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Properties of Calcined Clay and Limestone Powder Blended Bio-Self Compacting Concrete

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ABSTRACT

Self-Compacting Concrete is a revolutionary concrete that has taken Europe, America, and Asia by storm following its development by Prof. Okamura's research team in the late 20th century. Since then, it has been revolutionized into various improved forms either by tinkering with the constituents and mixing design towards perfecting it or by use of locally available secondary cementitious materials and fillers that are ecofriendly and economical, thereby reducing the carbon footprint as well as enhancing its properties. This research looks into the possibility of using Microbial Induced Calcite Precipitation on the engineering properties of calcined clay and Limestone powder blended ternary Self-Compacting Concrete, with emphasis on the evaluation of the fresh state properties as well as strength and durability. Calcined clay was used as Supplementary Cementitious Material at 15% replacement of cement and Limestone powder as filler, with *Sporosarcina Pasteurii* as MICP bacteria at different bacterial cell densities of 1.5x10⁸cfu/ml, 1.2x10⁹cfu/ml, and 2.4x10⁹cfu/ml, (McFarland turbidity scale of 0.5, 4.0 and 8.0 respectively) and calcium lactate (nutrient) concentration of 0.5%, 1.0% and 2.0% by weight of cement incorporated into the ternary blend. The strength is evaluated using compressive strength (at 7, 28, and 56 days of curing) and split tensile strength (at 7 and 28 days), while the durability characteristics are evaluated using water absorption (7, 28, and 56 days) and sorptivity (7, 28 and 56 days) and the microstructure investigated using Standard electronic microscopy. The result indicates an overall improvement in the properties of the Self-compacting concrete.

Keywords: Microbial Induced Calcite, Precipitation Micro-structural characterization, Permeation properties, Compressive Strength, *Sporosarcina pasteurii*

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1. INTRODUCTION

Self-Compacting Concrete (SCC) is a revolutionary concrete that has taken Europe, America, and Asia by storm following its development by Prof. Okamura's research team in the late 20th century [1]. Since then, this very important construction material has been revolutionized into various improved forms either by tinkering with the constituents and mix design towards perfecting it and reducing the number of trials and errors involved in designing SCC mixes or by use of locally available secondary cementitious materials and fillers that

are ecofriendly and economical, thereby reducing the carbon footprint involved in the production of concrete as well as enhancing its properties [2-9]. From small beginnings in the late 1980s, the self-compressed concrete market is expected to reach USD 18.4 billion by 2028 and grow at a compound annual growth rate of 6% over the forecast period from 2021 to 2027 [10]. This expected market growth in the use and production of SCC is due to the advantages inherent in this very important construction material, and research is ever ongoing, aimed at further

improving the properties and economy of SCC. Over the last decade, the application of bacteria in the construction industry has become a topic of worldwide research, with special emphasis on Microbial Induced Calcite Precipitation (MICP) which has been studied and applied in the field of civil engineering for surface protection of natural stone, soil improvement, crack remediation, strength and durability improvement [11]. MICP in concrete technology involves the use of calcite precipitating bacteria in concrete to remediate micro and even macro cracks as well as improve other properties of concrete. MICP has been used successfully by researchers in enhancing concrete properties such as Compressive Strength [12], Concrete durability [13] remediation of cracks [14], water absorption [15], surface consolidation [16], and Rebar corrosion inhibition [17] amongst other applications. In the same vein, extensive research has been put into the use of calcite precipitating bacteria in NVC to improve its properties in both the fresh and hardened states with very exciting and positive results [18-22] However, this technology has not yet been fully applied in SCC, with limited research in self-healing SCC restricted to strength characterization [23,24]. Simply assuming that the use of

MICP technology in SCC will have the same effect as in NVC may not be appropriate because SCC behaves differently from NVC in most areas due to differences in mix proportioning of the constituents, and mix design procedures, lack of mechanical vibrations, high volume of paste used and reduced amount and sizes of coarse aggregates used in SCC [25, 26]. It is, therefore, imperative to carry out this research on characterizing bio-SCC made by incorporating MICP bacteria in SCC and establishing the effect on the properties of SCC in the hardened states both in the short and long terms. This product, which will be referred to in this work as Sustainable Bio-SCC or bio-SCC for short (Comprising MICP Bacteria, a Large amount of SCM and filler, OPC, and Fine and coarse aggregates), will open up a new frontier of research that will in the long run aid in the production of more durable and environmentally friendly and sustainable SCC. This research is significant as it brings together these two exciting frontiers of research so as to improve the properties of SCC, especially its cost and pore characterization, thereby helping in its sustainability, strength, and durability.

2. MATERIALS AND METHODS

2.1. MATERIALS

The materials used for this research work include: 1 Cement: The cement used for the work is a grade 42.5N Portland cement and was characterized using XRF, setting times, consistency, and specific gravity in accordance with BS 12. 2 Fine Aggregates: The fine aggregates used are river sand obtained locally in Samaru, Zaria, and were characterized using sieve analysis and specific gravity tests in accordance with the provisions of the relevant BS 812. 3 Coarse Aggregates: The coarse aggregate used is crushed granite obtained locally in Zaria and was characterized using sieve analysis, specific gravity, aggregate crushing value, and aggregates impact value tests in accordance with the provisions of the relevant BS 812. 4 Calcined Clay: The Calcined clay used in this work was obtained at the Nigeria Building and Road Research Institute (NBRI) Pozzolana plant in Bokkos, Plateau state, and was characterized for setting times and specific gravity, as well as sieve analysis, oxide composition using XRF analysis and XRD as well as FTIR analysis. The clay

was calcined at 700 oC using the NBRI pozzolana furnace in Bokkos, Plateau state. 5 Limestone Powder: The limestone used in work was obtained from a limestone quarry site of the Dangote Cement factory in Yandev, Gboko. The solid limestone was pulverized and later ground to a fine powder using the ball mill in the Department of Chemical Engineering, Ahmadu Bello University Zaria. The Limestone powder was characterized using XRF, XRD, and FTIR analysis. 6 Water: The water used was obtained from the Civil Engineering Laboratory of the Ahmadu Bello University Zaria. 7 Super-plasticizer: Polycarboxylic ether (PCE) based high-range super-plasticizing, water-reducing super-plasticizer, known as SANDCRETE SPR-300, manufactured by Wafa manufacturing group Lagos was used for the work. 8 Bacteria: The Bacteria used for the work was *Sporosarcina pasteurii*, and it was cultured and inoculated in the laboratory of the Department of Microbiology, Ahmadu Bello University Zaria.

2.2. METHODOLOGY

2.2.1. FLOWABILITY/ DEFORMABILITY TESTS

The flowability/ deformability properties of SCC were determined by carrying out slump flow and V-Funnel tests. The slump flow tests were carried out to determine the flow time, the time taken to reach a diameter of 500mm, and the flow diameter in accordance with the procedure set

out in EFNARC 2005 and BS EN 12350-8:(2010) 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 in accordance with the provisions of BS EN 12350-9:(2010).

2.2.2. *PASSING AND FILLING ABILITY TESTING*

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 as well as to 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 BS EN 12350-12: (2010); BS EN 206-9:(2010), and EFNARC 2005; while the L-box tests were carried out in accordance to the procedure set out in BS EN 12350-10: (2010).

2.2.3. *WATER ABSORPTION AND SORPTIVITY TESTS*

The Sorptivity test was carried out using the procedure outlined in ASTM C 1585-13 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 test was carried out at 7, 28, and 56 days for each mix to evaluate the short and long-term

effects of the SCM and filler materials on the rate of water absorption through interconnected capillary poles. The water absorption test was carried out to determine, in line with the provisions of BS 1881-122:2011 and ASTM C642: 2006. The test was carried out at 7, 28, and 56 days to monitor the change in water absorption capacity of the SCC with changing quantities of SCM and filler.

2.2.4. *STRENGTH TESTS*

The tensile strength was determined using diameter 100mm by 200mm cylinders at 7 and 28 days in line with the provisions of BS-EN 12390-6, while the compressive

strength of the SCC was determined using 100 cubic millimeters concrete cubes cured at 7, 28 and 56 days in accordance with the provisions of BS EN 12390 – 3:2009.

3. RESULTS AND DISCUSSION

3.1. *MIX CONSTITUENTS AND DESIGNATION*

Ten mixes were designed and designated S0 to S9, with S0 the control (without bacteria and nutrients) and S1 to S9 at varying bacteria and calcium lactate concentrations. A total of 90 number 100x100x100 cubic millimeter cubes (for compressive strength), 70 number 100x200 cubic millimeters cylinders (tensile strength and sorptivity), and

90 number 50x50x50 cubic millimeter cubes (water absorption) were cast and used for the entire work. A constant water-cement ratio of 0.53 was used for all the mixes. [Table 1](#) gives the mix proportions/ quantities for the laboratory work of the research.

Table 1. Mix designation and proportions

Mix	Bacteria Conc. (Cfu/ml)	Calcium lactate	Cement (Kg)	Calcined Clay (kg)	Superplasticizer (kg)	Limestone powder (Kg)	Sand (Kg)	Coarse Aggregates
S0	-	-	11.56	2.04	0.083	4.1	25.9	28.3
S1	1.5x10 ⁸	0.5	11.56	2.04	0.083	4.1	25.9	28.3
S2	1.5x10 ⁸	1.0	11.56	2.04	0.083	4.1	25.9	28.3
S3	1.5x10 ⁸	2.0	11.56	2.04	0.083	4.1	25.9	28.3
S4	1.2x10 ⁹	0.5	11.56	2.04	0.083	4.1	25.9	28.3
S5	1.2x10 ⁹	1.0	11.56	2.04	0.083	4.1	25.9	28.3
S6	1.2x10 ⁹	2.0	11.56	2.04	0.083	4.1	25.9	28.3
S7	2.4x10 ⁹	0.5	11.56	2.04	0.083	4.1	25.9	28.3
S8	2.4x10 ⁹	1.0	11.56	2.04	0.083	4.1	25.9	28.3
S9	2.4x10 ⁹	2.0	11.56	2.04	0.083	4.1	25.9	28.3

3.2. *FRESH STATE SELF-COMPACTING CONCRETE PROPERTIES*

The flowability of the SCC is measured using the slump flow and V-funnel tests, and the result, as well as that of the passing and filling ability tests performed using the L-

box and J-ring equipment, is presented in [Table 2](#). The result indicates that the mix belongs to viscosity class VS1/ VF1 based on the result of the t500 test and Vfunnel time

tests, respectively, and SCC class SF2. EFNARC 2005 stipulates that for proper filling ability $0.8 \leq H_2/H_1 \leq 1.0$. It can be seen from Table 2 that the ratio holds true for all mixes, and it also met the passing ability criterion and showed no signs of segregation or blockage and show no visible blockage due to their high workability. The incorporation of *Sporosarcina Pasteurii* at different bacterial cell densities of 1.5×10^8 cfu/ml, 1.2×10^9 cfu/ml,

and 2.4×10^9 cfu/ml, (McFarland turbidity scale of 0.5, 4.0, and 8.0 respectively) and calcium lactate (nutrient broth) concentration of 0.5%, 1.0% and 2.0% by weight of cement had no significant effect on the fresh state properties of SCC. This is in agreement with research by various projects [27-30]. The fresh state properties are affected by cement content, water content, superplasticizer dosage as well as SCM and filler content [31].

Table 2. Flowability, filling and passing ability testing

% of CC	t ₅₀₀ (s)	Viscosity class		t _{flow} (s)	d ₁ (mm)	d ₂ (mm)	Slump flow(mm)	SCC class	V _{funnel} time (s)
15	0.33	VS1/VF1		4.20	730	730	730	SF2	5.84
L-Box Test results						J-Ring Tests results			
% of CC	H ₁	H ₂	H ₂ /H ₁	T ₂₀₀ (s)	T ₄₀₀ (s)	d _{Jx} mm	d _{Jy} mm	SF _J	
15	8.4	7.8	0.93	0.36	1.07	715	715	715	

3.3. PERMEATION AND STRENGTH PROPERTIES

3.3.1. PERMEATION (DURABILITY) CHARACTERIZATION

The 24 hours' water absorption of the SCC containing CC and LP and, incorporating *Sporosarcina Pasteurii* at different bacterial cell densities calcium lactate (nutrient

broth) concentration of 0.5%, after curing for 7, 28 and 56 days given in figure 1.

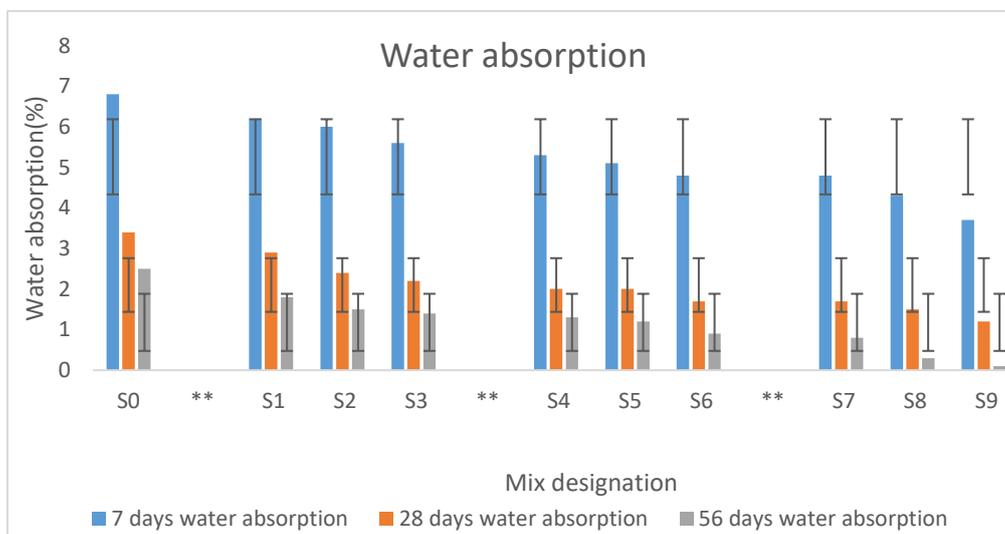


Figure 1. Variation of water absorption with age of concrete, bacteria density and calcium lactate concentration

Figure 1 shows that the water absorption decreases with the age of the concrete for all the samples tested. The initial water absorption reduces with age for all mixtures 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, as well as the deposition of calcium carbonate due to the MICP activity of the bacteria. The water absorption decreased as the bacteria concentration is increased. Also, for a given bacteria

concentration, the water absorption decreased with an increased percentage of calcium lactate. According to Raid et al. [32], the presence of nutrients in concrete specimens in which there are bacteria may have caused the bacteria to form calcite sediments deposited in the pores, which naturally reduces water penetration through the concrete. The result agrees with the works of Irwan et al. , Mondal & Ghosh and Riad et al [33-35].

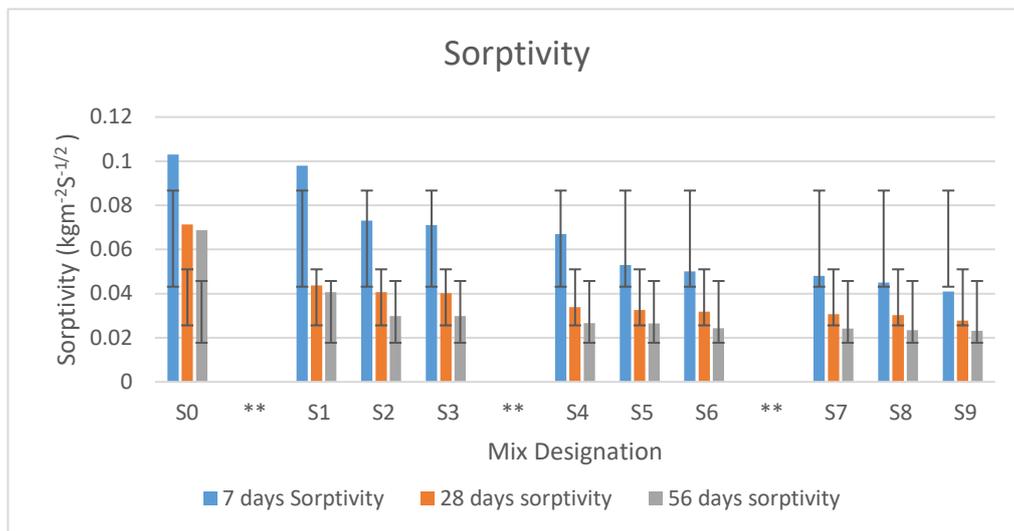


Figure 2. Variation of water absorption with age of concrete, bacteria density and calcium lactate concentration.

It can be seen from [Figure 2](#) that the rate of absorption (sorptivity) decreases with age, bacteria concentration, and calcium lactate content, with all the samples showing better resistance to capillary absorption at 58 days than at 7 and 28 days. What this means is that the concrete gets less porous as it ages. Also, according to Vaezi et al. research [\[36\]](#), the reduction of pore size as a result of calcium carbonate deposition is the main reason for reduced water absorption. The reduction in sorptivity with additional calcium lactate (calcium source) and bacterial concentration is due to the additional calcium lactate acting as a catalyst for further deposition of calcium carbonate [\[13\]](#). The increased precipitation of calcium carbonate further plugs out the continuous pores within the

concrete matrix, thereby further reducing the sorptivity. This result is in agreement with previous research on NVC [\[36, 37, 13\]](#). The filling of the pores by the products of cement hydration as well as the products of the pozzolanic reaction, could also account for some reduction in the sorptivity, hence the reduced absorption by S0 with age even though it contains no bacteria and nutrients. The evaluation of the standard deviation from the mean shows that all the samples containing bacteria and calcium lactate have sorptivity values less than the mean value, with the standard deviation increasing with bacteria cell concentration and calcium lactate content. This agrees with Pereira de Oliveira Dhandapani et al. and Karatas et al. [\[38-40\]](#).

3.3.2. STRENGTH CHARACTERIZATION

The compressive strength of the Bio-SCC after curing the

samples for 7, 28 and 56 days is presented in [figure 3](#).

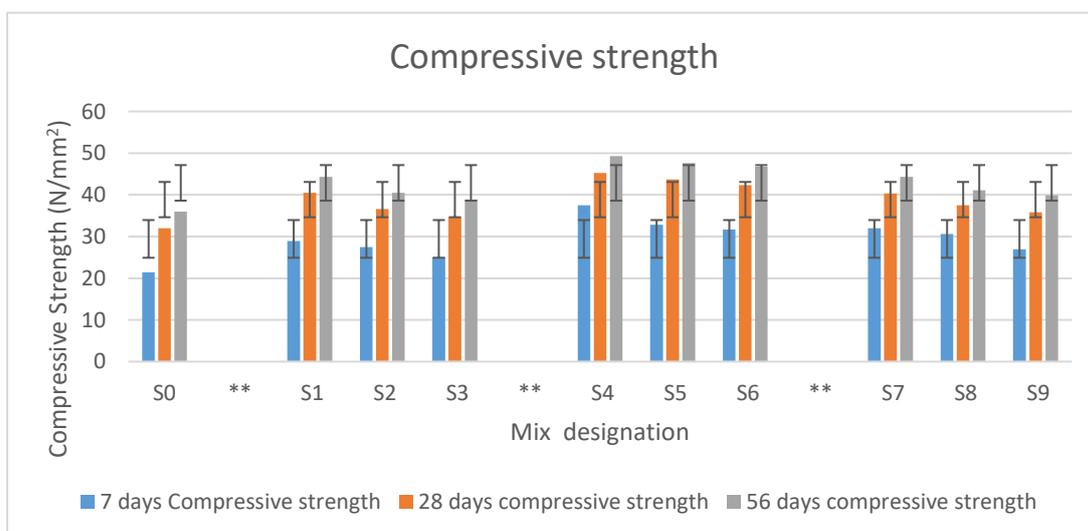


Figure 3. Variation of compressive strength with age of concrete, bacterial concentration and calcium lactate content.

The result of Compressive strength is presented in [Figure 3](#) shows the variation of compressive strength with curing

age, bacterial concentration, and calcium calcite content. It can be seen that the compressive strength increased with

age for all mixes (S0 to S9), with the highest strength recorded at 56 days. For a given bacteria concentration, using 0.5% calcium lactate maximized the compressive strength, with a decrease in the rate of strength development with increasing calcium lactate content. The over-production of calcium carbonate crystals, which lowers the quality of the micro-structure, resulted in a lower rate of strength gain at higher calcium content Vaezi et al. [36]. Also, Irwan et al. [33] Stated that materials added to concrete that do not contribute to the hydration process could impede strength development. The ultimate strength is obtained at 1.2×10^9 cfu/ml and lactate content of 0.5% of the weight of cement. Vaezi et al. [36] state that the gain in strength is due to the densification of the pore system due to the precipitation of calcium carbonate by the bacteria cells. According to Mondal & Ghosh [34], it can be inferred that adding different bacterial concentrations leads to two different types of healing in concrete, namely, surface healing and inner matrix healing. At the surface region, as the availability of water is equal for all the samples, the precipitation is only dependent upon the

bacterial concentration. Since a greater number of bacterial cells can precipitate a higher amount of calcite, the maximum amount of precipitation at the surface region of the mortar takes place at the highest cell concentration. Thus, all the test results directly related to the surface region of the mortar, such as surface crack and pore healing and reduction in water penetration depth, exhibit better performance at the highest cell concentration (2.4×10^9 cfu/ml). However, the high calcite precipitation almost blocks the surface pores, and that leads to lower availability of water inside the mortar matrix. This is correlated by Chahal et al. [41], who states that there is an optimal bacteria cell concentration beyond which the strength could be adversely affected. The results agree with the research of Siddique et al. [44], Nagarajan et al. [42], Mondal & Ghosh [34], Alisha et al. [30], and Tanyildizi et al. [43]. Also, an evaluation of the standard deviation shows that all the strength values for 1.2×10^9 cfu/ml fell above the mean value of the strength for all calcium lactate dosages. The result of tensile strength testing for 7 and 28 days is presented in figure 4

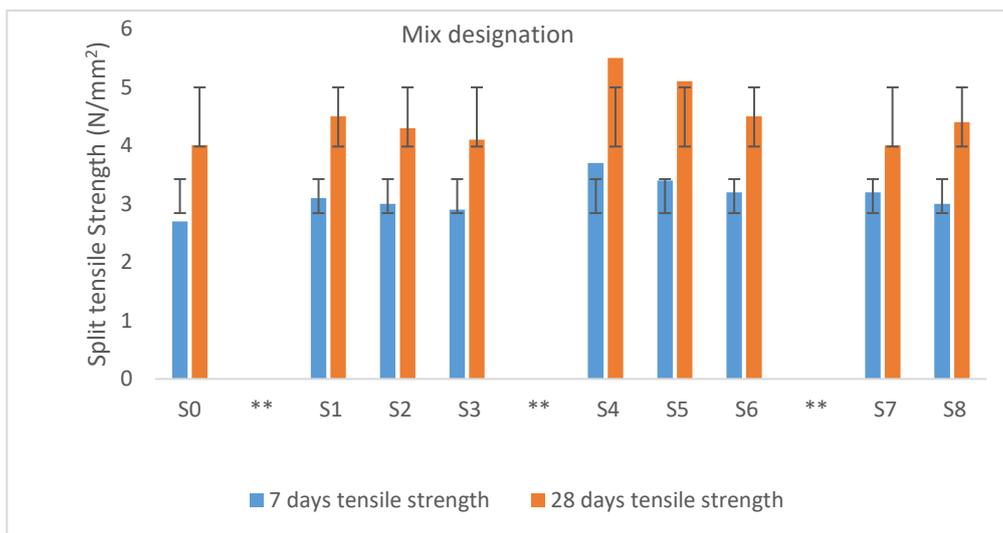


Figure 4. Tensile strength testing

As expected, the variation in tensile strength with curing age follows the compressive strength. This is because of the positive correlation between compressive and tensile strengths [45]. The same explanation for this trend holds for tensile strength as earlier explained

3.4. STATISTICAL EVALUATION

One-way Analysis of Variance (ANOVA) was used to evaluate the relationship between the bacteria dosage, nutrient content and the properties of the SCC. The result of one-way ANOVA on the compressive strength is given in Table 3, with the null hypothesis postulating that there is

no significant correlation between the bacteria content, the nutrient content and compressive strength at 7, 28 and 56 days and the alternate hypothesis proposing a significant relationship.

Table 3. ANOVA result for Compressive Strength

Summary						
Groups	Count	Sum	Average	Variance		
Bacteria Conc.	10	1.13E+10	1.13E+09	1E+18		
Calcium lactate	10	10.5	1.05	0.525		
7 days	10	294.3	29.43	20.45789		
28 days	10	388.6	38.86	17.87822		
56 days	10	428.7	42.87	18.19789		
ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	1.01E+19	4	2.53E+18	12.64045	5.64E-07	2.578739
Within Groups	9.01E+18	45	2E+17			
Total	1.91E+19	49				

The null hypothesis which states that there is no significant relationship between the parameters investigated is discarded due to the p-value being less than 0.05 and the Fcritical being less than the F-value. A post hoc test known as Turkey honestly significant difference (HSD) is carried out to determine where the exact statistical relationships lie. [Table 4](#) gives the result of Turkey’s HSD, calculated using Engineering statistical packages from the National Institute of Standards and Technology (NIST), US

Department of Commerce. A, B, C, D, and E are the Bacteria Density, Calcium lactate (nutrient content), 7 days, 28, and 56 days compressive strength, respectively. It can be seen that there is a positive and significant relationship between the bacteria density and each of the other parameters with a Tukey HSD p-value of 0.0010053, which is less than 0.01. the rest of the relationships are insignificant, with a p-value of 0.8999 which is greater than 0.01.

Table 4. Turkey HSD p-value for water absorption

Treatments pair	Tukey HSD Q statistic	Tukey HSD p-value	Tukey HSD inference
A vs B	7.9500	0.0010053	** p<0.01
A vs C	7.9500	0.0010053	** p<0.01
A vs D	7.9500	0.0010053	** p<0.01
A vs E	7.9500	0.0010053	** p<0.01
B vs C	0.0000	0.8999947	Insignificant
B vs D	0.0000	0.8999947	Insignificant
B vs E	0.0000	0.8999947	Insignificant
C vs D	0.0000	0.8999947	Insignificant
C vs E	0.0000	0.8999947	Insignificant
D vs E	0.0000	0.8999947	Insignificant

3.5. MICRO-STRUCTURAL CHARACTERIZATION

The Microstructural properties of the Bio-SCC were investigated using SEM analysis on SCC samples taken at 7, 28, and 58 days of curing. The result for a fixed calcium lactate content of 0.5 and different bacterial cell densities of 1.5E⁸cfu/ml, 1.2E⁹cfu/ml, 2.4E⁹cfu/ml are presented in

[Figure 5 \(a\) to \(c\)](#), respectively. As can be seen in all cases, the microstructure of the samples improves with age due to the pozzolanic reaction and the effect of the precipitation of calcite by the bacteria.

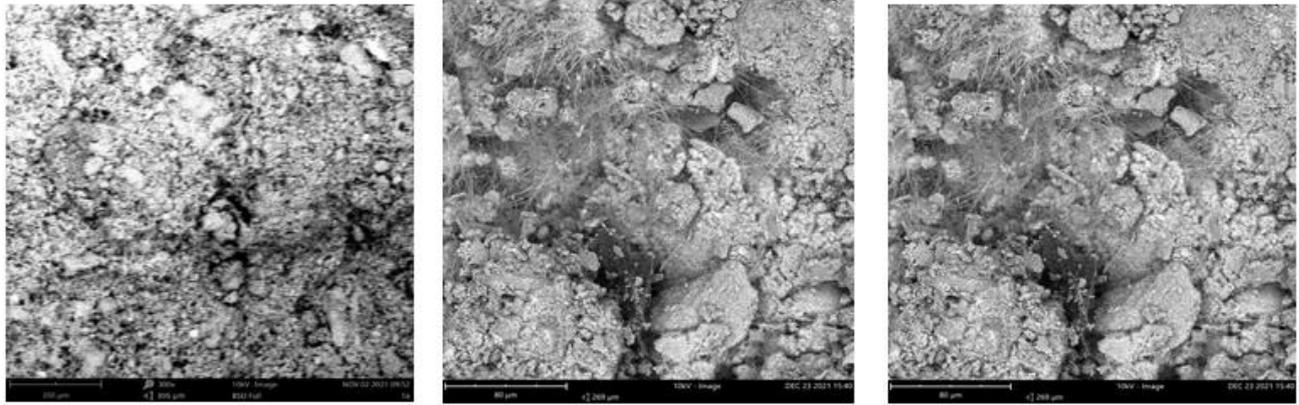


Figure 5(a). SEM for sample S1 at 7, 28 and 56 days respectively



Figure 5(b). SEM for sample S4 at 7, 28 and 56 days respectively



Figure 5(c). SEM for sample S7 at 7, 28 and 56 days respectively.

4. CONCLUSION

The Following conclusions are reached from this research:

1. The incorporation of *Sporosarcina Pasteurii* at different bacterial cell densities of 1.5×10^8 cfu/ml, 1.2×10^9 cfu/ml, and 2.4×10^9 cfu/ml, (McFarland turbidity scale of 0.5, 4.0 and 8.0 respectively) and calcium lactate (nutrient broth) concentration of 0.5%, 1.0% and 2.0% by weight of cement had no significant effect on the fresh state properties of SCC.
2. The incorporation of *Sporosarcina Pasteurii* at different bacterial cell densities (BCD) and calcium lactate (nutrient broth) positively impacted on the water absorption and sorptivity of SCC, with optimum values at BCD of 2.4×10^9 cfu/ml and calcium lactate content of 2.0%.

3. The incorporation of *Sporosarcina Pasteurii* at different bacterial cell densities and calcium lactate (nutrient broth) concentration impacted the compressive and tensile strengths of SCC with Optimum values at BCD of 1.2×10^9 cfu/ml and 0.5% calcium lactate content.

It is recommended that an optimal bacterial concentration of 1.2×10^9 cfu/ml (considering the McFarland scale of between 0.5 and 8.0) at calcium lactate content of 0.5% by weight of cement be used in SCC if the strength is the main consideration. If, however, the permeability characteristics are a major consideration, a higher BCD and calcium lactate content can be considered.

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AUTHORS CONTRIBUTION

This work was carried out in collaboration among all authors.

CONFLICT OF INTEREST

The author (s) declared no potential conflicts of interests with respect to the authorship and/or publication of this paper.

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