 10% RHA.

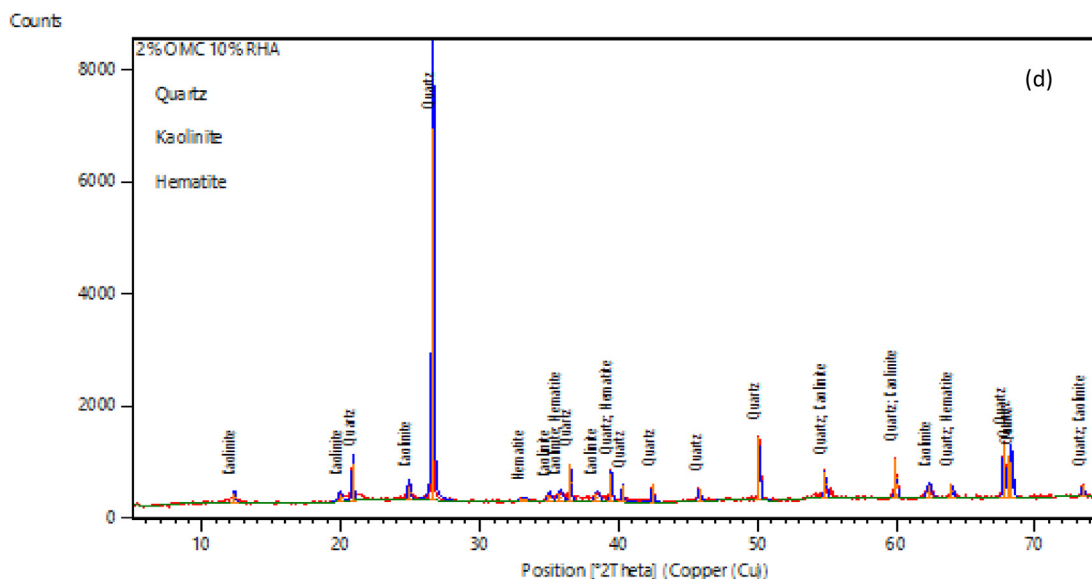


Fig. 9 (continued)

ing resistance to softening by water through bonding the soil particles together, waterproofing the particles, or a combination of the two. Soil stabilization can be accomplished by several methods, but all these methods fall into two broad categories, namely, mechanical and chemical stabilization. Mechanical Stabilization is a physical process that involves altering the physical nature of native soil particles by either inducing vibration or compaction or by incorporating other physical properties such as barriers and nailing. Chemical Stabilization involves initiating chemical reactions between stabilizers (cementitious material) and soil minerals (pozzolanic materials) to achieve the desired effect of improving the chief properties of a soil that are of interest to engineers namely volume stability, strength, compressibility, permeability and durability.

#### Lateritic soil

Fine-grained lateritic soil sample (see Fig. 3) obtained from a borrow pit in Umuahia (Latitude 5°32'39" N and Longitude 7°30'14" E) Abia State, Nigeria at about 1.2 m depth was used for this study presented in Fig. 3. Laterite is a soil and rock type rich in iron and aluminium and is commonly considered to have formed in hot and wet tropical areas. Nearly all laterites are of rusty-red coloration, because of high iron oxide content. They develop by intensive and prolonged weathering of the underlying parent rock. Tropical weathering (laterization) is a prolonged process of chemical weathering which produces a wide variety in the thickness, grade, chemistry and ore mineralogy of the resulting soils. Laterites are formed from the leaching of parent sedimentary rocks (sandstones, clays, limestones); metamorphic rocks igneous rocks (granites, basalts, gabbros, peridotites); and mineralized proto-ores; which leaves the more insoluble ions, predominantly iron and aluminium (Onyelowe et al. 2020a). The mechanism of leaching involves acid dissolving the host mineral lattice, followed by hydrolysis and precipitation of insoluble oxides and sulfates of iron, aluminium and silica under the high-temperature conditions of a humid subtropical monsoon climate. Tropical weathering (laterization) is a prolonged process of chemical weathering which produces a wide variety in the thickness, grade, chemistry and ore mineralogy of the resulting soils. Laterite contains a substantial amount of clay minerals, hence its strength and stability cannot be guaranteed under load, especially in the presence of moisture. Stabilization using stabilizing agents improves the engineering properties and makes it suitable for construction (Onyelowe et al. 2020b). This paper aims to present a review

of the effects of various stabilizing agents used for the stabilization of lateritic soil. The formation of lateritic soil involves the Physico-chemical breakdown of primary minerals and the release of constituent elements ( $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{K}_2\text{O}$ ,  $\text{Na}_2\text{O}$ , etc.), which appear in simple ionic forms; leaching (laterization) under appropriate conditions of combined silica and bases and the relative accumulation or enrichment of oxides and hydroxides of Sesquioxides ( $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{TiO}_2$ ) and partial or complete dehydration (sometimes involving hardening) of the Sesquioxide rich materials and secondary minerals (Onyelowe et al., 2019).

#### Experimental methods

##### X-ray diffraction (XRD)

X-ray diffraction (XRD), presented in Fig. 4, is a primary tool for probing the structure of a nanomaterial. XRD offers unparalleled accuracy in the measurement of atomic spacing and is the technique of choice for determining strain states in thin films. The intensities measured with XRD can provide quantitative, accurate information on the atomic arrangements at interfaces. Synchrotron radiation allows the characterization of much thinner films and for many materials, monoatomic layers can be analyzed (Onyelowe et al. 2019). XRD is non-contact and non-destructive, which makes it ideal for in situ studies. Nanomaterials have a characteristic microstructure length comparable to the critical length scales of physical phenomena, giving them unique mechanical, optical, and electronic properties. X-ray diffractograms of the nanomaterial provide a wealth of information - from phase composition to crystallite size, from lattice strain to crystallographic orientation. The main use of powder diffraction is to identify components in a sample by a search/match procedure. Furthermore, the areas under the peak are related to the amount of each phase present in the sample. Every crystalline substance gives a pattern; the same substance always gives the same pattern; and in a mixture of substances, each produces its pattern independently of the others. The X-ray diffraction pattern of a pure substance is, therefore, like a fingerprint of the substance. The powder diffraction method is thus ideally suited for the characterization and identification of polycrystalline phases.

##### Scanning electron Microscope (SEM)

A scanning electron microscope (SEM) presented in Fig. 5, scans a focused electron beam over a surface to create an image. The electrons

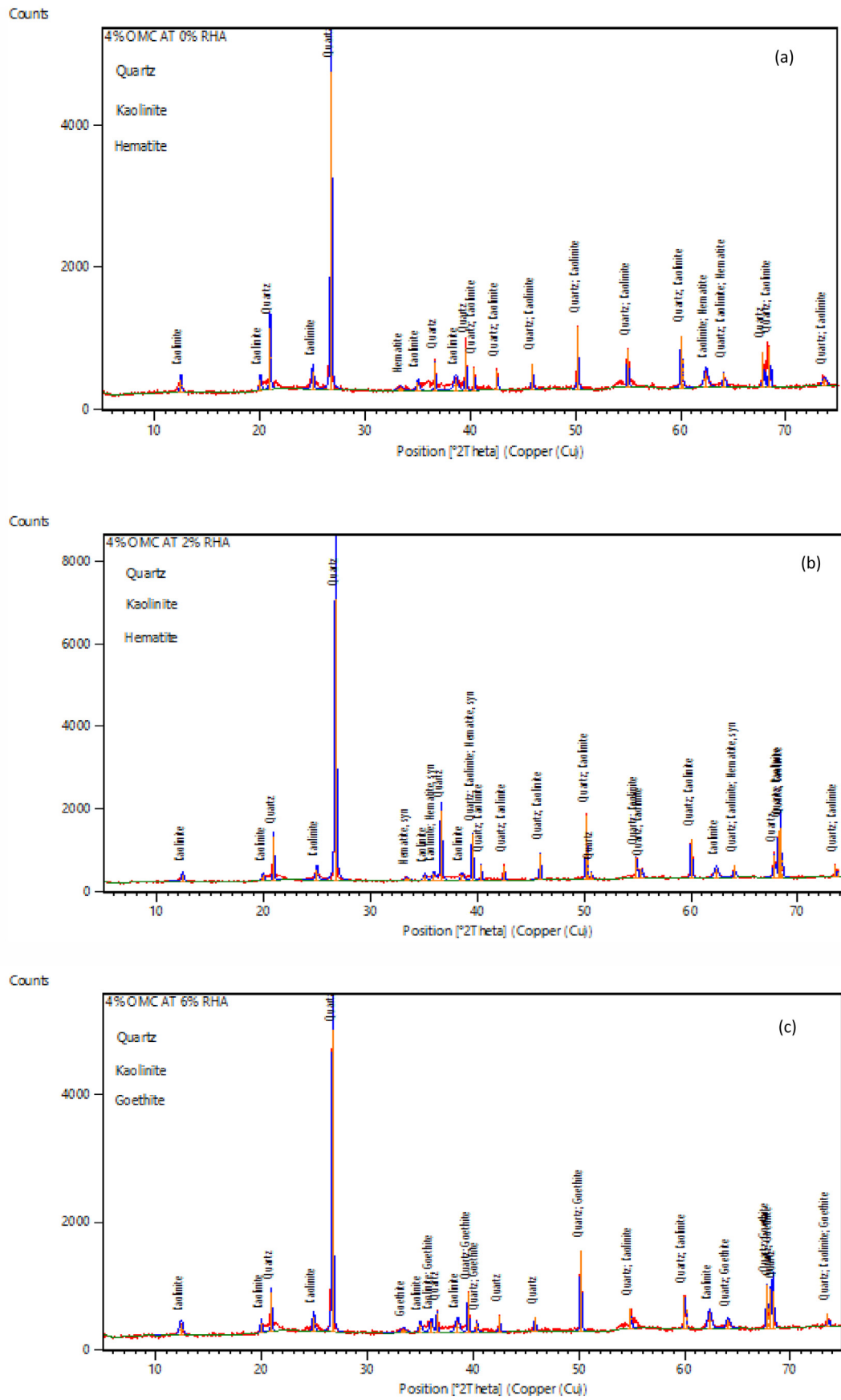


Fig. 10. XRD Spectra of treated soil (a) + 4% of OMC at 0% RHA, (b) + 4% of OMC at 2% RHA, (c) + 4% of OMC at 6% RHA (d) + 4% OMC at 10% RHA.

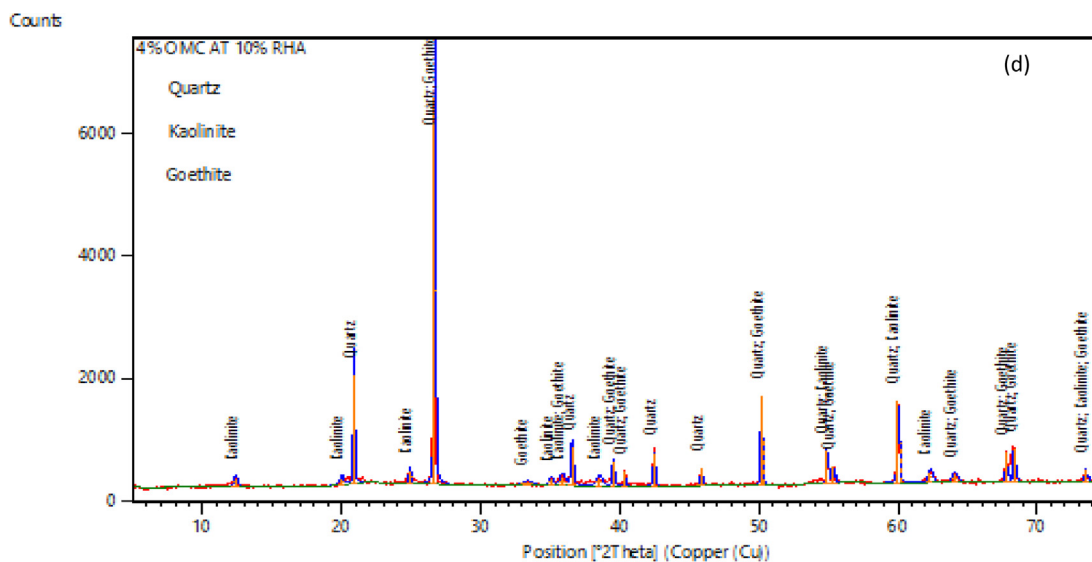


Fig. 10 (continued)

in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and composition. Given sufficient light, the human eye can distinguish two points 0.2 mm apart, without the aid of any additional lenses. This distance is called the resolving power or resolution of the eye. A lens or an assembly of lenses (a microscope) can be used to magnify this distance and enable the eye to see points even closer together than 0.2 mm. The scanning electron microscope (SEM) produces images by scanning the sample with a high-energy beam of electrons. As the electrons interact with the sample, they produce secondary electrons, backscattered electrons, and characteristic X-rays. These signals are collected by one or more detectors to form images which are then displayed on the computer screen. When the electron beam hits the surface of the sample, it penetrates the sample to a depth of a few microns, depending on the accelerating voltage and the density of the sample. Many signals, like secondary electrons and X-rays, are produced as a result of this interaction inside the sample. Electron microscopes utilize the same basic principles as light microscopes, but focus beams of energetic electrons rather than photons, to magnify an object. Electron microscopes use electrons for imaging, in a similar way that light microscopes use visible light. SEMs use a specific set of coils to scan the beam in a raster-like pattern and use the electrons that are reflected or knocked off the near-surface region of a sample to form an image. Since the wavelength of electrons is much smaller than the wavelength of light, the resolution of SEMs is superior to that of a light microscope. Geotechnical engineers are trying to include some of the advanced technologies in nondestructive testing methods. X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) are two of them. XRD is used for the mineralogical analysis of the stabilized soil and SEM is used for the morphological observation and surface analysis of the soil. A mineralogical analysis is the study of materials to determine the mineral composition and mineral structure.

## Discussion of results

### General characteristic results

Preliminary investigations show that the reference soil is classified as A-7 soil and poorly graded (CL) according to AASHTO and USCS, respectively, as presented in Figs. 6 and 7. Fig. 6 shows that the reference soil is highly plastic and high liquidity. Fig. 7 shows the improvement recorded in the particle size arrangement with the increased

addition of rice husk ash from poorly graded to improved grading arrangement. Fig. 8 shows the compaction results of the natural soil. Table 1 presents the effect of the increased addition of rice husk ash on the Atterberg limits and specific gravity of the treated soil.

### Effect of moulding moisture and rice husk ash on the mineralogical behaviour of the treated soil

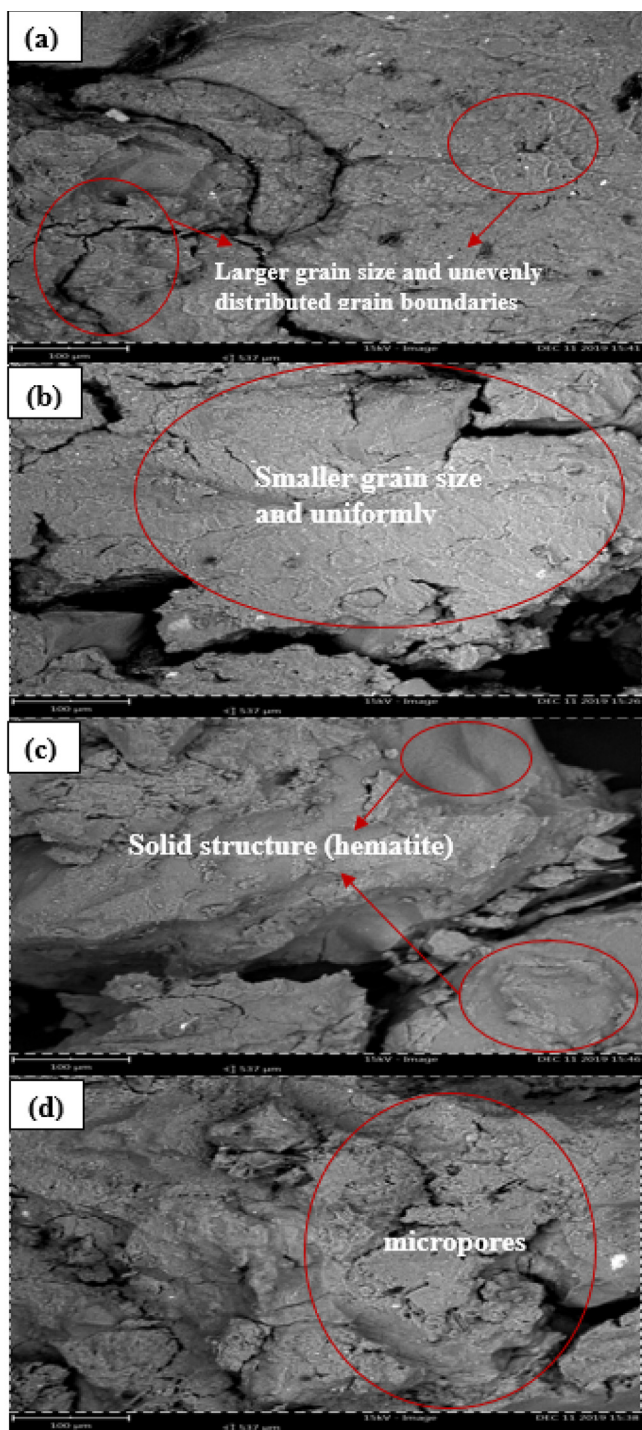
The XRD spectra indicating the mineralogical compositions of both RHA treated and untreated soils are shown in Figs. 9 and 10. At 2% OMC, the XRD spectra of the soil samples treated with RHA indicate the presence of quartz, kaolinite, and hematite as shown in Fig. 9(b, c & d). The untreated soils which indicate the presence of quartz and kaolinite only as shown in Fig. 9a contradict the results of the SEM analysis shown in Fig. 11a which indicates that the soil sample contains 32.60% of Si, 30.55% of Fe, and 19.67% of Al. This could be as a result of the error arising from the inability of the XRD machine to detect the presence of iron oxide or as a result of the molding moisture. The presence of hematite (oxides of iron) in combination with quartz and kaolinite in the treated soil samples implies that the composite (RHA and lateritic soil) is a pozzolanic material and will likely become cementitious when it reacts with  $\text{Ca}(\text{OH})_2$  in cement which makes it a good material for landfill liner. However, at 4% OMC, both the XRD spectra of RHA treated and untreated soil contained quartz, kaolinite, and hematite as shown in Fig. 9(a & b) and this was confirmed by the result of SEM analysis shown in Fig. 12(a & b). The presence of goethite alongside quartz and kaolinite were observed in the XRD spectra of 6% and 10% RHA treated soil as shown in Fig. 9(c & d). The Goethite possessing an inner needle-like structure with a closed packed striated structure makes the composite a promising material for constructing landfill liners. This is because the closed packed striated structure of the goethite present in the composite will slow down the vertical seepage of leachate to allow its collection and removal by the leachate collection system and this agrees with the results of Vodyanitskii (2016). The composite will form a barrier between groundwater, soil, and substrata, and waste.

### Effect of moulding moisture and rice husk ash on the morphological and elemental disposition behaviour of the treated soil

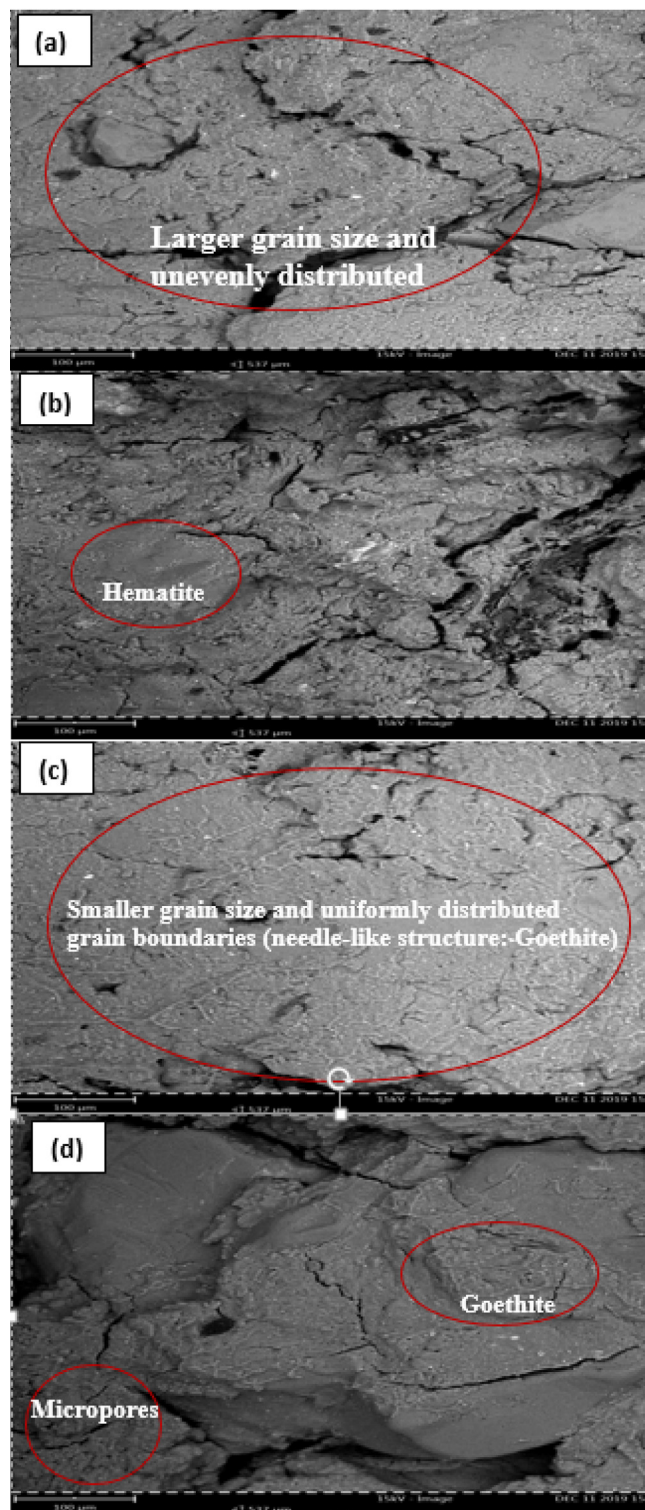
The microstructures of both RHA treated and untreated soils are shown in Figs. 11 and 12. The presence of micro-pores was observed

in the composites treated with 10% RHA as shown in Fig. 11(d) and Fig. 12(d). This could be as a result of stabilizing the soil to an excess of the optimum percentage of RHA. In Fig. 11(b-d) and Fig. 12(b-d) stabilized with RHA, there are indications of an increase in the surface area of the particles compared to Fig. 11(a) and Fig. 12(a) without RHA due to the agglomeration between the lateritic soil and the stabi-

lizer (RHA) during pozzolanic reaction and binding. Additionally, high numbers of monocrystalline grains were observed in all microstructures of the samples indicating that the soil has undergone extensive



**Fig. 11.** (a) 2% wet of OMC with 0% RHA {Si of 32.60%, Fe of 30.55%, Al of 19.67%} (b) 2% wet of OMC with 2% RHA {Si of 36.34%, Fe of 23.21%, Al of 20.53%} (c) 2% wet of OMC with 6% RHA {Si of 36.22%, Fe of 27.40%, Al of 20.87%} (d) 2% wet of OMC with 10% RHA {Si of 37.93%, Fe of 21.38%, Al of 18.95}



**Fig. 12.** (a) 4% wet of OMC with 0% RHA {Si of 36.99%, Fe of 21.96%, Al of 18.84%} (b) 4% wet of OMC with 2% RHA {Si of 39.32%, Fe of 20.01%, Al of 20.27%} (c) 4% wet of OMC with 6% RHA {Si of 39.50%, Fe of 19.92%, Al of 21.51%} (d) 4% wet of OMC with 10% RHA {Si of 42.86%, Fe of 19.87%, Al of 18.08%}

weathering during erosion. The presence of a solid structure mineral (hematite) and an inner needle-like structure with a closed packed striated structure (Goethite) was observed in Figs. 11 and 12. The resulting composite is a good material for constructing a landfill liner system. This is because the closed packed striated structure of the goethite and the solid structure of the hematite will retard the lateral movement of landfill gas from the landfill and will ultimately prevent its infiltration into the groundwater. This protects the groundwater from the impacts of the leachate. The size of the grains becomes smaller and the grain boundaries are uniformly distributed after stabilization with RHA as shown in Fig. 11 (b-d) and Fig. 12c, when compared to Fig. 11, (a) with larger grain size and grain boundaries that are unevenly distributed. Uniformly distributed grain boundaries and smaller grain size of the composite (lateritic soil and RHA) will serve as a barrier to the movement of contaminants and other leachates to the groundwater and thus, making the composite a viable material for the landfill liner system. Figs. 11 and 12 indicates that the two different molding moisture percentages explored in this study did not have a significant effect on the morphology and elemental disposition of the treated soil. The summation of the percentage oxides of silicon, aluminium, and iron is up to 70% as shown by the results of the elemental analysis presented alongside Figs. 11 and 12. This implies that the materials are pozzolanic and will become cementitious when it reacts with CaO in the presence of water (Obianyo et al. 2020). The Ca (OH)<sub>2</sub> in cement will bind the soil particles together and will prevent the seepage of leachate into the soil. These behavioral changes are in line with the findings of Liu et al. (2021) and Onyelowe et al. (2019) where moisture changes affect the morphological arrangement of untreated and treated soils, especially problematic soil. As the moisture impregnation index changes, the surface microstructures are affected thereby rearranging the mass of soil inter-particle structure at the micro and nano levels. The increase in molding moisture on one hand increases the microstructural bond in soil mass reducing to zero the leachate flow effect of the compacted landfill liner, while the addition of binder materials like the RHA in this case on the other hand improved the cementing surface and closing the gaps between particles from transmitting leachate and this agrees with the findings of Sunil et al. (2008) and Harun et al. (2014).

## Conclusions

The effect of molding moisture and rice husk ash treatment on the morphological structure and mineralogical composition of soil utilized as landfill liner has been studied using Xray diffractometer and scanning electron microscopy methods under laboratory conditions. From the foregoing, it can be concluded that increased moisture improved the agglomeration reaction between the green material composition of silicate and aluminates to form the hydrates of these compounds. This development further enhanced the filling of the micro-pores within the treated soil by producing a gel in an aqueous form thereby yielding a mesh of polymer chains, which forms interlinking solid masses. Micro-fillings as observed in the microstructural configuration changes were also due to the elemental particles, which were enhanced by increased molding moisture and admixture content. With the above results achieved in this exercise, molding moisture increase and rice husk ash addition have shown to improve the pores by micro-filling. This has shown that rice husk ash as a product of agricultural processing is a good green construction material to improve the leachate barrier of landfill clay liner for sustainable construction.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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