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Appraising the impacts of binary blends of limestone powder – metakaolin mixtures on the geomechanical behaviour of black fine-grained soil and its microstructural evolution

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Abstract

This study strives for the usage of zero - cement/lime binders on the geomechanical performance of black fine-grained soil ameliorated with the mixtures of waste derivatives. In a nutshell, this study is aimed at appraising the impacts of limestone powder (LP) and metakaolin powder (MP) on black expansive soil (BES) via mechanical and microstructural means. During the compaction testing, an increment in the dosage of additive materials resulted in a gradual increase in maximum dry density (MDDs) with a corresponding decline in optimum moisture contents (OMCs). The strength testing revealed that the incorporation of the LP-MP prompted a significant rate of improvement and it was affirmed through qualitative analysis. Furthermore, both the original and additive-treated soil materials were examined qualitatively via the means of scanning electron microscopy (SEM) and Energy dispersive spectroscopy (EDS) techniques. The result from the SEM testing indicates well-compacted soil chemistry whereas the EDS revealed higher peaks of aluminosilicate minerals which is a good pointer towards soil improvement.

Keywords: Fine-grained soil, Limestone powder, Metakaolin powder, Mechanical performance, Microstructural chemistry, Soil re-engineering

1. Introduction

Civil infrastructure such as roads remains one of the most critical success factors in modern-day society [1]. A large number of these road infrastructures are built with the intent that they aid in the rapid and safe transportation of goods and services during their design life. During the execution of these civil infrastructures, soils are the most consumed materials and they form the subgrade layer of roads. The subgrade layer is the foundation for road infrastructures and as such, it is of great importance the subgrade materials should be of good geotechnical standing. It is worth stating, that a good number of subgrade materials found in tropical zones like Nigeria contain some fractions of fines as well as fusions of clay minerals. The manifestation of montmorillonite minerals or other clay minerals is a strong indicator of weak/expansive soil. Black cotton soil is a prototype of expansive soil and it is known for its swell-shrink actions, especially during seasonal changes. As a result of their swell-shrink actions, low strength etc. they are cancerous and not suitable in their natural form for usage in civil engineering applications. Thus, the presence of this soil underneath the foundation of road infrastructures is normally linked with stability issues which have also been a source of worry for civil engineers. For instance, the existence of such soil has led to colossal damage to road pavements, buildings, railways and retaining walls due to their low strength and cyclic swell-shrink actions [2-5]. For that reason, the mitigation strategies to overcome such type of soil are very crucial to materials and construction engineers. In a bid to mitigate this undesirable behaviour of fine-grained soil, a host of amendment strategies have been evolved over the years by soil scholars [6-8].

The choice of the amendment strategy is governed by the achievability of a particular strategy, the intended properties of the marginal soil to be amended and the associated costs. The use of solid waste residue in soil amendment studies is one of the strategies that involve the systematic integration and admixing of additives with soil materials. The main aim of amending the geomechanical parameters of feeble soil comprises lessening the plasticity, swelling potentials and increment in strength and as well as durability. Both Nigerian and foreign soil investigators such as [9-12] have intensified efforts in the quest to focus on the utilisation of solid waste residue in strengthening protocols of marginal soil. However, the effective utilization of these waste residues could be beneficial to man, and the environment and it will also act as a cost-effective and eco-friendly soil strengthening strategy. The utilization of waste residue as supplementary cementitious materials has been in vogue lately and it has become a research hotspot in modern times. Thus, the use of this waste residue being the major source of calcium-silicate needed in soil amelioration has been reported by materials and construction experts [10, 12-14]. The experimental outcomes of waste residue such as oyster shell ash [15-17] limestone powder [10, 18-20], groundnut shell ash [21, 22], periwinkle shell ash [23, 24], metakaolin [12, 25], cement kiln dust [26-28], rice husk ash [29, 30], bambara nut shell ash [31], quarry fines [32, 33], lime–rice husk ash [34, 35], lime-periwinkle shell ash [36], cement kiln dust-rice husk ash [37, 38], quarry fine–cement kiln dust [39], rice husk ash-sisal fibre [40], cement kiln dust-metakaolin [41, 42], sawdust ash – quarry dust [43], cow bone ash – waste glass powder [44], groundnut shell ash – metakaolin [45], scrap tyre crumb rubber [46]

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either as single or binary additives have shown some level of potency both in mitigating unstable fine-grained soil or as cement replacement in concrete works. A good number of experimental works have proven that the mixtures of silicate and aluminate establishment are the brain behind the mechanical property improvement of soil treated with additive, lime-additive and cement-additive [47, 48]. In this regard, field practitioners [12, 20] have reported the potency of limestone powder (LP) and metakaolin powder (MP) in soil r-engineering. With the advancement in soil strengthening strategy, material and construction investigators are enticed to utilise solid waste derivatives (limestone powder and metakaolin powder) as additives which offer cost-effective solutions and promote a sustainable environment. Owing to the numerous benefits of the use of solid waste derivatives in soil strengthening protocols, investigators and soil practitioners have meticulously carried out experimental works in this field. The main advantages of using solid waste derivatives include a decrease in the cost of stabilization [49], a lessening of deformability of weak clayey soil [18] and so on. From the overview of the literature cited, it is evident several authors have explored the feasibility of limestone powder or metakaolin powder for soil amelioration studies either as a standalone or combined with other additives.

In this regard, Attah et al. [25] modelled and predicted CBR values of marginal soil amended with metakaolin powder and found metakaolin as a potent supplementary cementitious material for the amelioration of soils for use as construction material. Recently, Dao et al. [12] used varying contents of metakaolin in the treatment of lateritic soil and concluded that 6 % metakaolin instigated the peak response in terms of mechanical performance. On the other hand, recent experimental works have shown that incorporating limestone powder in cement-treated saline soil decreases soil plasticity, increases maximum dry density, improves strength parameter, reduces volumetric swelling and moisture susceptibility of soil [19]. Also, an increase in the strength and a reduction of its deformability properties of the mixed soil was found by Pastor et al. [18] when limestone powder was incorporated.

Howbeit, a plethora of these studies have been documented on the strengthening of soil's mechanical properties, to the best of the author's knowledge there exists minute or no known research work documented in the public domain on the microstructural chemistry of fine-grained soil treated with limestone powder – metakaolin powder. It is against this backdrop, that there is a need to appraise the impacts of the mixtures of limestone powder and metakaolin powder on the geomechanical response of fine-grained soil. Secondly, this study explored the microstructural machinery via scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX). Thus, it is well hoped that this present investigation will make available new intuitions in the field of soil strengthening protocols.

2. Materials and methods

2.1 Constituent materials

In the course of this investigational study, locally sourced soil material known as: black expansive soil (BES) together with two locally sourced additive materials also known as: metakaolin powder (MP) and limestone powder (LP) were utilised (see Figure 1). Black cotton soil which is a prototype of black expansive soil (BES) sourced within the Northern-Eastern geopolitical zone of Nigeria, specifically from Deba province, Gombe State, Nigeria (on GPS co-ordinates of 10° 12′ 42.73″ N and 11° 23′ 13.56″ E) was utilised for this investigational research. The BES was sourced from its deposit at an approximate depth of 1.5 m via means of a disturbed sampling technique and transported to the laboratory. Kaolin clay materials were sourced from a deposit in Ohiya, Umuahia South Local Government Area, Abia State (on GPS co-ordinates of 5° 29′ 19″ N and 7° 30′ 16″ E). Thereafter, to produce the MP needed for soil amendment studies, the raw kaolin samples were broken into smaller particles, sundried and subjected to a calcination process at a temperature setting of 800°C. The visual inspection MP used in this study showed that it is odourless and fine in texture with an off-white colour. The exploited LP in this investigation was obtained from a natural quarry site located at Mfamosing, Akamkpa, Cross River State (on GPS co-ordinates of 5° 6′ 41″ N and 8° 30′ 35″ E).

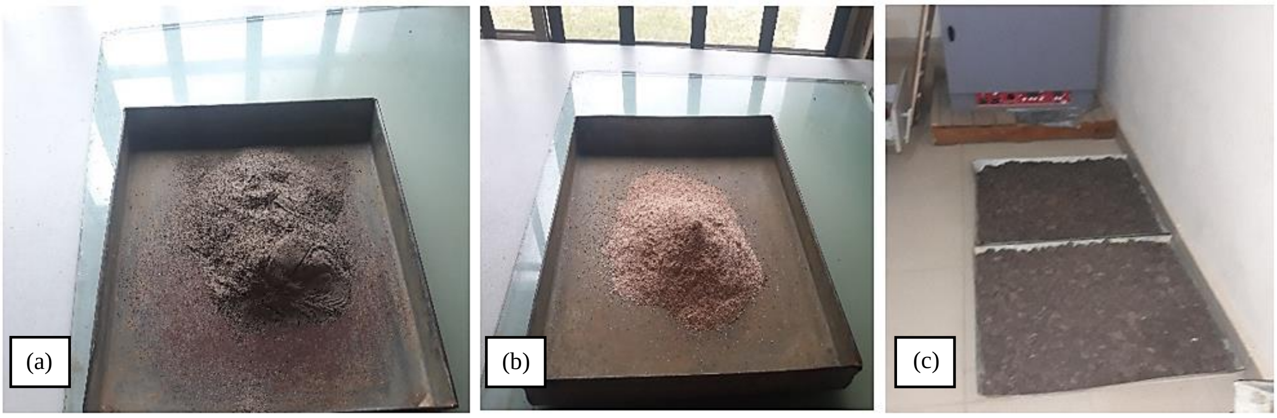

Figure 1 (a) Limestone powder, (b) metakaolin powder and (c) black expansive soil.

2.2 Experimental approach

The elementary set of experiments comprised of basic geotechnical testing such as: grain size distribution, specific gravity and Atterberg limits were executed based on the conditions of BS 1377 [50] to classify and characterize the unaltered BES. A step further, mechanical testing including compaction, CBR, UCS and microstructural investigations like SEM and energy dispersive X-ray (EDX) were employed on both the unaltered and the additive-altered soils. The soil materials (BES) considered for the experimental protocols were prepared by incorporating dosages of 0-8 % of LP and 0-10 % of MP in an increment dosage of 2 %, respectively by dry weight of soil to amend the deficient soil. This amendment protocol was executed with reference to the regulations stipulated in BS 1377 [50] and 1924 [51], respectively. After the characterization of BES in its unaltered form, the additive materials (MP and LP) were measured in their required dosages and admixed with the two studied soils independently until a homogenous blend was accomplished. It is worth stating, that all mechanical testing in this study was accomplished on both untreated BES and BES admixed with various dosages of the additive materials. To attain the maximum dry density (MDD) and optimum moisture content (OMC) for each soil mixture, the British Standard Light (BSL) approach was engaged and it involves the use of 2.5 kg rammer and a standard proctor mould. Subsequently, to appraise the strength capacity of both the unaltered and additive-altered BES specimens, CBR testing was engaged based on the conditions postulated in the Nigerian General Specification [52]. As indicated earlier, the compaction of soil samples for the CBR testing was based on BSL energy under both soaked and unsoaked conditions, respectively. The CBR test was done in accordance with BS 1377 [50] and 1924 [51] for the natural and treated soils. Curing of the specimens used for the CBR test was done for 6 days and after the sixth day, the specimens were submerged in water for 48 hours before testing. The curing period used was as per the requirements of the NGS [52]. The UCS testing was executed as established by the principles recorded in BS 1377 [50] and 1924 [51], respectively for both the unaltered and additive-altered BES specimens. For the period of the UCS experiment, the soil mixtures were achieved utilizing their corresponding OMCs arrived at during compaction and were later subjected to a curing exercise for a period of 7 and 14 days before carrying out the test. In this present-day manuscript, for us to properly appraise the impact of the additive materials on the micro-fabric chemistry of the marginal soil, non-destructive mechanisms such as scanning electron microscopy (SEM), energy dispersive X-ray (EDX), fibre histograms and X-ray diffraction (XRD) techniques were engaged on both the unaltered and additive altered soil. The underlying disparities in terms of soil morphology were understudied using the SEM technique whereas the features of chemical analysis being discovered in the SEM examination were provided by the EDX testing. The non-destructive mechanisms were engaged because of the verdict by Choobbasti and Kutanaei [53], who reported that the geotechnical response of soil material is a function of the soil's microstructure.

3. Results and discussion

3.1 Outcomes of unaltered soil characterisation

Both the outcome of the characterisation protocols and as well as the particle sieve fraction plot carried out on the tested soil are accessible in Table 1 and Figure 2, respectively. Looking at Table 1, more than 35 % of the tested soil in its natural state used for the grain size gradation passed through 75 microns sieve and this is a pointer that the understudied soil is a fine-grained soil material. As per AASHTO [54] characterisation, the soil's plasticity index (PI) of 28.70 % by far exceeds the 11 % maximum requirement and soil BES belongs to the A-7–6 (14) whereas USCS systems [55] rate it as CH soil material. However, the three constituent materials which include BES, MP and LP had a specific gravity of 2.40, 2.58 and 2.65, respectively and their chemical oxides can be seen in Table 2. The specific gravity of the additive materials (MP and LP) was found to be far less than that of Portland limestone cement (3.15). The X-ray fluorescence used in analysing the chemical oxide of the materials proves the high dominance of oxides such as silica, calcium and alumina which are answerable for imparting pozzolanic interplay.

Table 1 Characterisation of BES.

Property of soil material	Standards	Values
NMC; %	ASTM D 2216-10	20.20
Percentage passing 75 microns	ASTM D 2487-11	71.99
LL; %	ASTM D 4318-10	56.30
PL; %	ASTM D 4318-10	27.60
PI; %	ASTM D 4318-10	28.70
LS; %	BS 1377	18
FS; %	-	53.50
Gs	ASTM D854-15	2.40
MDD; Mg/m^3	ASTM D 698-15	1.61
OMC; %	ASTM D 698-15	18
CBR; %	ASTM D 1883-05	3
7 days UCS, kN/m^2	ASTM D 2166-16	105
AASHTO classification (Group Index)	AASHTO	A-7-6 (14)
USCS classification	ASTM D 2487-11	CH
Colour	-	Greyish black

Table 2 Chemical oxides of the BES, MP and LP.

Oxides		SiO_2	CaO	SO_3	MgO	TiO_2	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
Mass fraction (%)	BES	48.50	0.90	-	2.22	-	2.20	18.60	1.55	0.70	10.10
	MP	52.72	0.18	0.99	0.09	-	1.72	42.20	-	-	0.25
	LP	7.25	60.50	0.24	2.58	0.03	0.04	0.95	0.19	0.10	21.65

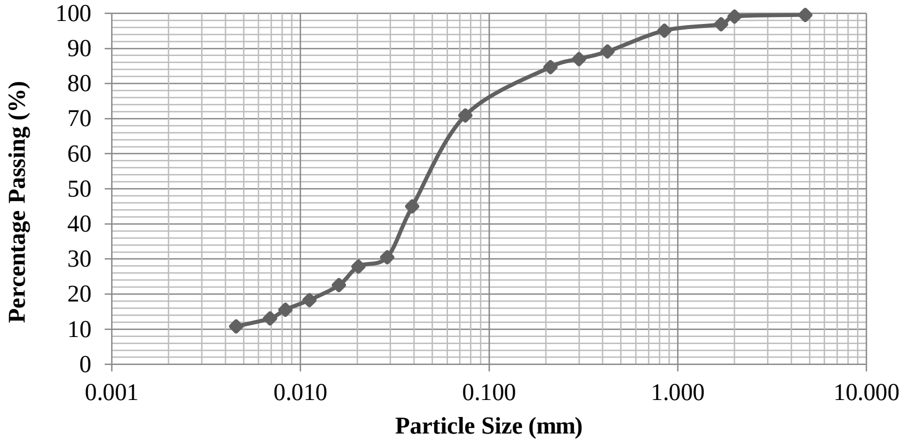

Figure 2 Particle sieve distribution curve of BES.

3.2 Impacts of MP-LP on compaction behaviour of BES material

3.2.1 Maximum Dry Density (MDD)

The relationship between the maximum dry density (MDD) of BES material and the MP for the various doses of LP via British Standard Light (BSL) compaction energy is shown in Figure 3. For the no additive soil sample, the MDD value of 1.615 Mg/m^3 was documented. The incorporation of these additives to ameliorate the geomechanical properties of the soil portrayed a sizeable impact on altering the MDD of the soil sample. It is unmistakably seen that the MDD was improved until the utmost values were achieved by the incorporation of MP at different BES-LP mixtures. Moreover, the inclusion of additives into the soil prominently enhanced the compaction behaviour as an upsurge in MDD was realized up to 8% LP – 8% MP dosage. Further than 8% LP – 8% MP, there was a decline in MDD value which is a pointer towards an optimum content of additive for compaction exercise [56]. As seen in Figure 2, the increments in MDD might not be unconnected to the useability of additive materials with higher specific gravity compared to the natural BES [57]. In addition, the increasing trend noticed in the MDD of the additive–soil materials could also be enlightened in terms of the additive mixtures filling the soil structure which in return causes a rise in the weight of the soil mixtures. It is also perceived that the rise in MDD might be attributable to increased workability instigated by the additive materials. Thus, it is worth stating that the inclusion of these additive materials steered the flocculation and agglomeration of soil mixtures, accompanied by cation exchange interplay within their surface. On a general note, there are some level of contradictions in the literature concerning the influence of additives on compaction performance but our upshots are comparable to the discoveries by other field practitioners [25, 58].

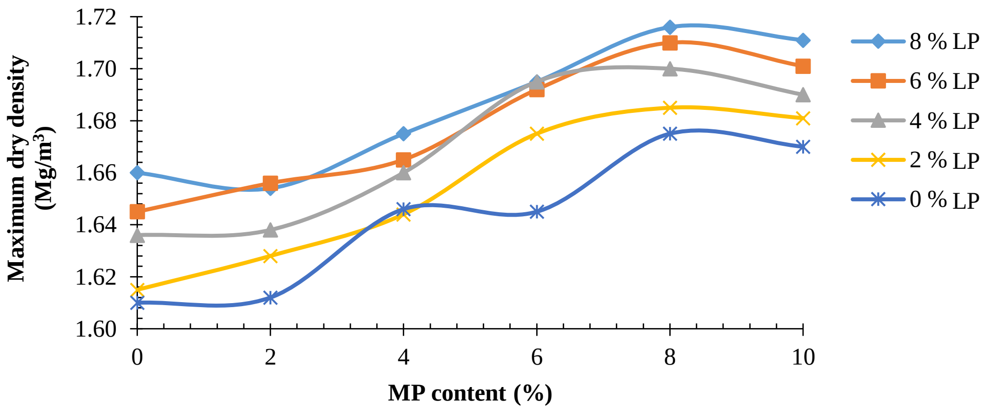

Figure 3 Relationship between MDD and MP at different LP content.

3.2.2 Optimum Moisture Content (OMC)

The chart depicting the OMCs generated as a result of the melioration exercise is revealed in Figure 4. The chart indicates generally an increasing tendency in OMCs as the dosage of LP augmented followed by a resultant depreciating tendency with the dosages of MP increased. Probably, the motive behind the increase in OMCs with increased LP concentration may not be far from the non-plastic nature of LP [20], as well as the build-ups of fine particles which facilitates the expansion of surface area [38]. In addition, the OMCs progress with an increase in LP dosages as the flocculated particles inhabit the larger space within the soil matrix [59]. On the other hand, the soil-additive OMCs depreciated continuously with an increase in MP concentrations, this might be linked with the increasing portion of coarser material which in return diminishes both the silt and clay portion, respectively. Secondly, the depreciation of OMCs might be due to the quest for a small quantity of water desirable for pozzolanic interplay with the clay portions of the soil [60]. This witness interplay might as well be answerable to the modification of clay particles from face-face alignment to a more compact edge-face alignment [61]. Furthermore, the witnessed upshot might perhaps be a consequence of cation exchange that triggered the alteration in the orientation of the clay particles. Even though there are discrepancies in the trend of additive-treated soils, the upshots of this paper are corroborated by the findings of numerous soil scientists including [24, 61].

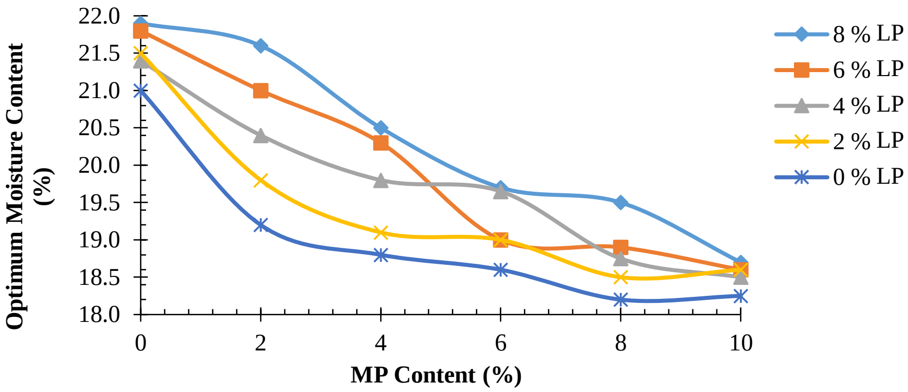

Figure 4 Relationship between OMC and MP at different LP content.

3.3 Impacts of MP-LP on California Bearing Ratio (CBR) of BES material

In the process of designing pavement for civil engineering infrastructures, the CBR value is an essential factor that aids in appraising the appropriateness of materials for usage either as a sub-grade, sub-base or base course material. The upshots of the CBR (soaked and unsoaked) testing on the BES-LP-MP mixtures are publicised in Figure 5 (a) and (b). The exploration of Figure 5 (a) demonstrates that the CBR testing for the soaked condition alternated from 3 to 29 % for all MP contents at all MP doses. The effect of blended LP-MP on the CBR samples disclosed a weighty strength enhancement as the additive materials were added. From the plots, it is witnessed that the addition of additives played an influential part in the development of strength. At the dosage level of 8% LP – 8% MP, there is a weighty increment in strength to a peak value of 29 %, beyond this point there was a noticeable depreciation and this trend of result is not at variance with other scholars [62, 63]. In the same way, Figure 5 (b) depicts the unsoaked CBR performance of BES ameliorated with LP – MP dosages using British Standard Light compaction energy. From the plots, the CBR performance of BES in its unaltered stage was 5 %, whereas the maximum upsurge in strength performance of 41 % was witnessed at an additive blending dosage of 8% LP – 8% MP. This detected rate of amelioration could be interlinked with the high dominance of silica supplied by the BES undergoing some chemically activated interplay with some amount of calcium hydroxide established after the hydration of cement compounds. The strength development of the unsoaked CBR outcome was not dissimilar to the soaked CBR even though with lower values and this behaviour might be interlinked with the soaking condition where water percolates into the compacted samples which in return depreciates its strength performance. Comparable verdicts were documented by preceding soil mechanics experts [38, 64]. At this juncture, it is worth saying that from the CBR plots, BES in its unaltered form had CBR (soaked and unsoaked) values of 3 and 5 % and adding the mixtures of 8% LP – 8% MP steered an optimal CBR performance of 29 and 41 % which translates to about 9 and 8 % increment. However, looking at the topmost value of the soaked CBR, it is found to surpass the minimum 20 % and maximum 30 % requirement for sub-base materials (Nigerian General Specification [52]. For the unsoaked CBR, the peak CBR value fell far beneath the minimum 60 % and maximum 80 % for base materials for materials compacted at the optimum moisture content but also overlapped the earlier adopted benchmark (Nigerian General Specification [52].

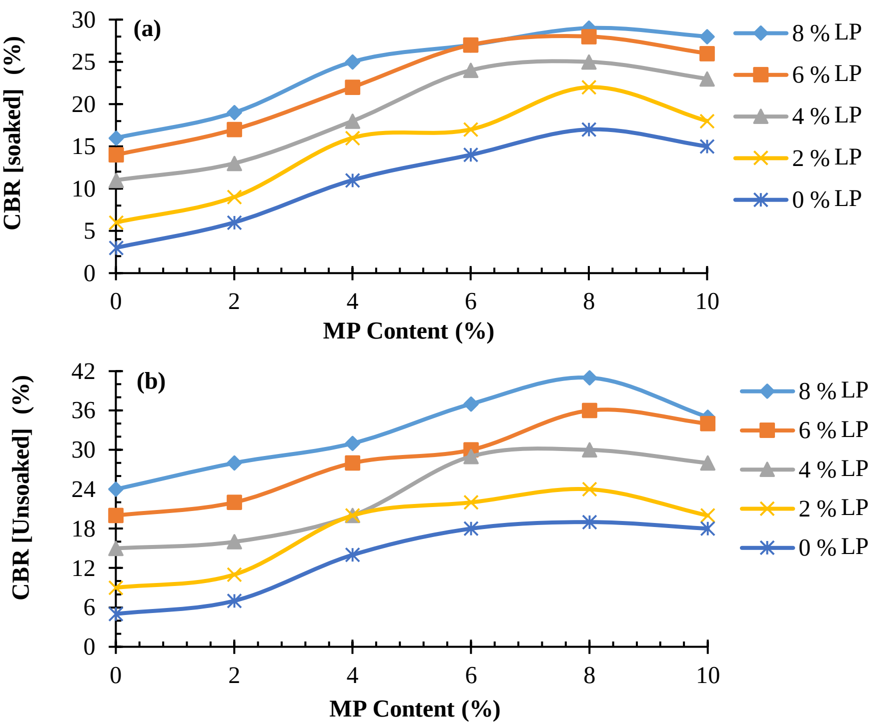

Figure 5 Plots of (a) soaked CBR and (b) unsoaked CBR of soil-MP-LP mixtures

3.4 Impacts of MP-LP on the Unconfined Compressive Strength (UCS) of BES materials

In the course of executing soil re-engineering protocols, mechanical testing such as unconfined compressive strength (UCS) can be utilised to achieve the anticipated quantity of additive material. The UCS experiment of LP against the percentage of numerous MP amelioration levels cured for 7 and 28 days via the British Standard Light (BSL) compaction energy is described graphically in Figures 6 (a) and (b). In the course of the 7 days UCS experimentation, a substantial enhancement in strength was recorded as the blends of LP-MP were added. Secondly, from the UCS plots, it is also noticeable that the curing duration is a critical success factor in the development of strength within the soil mixtures. The information in Figure 6 (a) points toward the fact that as the dosage level of additives rises, the UCS upsurges progressively. At this juncture, it can be seen that the 7 days UCS values altered from 105 to 545 kN/m^2 for natural black fine-grained soil and additive blended black fine-grained soil, respectively. This trend of response could be adduced to the fact that the binary additive materials replace the finer particles sourced from the fine grain soil during compaction which thereby imparts higher mechanical performance of treated soil. Exploring the result of 28 days UCS as shown in Figure 6 (b), it indicates that there is a parallel behaviour compared to the 7 days UCS. However, the addition of additives into the soil reflected an elevation from its lowest UCS value of 189 kN/m^2 for soil without additives to 720 kN/m^2 for soil additive composite. On a second thought, it is also noticeable that the curing period is a critical success factor in the development of strength. In a nutshell, the curing duration correlates positively with the UCS values for all soil composites and this is a strong indicator of pozzolanic interplay taking place between the additives and soil material. It is believed that this interplay led to the establishment of new cementitious products which was established by the soil's microstructural chemistry. The interplay within the soil composite is explainable in the equation below:

$$Ca^{2+} + 2(OH)^{-} + SiO_2 \rightarrow CSH \quad (1)$$

$$Ca^{2+} + 2(OH)^{-} + SiO_2Al_2O_3 \rightarrow CASH \quad (2)$$

The equation shows that Ca^{2+} in the presence of SiO_2 combined with Al_2O_3 stimulates C-S-H and C-A-S-H gels which is responsible for the binding forces within the soil composite and increases soil strength [65, 66]. However, the combined impacts of additive materials unquestionably indicated a significant upsurge in the UCS upshots. This tendency of upshots is not new in transportation geotechnique and it might not be too far from the fact that the additive materials act as a good micro filler material thereby occasioning in diminishing the plasticity of the soil which therefore translates to an enhancement in soil's mechanical behaviour. This kind of outcome is not in disagreement with other soil material experts who engaged solid waste derivatives in soil strengthening protocols [67]. Additionally, the increase in additive dosages triggered the values of both 7 and 28 days UCS to noteworthy levels of 545 and 720 kN/m^2 with 8% LP – 8% MP mixture and beyond this optimum additive combination, there was a slight decline in UCS. In a nutshell, this depicts the optimal additive combination for re-engineering black fine-grain soil material. As for the general rating of the soil materials, the peak 7 and 28 days UCS values of the optimally treated soil were found far below the strength rating of 1034 kN/m^2 as postulated for adequate lime stabilization [68]. Comparatively, the uttermost UCS values for both curing durations were 545 and 720 kN/m^2 and were within the boundary conditions of 687– 1373 kN/m^2 for the sub-base material in highway infrastructure as documented by Ingles and Metcalf [69].

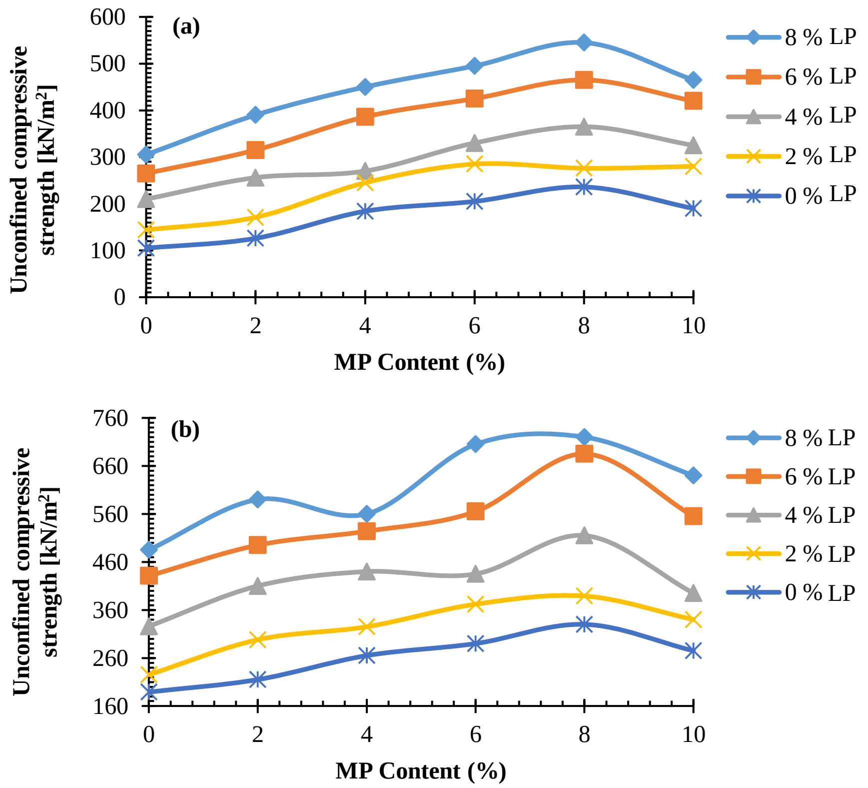

Figure 6 Plots of (a) 7 days UCS and (b) 28 days UCS of soil-MP-LP mixtures

3.5 Impacts of MP-LP on microstructural chemistry of soil materials

With the resolve to appraise the microstructural and elemental chemistry stimulating the mechanical behaviour of soil materials, scanning electron morphology (SEM), energy dispersive spectroscopy (EDS), fiber histogram and X–ray diffraction (XRD) tests of soil materials before and after amelioration protocols were conducted, respectively.

3.5.1 Scanning Electron Morphology (SEM)

The scanning electron microscopy (SEM) technique was deployed to establish the disparities in soil's microstructural/ morphological arrangement as a result of incorporating solid waste derivatives. Lately, soil experts such as [70-78] had engaged SEM characterizations to document essential information regarding the elemental compositions and mineralogy of studied soils. In view of this, the morphological characterizations at magnification levels of 300X and 500X for both the unaltered and additives-modified soil composite are revealed in Figures 7 and 8. Viewing the micrographs of these soil specimens, noticeable disparities exist in terms of pore size, particle arrangement and smoothness. As revealed in Figure 7, it appears there exists a fair connexion and a good number of pores within the soil structure. This observed morphology could be accredited to the non-existence of additive materials which also act as soil reinforcements and fillers. Generally, the upshot of the microstructural characterization depicts a porous microstructure and this demands the amelioration of the soil's mechanical properties. In summary, the micro fabric of the unmodified soil displayed some prominent fissures with a minute observable occurrence of inter-grain linkage within the soil structure. The noticeable fissures as seen in the soil fabric might be attributed to the high clay dominance which makes their affinity for water more noticeable. On the other hand, Figure 8 depicts that the additive-structured soil experienced some levels of micro-macro structural alterations which climaxed in the additive materials (filler) occupying the noticeable cavities in the untreated soil and also led to the formation of a well-dense-like structure. Also, the additive-structured soil showed the manifestation of whitish formation with the soil's structure and this might not be unconnected with the pozzolanic interplay thereby supporting the birth of CSH and C(A)SH products. It is believed that the incorporation of additive materials triggered the disparities in the soil's macrostructure and this is linked with the mechanical response of the ameliorated soil. There is no inconsistency in the material's reaction to this investigation when compared to other soil scientists who worked with related materials [79].

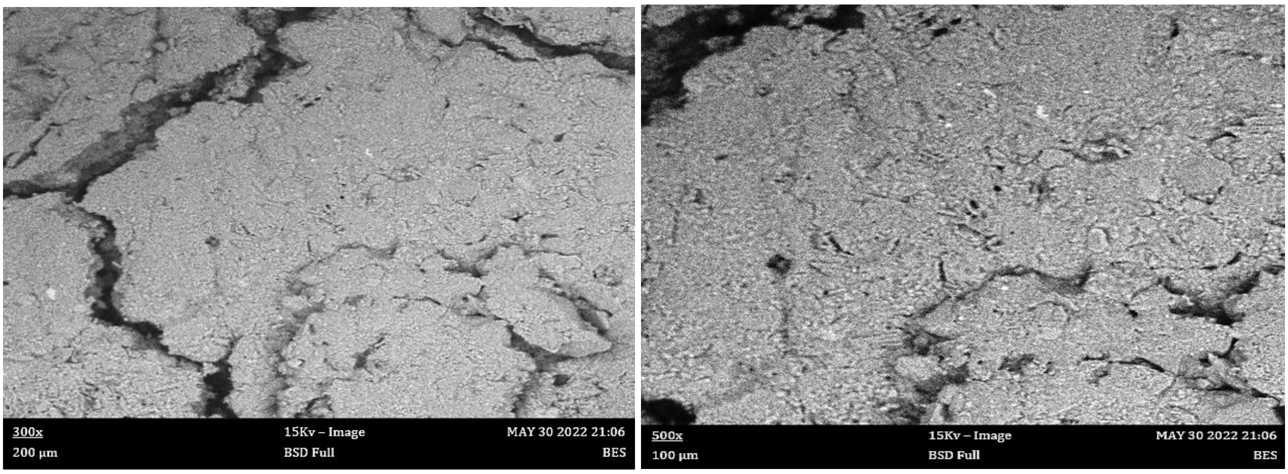

Figure 7 SEM appearance of unstructured BES

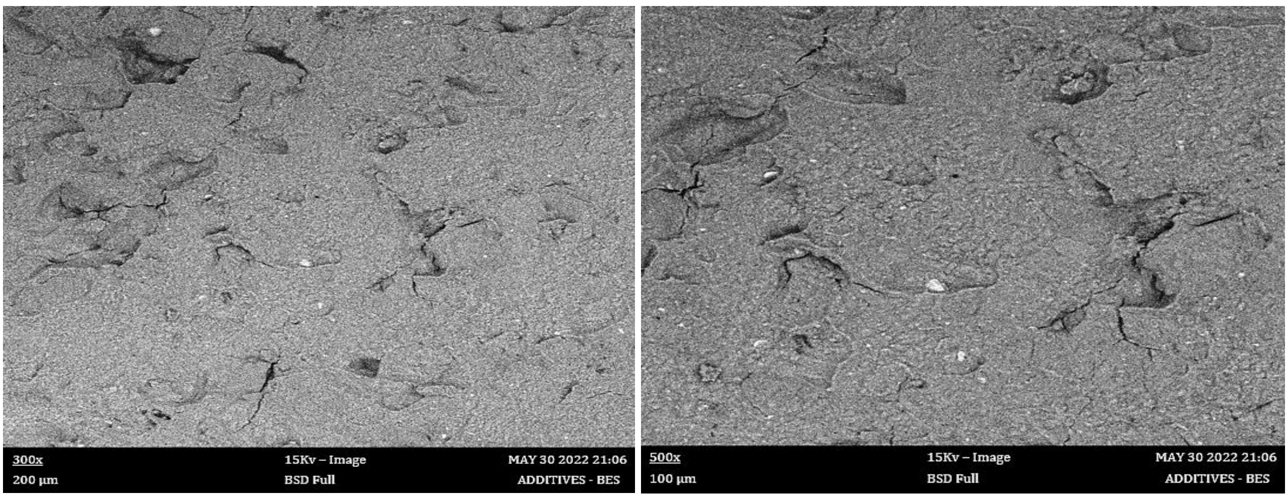

Figure 8 SEM appearance of additives structured BES

3.5.2 Energy Dispersive Spectroscopy (EDS)

Energy dispersive spectroscopy (EDS) is a strategy utilised for assessing soil's elemental composition and structural assessment. The spectra of additive materials (LP and MP) used in the study are displayed in Figures 9 – 10 and they signify a higher peak of calcium, silicon and aluminium. These results are in consonance with the chemical composition result of the materials as presented in Table 2 and it validates the feasibility of the additive materials in soil improvement studies. Viewing the EDS spectra of the raw BES depicts a major composition of calcium, silicon and aluminium which are also known as the aluminosilicate minerals [80], with a minor presence of elements such as iron, titanium, silver, potassium etc. The comparison between the spectrums of Figures 11 – 12 depicts some levels of disparities in the elemental composition. However, the noticeable disparities between the unstructured BES (see Figure 11) and the additives-structured BES (see Figure 12) might not be unconnected with the existence of additives in the soil, which in turn activated the pozzolanic interplay within the soil mixtures. This pozzolanic interplay is the fingerprint towards the mechanical response of the soil which was also affirmed by scanning electron microscopy testing. This discovery is not at variance with the study performed by Odumade et al. [57].

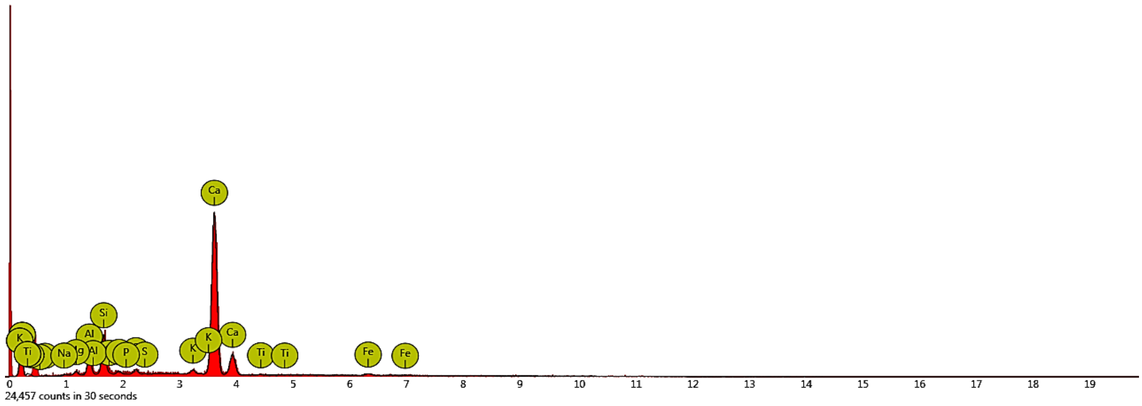

Figure 9 Energy-dispersive x-ray spectroscopy of LP

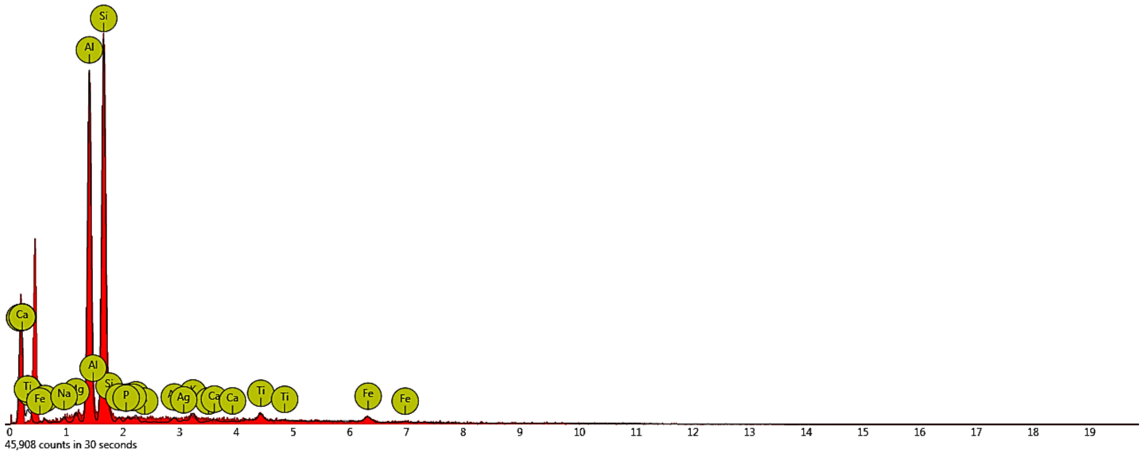

Figure 10 Energy-dispersive x-ray spectroscopy of MP

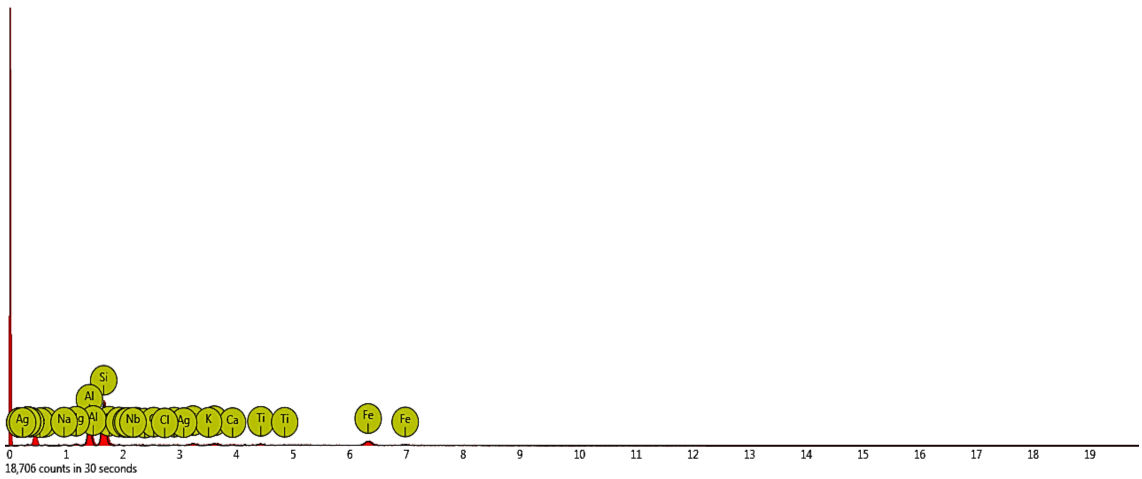

Figure 11 Energy-dispersive x-ray spectroscopy of unstructured BES

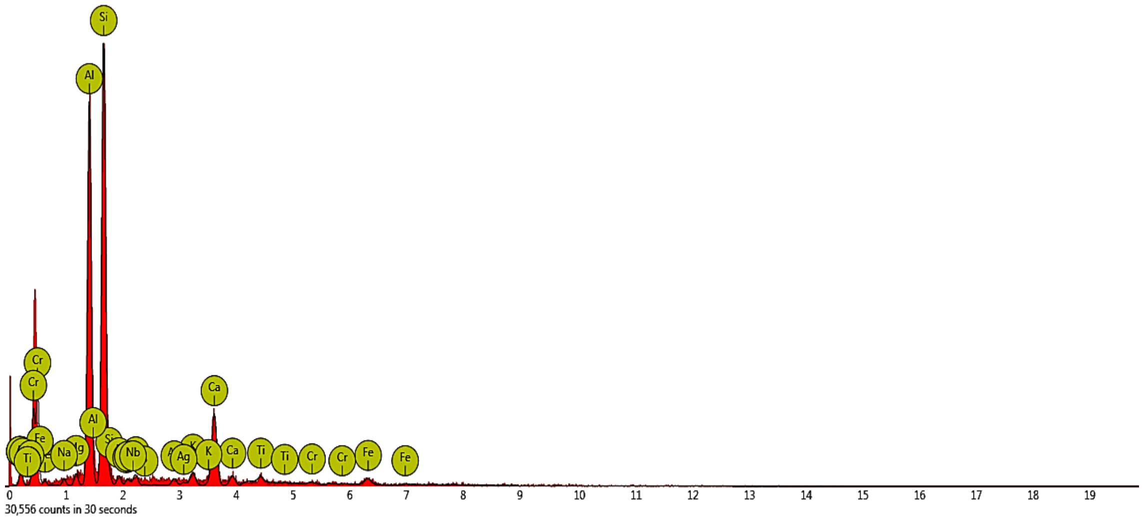

Figure 12 Energy-dispersive x-ray spectroscopy of structured BES

3.5.3 Fibermetric interface

The fibermetric interactive tool is a statistical component embedded in the SEM equipment utilised in unravelling the rationale responsible for the qualitative disparities such as pore sizes perceived within morphologies of the untreated and treated soil mixtures, respectively. The fabrics of the individual feeble soil and the additive-treated feeble soil in the form of fibre histograms are shown in Figures 13 (a) and (b). As seen in the fibre histograms, the pore length of the natural feeble soil alternated from its lowest value of 2.17 to the utmost value of 19.05 μm, whereas in the case of the additive-treated soil, it ranged between 1.28 and 44.65 μm. In contrast, this depicts approximate fabric lengths of 89 and 97 % for both the natural and additives treated soil, respectively. From the fibre histogram views, it can be seen that some noticeable dissimilar patterns exist by comparing the fibre lengths of the natural feeble and additive-treated feeble soil. Howbeit, the noticeable dissimilar patterns might not be too far from the distortion of the soil's chemistry as reported in the SEM analysis. Secondly, the alteration in fibre lengths of the soil materials could be a result of the migration of calcium silicate hydrate (CSH) and calcium aluminate hydrates (C(A)SH) from the additives which thereby activates pozzolanic interplay within the soil matrix. In soil mechanics, the documented upshots are indicative of the formation of cementitious compounds built in the course of chemical interplay. The discoveries in this study are not at variance with other soil practitioners who applied fiber histograms in soil re-engineering [24, 28, 81].

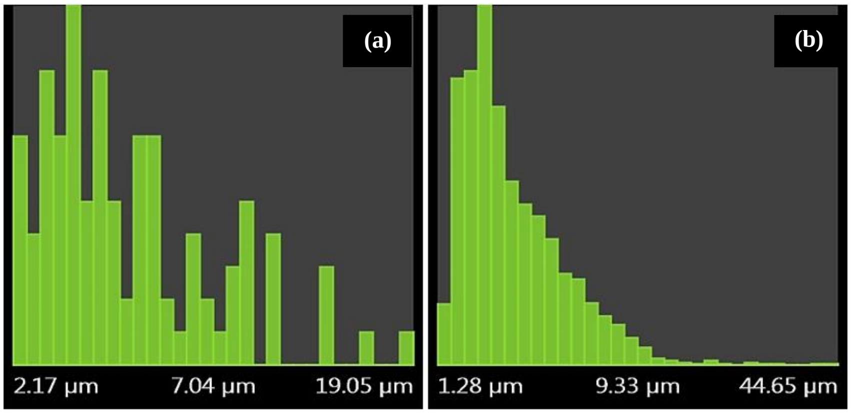

Figure 13 Fibre histogram outcomes of (a) unstructured feeble soil (b) additives structured soil

3.5.4 X-ray Diffraction (XRD) examination

The XRD testing enables a better understanding and identification of mineral crystallinity present in both the unaltered and optimally treated soil. The XRD pattern of the unstructured black expansive soil alone is shown in Figure 14 (a) whereas Figure 14 (b) shows the XRD pattern of the treated soil with mixtures of limestone powder and metakaolin powder. The major mineral present in the unstructured soil BES alone based on pronounced peaks in Figure 14 (a) is the montmorillonite with traces of quartz (Q) and kaolinite (K). The pronounced peaks of montmorillonite at several d spacing in the soil confirm the expansive tendency of the soil. According to Jha and Sivapullaiah [82], montmorillonite belongs to the smectite group with weak interlayer bonding which is responsible for the swelling and shrinkage tendency. Similarly, Figure 14 (b) reveals the minerals formed due to pozzolanic interplay between soil, limestone powder and metakaolin powder. It can be seen that the addition of limestone powder-metakaolin powder mixtures boosts the availability of alumina-silica compounds which thereby aids the interplay leading to the establishment of new cementitious compounds of calcium silicate hydrate (CSH) and calcium aluminate hydrate (C(A)SH) [33]. According to Richardson [83] and Li et al. [84], the structural CSH and CASH are model structures for the binding phase of samples, which are responsible for the high mechanical strength earlier reported from experimental observation. Interestingly, the high dominance of intensity peaks of montmorillonite in the untreated soil reduced drastically in the treated soil and this could be a pointer towards soil improvement.

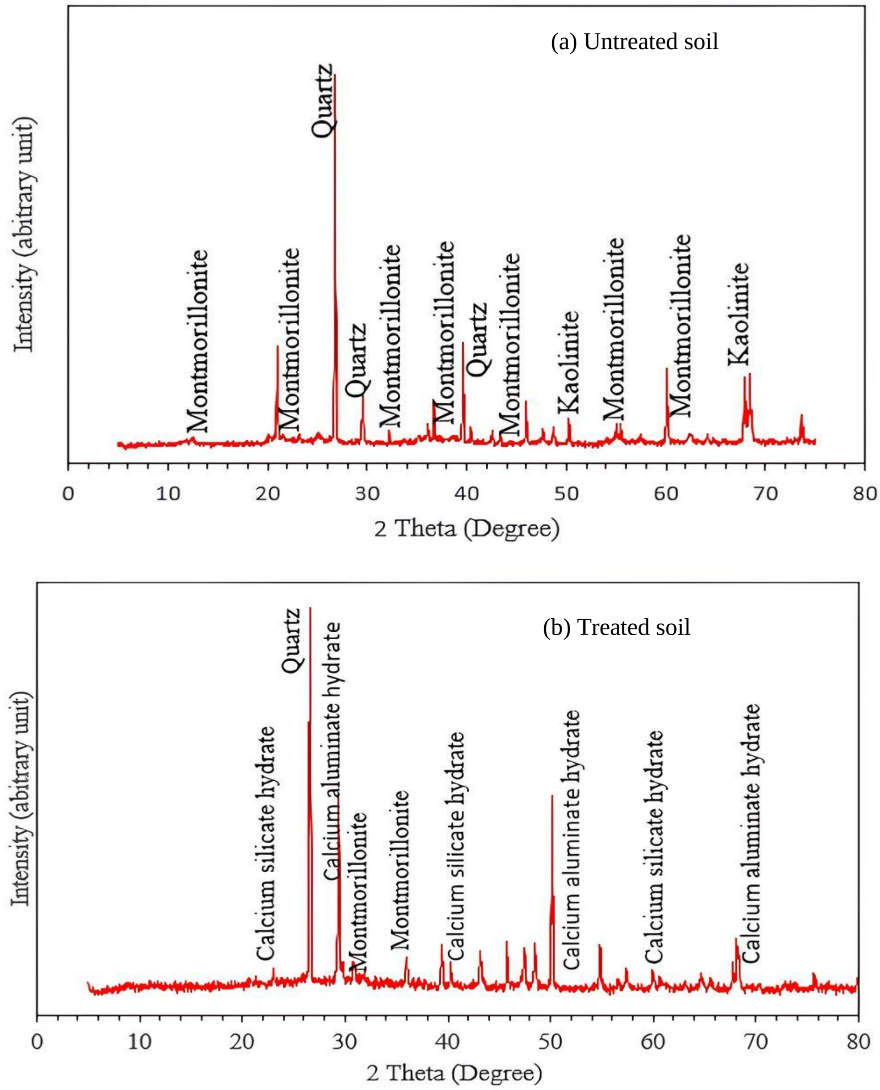

Figure 14 XRD pattern of treated black cotton soil

3.6 Analysis of Variance (ANOVA)

The application of ANOVA to ascertain the influence of additives and other parameters in soil re-engineering has been documented by [16, 17, 85]. The Statistical analysis technique known as analysis of variance (ANOVA) two-factor without replication was implemented to elucidate the level of significance of the additive materials on the tested mechanical property. The summary of the ANOVA exercise is documented in Table 3. Interestingly, the basis for accepting or rejecting the hypothesis is that in a case where Fcal was greater than the corresponding Fcrit (Fcal > Fcrit), we do not accept the null hypothesis. From Table 3, it can be inferred that the additives (MP and LP) had a significant influence on the tested property. More importantly, the p-values for both additives were found to be far less than 0.05 ($p < 0.05$). This also connotes a good statistical significance of the additive materials.

Table 3 Two-way analysis of variance

Property	Variation source	df	Fcal-value	p-value	Fcrit-value
MDD	MP	5	106.80	1.01E-13	2.71
	LP	4	50.97	3.27E-10	2.87
OMC	MP	5	55.28	5.04E-11	2.71
	LP	4	13.10	2.13E-05	2.87
CBR (S)	MP	5	237.39	4.33E-17	2.71
	LP	4	257.72	6.74E-17	2.87
CBR (US)	MP	5	86.13	7.9E-13	2.71
	LP	4	162.06	6.17E-15	2.87
UCS (7 Days)	MP	5	56.45	4.16E-11	2.71
	LP	4	189.96	1.32E-15	2.87
UCS (28 Days)	MP	5	29.16	1.56E-08	2.71
	LP	4	172.46	3.38E-15	2.87

df = Degree of freedom, Fcal = F calculate, p-value = Probability of failure, Fcrit = F critical

4. Conclusion

In this paper, the appraisal of the combined impact of additive materials (limestone powder and metakaolin powder) on the geomechanical and macrostructural behaviour of black fine-grained soil was evaluated. In the aftermath of the experimental work, the following discoveries were pinpointed:

- Characterising the unmodified BES using the AASHTO and USCS classification schemes respectively, the understudied soil is classified as an A-7-6 material with a group index of 14.
- Incorporating the blends of additive materials resulted in an increase in maximum dry density with a corresponding decline in optimum moisture content when compared to the virgin soil material.
- The mixing of the binary additives displayed a great impact on the mechanical properties of the studied soil. At mixture blending of 8% LP – 8% MP, the uttermost CBR (soaked and unsoaked) and UCS (7 and 28 days) values of 29 %, 41 %, 545 kN/m^2 and 720 kN/m^2 were achieved. Interestingly, the improvement in soil's mechanical properties was further confirmed microstructurally via the use of qualitative techniques.
- By adding the dosages of the additives it triggered some levels of alteration within the microstructural orientation and elemental configuration of the soil material. The optimally treated soil mixtures also witnessed some morphological and elemental modifications which were confirmed using scanning electron microscopy and Energy dispersive spectroscopy testing strategies. From the SEM analysis point of view, the micro fabric assessment revealed the influence of crystalline products of CSH and CAH built within the soil matrix which were believed to be responsible for strength improvement.
- Additionally, the fibre histogram outcomes for both the treated and untreated soils shows clearly the distortion in the pore length of soil materials which affirms the SEM results.

In conclusion, this current paper has shed the feasibility of utilising solid waste residues such as limestone powder and metakaolin powder in soil re-engineering and this will promote greener environmental practices and lessen the purchasing power of road construction materials.

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