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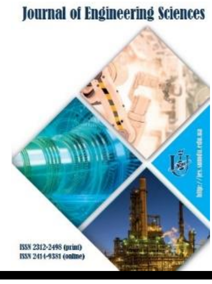


# Durability Evaluation of Calcined Clay and Limestone Powder Blended Ternary Self-Compacting Concrete

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**Abstract.** This research investigates the durability-based properties of a ternary calcined clay and limestone powder blended Self Compacting Concrete by measuring the short- and long-term permeation properties using water absorption and sorptivity properties testing. Also, the variation of compressive strength with age was evaluated at 7, 14, 28, and 56 days, while the split tensile strength was determined at 7 and 28 days curing. The ternary SCC's mineralogy and morphology were evaluated using FT IR Spectroscopy, SEM imaging, and EDS. The results obtained show that the ternary SCC showed improved durability and strength properties with dense and improved microstructure.

**Keywords:** ternary concrete, self-compacting concrete, durability properties, calcined clay, limestone powder.

## 1 Introduction

Sustainable construction requires the use of durable and economical construction materials locally available and can perform optimally throughout the design life of the structure for which they are used. One measure of the durability of concrete is the permeation property, which is a function of the pore structure of the concrete and controls the ingress or otherwise of water and other substances into the concrete. Durable concrete should have minimal amounts of water and other minerals ingress as it ages.

Due to its non-vibrated nature, self-compacting concrete (SCC) could be quite porous, especially if the mix does not have adequate workability. This workability can be enhanced using Supplementary Cementitious Materials (SCM) and fillers [1]. This work investigates the properties of a ternary SCC comprising Calcined Clay (CC) and Limestone Powder (LP) as Supplementary Cementitious Material (SCM) and filler, respectively.

## 2 Literature Review

The past decade has witnessed rapid breakthroughs in the development and application of SCC in the construction industry [2]. A lot of research has been put into the design and production of SCC with very positive results. However, there is no limit to improving this very viable construction material further to improve further its properties in the fresh and hardened states [3].

Different research works have been carried out on self-compacting concrete to either characterize this essential construction material and improve it. [4] presented an overview of various research works on SCC to investigate the new properties, strength, permeability, diffusivity, tribological behavior and thixotropy of SCC incorporating different CRMs and the effects on the interfacial Transition Zone (ITZ). He also reported on different mix design methods and the performance of SCC for different applications. [5] investigated the flow of SCC with and without steel fibers and carried out a 3D modelling of the flow of SCC in slump and L-box tests using a Lagrangian particle-based method known as the Smooth Particle Hydrodynamics (SPH). [6] investigated and modelled the bond strength between SCC and reinforcement using the RILEM pull out method, developing a model for evaluating the bond stress and ultimate slip for SCC. [7] reported on recent updates and developments on SCC as presented at the 2016 RILEM conference on SCC in the areas of mix design methods, materials, test methods, durability, and sustainability. It is interesting that the report contains novel research on the production of eco-efficient SCC (eco-SCC) produced by optimizing aggregate gradation with good results by [8].

One way of improving SCC is the incorporation of SCMs and fillers into the concrete. The absorption of cement composites is principally controlled by the connectivity, pore size, and total porosity, which provides the moisture pathways. This is expected because Concretes with blended binder systems exhibit a more refined pore structure than OPC systems because of the additional/prolonged hydraulic and pozzolanic reactions [9]. Other properties that may be improved include strength [10], durability [11], and microstructural properties [12] of SCC.

Incorporating different mineral admixtures to replace cement or as fillers improve the workability of concrete; thus, helping to improve the concrete microstructure and durability [11]. [13] reports that ternary blends in concrete help reduce/ eliminate the drawbacks of a particular SCM or filler. [14] also investigated ternary blended SCCs with good results. In a similar vein, [15] used different mineral powders in a ternary blend of SCC and reported improved durability. Simoes et al. (2012) reported improved workability in the paper "Ternary mixes of self-compacting concrete with fly ash and municipal solid waste incinerator bottom ash". Thus, the use of Ternary blends in SCC helps to improve the durability of SCC. This work investigates the durability of a ternary SCC using locally available materials (calcined clay and Limestone powder) as supplementary cementitious material and filler, respectively.

## 3 Research Methodology

### 3.1 Materials testing

The fine aggregate- river sand, and the coarse aggregate – crushed granite rock of maximum size 20mm, both obtained locally in Zaria, Nigeria, and the CC was characterized for their gradation using sieve analysis. At the same time, the chemical, mineralogical and morphological properties of the cement, CC and LP were determined using SEM, XRF, FTIR and XRD analysis.

### 3.2 Testing of fresh state properties of SCC

The flowability/ deformability properties of SCC were investigated by carrying out slump flow and V-Funnel tests. The slump flow tests were carried out to determine the flow time, time taken to reach a diameter of 500 mm, and the flow diameter under the procedure set out in [16, 17] while the V-Funnel test was carried out to determine the time taken for the SCC to flow out of the funnel and is carried out under the provisions of [18]. According to the European guidelines for SCC (EGSCC 2005), the slump flow value gives the flowability of a fresh SCC, and SCC class SF2 is suitable for most standard applications.

The passing and filling ability of fresh SCC was evaluated using the L-Box and the J-Ring to determine the ability of the SCC to pass through reinforcements and fill the formwork without segregation. The J-ring was used to determine the flow spread and the blocking step in line with the provisions of [19], while the L-box tests were carried out under the procedure set out in [22]. Both the J-ring and L-box tests were used to determine how well a specific batch of SCC will flow through restricted spaces without blocking. The filling ability, determined using the L-box, gives an idea of how well an SCC mix batch can flow into and fill formwork under the action of gravity alone [20].

### 3.3 Permeation and strength properties

#### 3.3.1 Water absorption and sorptivity tests

The durability of the SCC in this research was measured using the rate at which water and other substances can ingress into the SCC, which is a function of the pore structure of the concrete. While water sorptivity measured the rate of water ingress through interconnected pore spaces, the water absorption that measures the general water ingress through all kinds of pore spaces The Sorptivity test was carried out using the procedure outlined in [21] to determine the susceptibility of the unsaturated concrete to the penetration of water through capillarity by determining the increase in the mass of the specimen resulting from absorption of water as a function of time when only one surface is exposed to water. The initial rate of water absorption (or sorptivity, $mm/s^{1/2}$) is defined as the slope of the line that is the best fit of absorption, *I* plotted against the square root of time [21]. The value of the initial rate of absorption for each sample is the slope of the plot of the least squares, linear regression analysis of *I* vs. the square root of time in seconds. The test was carried out at 28 and 56 days for each mix to evaluate the short and long-term effects of the SCM and filler materials on water absorption rate through interconnected capillary poles as a measure of the concrete durability. Three numbers diameter 100 by 50 discs, cut from 100 by 200 concrete cylinder specimens, were used for each test and the average result calculated, while the water absorption test was carried out to determine, in line with the provisions of [24,28], the change in water absorption capacity of the specimens with age after being submerged for 24 hours. The test was carried out at 7, 14, 21, 28, and 56 days to monitor the change in water absorption capacity of the SCC with age. For each test, three 50x50x50 cubes were used and the average value was taken.

#### 3.3.2 Strength tests

The compressive strength test was carried out to measure the effect of CC and LP on the long-term strength development of the ternary SCC and was determined using 100 cubic millimeters concrete cubes cured at 7, 14, 28, and 56 days in accordance with the provisions of [27].

Granted that although concrete is not typically designed to resist direct tension, the knowledge of tensile strength is used to estimate the load under which cracking will develop. This is due to its influence on the formation of cracks and its propagation to the tension side of the reinforced concrete flexural members [25, 26], hence the importance of tensile strength testing. This test was carried out using diameter 100 by 200 cylinders at 7 and 28 days using the split tensile strength test by crushing the cylinders longitudinally on a compressive strength test machine in line with the provisions of [27]. Three specimens were used for each test, and the average value was taken.

### 3.4 Microstructural analysis

The micromechanical properties of the SCC produced were investigated to determine the morphological and mineralogical properties of the ternary SCC after curing for 56 days and then kept outside the water for 90 days. This was to evaluate the effect of the pozzolanic reaction and the filler effect on the microstructural properties of the ternary. This characterization was carried out using SEM, EDS, and FTIR analysis. While the SEM was used to study the morphological properties of the SCC, EDX gave the quantitative and qualitative mineralogical properties thereof, and the FTIR gave the qualitative mineralogical properties of the SCC.

## 4 Results and Discussion

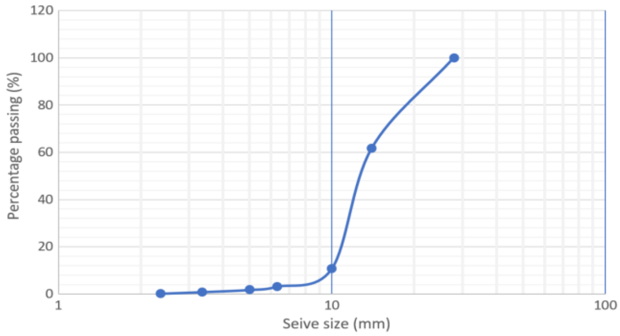
### 4.1 Materials characterization

Figure 1 gives the gradation curves for coarse and fine aggregates and calcined clay, respectively.

The fine and coarse aggregates are well graded, with the coarse aggregates having a maximum aggregates size of less than 20mm as specified in [16] for aggregates and are thus suited for SCC application. It can also be seen that more than 50 % of the clay is finer than and passes through the 75 μm sieve, and over 99 % of the clay particles are finer than 300 μm. That shows that the clay particles have a large surface area for reactivity and can also fill the pore spaces in the concrete. All the LP particles passed through the 75μm sieve and thus should be well filled for filling the pore spaces in concrete.

The result of XRF analysis is presented in Table 1, and it shows that Calcined clay contains more than 70 % $SiO_2 + Al_2O_3 + Fe_2O_3$ stipulated by ASTM C618 for class F pozzolana and so have pozzolanic potential. Also, limestone powder contains more than 50 % CaO and less than 50 % $SiO_2 + Al_2O_3 + Fe_2O_3$, which means even though it is not pozzolanic, it has the potential of being a good filler in concrete and has been used severally in SCC and NVC with positive results [29–31] The high loss on ignition content (LOI) was assumed to be due to the water chemically combined in kaolinite (11.0 % wt.), which would be lost during the calcination process [32].

The results of XRD analysis on the calcined clay and Limestone powder are presented in Figures 2–3 for the qualitative and quantitative analysis respectively of the CC and LP minerals, respectively. It can be seen from the result of the XRD analysis of Calcined clay as presented in Figure 2 a that the CC is made up principally of quartz which is basically $SiO_2$, and thus shows that the material is pozzolanic in nature and can be used as supplementary cementitious material. It is evident that LP comprises calcite principally with quartz, Dolomite, Lime, and Garnet making up the other peaks, as seen in Figure 2 b. The quantitative analysis (Figure 3 a) shows that the calcite makes up the bulk of the LP (77 % of the total composition) with quartz (10 %), dolomite (7.5 %), Lime (5.2 %), and Garnet (0.5 %) taking up the rest of the composition. This result agrees with [33, 34].

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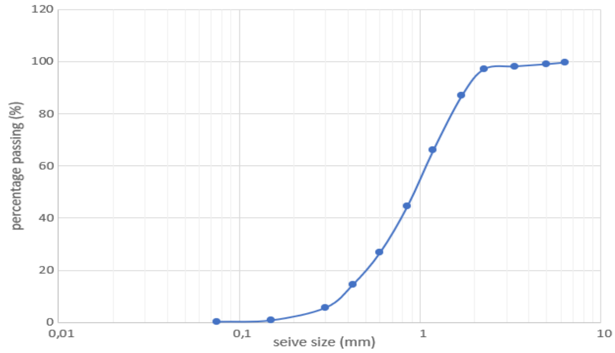


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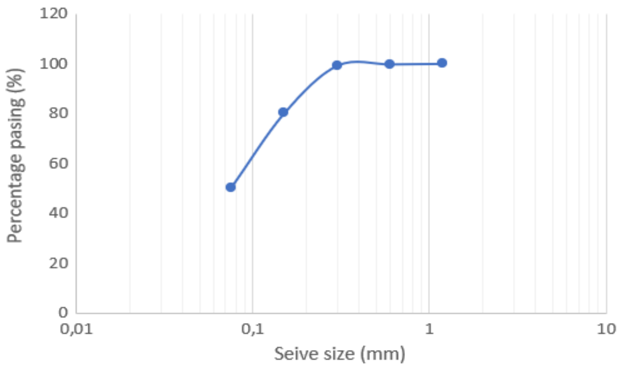
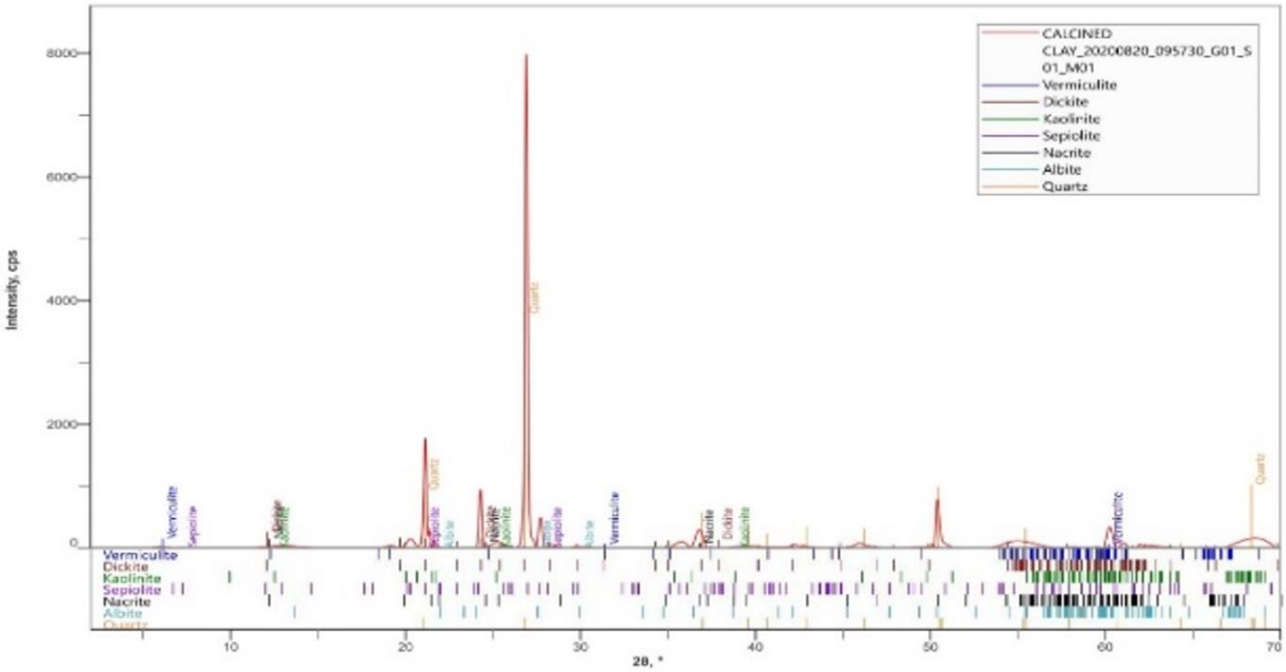



c

Figure 1 – Gradation curve for coarse (a) and fine (b) aggregates, and calcined clay (c)

Table 1 – Oxide composition of PLC, limestone (L), and calcined clay (CC) using XRF analysis

| Oxide | CC | Cement | LP |
|---|---|---|---|
| CaO | 1.50 | 66.5 | 53.2 |
| $Fe_2O_3$ | 0.90 | 6.83 | 1.4 |
| $Al_2O_3$ | 25.1 | 5.60 | 2.13 |
| $SiO_2$ | 59.2 | 16.2 | 3.96 |
| TiO | – | 0.20 | – |
| $K_2O$ | 1.80 | 0.48 | 0.46 |
| $Na_2O$ | 0.03 | 0.78 | 0.05 |
| $SO_2$ | 0.20 | 2.51 | 0.08 |
| BaO | 0.11 | 0.12 | – |
| $V_2O_3$ | 0.10 | – | – |
| $Cr_2O_5$ | 0.03 | – | – |
| MnO | 0.04 | – | – |
| LOI | 11.0 | 1.71 | 37.7 |

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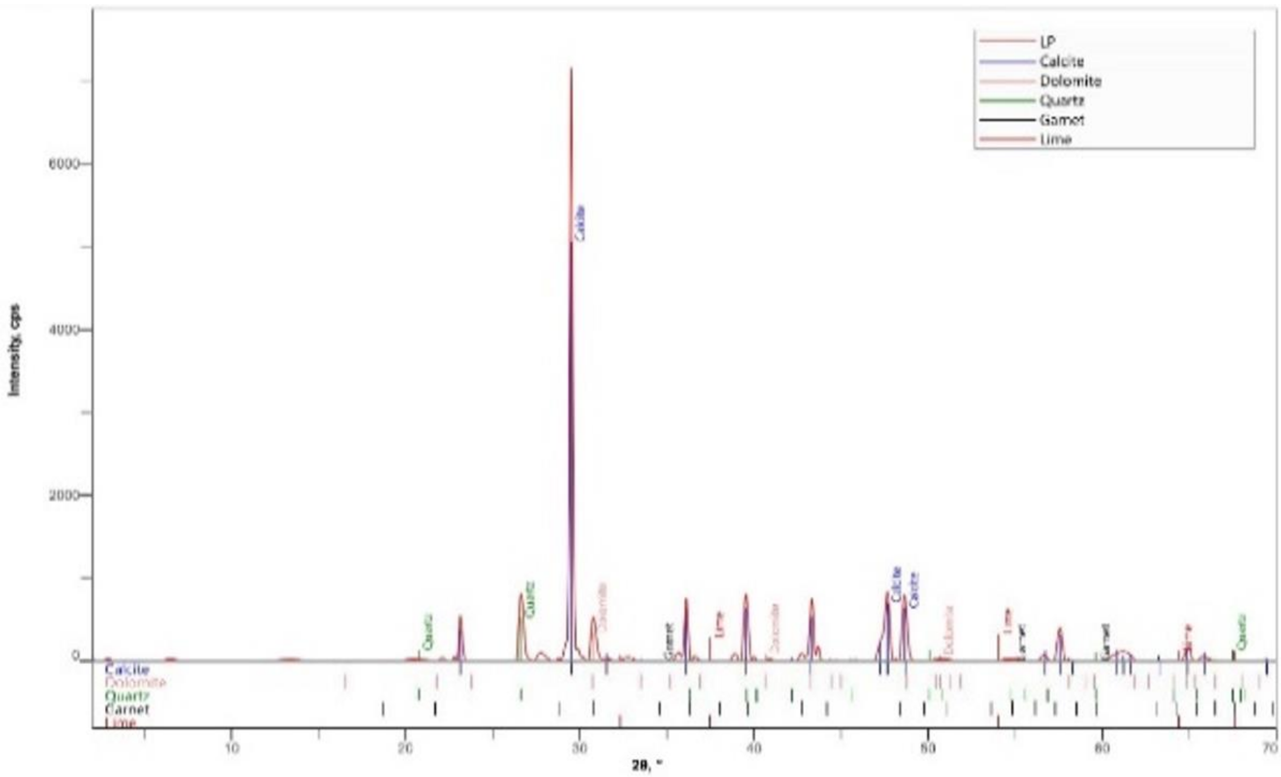


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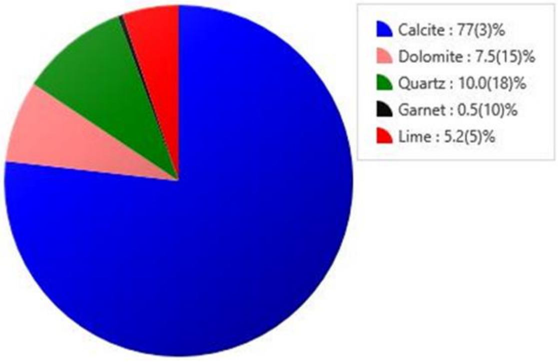
Figure 2 – XRD result (qualitative) of calcined clay(a) and limestone powder (b)

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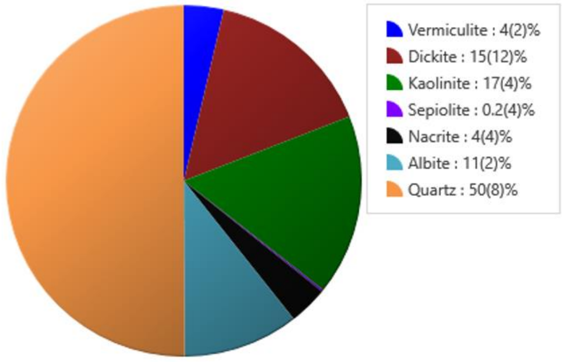


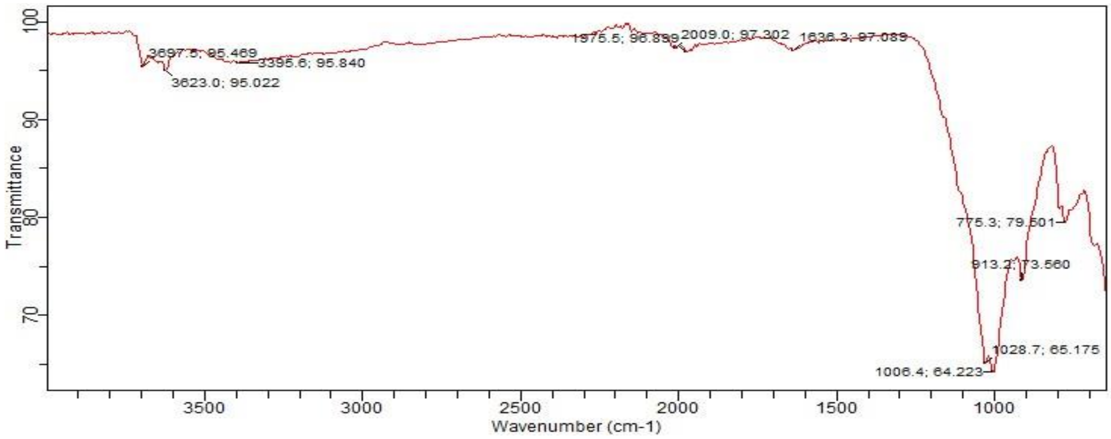

b

Figure 3 – Quantitative analysis of limestone powder (a) and calcined clay (b) minerals

It can also be seen from the result of XRF and XRD analysis of LP that it is not pozzolanic. Limestone powder has no pozzolanic activity and is still unhydrated at the age of 28 days. However, its filling effect can make the paste matrix and the interfacial transition zone between matrix and aggregate denser, which will improve the performance of concrete. Thus, using LP combined with CC in ternary concrete will significantly improve the overall concrete properties.

The result of the XRD analysis (quantitative) analysis report for CC presented in Figure 3 b shows that calcined clay comprises up to 50 % quartz and other clay minerals of the kaolinite group, making 36 % of the calcined clay. Kaolinite, dickite, and nacrite are clays belonging to the kaolin group, which are 1:1 clay made up of Si-O tetrahedral and Al-O(OH) octahedral sheets. Together they form a "composite kaolin layer" with distorted Si-O and Al-O distances.

Figure 4 gives the results of FTIR spectroscopic analysis for Calcined clay and Limestone powder, respectively, using transmission spectroscopy. Major assignments of the bands as compared to literature are Si-O-Si, Si-O Stretching, and H-O-H.

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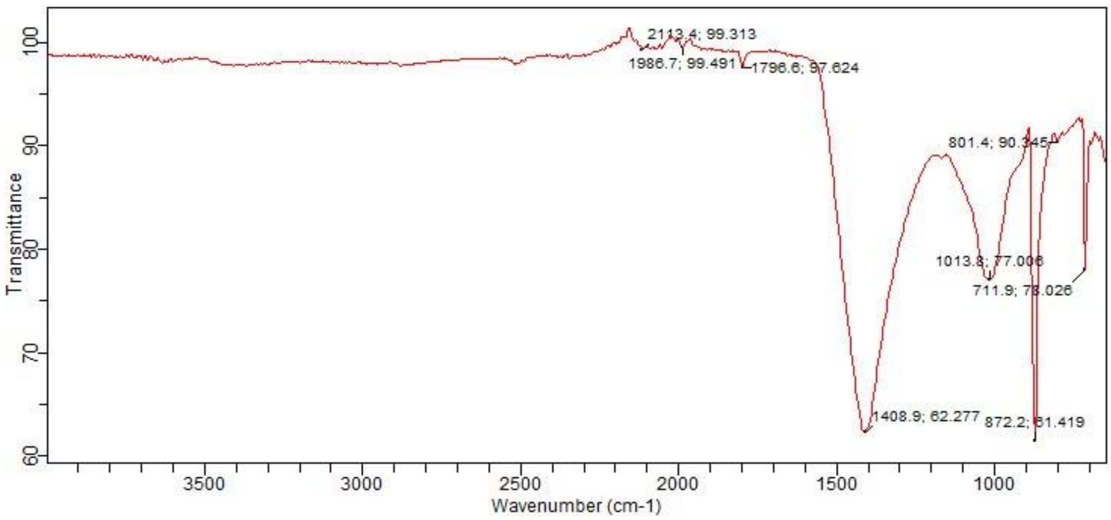



b

Figure 4 – FT IR Spectroscopic analysis for calcined clay (a) and limestone powder (b)

In the IR studies of the clays, Si-O stretching vibrations were observed at 773.3 cm$^{-1}$ (775.3 cm$^{-1}$); 909.5 cm$^{-1}$ (913.2 cm$^{-1}$), 998.9 cm$^{-1}$ (1028.7 cm$^{-1}$), for clay (and calcined clay), showing the presence of quartz [35]. A strong band at 3,693.3 cm$^{-1}$ (3,697.5 cm$^{-1}$) and 3,649.1 cm$^{-1}$ (3,623.0 cm$^{-1}$) indicate the possibility of the hydroxyl linkage (Messaoud et al, 2018), while the interlayer hydrogen bonding is assigned by the characteristic band of 3,620.0 cm$^{-1}$ [36]. Most of the bands present in the clays show the presence of the Kaolintes [35].

A study of the FTIR spectroscopic analysis presented in Figure 4 b shows a dissimilar pattern from the clays, with the characteristic bands of calcite near 1,408.9, 872.2, and 711.9 cm$^{-1}$. The IR peaks appearing at 1,793 and 2,508–2,512 cm$^{-1}$ are also an indication of the presence of calcite [37]. Other vibrations at 1,164.8 and 1,035.6 cm$^{-1}$ appearing as shoulders are also characteristic of quartz. quartz also gives two other characteristic bands at 800.3 and 781.0 cm$^{-1}$. The result agrees with [38] and correlates with the result of XRF and XRD analyses.

### 4.2 Fresh state SCC properties

The flowability of the SCC is measured using the slump flow and V-funnel tests, while the filling/ passing ability of the SCC was measured using the L-Box and J-ring tests, respectively. The results are presented in Table 2.

Table 2 – Flowability and filling/passing ability testing

| Slump test/ V-funnel test | | L-Box/ J-Ring | |
|---|---|---|---|
| $T_{500}$ (s) | 0.33 | $H_1$ | 8.4 |
| Viscosity class | VS1/VF1 | $H_2$ | 7.81 |
| $T_{flow}$ (s) | 4.20 | $H_2/H_1$ | 0.93 |
| $d_1$ (mm) | 730 | $T_{200}$ (s) | 0.36 |
| $d_2$ (mm) | 730 | $T_{400}$ (s) | 1.07 |
| Slump flow (mm) | 730 | $d_{Jx}$ (mm) | 715 |
| SCC class | SF2 | $d_{Jy}$ (mm) | 715 |
| $V_{funnel}$ time (s) | 5.84 | SF J-Ring (mm) | 715 |

The result of the flowability test indicates that the mix belongs to viscosity class VS1/ VF1 based on the result of the $T_{500}$ and $V_{funnel}$ time tests, respectively, and the flowability characteristics are like [16] specification for SF2. [16] stipulates that, for proper filling ability $0.8 \leq H_2/H_1 \leq 1.0$; and this ratio holds for the ternary SCC. The mix also met the passing ability criterion and showed no signs of segregation or blockage. Compared with the provisions of [16], the sample shows no visible blockage due to the high workability. This result agrees with [29, 39, 40].

### 4.3 Durability characterization

#### 4.3.1 Permeation properties (water absorption and sorptivity)

Figures 5, 6 gives the result of water absorption and sorptivity testing, respectively. The 24 hours water absorption after curing for 7, 14, 28, and 56 days given in Figure 5 show that the water absorption decreases with the age of the concrete because of the microstructural evolution related to the continuous cement hydration as well as the filling of the pore spaces by the very fine CC and LP filler.

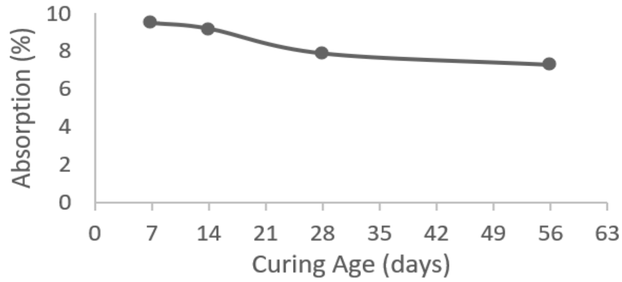



Figure 5 – Variation of water absorption with age

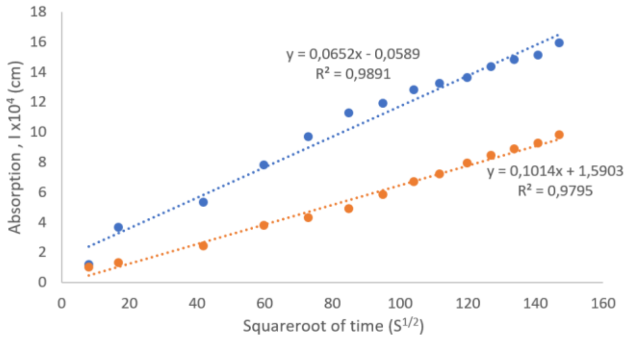



Figure 6 – Initial absorption rate vs square root of time

The incorporation of mixed LS and CC can make more dense interfacial transition zones between cement matrix and aggregates and refine the pore structures of the bulk [41]. Also, the rate at which water is absorbed by the discontinuous pore spaces in the concrete decreases with age. This is due to the pozzolanic reaction, which increases with age as the hydration of cement releases the CH for the pozzolanic reaction, the products thereof filling the pore spaces in the concrete [42, 13]. The primary reason for the better performance was attributed to the more compact and denser microstructure of the system, and the result agrees with [9, 13, 43].

The water sorptivity decreases with age, with the concrete showing better resistivity to capillary absorption at 58 days than at 28 days. What this means is that the concrete gets less porous as it ages. This is due to the filling of the pores by the products of cement hydration and the products of the pozzolanic reaction, and the additional filling up of the pore spaces by the filler effect of the LP [42]. Thus, the ternary blended SCC gets more durable as it ages. The pattern of the result agrees with [43, 46].

#### 4.3.2 Strength Development

The compressive strength 7, 14, 28, and 56 days and the tensile strength at 7 and 28 days are plotted in Figure 7. Both the compressive and tensile strengths increased due to the pozzolanic reaction that starts after lime (CH) is released from cement hydration and the effect of the LP. In addition to its filler effect, LP also has a chemical effect; the calcium carbonate of the limestone powder can interact with the aluminate hydrates formed by the hydration reactions of Portland cement, leading to the stabilization of the ettringite and could increase the total volume of the hydration products, decrease the porosity of the concrete, and consequently increase its strength.

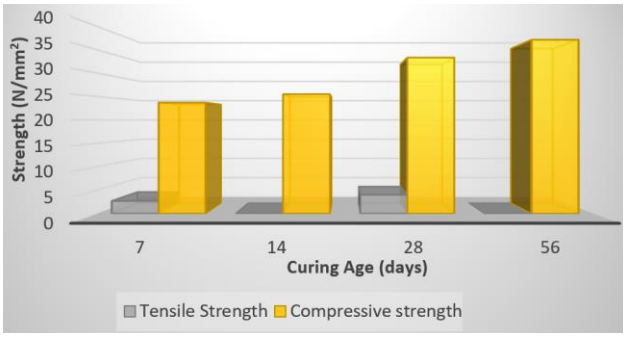
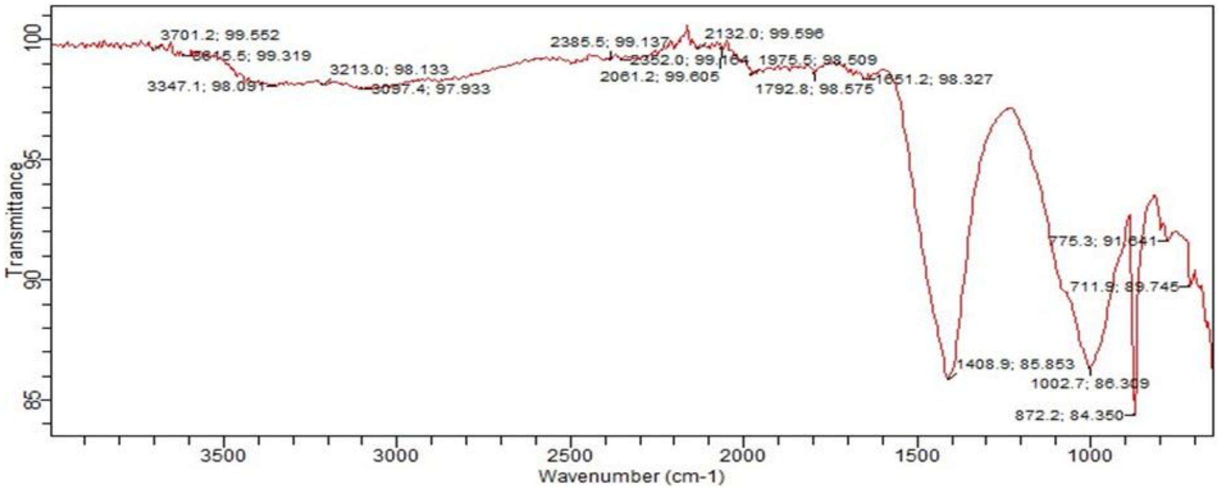



Figure 7 – Strength development of Ternary SCC

Limestone powder could also interact with the AFm and AFt hydration phases, leading to the formation of carboaluminates at the expense of monosulfate, thereby stabilizing the ettringite [39] reported that a ternary cementitious system containing 20 % LS filler and 30 % natural pozzolans exhibited improved early and long-term compressive and flexural strengths and enhanced durability against sulfate, acid, and chloride ion ingress. The trend in this result agrees with findings by [47, 48]. Thus, the pozzolanic properties of calcined clay and the filler effect of LP are combined to more significant advantage, aiding in the durability of SCC measured in terms of long-term strength development.

### 4.3.3 Microstructural Properties

The result of the microstructural investigation using FTIR Spectroscopy and SEM imaging of the ternary SCC is given in Figure 8.




a


b

Figure 8 – Microstructural analysis of SCC using FTIR (a) and SEM imaging (b)

It can be seen from Figure 8 that the microstructure of the ternary SCC has a dense microstructure, which is due to the combination of the pozzolanic reaction from the CC and the filler effect from the LP, hence the denser, more compact, and overall improved microstructure containing CSH, crystalline CH, and fewer voids than the other samples. The result of the microstructural analysis is like research works by [49, 50].

The result of the mineralogical analysis from the SEM micrographs using EDS (Energy Distribution Spectroscopy), at x320 magnification is presented in Figure 9 and summarized in Table 3.

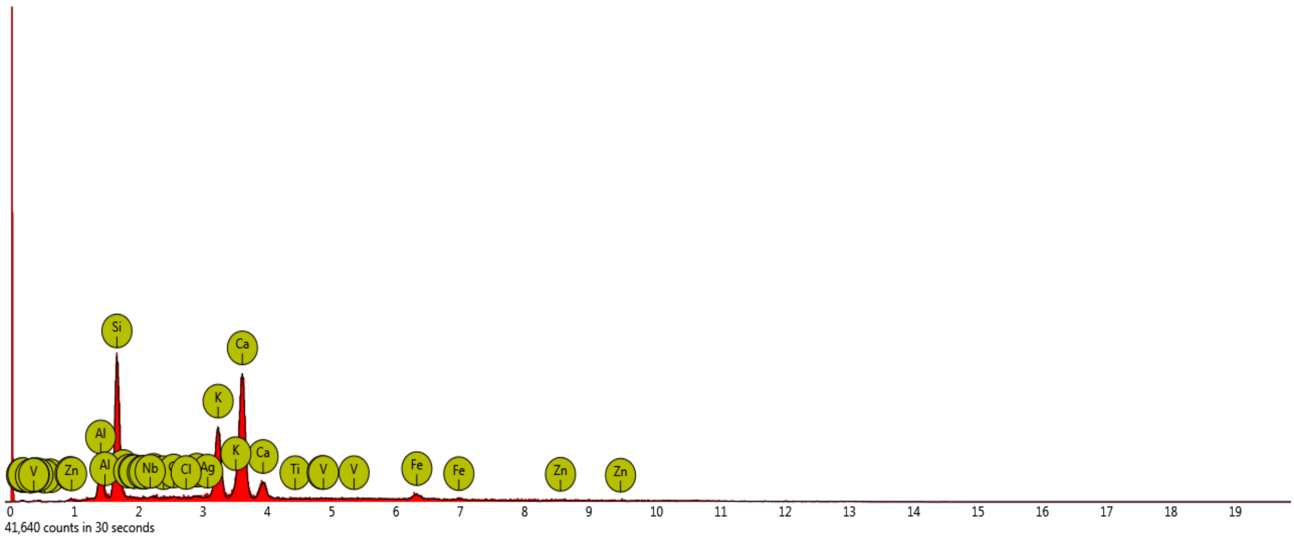



Figure 9 – Mineralogical analysis from SEM Micrographs

Table 3 – Resulting data of mineralogical analysis

| Element Number | Element Symbol | Element Name | Atomic Conc. | Weight Conc. |
|---|---|---|---|---|
| 20 | Ca | Calcium | 38.23 | 41.58 |
| 14 | Si | Silicon | 26.93 | 20.52 |
| 19 | K | Potassium | 18.93 | 20.08 |
| 13 | Al | Aluminium | 8.38 | 6.14 |
| 26 | Fe | Iron | 3.18 | 4.82 |
| 30 | Zn | Zinc | 1.53 | 2.72 |
| 47 | Ag | Silver | 0.47 | 1.37 |
| 39 | Y | Yttrium | 0.32 | 0.76 |
| 41 | Nb | Niobium | 0.18 | 0.46 |
| 16 | S | Sulfur | 0.51 | 0.44 |
| 17 | Cl | Chlorine | 0.39 | 0.38 |
| 23 | V | Vanadium | 0.18 | 0.24 |
| 22 | Ti | Titanium | 0.18 | 0.23 |
| 6 | C | Carbon | 0.48 | 0.16 |
| 15 | P | Phosphorus | 0.13 | 0.11 |

The ternary concrete has an even distribution of calcite, silicate, aluminate, and ferrite-based content due to the pozzolanic reaction and CC and LP filler effect, respectively.

## 5 Conclusions

In terms of recovery of permeation properties such as Sorptivity and water absorption, the durability performance of the ternary SCC is enhanced with the age of concrete. Also, the strength increased as the concrete ages due to the hydration of cement, the pozzolanic reaction, and the improved permeability because of the combined effect of the pozzolanic reaction and the filler effect, with a dense microstructure as well as better mineralogical distribution. Overall, the use of Calcined clay and Limestone powder blend in self-compacting concrete produces durable concrete.

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