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Utilization of Sem-EDX and FTIR In the Characterization of Selected Soils and Periwinkle Shell Ash

[a]A.B. Fajobi, and [b]O. A. Agbede

[a]*Department of Civil Engineering, Obafemi Awolowo University, Ile-Ife, Osun State, Nigeria*

[b]*Department of Civil Engineering, University of Ibadan, Ibadan, Oyo State, Nigeria*

Abstract

This study presents an integrated analytical framework for characterizing selected clay soils and periwinkle shell ash (PSA) using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX) and Fourier-transform infrared spectroscopy (FTIR), with a view to assessing the potential of PSA for enhancing the geotechnical properties of clay soils. Soil samples were collected from six locations within the Ile-Ife and Ede areas of Osun State, southwestern Nigeria, while periwinkle shells used for PSA production were obtained from a dumpsite in Egbeda, Lagos State. The shells were soaked in distilled water to remove adhering impurities and subsequently sun-dried before processing. SEM analysis was employed to characterize the morphology and microstructural features of the materials, while EDX was used to determine their elemental compositions. FTIR spectroscopy was utilized to identify the characteristic functional groups and chemical bonds. The results revealed that the investigated clay soils are predominantly aluminosilicate materials characterized by hydrogen bonding, with iron occurring in minor quantities. The PSA contained phosphate-bearing phases, while FTIR and EDX analyses confirmed the dominance of calcium and oxygen in the fired periwinkle shells, with residual sodium and magnesium. The occurrence of phosphates in PSA, coupled with the relatively high aluminium content of the soils, suggests potential chemical reactions leading to the formation of a hard, highly insoluble cementitious compound [Al $(OH)_2H_2PO_4 \cdot XH_3O$], which could contribute to soil stabilization. The integrated SEM-EDX-FTIR approach therefore provides an effective screening technique for evaluating mineralogical reactivity and the potential engineering application of PSA in soil modification. However, these interpretations require validation through complementary geotechnical performance and long-term durability testing.

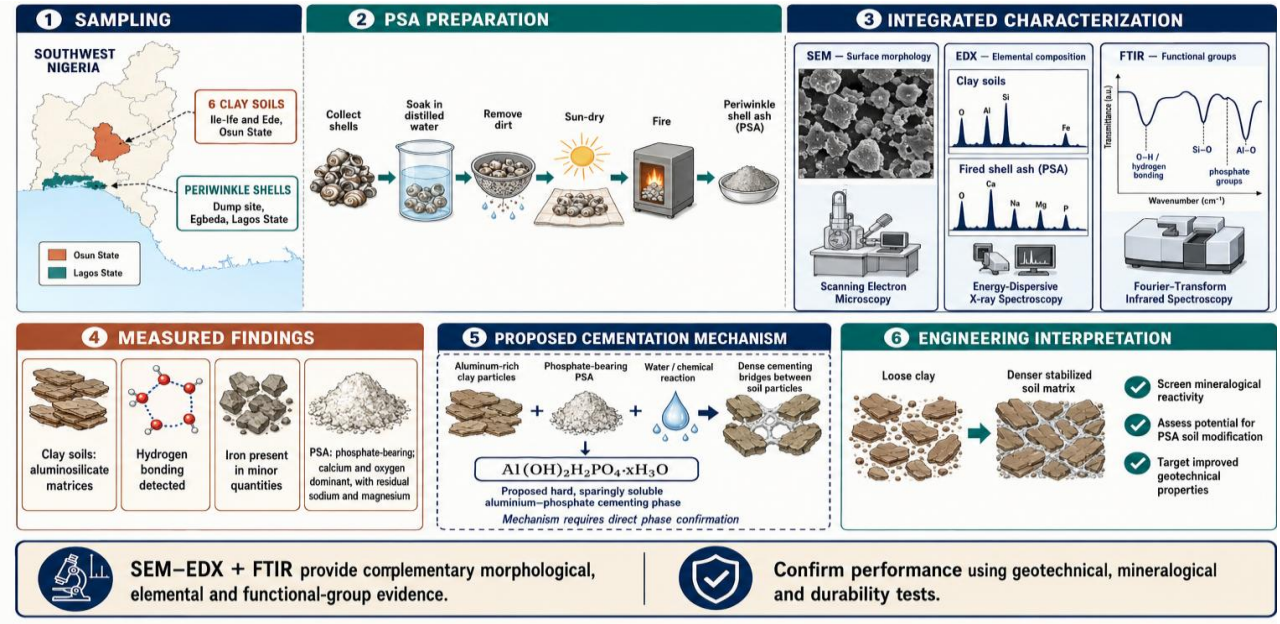
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Scanning Electron Microscopy;
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Corresponding Author: A.B. Fajobi, Department of Civil Engineering, Obafemi Awolowo University, Ile-Ife, Osun State, Nigeria.
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Graphic of Abstract



INTRODUCTION

The performance of soils in foundations, embankments and pavement subgrades depends not only on index and engineering properties and particle-size distribution, but also depend on particle morphology, elemental composition, mineral bonding and the microstructural changes produced by treatment. Traditional geotechnical tests that describe the engineering behaviour of soil at the bulk scale; microscopic and spectroscopic procedures help explain the material mechanisms underlying that soil behaviour. This distinction is important and essential for tropical and clay-rich soils, whose response can vary with soil parent material, degree of weathering, iron and aluminium oxides, clay-mineral assemblage and pore structure (Kumar et al., 2022; Oyebanjo et al., 2021). Soil performance depends on two interrelated characteristics namely: load bearing capacity and changing in volumes.

A soil with low strength needs soil treatment which is known as soil stabilization (Adeyemi and Wahab, 2008). A chemical approach of soil stabilization is preferred, and it is the concept of soil improvement or modification through stabilization with the use of additives. The system has been in practice for several thousand years (Bell, 1993). Researches have been conducted on tropical soils utilising various stabilizing agents, it has been established that Portland cement, lime and bitumen are the most utilised stabilizing agents for wide range of soils, such as granular materials, silts, clays and lateritic soils (Mesida, 1985; Gidigasu, 1991; Agbede, 1992; Ouf, 2001; Adeyemi et al., 2003; Ayininuola and Agbede, 2009; Kalantari, 2010). It has been established that cost is an important or vital factor in the equation that determines stabilising agent (Tomlinson, 1995).

As these conventional road and civil construction materials become threatened or more expensive, there is the need to turn to alternatives. The global trend is to develop soil additives from industrial, agricultural and natural by-product or waste materials. Some products such as sea shale, pulverized fuel ash, furnace clinker, pulverized egg shell, blast furnace and steel slag, incinerator waste, agricultural wastes (Osinubi et al., 2009; Barazesh et al., 2012; Malu and Bassey

(2003); Umoh, 2012; Ojuri and Ogundipe, 2012: Umar et al 2023; Manmatharajan et al2023; Almuaythir et al 2024; and Ayodele et al 2024;) have been utilised to improve and enhance geotechnical engineering properties of soil. It is known that Nigeria has a large deposit of periwinkle shells, which is under-exploited and, more importantly, the by-product constitutes environmental hazards. It has been established that these periwinkle shells contain a high percentage of calcium oxide (lime) which made it suitable for use as pozzolan materials for road stabilization (Malu and Bassey, 2003; Umoh, 2012). It is on this basis that periwinkle shell ash is being considered as a soil stabilizer in the southwestern part of Nigeria.

Some previous studies reported the use of periwinkle shell as partial replacement for aggregates in concrete at various times with various successes [(Dahunsi and Bamisaiye, 2002; Adewuyi and Adegoke, 2008; Agbede and Manasseh, 2009; Umoh, 2012)]. The information on the exploration of Periwinkle shell ash (PSA) as a potential stabilizer on the geotechnical properties of clay soil for road construction is very scanty. A recent study assessed the potential of periwinkle shell ash on lime-stabilized lateritic soil and concluded that it can act as a lime supplement in soil stabilization (Nnochiri, 2017). In addition, SEM forms high-resolution images from electron–sample interactions and is useful for observing particle shape, surface roughness, pores, aggregation, coatings and cementitious products. When SEM is coupled with EDX, characteristic X-rays generated by the electron beam provide semi-quantitative elemental information from selected points or mapped areas. The combination of these tools can therefore associate a morphological feature with its local chemistry.

In the literature, EDX reports elements rather than compounds, has limited sensitivity to light elements, and its results depend on surface preparation, interaction volume, accelerating voltage, particle geometry, and the area selected for analysis (Markewich et al., 2023).. In the case of FTIR, this technique records absorption associated with molecular vibrations and supplies a chemical overview of crystalline and poorly crystalline components. In soils and clays, O–H stretching and bending, Si–O and Al–O vibrations, carbonate bands and water-related bands can help screen for kaolinite, quartz, aluminosilicates, iron-bearing phases and carbonates. Band overlap, particle size, moisture and preparation method can complicate assignments; consequently, FTIR is most defensible when interpreted with reference spectra and complementary X-ray diffraction (XRD), chemical and thermal analyses (Nayak and Singh, 2007; Volkov et al., 2021).

Periwinkle shells are abundant molluscan residues in many coastal and riverine communities in West Africa. Poor disposal can create a nuisance and occupy landfill space, whereas controlled processing can convert the shells into a calcium-rich powder or ash for use as filler, adsorbent, catalyst precursor or construction material. Abdelmalik and Sadiq (2019) reported calcium oxide as the dominant oxide in periwinkle shell powder and observed micrometre-scale particles by SEM. In geotechnical application, Etim et al. (2021) showed that PSA used with cement altered the strength and microstructure of lateritic soil, with FTIR and SEM evidence consistent with calcite and pore-filling reaction products.

Generally, the choice of PSA as a soil stabilizer in Nigeria is supported by its low cost and environmental concern, waste-to-wealth philosophy, and increased local content. There is a reason to take the issue of PSA chemical constituents as there is no consensus; the dominating argument is that it has high

percentage of calcium and possibly calcium oxide (CaO). In literature such as Offiong and Akpan (2017), which evaluated the possibility of using PSA as a replacement for cement in concrete, PSA was chemically analyzed with the aid of x-ray fluorescent machine. The results showed that silica and calcium oxide are very high, which supports pozzolanic reactions. In the case of Malu and Bassey (2003), lime and magnesium are found in large quantities in periwinkle shell. These studies indicate promise but also show that shell species, washing, calcination temperature, retention time, cooling, grinding and storage can materially alter the final product. Hence, the aim of this research is to conduct microstructural evaluation of some selected Nigerian soil samples and periwinkle shell ash.

The specific objective of this study is to utilize SEM-EDX and FTIR to characterize selected soil samples and PSA, compare their microstructural, elemental and functional-group signatures, and assess the implications for potential engineering use. The study specifically seeks to: (i) examine particle morphology and pore features; (ii) identify major and minor elements at selected micro-areas; (iii) assign major FTIR absorption bands; (iv) evaluate agreement among the three evidence streams; and (v) define the limitations and complementary tests required for reliable material selection.

MATERIALS AND METHOD

In the preliminary study, a desktop review was conducted on the previous studies between 2000 and 2025. On the previous studies comparative were made on the findings, techniques used, and the limitations. In the field study, clay subgrade soils used in this study were collected from six different locations within the Ile-Ife and Ede areas of Osun State, Southwest Nigeria. The identities of the locations are as presented in Table 1. The clay soil samples were collected at a depth of 100 cm to 150 cm, after removal of the topsoil. The periwinkle shells were obtained from a dump site in Egbeda, Lagos State, Nigeria. The periwinkle shells were immersed in water to remove the dirt, and thereafter sun-dried. The methods used in this study include the analysis of the soil samples in their natural states, preparation of periwinkle shells into ash form, and characterization of the soil samples and PSA.

The natural moisture contents of the soil samples were determined, after which the samples were air dried in open inside the laboratory. The grain size distribution of the soil samples was determined by using wet sieve analysis. The soil samples passing sieve opening 7.5mm (with sieve analysis used for particles larger than 0.075mm, while hydrometer analysis was used for particles passing through 0.075mm). Some of the sun-dried periwinkle shells were milled into powder form with the particle size passing sieve 0.425mm. Another set was heated in an electric oven at 1000° C, allowed to cool, and then ground into ash form. Zeiss EVO MA15 Scanning Electron Microscope was used to assess the samples' morphology. The SEM was coupled with EDX Oxford Instruments-Aztec version 1.0 to provide information on the samples' chemical composition in terms of the elemental constituents. The functional groups were determined with the aid of a Perkin-Elmer 8000 FTIR spectrophotometer. The powdered samples (clays and PSA) infrared spectra were acquired using air as the reference, at a resolution of 4 cm^{-1}. The spectra were collected and analyzed within the region 4000 - 650 cm^{-1}. The experiment was carried out at room temperature (25 °C). Figure 1 presents the flowchart of the materials and methods.

Table 1: Soil Samples Locations and GPS coordinates

Sample	Location	Latitude	Longitude	Accuracy	Elevation
OS1	Mokuro	N07°30'07.3"	E04° 35' 42.7"	3m	335m
OS2	Ipetumodu	N07°31'51.7"	E04° 26' 10.5"	4m	239m
OS3	Ede-Ido Osun	N07°46'28.4"	E04° 29' 15.5"	4m	322m
OS4	Adesanmi	N07°29'49.9"	E04° 30' 57.6"	4m	252m
OS5	OAUTH Link Rd	N07°30'44.7"	E04° 32' 59.8"	4m	254m
OS6	New Market OAU	N07°31'01.8"	E0 4° 30'51.1"	3m	278m

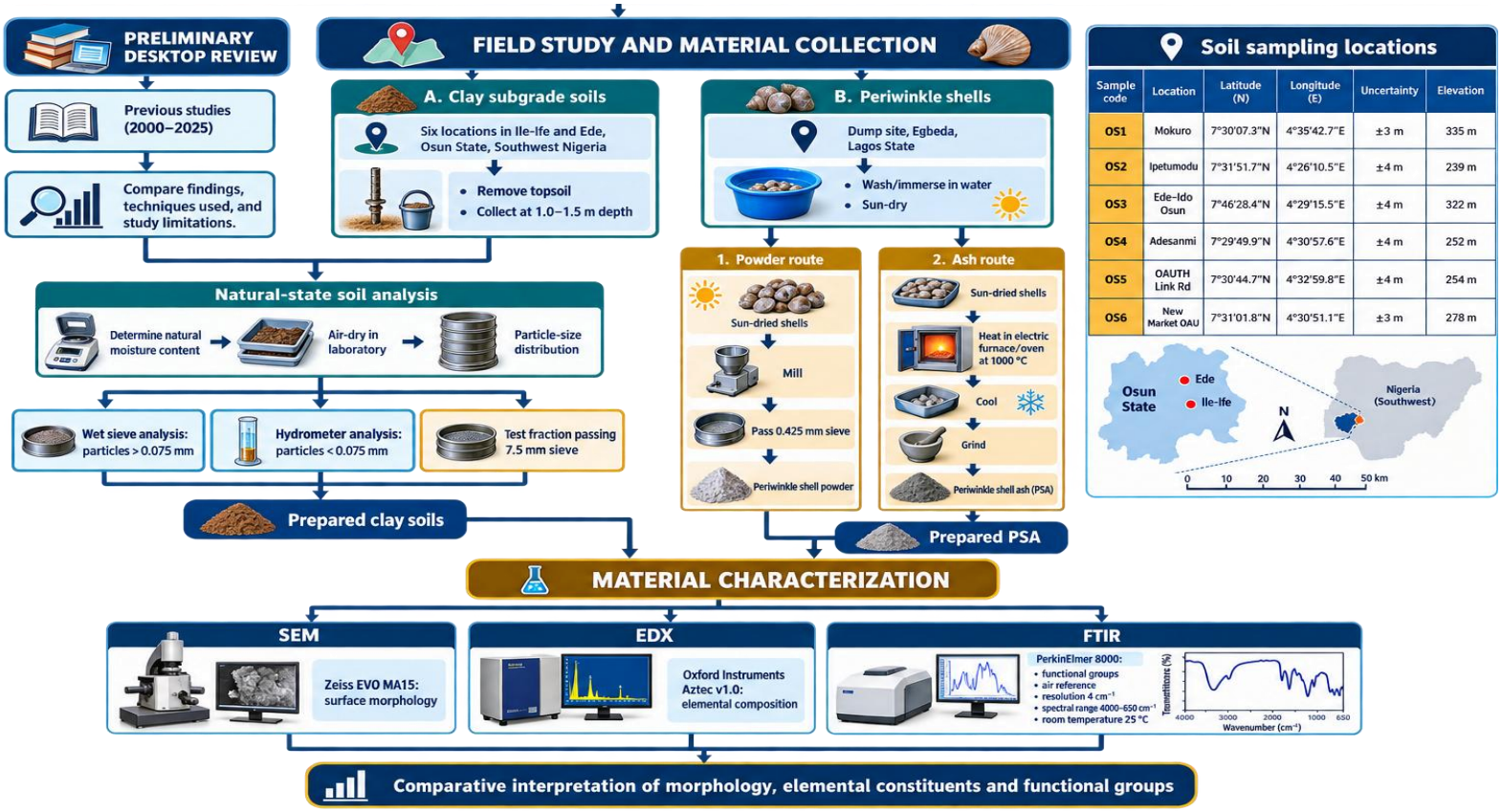


Figure 1: the flowchart of the materials and methods

RESULTS AND DISCUSSION

Results from the study are presented and discussed as follows:

a. **Desktop Review on the Previous Studies Between 2000 and 2025:** The results of the desktop review conducted on the previous studies are presented in Table 2. The table provides an overall trend from 2000 to 2025. The \Table revealed that there is a gradual progression (Figure 2) as follows:

- **2000–2010**:SEM and FTIR were principally employed as independent characterization techniques for soils, clays and mineral materials.
- **2011–2015:** The techniques increasingly became integrated with geotechnical stabilization research. The 2015 Fajobi work is particularly important because it combined FTIR + SEM + EDX for both soil and PSA.
- **2016–2020:** Characterization increasingly focused on understanding the relationship between mineral composition, surface morphology and engineering behavior. Studies of Nigerian clay minerals demonstrated the effectiveness of combining SEM, EDX and FTIR with XRD/XPS.
- **2021–2022:** There was stronger emphasis on mechanistic interpretation. SEM and FTIR were used to explain how PSA, lime and cement interact with lateritic soil, including formation of new microstructures and cementitious compounds.
- **2023–2025:** The application expanded toward sustainable construction materials, marine-shell waste utilization, adsorption materials and advanced composites. PSA was increasingly investigated using SEM-EDX and FTIR alongside XRD, BET, TEM and AFM.

It can be established from the Table that despite the progress between 2000 and 2025, there are several gaps that remain. The gaps are

- limited combined SEM–EDX/FTIR studies: Many PSA soil-stabilization investigations report engineering properties but do not provide comprehensive SEM–EDX and FTIR characterization,
- limited comparison of different soil types: More comparative studies are required involving lateritic, clayey, sandy-clay, silty and expansive soils treated with the same PSA.
- insufficient elemental mapping: EDX is often reported as point analysis rather than elemental mapping. Mapping could show the spatial distribution of Ca, Si, Al, Fe and other elements.
- limited quantitative FTIR analysis: Many studies identify peaks qualitatively without quantifying changes in peak intensity, position or area after PSA addition.
- limited correlation between microstructure and engineering properties: There is a need to statistically correlate SEM–EDX/FTIR results with CBR, UCS, MDD, OMC, plasticity index and durability.
- variation in PSA production: Shell washing, drying temperature, calcination temperature, duration and particle size can strongly affect PSA chemistry. The Fajobi (2015) for example, used calcination at 1000°C, demonstrating the importance of clearly reporting preparation conditions, and
- need for standardized characterization: Different researchers use different

FTIR ranges, calcination temperatures, magnifications and EDX operating conditions, making direct comparison difficult.

b. **SEM Results:** The SEM images were evaluated on the basis of their particle morphology vis-à-vis their particle sizes and particle shapes. Particle shapes have to do with form, roughness, and roundness (Barrett, 1980), and the particle roughness is determined by the scale employed (Santamarina *et al.*, 2001). Figure 3a shows the unfired periwinkle particles having varied size and shape. The large particle sizes were in the region of 250 μm and those of small particles are between 10 – 40 μm. In respect of the shapes, the large particles have flat and lath-like shapes; whereas the smaller ones are cubical. The particle surface roughness is found to decrease with a decrease in particle size.

On the other hand, the fired periwinkle samples image (Figure 3b) was characterized with large particles having an average size of 140 – 180 μm and the small particles are less than 5 μm. The small particles are mainly spherical in shape, while the large particles are elongated. Generally, all the particles have distinct sharp edges and corners. It must be noted that the particle shape is the external morphology of a particle and that the particle size is related to the packing factor and the void ratio. It is ascertained that the particle geometry has a significant effect on the magnitude of the forces of attraction, and this is a function of the separation distance of the atoms in each particle (Barrett, 1980; Samtamarina *et al.*, 2001). Figure 4a is the OS1 sample clay morphology, which shows bulky, rough, and irregular particles that are near spherical in shape. The particle sizes are varied, with the larger ones having a size of about 100 μm, and the smaller particles are almost 8 μm. Meanwhile, the OS2 SEM (Figure 4b) image has uniformly distributed and closely packed particles. The particles are in the form of platelets, smooth, and grain–like with varied particle sizes ranging from 2 – 40 μm. Figure 4c (OS3) revealed coarse, porous, and rough–edged large particles, which have small seed – like particles on the surface. The clay obtained from Adesanmi Road (Figure 4d) had particle shapes that are elongated, pencil-shaped, and spherical in geometry.

Table 2: Comparative literature review, 2000–2025

Year	Study/material investigated	SEM/SEM–EDX utilization	FTIR utilization	Major findings	Relevance to PSA–soil characterization
2000–2010	Soil/clay and mineral characterization studies	SEM increasingly used to observe particle morphology, surface texture, pores and aggregation; EDX used to determine major elemental constituents	FTIR used to identify Si–O, Al–O, O–H and other mineral-associated bonds	Established the usefulness of combining morphology, elemental chemistry and vibrational spectroscopy for clay/mineral identification	Provides methodological foundation for later PSA–soil studies
2015	**Clay subgrade soils + periwinkle shell ash**, Fajobi	SEM used to examine morphology; EDX identified elemental composition of soils and PSA	FTIR identified characteristic functional groups/mineral bonds	Soils were classified mainly as A-2-7 and A-7-5; clay samples were aluminosilicate-rich; PSA was dominated by Ca and O with traces of Na and Mg	**Directly relevant.** Demonstrates simultaneous use of FTIR, SEM and EDX for soil and PSA characterization.
2017	Lime-stabilized lateritic soil + PSA, Nnochiri	Microstructural/engineering assessment associated PSA with improved soil behavior	Used as part of the broader characterization of stabilization reactions in PSA-treated soil	PSA acted as a complementary stabilizer to lime and improved CBR and UCS characteristics	Demonstrates that chemical characterization is important for explaining engineering improvement.
2020	Nigerian clay minerals, Ekpe et al.	SEM determined morphology; EDX, XPS and XRD quantified chemical/mineral composition	FTIR investigated functional groups and chemical behavior of adsorbed species	Seven Nigerian clays were characterized; samples were predominantly kaolinite/illite, with gibbsite and quartz also identified	Important methodological comparison showing the value of combining SEM–EDX and FTIR for Nigerian soils/clays.
2021	Lateritic soil + Portland limestone cement + PSA	SEM used to identify morphological changes and microstructural development	FTIR identified functional groups/mineralogical	Optimum PLC–PSA treatment produced improved strength; SEM showed new	**Highly relevant.** Demonstrates that SEM and FTIR can explain the mechanism behind

Year	Study/material investigated	SEM/SEM–EDX utilization	FTIR utilization	Major findings	Relevance to PSA–soil characterization
			changes in natural and treated soils	microstructural arrangements and FTIR showed characteristic functional groups	engineering improvement.
2021/2022	Lateritic soil + lime + PSA, Attah et al.	SEM examined morphological changes after stabilization	FTIR identified characteristic functional groups in natural and stabilized soil	At 8% lime + 8% PSA, UCS reached about 2670.45 kN/m² at 28 days and CBR increased substantially; SEM indicated new microstructural arrangements	**Highly relevant** for explaining formation of cementitious products and soil densification.
2022	Lateritic soil stabilized with oyster shell ash + PSA	Microstructural characterization used to support strength improvement	Chemical/mineral characteristics associated with shell ash stabilization	PSA and oyster shell ash were investigated as environmentally friendly alternatives to conventional binders	Demonstrates comparative use of marine-shell wastes in soil stabilization.
2023	Marine debris and soil stabilization review	SEM reported as a useful technique for identifying morphological changes in stabilized soils	FTIR/other mineralogical methods used to understand reaction products	Review confirmed that marine-derived wastes, including shell materials, can generate new compounds responsible for improved soil strength	Provides broader evidence supporting SEM/FTIR characterization of marine-shell-based stabilizers.
2024	PSA + quarry dust	SEM images used to investigate morphology of PSA and quarry dust	FTIR spectra of PSA examined between 4000 and 400 cm^{-1}	PSA exhibited characteristic bands around **1394, 872 and 711 cm^{-1}**, associated with carbonate-related vibrations, while additional bands were reported	Demonstrates the ability of FTIR to chemically fingerprint PSA before its use as a construction material.

Year	Study/material investigated	SEM/SEM–EDX utilization	FTIR utilization	Major findings	Relevance to PSA–soil characterization
				around 656, 611 and 533 cm^{-1}	
2024	Lateritic soil + PSA, Kano, Nigeria	Characterization complemented engineering tests of PSA-treated lateritic soil	Used within the broader assessment of PSA effects	Lateritic soil was treated with 0–10% PSA at 2% increments to investigate changes in engineering properties	Shows continued application of PSA as a local stabilizer up to 2024.
2025	Zeolite/periwinkle-shell ash nanocomposite	SEM–EDX used with TEM, AFM and BET to characterize morphology and elemental/surface characteristics	FTIR used to identify chemical bonds and surface functional groups	Zeolite/PSA nanocomposite was developed for Pb^{2+} adsorption; characterization demonstrated the complementary nature of SEM–EDX and FTIR	Although not a soil-stabilization study, it demonstrates the advanced 2025 application of SEM–EDX/FTIR to PSA-derived materials.
2025	Periwinkle-shell-containing AA6061 composite	SEM–EDS examined morphology and elemental distribution	FTIR characterized chemical interactions	Advanced SEM–EDS and FTIR were used to evaluate a composite containing periwinkle shell material	Demonstrates expansion of PSA/periwinkle characterization beyond geotechnical applications.

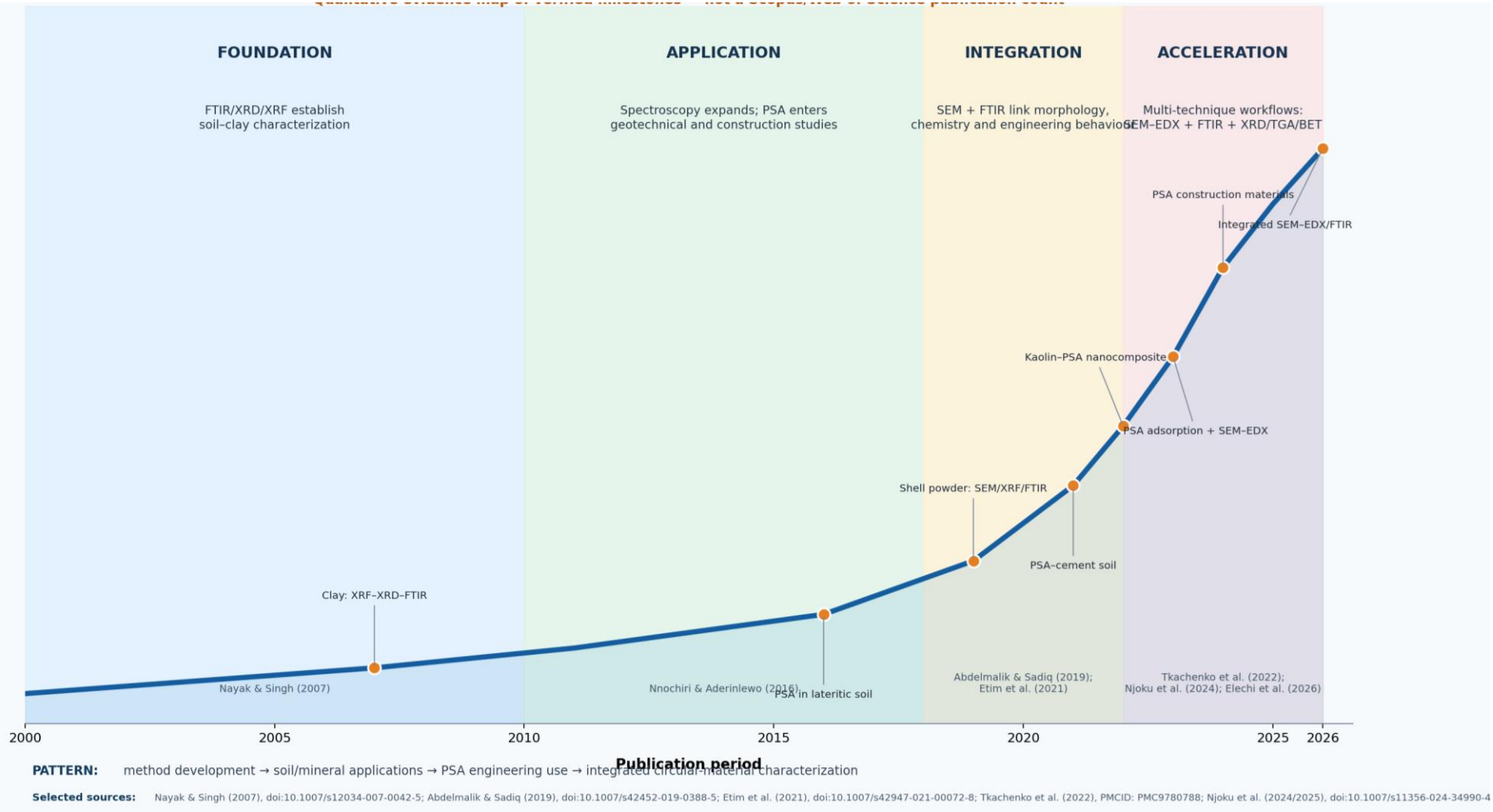


Figure 2a: Trend and Pattern of global publications on SEM-EDM and FTIR characterization of soils periwinkle shell ash

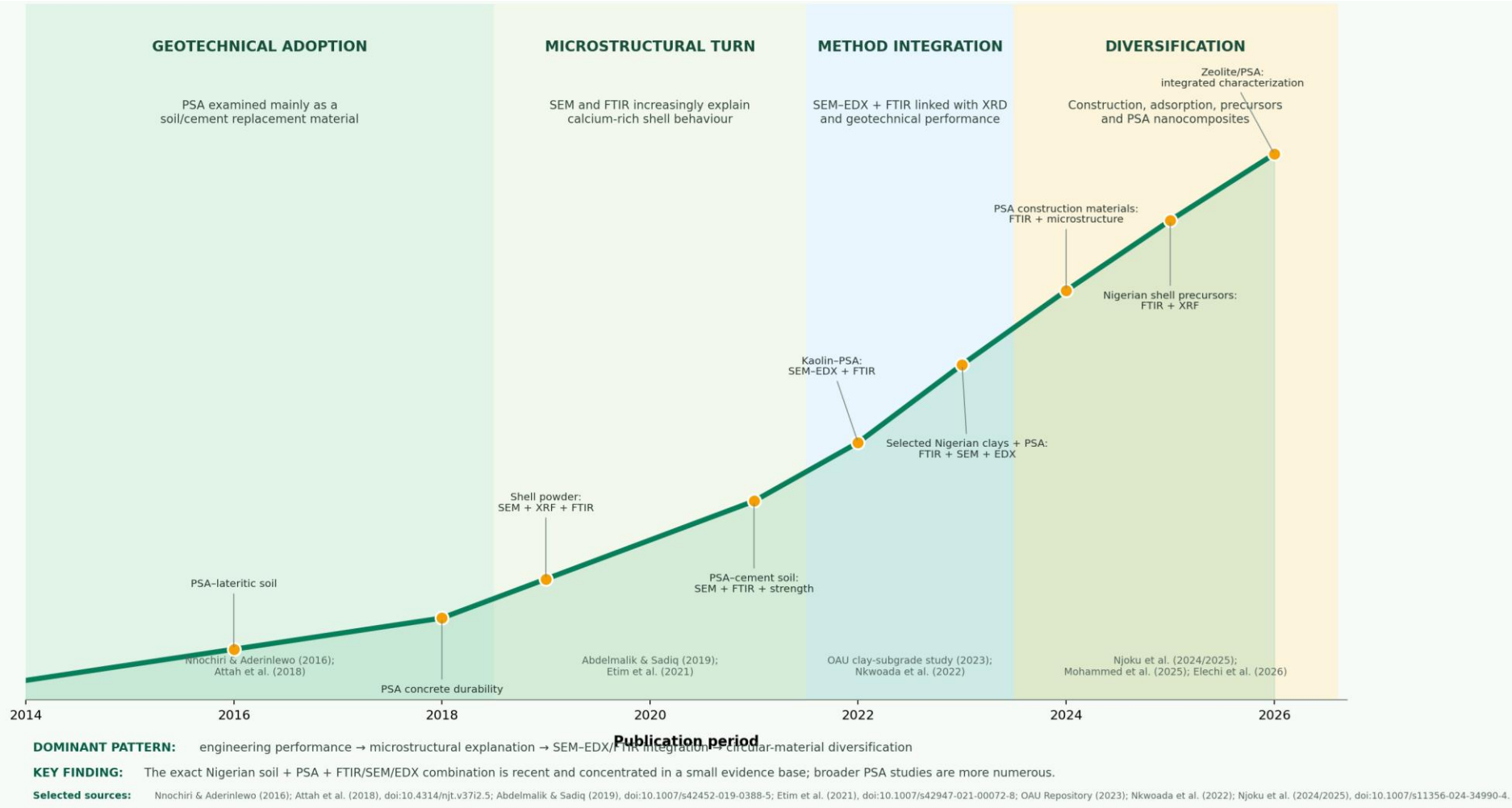
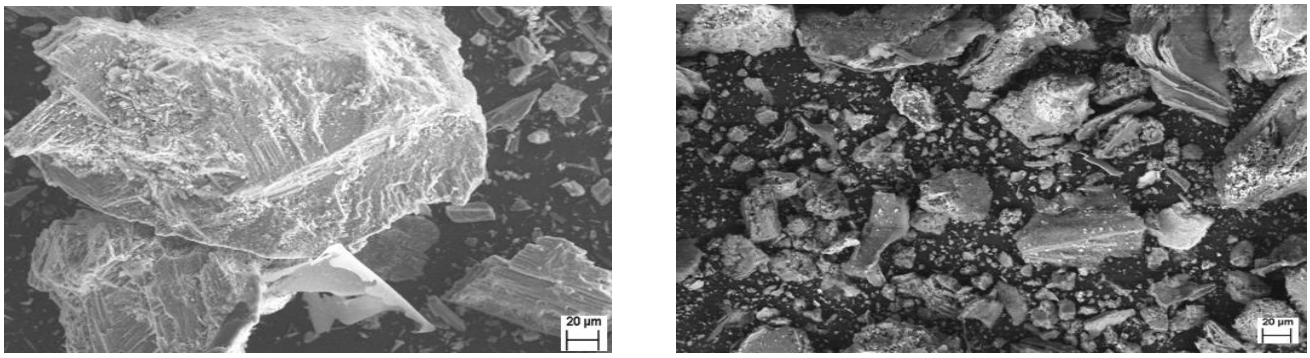
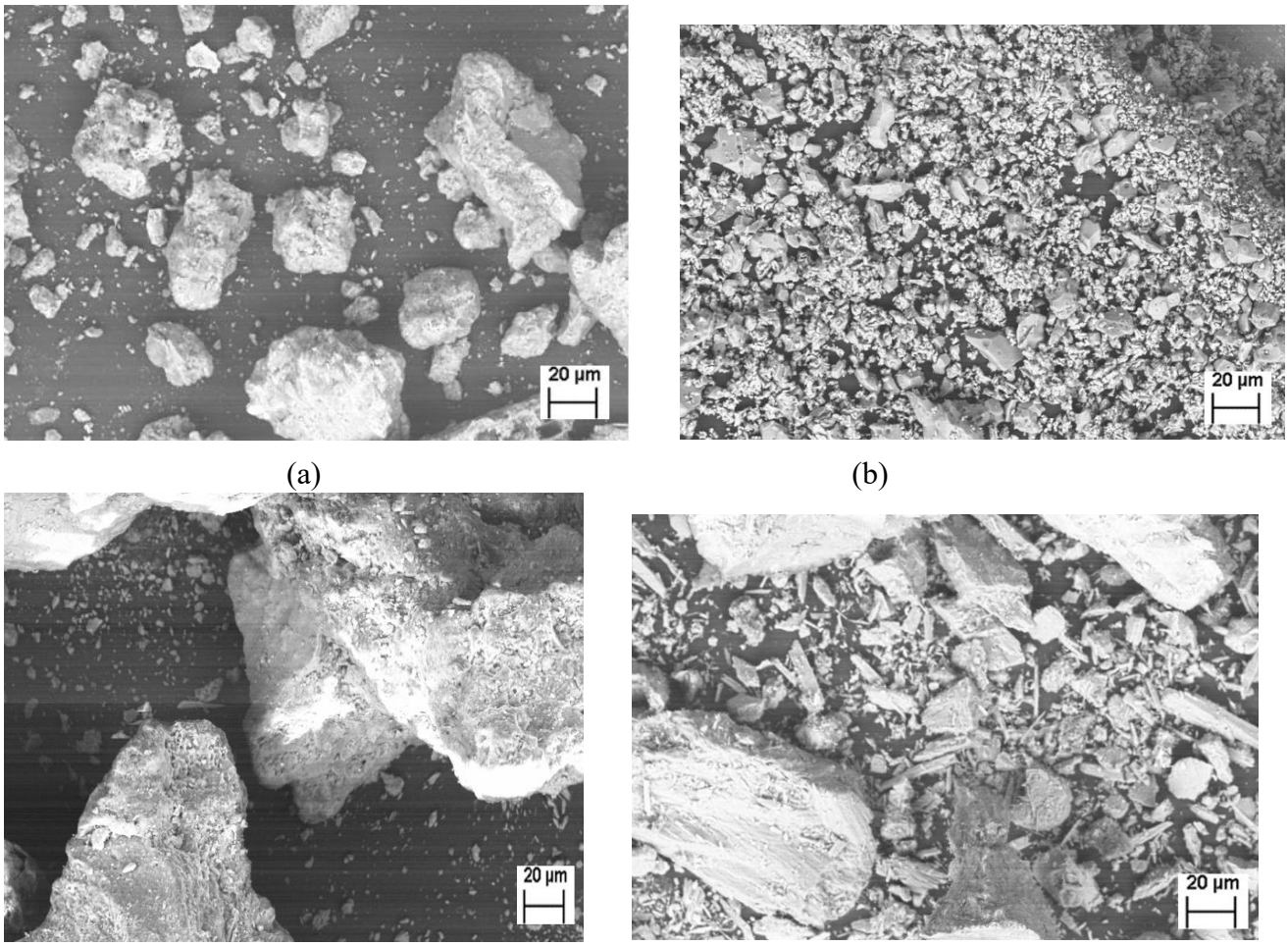


Figure 2b: Trend and Pattern of global techniques on SEM-EDM and FTIR characterization of soils periwinkle shell ash



(a) (b)

Figure 3: SEM micrograph of Periwinkle samples (a) Unfired (b) Fired at 1000 °C.



(a) (b)

(c) (d)

Figure 4: SEM micrographs of the different clay samples (a) Mokuro Road (b) Ipetu Road (c) Ede Road (d) Adesanmi Road

Also, it had large particle sizes with rough surfaces and varied angularity. According to Dodds (2003) the surface roughness increased with the coefficient of friction and particle adhesion. The particle angularity has been argued to change the void ratio (Miura *et al.*, 1999) and the way particles move in relation to each other. Figure 4e (OS5) had scattered and non-uniform particle sizes and shapes but with smooth surfaces. It is known that particle size and shape play a critical role in the behaviour of materials due to the interaction of individual particles (Dodds 2003).

The particle sizes ranged between 4 – 60 μm, and the particle shapes are highly angular, while some are elliptical. Large sizes with virtually no smaller ones are observed for New Market (Figure 4f). The particle shape is bulky, relatively angular, and rough - edged. The particle size showed that there will be minimum forces of attraction between the particles. It must be realised that as the angularity increased, the energy required for dilation increased and therefore the internal friction increased due to particle interlocking. Also, increasing the particle angularity leads to greater shearing resistance (Dodd, 2003).

c. **EDX:** The qualitative results of the samples evaluated by energy dispersive X-ray (EDX) are presented. The quantitative results for the periwinkle samples are listed in Table 3. The calcium contents for the unfired and fired periwinkles are 40.11 and 51.19%, respectively. The oxygen compositions were 59.10 and 48.30% respectively for unfired and fired periwinkle. Simply, it indicates that oxygen concentrations decreased after firing the periwinkle, whereas the calcium increased correspondingly. There is also the presence of magnesium in the fired periwinkle. The results are in agreement with expectations based on the fact that the periwinkle shells are supposed to act as calcium sources. This will enhance its substitution as lime in soil stabilization. The presence of calcium increased the long-term strength by minimizing plasticity, increasing shear strength, reducing their compressibility, and improving hydration characteristics (Cerato et al., 2009). It is believed that the hydration and pozzolanic reactions (the reaction between CaO, SiO_2, and Al_2O_3) will greatly influence the behaviour of calcium in soil stabilization. The suspected chemical reactions are as follows:

$$Ca + O \rightarrow CaO \quad (1)$$

$$CaO + H_2O \rightarrow Ca(OH)_2 \quad (2)$$

$$Ca(OH)_2 + H_4SiO_4 \rightarrow Ca^{2+} + H_4SiO_4^{2-} + 2H_20 \rightarrow CaH_2SiO_4.2H_2O \quad (3)$$

While the cement chemistry is represented as:

$$CH + SH \rightarrow C - S - H \quad (4)$$

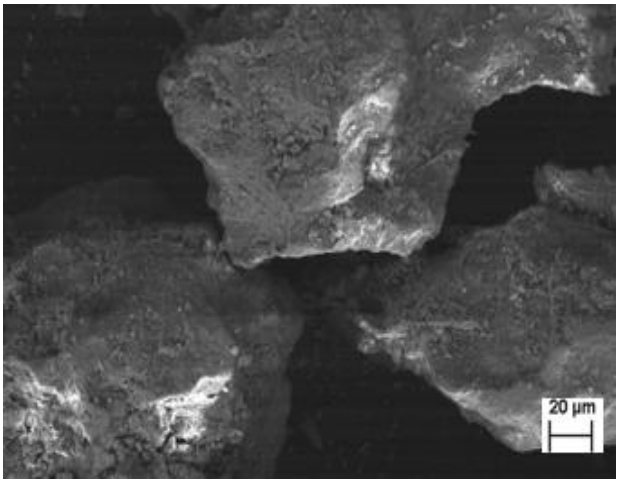
Where: C = CaO; S = SiO_2; H = H_2O.

The table also shows that the various clay samples are aluminosilicates with different aluminium, silicon, and oxygen concentrations. For example, the aluminium content for the OS2 sample was the lowest (8.5%), while OS4 had the highest aluminium values (20.59%). Whereas the silicon composition of Ipetu road (OS2) is 32.77% which is the maximum values obtained, and the least is recorded for OS1 with 13.27%. The duo of OS1and OS5 samples had other elements in small quantities and some of these are phosphorus and magnesium. The phosphorus and magnesium for the Mokuro sample are 0.56 and 0.14%, respectively, while those of Link Road were 0.11 and 0.16%, respectively. All the clay samples have various amounts of iron with the exception of the New Market Road sample (OS6). It is generally believed that iron increases the sorption capacity and therefore influences soil stabilization by increasing the soil hydration characteristics

d. **FTIR:** Figure 5 is the Fourier Transform Infrared (FTIR) spectra for the periwinkle samples. The following functional groups can be suggested for the periwinkles as obtained from Table 4; Al-OH, organic phosphates, PO_4^{2-}; CO_3^{2-}; (Si-O-Si); SiO_3^{2-} . It was noticed that the fired samples have weaker bands, and this can be attributed to removal of oxygen during the heating process. Another unique feature occurred at 1000 cm^{-1} and 1400 cm^{-1} regions, whereby there was a conspicuous difference between the spectral band intensity for unfired and fired samples. An explanation can be offered that the carbonate and organic phosphate wave bands are eliminated, although it is assumed that the phosphate and carbonate ions occur in minute quantities in the periwinkle shells. It must be stated that compared to other studies, this study is the first to identify the presence of phosphates in periwinkle shell ash (Offiong and Akpan, 2017; Malu and Bassey, 2003; Umoh, 2012). The presence of phosphates is expected to enhance soil stabilization and make the soil more suitable for engineering applications (Hemwall and Scott, 2018). Literature has reported the presence of silica in substantial amounts (Offiong and Akpan, 2017), but this study can only conclude that it is in small quantities. There is a plan to further examine the samples with the aid of the X-ray diffraction technique to determine the phases. Figure 6 displays all the clays' FTIR spectra, with their wavelength and suggested assignments listed in Table 5.



(e)



(f)

Figure 4: SEM micrographs of the different clay samples (e) Link Road (f) New Market Road

Table 3: The samples' elemental constituents as obtained from EDX

Elements	Grinded Periwinkle	Fired Periwinkle	OS1	OS2	OS3	OS4	OS5	OS6
O	59.10	48.30	41.33	55.90	48.99	35.74	47.88	53.33
Al			15.94	8.50	12.88	20.59	15.16	11.91
Si			13.27	32.77	23.28	20.47	17.30	29.79
K			0.11	0.34	0.27	0.28	0.11	0.82
Ti			0.79	0.25	1.43	2.93	0.52	4.15
Fe			27.58	2.24	13.15	19.99	18.64	
Cu							0.12	
P			0.56				0.11	
Na	0.53	0.34						
Ca	40.11	51.19	0.19					
Mg		0.26	0.14				0.16	
Mn			0.09					

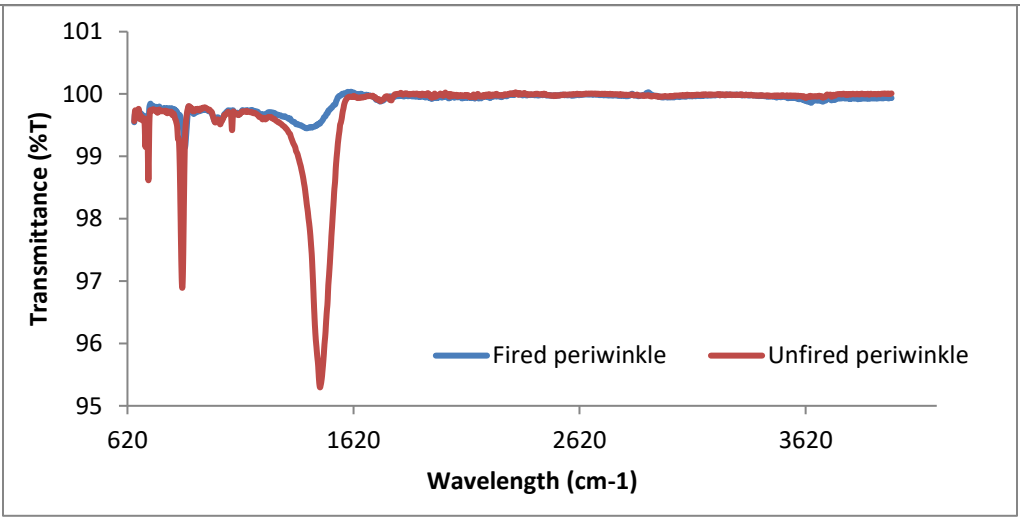


Figure 5: The FITR spectra for periwinkle shell

Table 4: The FTIR band positions for the unfired and fired periwinkle and suggested interpretation

Wavelength (cm-1)	Unfired Periwinkle	Fired Periwinkle	Suggested assignment
700	713 (w)	713 (w)	Al – OH
800	864 (s)	864 (m)	CO_3^{2-};
1000	1085 (w); 1071 (w, dp)	1085 (w)	PO_4^{2-}; CO_3^{2-}; (Si-O-Si); SiO_3^{2-}
1200	1273 (w)	1273 (w)	Organic phosphates (P=O stretch)
1400	1464 (s)	1464 (w, broad)	Organic phosphates (P=O stretch)

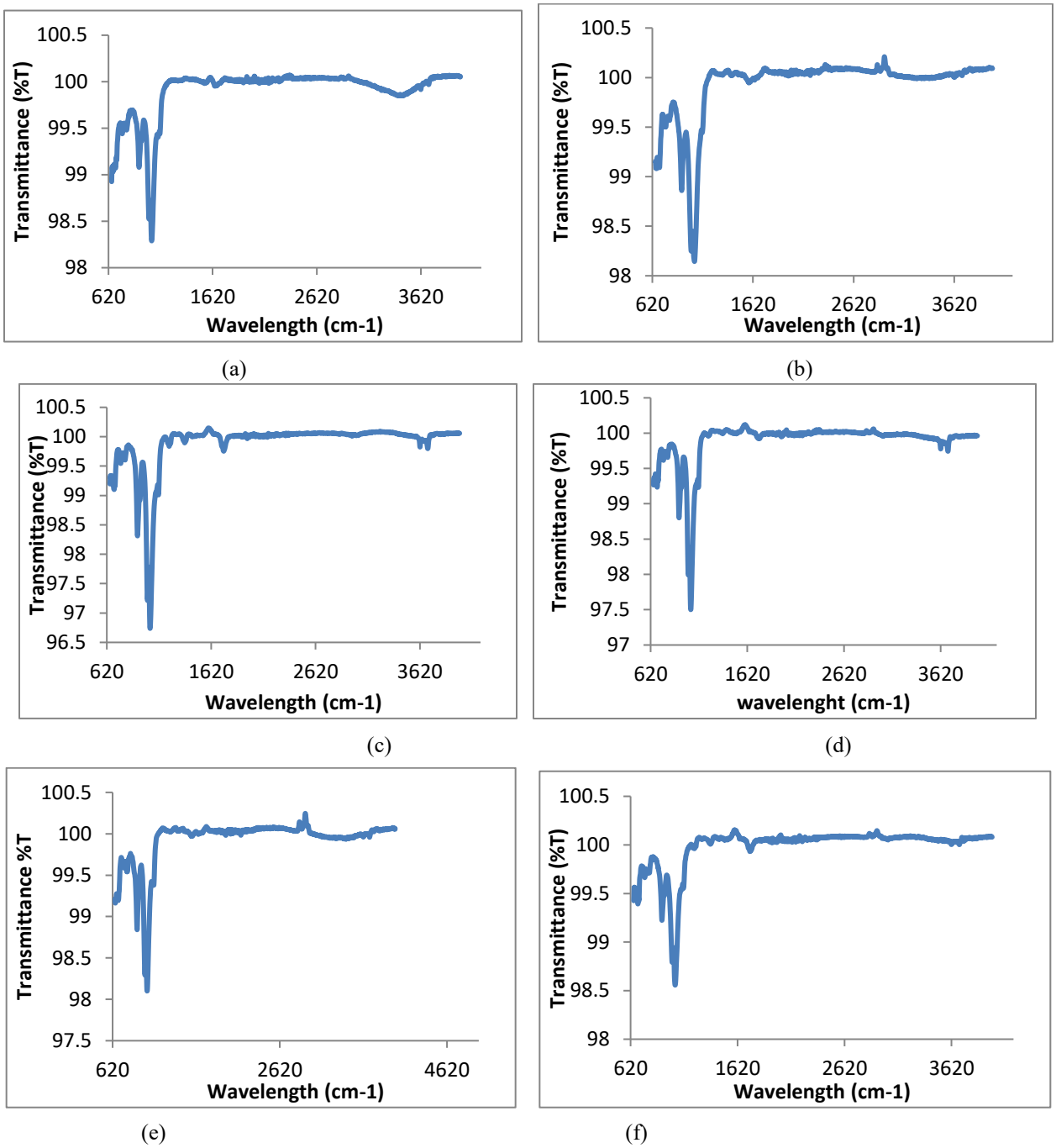


Figure 6: The FITR spectra for the various clay (a) Mokuro Road (b) Ipetu Road (c) Ede Road (d) Adesanmi Road (e) Link Road (f) New Market Road

Table 5: The FTIR bands position for various clay samples and suggested interpretation

Wavelength (cm-1)	Adesanmi	Ede	Ipetu	Link	Mokuro	New Market	Suggested assignment
600			694 (w,dp)		651 (m)		Al-OH or Mg-OH translation; CO_3^{2-}; Si – O – Si bending
700	701 (w)	701; 799 (w)	792 (w)	700 (w)		700 (w)	Al-OH vibrations of surface hydroxyl (Farmer, 1974; Russell,, 1970); Si – O quartz
800		807 (w)	863 (w)	813 (w,dp)	802 (w,dp)	803 (w,dp)	CO_3^{2-}; OH deformation linked to Al^{2+}
900	919 (m)	917 (m)	915 (m)	918 (m)	917 (m)	918 (m)	-OH; doublet Al-OH deformation; OH deformation linked to Mg^{2+}
1000	1041 (s,dp)	1039 (s,dp)	1046 (s,dp)	1038 (s,dp)	1040 (s,dp)	1043 (s,dp)	Organic siloxane or silicone (Si-O-Si); SiO_3^{2-}
1100				1119 (w)	1118 (w)	1120 (w)	Si-O vibrations
1200	1267 (w)	1238 (w)				1236 (w)	No assignment
1300			1339 - 1353 (w)	1376 (w, broad)			No assignment
1400	1430 (w)	1411 (w)				1411 (w)	CO_3^{2-};
1500	1565 (w, broad)						No assignment
1600			1613 (w)	1623 (w)	1604 (w)		H-O-H bending of H_2O
1700		1765 (w)			1700 (w)	1764 (w)	No assignment
1800	1806 (w)						No assignment
3400					3451 (w, broad)		-OH stretching; weak OH
3600	3663 (w)	3691 (w)	3682 (w)	3695 (w)	3663 (w)	3682 (w)	-OH stretching; M-OH
3700	3702 (w)	3704 (w)	3749 (w)	3766 (w)	3759 (w)	3749 (w)	-OH stretching; M-OH;

It was only Mokuro sample (OS1) that was not characterized with the Al-OH functional group with vibrations of surface hydroxyl at a bandwidth of 700 cm^{-1} among the studied clays. The clays are suspected to have Si – O quartz band. Also all the clay samples, with the exception of the Adesanmi sample (OS4) have 800 cm^{-1} wavelength spectra which are assigned to carbonate and hydroxyl ions deformation linked to Al^{3+}. There are -OH; doublet Al-OH deformation; OH deformation linked to Mg^{2+}, and organic siloxane or silicone (Si-O-Si); and SiO_3^{2-} at the wavelength of 900 – 1000 cm^{-1} for all the clay samples. Another carbonate ion occurred at 1400 cm^{-1} for OS3, OS4 and OS6 samples, while H-O-H bending of H_2O molecular bonds is suspected for Mokuro (OS1), Ipetu (OS2) and Link road (OS5) 1600 cm^{-1}. Obviously, the results indicated that all the clay samples are aluminosilicate with hydrogen bonding. In

summary, Akinwumi (2012), Ismail and Belal (2014), and Rakaraddi and Gomarsi (2015), all established that the quantity of moisture absorbed by soil before it reaches its plastic limit depends on the quantity of fines (particles passing the 0.075 mm sieve) it contains.

It therefore follows that the higher the quantity of particles coarser than 0.075 mm, the lower the quantity of moisture required by the soil to reach its plastic limit. Ika Putra (2014) opined that a stable subgrade can be achieved by increasing the soil particle size through a reduction in the plasticity index, reducing the shrink / swell potential and cementation. The type of clay minerals and their proportional amounts in a soil will affect the liquid and plastic limits (Das, 2011). Skempton (1953) observed that the plasticity index of a soil increases linearly with the percentage of clay-size fraction present. Bojana and Stanislav (2013) suggested that the interactions between the clay minerals and water affect soil's water-holding capacity. Soil composition influences the water content at the liquid and plastic limits; the quantity of water at the Atterberg limits depends mostly on the size and the portion of clay minerals in non-swelling soils, while in swelling soils it also depends on the quantity of interlayer water, which is mostly dependent on the type of clay minerals, exchangeable cations and the chemical composition of the pore water.

From all the foregoing, it can be reasonably inferred that the number of fine particles contained in a particular soil (and the mineralogy of these particles); dictates its moisture content and change from plastic to liquid to semi solid etc. Hence, the Atterberg limits of soil depends on the coarseness or fineness of the soil particles i.e. its texture. It has been argued that grain size affects the soil packing density and the friction internal angle (Kim, 2003). Due to the frictional resistance around the particles it leads to higher resistance to particle rearrangement. The presence of pozzolanic material such as SiO_2 and Al_2O_3 result in the formation of strong bonding in the geomaterial (Geetha and Ramamurthy, 2011). An instable tendency which may be reflected in slight increase or decrease in performance may be due to size effect. The reasons may be varied but a plausible one is that the physical properties like morphology, particle size, surface properties, and amorphous phase content may affects the geomaterial reactions(Jaturapitakkul and Cheerarot, 2003). With the minute presence of phosphates and possible chemical reactions envisaged, there is formation of a hard and highly insoluble cementing compound $[Al(OH)_2H_2PO_4.XH_3O]$ which is due to occurrence of aluminum in high content in the selected soils. This product will result in effective soil solidification and enhanced soil treatment.

e. **Integrated interpretation and engineering significance:** The principal value of the combined approach lies in triangulation. For example, a plate-like particle that yields Si–Al–O by EDX and occurs in a sample with compatible hydroxyl and silicate FTIR bands supports, but does not prove, a clay-mineral interpretation. A porous PSA particle dominated locally by Ca and O, together with carbonate or hydroxyl FTIR bands, supports a calcium-bearing shell-derived material. XRD must identify the crystalline phases, while XRF or ICP methods must determine bulk chemical composition. If the selected soils are to be stabilized with PSA, calcium availability, fineness, surface area and phase assemblage will govern possible cation exchange, flocculation and cementitious reactions. PSA that is predominantly unreacted carbonate may behave mainly as filler or nucleation material; ash containing available lime may increase pH and promote reactions with reactive silica and alumina. These mechanisms cannot be inferred from SEM-EDX and FTIR

alone. These tools should be tested through pH, electrical conductivity, lime-consumption or strength tests, compaction, unconfined compressive strength, California bearing ratio, swelling, durability and curing-age microstructural analysis. Etim et al. (2021) found that PSA combined with cement improved lateritic-soil strength and produced pore-filling microstructural changes, illustrating why chemical evidence should be connected directly to measured engineering performance. Table 6 presents the selected comparative studies relevant to SEM-EDX and FTIR characterization. The table summarizes the complementary roles of mineralogical, chemical, spectroscopic, and microscopic techniques in characterizing soils and periwinkle-shell-derived materials. Nayak and Singh (2007) demonstrated that FTIR interpretations should be supported by XRD or XRF because FTIR identifies functional groups and characteristic bonds but may not conclusively establish crystalline mineral phases. Abdelmalik and Sadiq (2019) reported fine periwinkle shell powder particles and a calcium-rich, CaO-dominant composition, indicating its potential as a cementitious or soil-stabilizing material. However, the composition of unburnt shell powder should not be assumed to represent every periwinkle shell ash because combustion conditions can significantly alter particle structure, mineral phases, and oxide composition. Etim et al. (2021) provided the most direct engineering connection by showing that calcite-related chemical signatures and pore filling were associated with improved strength in cement-stabilized lateritic soil containing periwinkle shell ash.

This demonstrates how SEM and FTIR findings can help explain observed geotechnical performance. Oyebanjo et al. (2021) showed that soil mineralogy and geochemistry depend strongly on parent rock. Therefore, soils collected from different locations should be characterized separately rather than regarded as compositionally identical. Finally, Markewich et al. (2023) demonstrated the usefulness of SEM–EDS for identifying minerals and elemental variations at the individual-particle scale, while emphasizing the importance of examining a representative number of particles. In summary, these studies support the combined use of **SEM–EDS, FTIR, XRF and XRD**. SEM–EDS reveals morphology and localized elemental composition; FTIR identifies important chemical bonds and functional groups; XRF provides bulk elemental or oxide composition; and XRD confirms crystalline mineral phases. Their combined application provides a more reliable explanation of the relationship between material composition, microstructure and engineering behaviour

f. **Methodological limitations:** SEM images a very small fraction of a heterogeneous soil and may over-represent visually distinctive particles. Drying, disaggregation, coating and vacuum exposure can alter fabric. EDX is localized and usually semi-quantitative; it is less reliable for light elements and cannot distinguish oxidation states or mineral compounds. Carbon tape and conductive coatings can contaminate spectra. FTIR bands may overlap and are affected by moisture, particle size, baseline treatment, and acquisition mode. Calcination history strongly influences PSA phase composition, and exposure to air can cause hydration and re-carbonation. Accordingly, representative sampling, replicate fields, transparent spectral processing, blanks, certified standards where available, and complementary XRD/XRF/TGA analyses are necessary.

Table .6: Comparison with previous studies

Study	Material	Methods	Main contribution	Relevance
Nayak and Singh (2007)	Clay	XRF, XRD, FTIR	FTIR supported quartz, alumina/haematite and mineral-matter interpretation; XRD supplied phase confirmation.	Shows the need for complementary phase analysis.
Abdelmalik and Sadiq (2019)	Periwinkle shell powder	SEM, XRF, FTIR	Particles of about 2–34 μm; reported CaO-dominant bulk oxide composition (81.8 wt%).	Supports calcium-rich potential; powder is not identical to every PSA.
Etim et al. (2021)	Cement-stabilized lateritic soil with PSA	Geotechnical tests, SEM, FTIR	Calcite-related signatures and pore filling accompanied strength improvement.	Directly links spectroscopy/microstructure with engineering behaviour.
Oyebanjo et al. (2021)	Soil clay fractions from different parent rocks	Mineralogical and geochemical methods	Mineralogy and chemistry varied with parent material.	Warns against treating all selected soils as compositionally equivalent.
Markewich et al. (2023)	Sand and silt samples	SEM-EDS	Particle-scale mineral identification and grain counts were achieved.	Demonstrates spatially resolved capability and need for representative counting.

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CONCLUSIONS

This study concluded that

- the combined FTIR, SEM and EDX analyses provided useful insights into the mineralogical, elemental, and microstructural characteristics of the selected soil samples and periwinkle shell ash (PSA).
- FTIR and EDX analyses confirm the dominance of calcium and oxygen in the fired periwinkle shell, with residual sodium and magnesium.
- Fired periwinkle shell ash particle size becomes smaller when compared with the unfired sample and the calcium content increased after firing, which enhanced the possible pozzolanic reactions.
- The presence of phosphate in the periwinkle shell ash, although in minute quantities, improves the soil strength. The clay samples are aluminosilicates with hydrogen bonding, and iron was found in all the samples with the exception of OS6 sample.
- The presence of iron enhances soil hydration characteristics.
- SEM-EDX and FTIR are complementary tools for the characterization of selected soils and PSA.
- SEM establishes particle morphology, aggregation, porosity and microstructural relationships; EDX associates those features with localized elemental composition; and FTIR screens functional groups and mineral-bond environments in the bulk powder.
- Applied together, these tools can discriminate soil fabrics, reveal elemental heterogeneity and evaluate whether PSA is a calcium-bearing, potentially useful engineering material.
- The tools' evidential limits must be respected: EDX does not identify compounds, FTIR bands are not always unique, and neither method alone quantifies crystalline mineral phases.
- The strongest characterization therefore integrates these techniques with XRD, bulk chemical analysis and geotechnical performance tests. On insertion of the actual spectra and measurements, the studies should conclude specifically which soils are clay-, silica- or iron-rich, which calcium phases occur in PSA, and the relationship of these features in PSA to stabilization performance.

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