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ARTICLE

Physio-Chemical Characteristics and Acid-Sulphate Reactions of Moringa Oleifera Seed Powder Cement Paste and Concrete

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ABSTRACT

The evaluation of the effect of using moringa oleifera seed powder (MOSP) to partially replace cement by wt. % has been carried out. A mix parameter of 1: 1.7: 2.5, with designed strength of 20 kN-m^2, and a cement content of 420 kg-m^3, water-cementitious ratio of 0.5, to produce concrete specimens to which percentages of MOSP by wt. % of cement were added and cured for 90 days. Basic characteristics of the MOSP material were determined (Consistency and Setting times), and the concrete parameters workability, density, water absorption and compressive strength were also determined. The analysis of the experimental data collected on MOSP and MOSP-concrete confirmed that MOSP is substantially silicate (Quartz and Cristobalite). These have greatly to a large extent imparted on the quality of MOSP-concrete produced good quality concrete. The optimum replacement was at 0.2 % wt. % of cement.

1. Introduction

The construction industry is evolving, and the need to address demands for constructed facilities are increasing and becoming more complex with the dwindling conventional materials, and climate change. Cost and safety have been the underlying thrust in the construction industry, and therefore, the need for improved Research and Development (R & D) becomes very imperative. The importance of admixtures and additives in the concrete industry is gaining wide recognition because of proven advantages in ease of concreting, strength gains and durability issues. Water-reducing additives such as superplasticizers make it possible to achieve good workability of fresh concrete and correspondingly a dense concrete microstructure in the hardened state [1].

Recent advances in researches on new chemical additives have arisen because of the great advances in concrete technology. These new chemical additives, applied in small quantities can drastically improve crucial properties of concrete in its fresh and/hardened state [1]. Moringa oleifera seeds and leaves have been used extensively for water treatment, and in the health sector. They have not been researched upon for possible use as additives in the construction industry. To this extent, not much is said about the use of Moringa oleifera for concrete production in the literature. Therefore, it becomes imperative to carry such research works on Moringa oleifera for possible application in the construction industry. These research works will help determine the advantages and disadvantages in the use of the material. The introduction of Moringa oleifera seed powder as a

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new component for concrete production makes available a number of new possibilities with respect to additive materials for concrete production. This will improve workability and the reheological properties of the fresh concrete.

A preliminary work on Moringa oleifera seed powder was undertaken by Susilorini et al [2]. They worked on the "compressive strength optimization of natural polymer modified mortar with Moringa oleifera in various curing media" using a mortar mix of 1: 1: 0.6, that is, cement, sand and water, with moringa oleifera seed powder dosage of 0, 0.1, 0.2, 0.5, 1, 2, and 5 % by wt. % of cement. Mortar cubes of 50 mm dimensions were made and cured for 7 days, 14 days, and 28 days, before testing to failure. The results of their study showed that the use of moringa oleifera seed powder improved the quality of the concrete, and the optimum dosage that gave the highest compressive strength was at 0.2 %.

A conference proceeding paper on the improved work on moringa oleifera seed powder and husk was carried out by Susilorini et al [3]. They made several concrete cylinder specimens of natural polymer modified concrete with dimensions 10 cm x 20 cm for a concrete design strength of 30 MPa and cured for 7, 14, and 28 days, using a moringa oleifera dosage of 0.2 wt. %. The results also showed that using the powder and husk improved the concrete strength.

The present work arose because of the very little knowledge of the spectrum of moringa oleifera seed powder applications as additives in concrete production. Equally of concern was the fact that Susilorini et al works were not in depth [2,3]. In the present study experiments are mounted to characterize the behavioral pattern of the moringa oleifera seed powder in cement paste and concrete. The basic characteristics of the material (water affinity and setting times) are established, and the effects of the hydration process on the physical, chemical, and morphological composition of MOSP on concrete characteristics such as workability, density, water absorption and compressive strength are evaluated. The work also assessed the behavior of this material in concrete, in acid and sulphate environments.

2. Materials

The materials used for this research are grade 43 'Ashaka' Portland cement, fine aggregate (river sand), coarse aggregate, Moringa oleifera seed powder (MOSP) and potable drinking water. The physical and chemical properties of the cement are shown in Table 1, while, the physical properties and sieve analysis of the fine and coarse aggregates are shown in Tables 2 and 3. Both the cement and aggregates conform to BS EN 196-1 [4] for testing cement, and BS EN 12620 [5] and BS 812-2 [6] for the testing of aggregates. The size of the coarse aggregate was 20 mm and both the fine and coarse aggregates were of zone 2 in the grading system.

Table 1. Physical and Chemical Properties of Cement

Physical Properties of Cement	
Specific Gravity	3.15
Lose Bulk Density (kg-m^3)	3150
Fineness Modulus	<10
Chemical Properties	
Oxide	Percentage by weight (%)
CaO	63.7
SiO_2	19.90
Al_2O_3	5.60
Fe_2O_3	2.90
MgO	1.50
Na_2O and K_2O	0.92
SO_3	2.30

Table 2. Physical Properties and Sieve Analysis of the Fine Aggregate

Physical Properties of the Fine Aggregate		
Test type		Results
Specific Gravity		2.32
Moisture Content (%)		1.18
Bulk Density (kg/m^3)		1611.32
Particle Size Distribution		
BS Sieve	Mass Retained (%)	% Passing
5 mm	-	
3.35 mm	0	100
2 mm	42.2	91.76
1.18 mm	60.9	79.58
600 µm	230	33.59
425 µm	87.7	16.06
300 µm	29.7	10.12
212	32.9	3.54
150	12.9	0.96
75	3.5	0.26
Pan	1.3	0.00
Total	500	

The Moringa Oleifera seed used for this work was sourced from the local markets in Bauchi State, Nigeria. Bauchi State is in the Sahel region of the Northern part of Nigeria, and characterized by extreme weathers. The seeds were grinded and sieved using sieve size 212 µm to obtain the desired fineness. The physical characteristics of the powder had a moisture content of 8.33%, bulk density, 8.42 g-cm^3, and a specific gravity of 0.28. The chemical compositions of the MOSP were determined by X-ray fluorescence (XRF) according to NF P15-467 [7], and the results are shown in Table 4. Figure 1 shows the x-ray diffraction of the MOSP.

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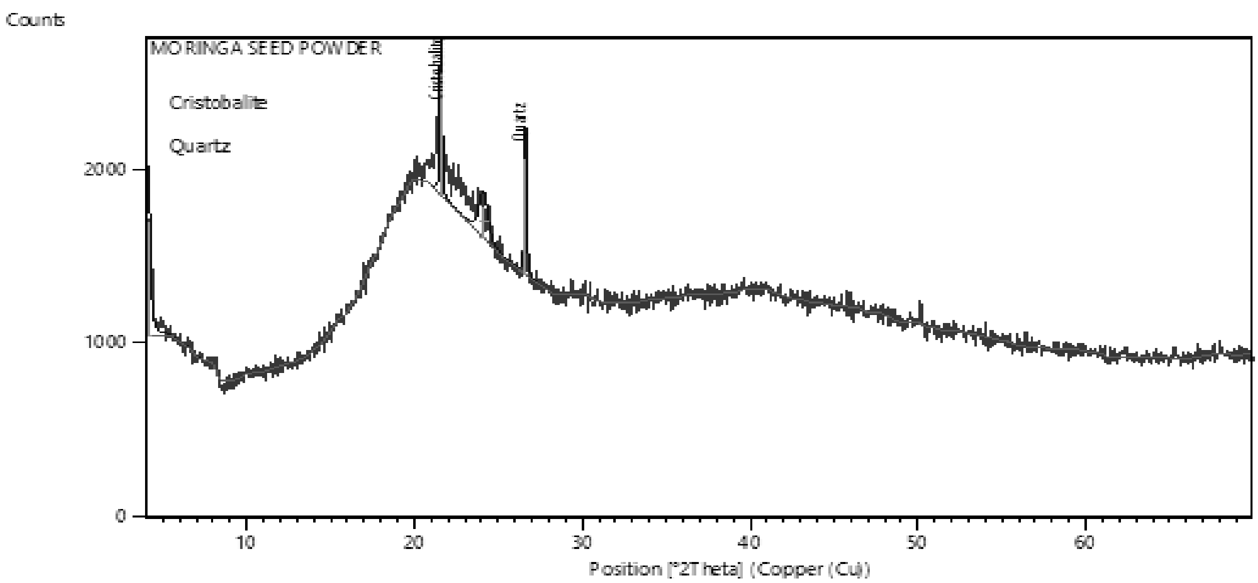
Table 3. Physical Properties and Sieve Analysis of Coarse Aggregates

Physical Properties of the Coarse Aggregate		
Test type		Results
Specific Gravity		2.74
Moisture Content (%)		0.15
Bulk Density (kg/m^3)		1611.32
Aggregate Impact Value (AIV)		12.85
Aggregate Crushing Value (ACV)		26.85
Particle Size Distribution		
BS Sieve (mm)	Mass Retained (%)	% Passing
37.5	-	
28	0	100.00
20	364.3	87.86
14	2159.2	15.89
10	390.8	2.86
6.3	75.1	0.36
5	9.5	0.04
3.35	1.1	0.00
Pan	0.1	0.00
Total	3000	

Table 4. Oxide composition of Moringa seed

Oxide	Percentage by weight
SO_3	28.3
P_2O_5	8.2
CaO	7.8
K_2O	7.5
SiO_2	5.8
MgO	2.1
Nb_2O_5	0.03
ZnO	2.0
Na_2O	1.4
TiO_2	0.1
MnO	0.5
Al_2O_3	6.2
NiO	3.8
Cr_2O_3	3.1
Fe_2O_3	17.6
Ag_2O	0.03



Pos.[°2Th.]	Height [cts]	FWHMLeft	[°2Th.] d-spacing [Å]	Rel. Int. [%]
4.1191	650.54	0.3070	21.45196	92.21
21.5700	705.51	0.1279	4.11992	100.00
24.1064	182.77	0.6140	3.69188	25.91
26.6326	673.4	0.1279	3.34714	95.45

Pattern List

Visible Ref.Code	Score	Compound Name	Displ.[°2Th]	Scale Fac.	Chem. Formula
*96-900-8233	35	Cristobalite	0.000	0.624	Si8.00 O16.00
*96-900-9667	23	Quartz	0.000	0.819	Si3.00 O6.00

Figure 1. Details on the Moringa Oleifera Seed Powder

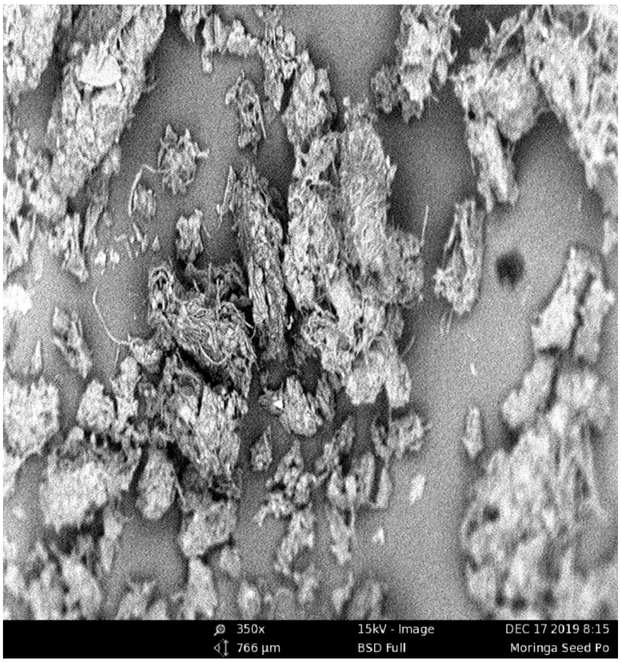


Figure 2. Microstructure of MOSP

3. Experiment

The experiments carried out were on the paste and concrete samples made using moringa oleifera seed powder in various proportions of 0.1% to 5.0% by wt. % to replace cement. The MOSP-paste tests were on the consistency and setting times of the paste. These were tested in accordance with BS EN 196-3 [8] using 300 g of cement with the MOSP in proportions of 0%, 0.2%, 0.5%, 2%, 3%, and 5% by wt. % of cement, and mixing kept within the stipulated 5 minutes. The results are shown in Table 5.

Table 5. Consistency and Setting Time of MOSP-Paste

Mix No	Consistency (%)	Initial setting time (Mins)	Final setting time (Mins)
M-0.0 MSP	34	105	253
M-0.2 MSP	34	118	274
M-0.5 MSP	34	121	293
M-1.0 MSP	34	142	368
M-2.0 MSP	35	179	457
M-3.0 MSP	37	203	552
M-5.0 MSP	38	365	703

While the MOSP-concrete was on the slump, density, water absorption, and compressive strength. The studies on the MOSP-concrete were done using a concrete mix of 1: 1.7: 2.5 with a cement content of 420 kg-m^3, fine and coarse aggregates of 708 kg-m^3 and 1062 kg-m^3, respectively, and a water-cementitious ratio of 0.5. The slump of the MOSP-concrete was in the fresh condition, and carried out in accordance with BS EN 12350-2 [9], using a truncated cone of 300 mm high with the bottom and top diameters as 200 mm and 100 mm, respectively. This was studied using replacement levels of MOSP by wt. % of cement from 0% to 5%. The results are shown in Table 6.

The density, water absorption and compressive strength were conducted in accordance with BS 1881 Part 122 [10], BS EN 12390-7 [11], and BS EN 12390-3 [12], respectively, The cube compressive strength was studied using a cube mold of 100 mm, and a total of seven (7) mixes were used and designated as 0.0% MOSPC, 0.2% MOSPC, 0.5% MOSPC, 1.0% MOSPC, 2.0% MOSPC, 3.0% MOSPC, and 5.0% MOSPC. The 0.0% MOSPC was the control, containing 0 % MOSP, while other mixes contained MOSP in proportions of the cement by wt. %. The total number of cube samples cast and cured from 3 days to 90 days was 105, three for each age, and at the end of each curing regime, three of the specimens were tested to failure and the average recorded. The compressive strength testing was done using the ELE Compression Machine with 2000 kN capacity and tested in accordance with 12390-3 [12]. The results on the slump, density, water absorption and compressive strength are shown in Tables 6 and 7.

Table 6. Slump of MOSP-Concrete

Mix No	Slump
M-0.0/ MOSPP	23
M-0.2 /MOSPP	26
M-0.5/ MOSPP	26
M-1.0 /MOSPP	29
M-2.0 /MOSPP	35
M-3.0/ MOSPP	45
M-5.0/ MOSPP	48

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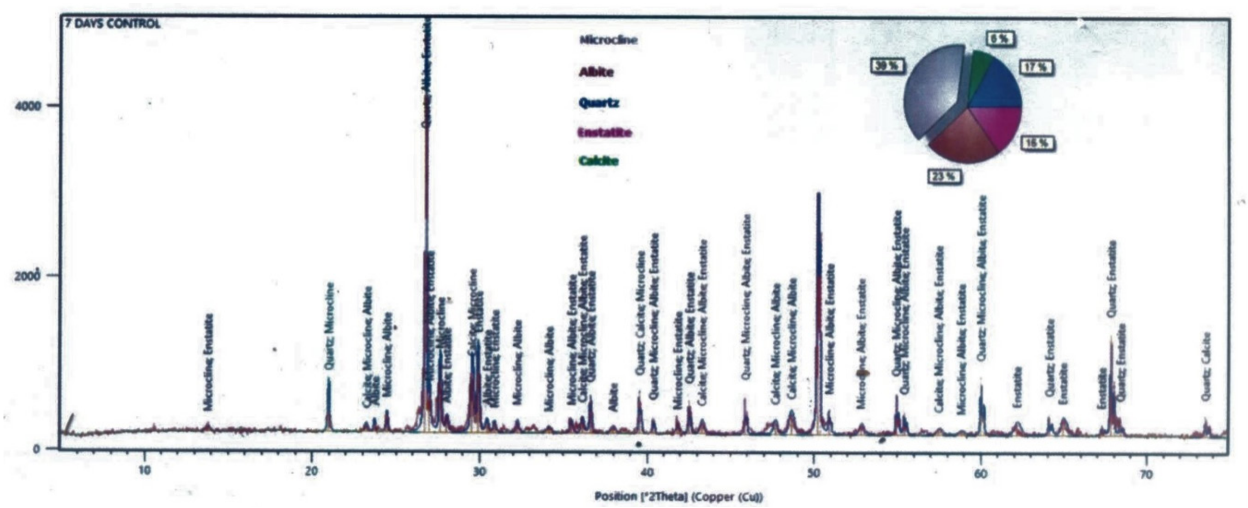
Table 7. Characteristics of MOSP-Concrete

Property	Mix No	Age (Days)				
		3	7	28	60	90
Water Absorption (%)	M-0.0/ MOSPC	0.7	1.0	1.5	1.8	2.3
	M-0.2/ MOSPC	0.8	1.1	1.6	1.9	3.0
	M-0.5 /MOSPC	0.8	1.1	1.7	2.2	3.7
	M-1.0/ MOSPC	0.9	1.2	1.8	2.6	4.3
	M-2,0/ MOSPC	1.0	1.8	2.0	2.7	4.7
	M-3.0/ MOSPC	1.2	1.6	2.1	2.8	5.1
	M-5.O/ MOSPC	1.4	2.0	2.2	3.2	6.0
Density(kg/m^3)	M-0.0 /MOSPC	2583	2613	2637	2607	2590
	M-0.2 /MOSPC	2650	2567	2587	2513	2527
	M-0.5 /MOSPC	2510	2667	2587	2617	2590
	M-1.0 /MOSPC	2590	2650	2610	2553	2547
	M-2,0 /MOSPC	2627	2597	2590.	2523.	2543.
	M-3.0 /MOSPC	2587	2583	2597	2530	2533
	M-5.O /MOSPC	2510	2503	2513	2513	2507
Compressive Strength (kN/m^2)	M-0.0 /MOSPC	14.0	18.0	20.7	22.0	24.0
	M-0.2 /MOSPC	17.0	19.9	25.3	27,0	27.1
	M-0.5 /MOSPC	14.4	16.3	24.0	25.3	23.9
	M-1.0 /MOSPC	12.5	15.0	18.4	19.5	17.6
	M-2,0 /MOSPC	12.2	14.7	17.9	18.9	17.5
	M-3.0 /MOSPC	11.7	14.5	16.1	17.6	14.8
	M-5.O /MOSPC	0.50	0.60	3.9	5.6	4.9

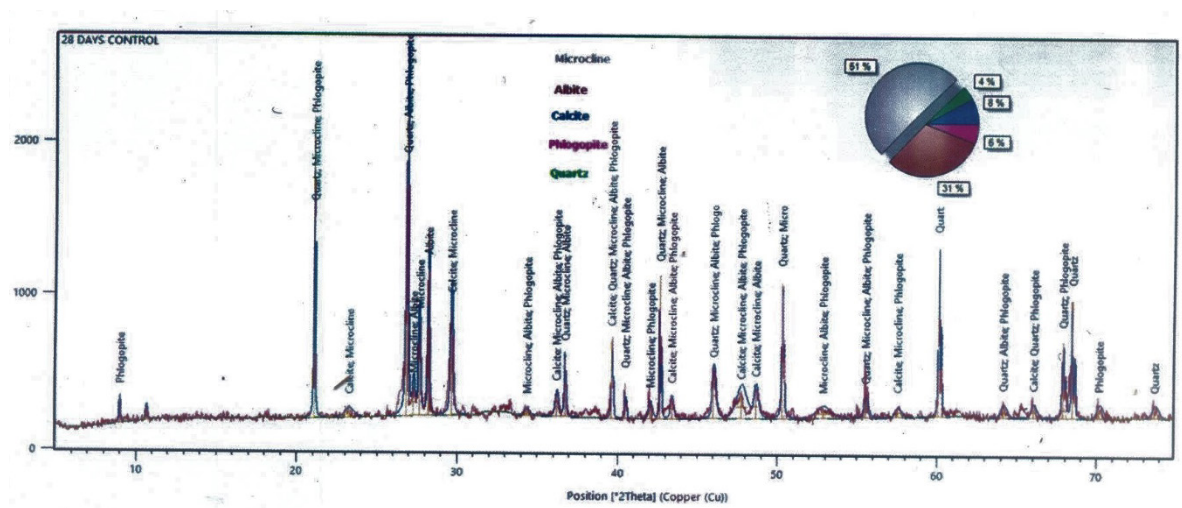
X-ray diffraction characterization of moringa oleifera seed powder and concrete

The second phase was the x-ray diffraction analysis on crushed cube compressive strength of the MOSP-concrete samples used for the investigation. This was taken at the replacement that gave the optimum strength, that was at 0.2% by wt. % of cement and cured from 3 to 90 days. These were matched with the control samples containing 0.0% MOSPC, and cured for the same period of days. The moringa oleifera seed was grinded into powder and sieved using sieve size 150μm. XRD is a nondestructive technique that provides detailed information about the crystallographic structure, chemical composition, and physical properties of materials. A Brunker-AXS D8 advanced equipment was used for the x-ray diffraction analysis. This was based on Bragg's law (nλ = 2dsinλ), and recorded on an X-ray diffractometer operating at known voltages and current using a Cu Kα X-rays (α = 0.15406 nm) over the 2θ range from 10 to 100 degrees in the steps of 0.01 degree at room temperature in open quartz sample holders. The results are shown in Figures 3 and 4. The characteristics of x-ray diffraction peaks are shown in Figure 5.

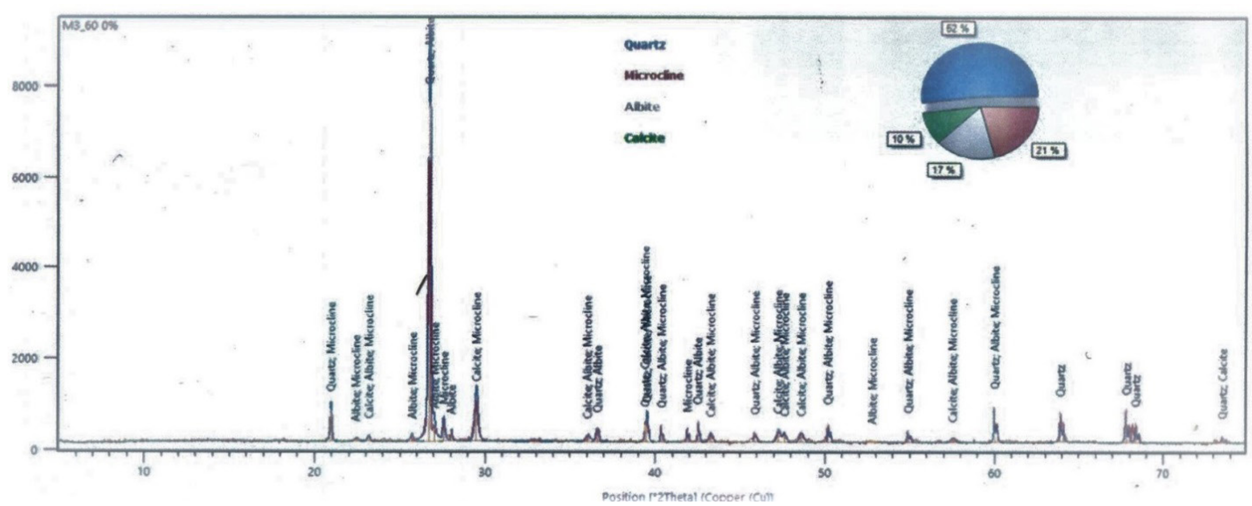
X-ray diffractogm of moringa oleifera seed powder Concrete



7 days



28 days



60 days

Figure 3. Control Samples of XRD MOSPC

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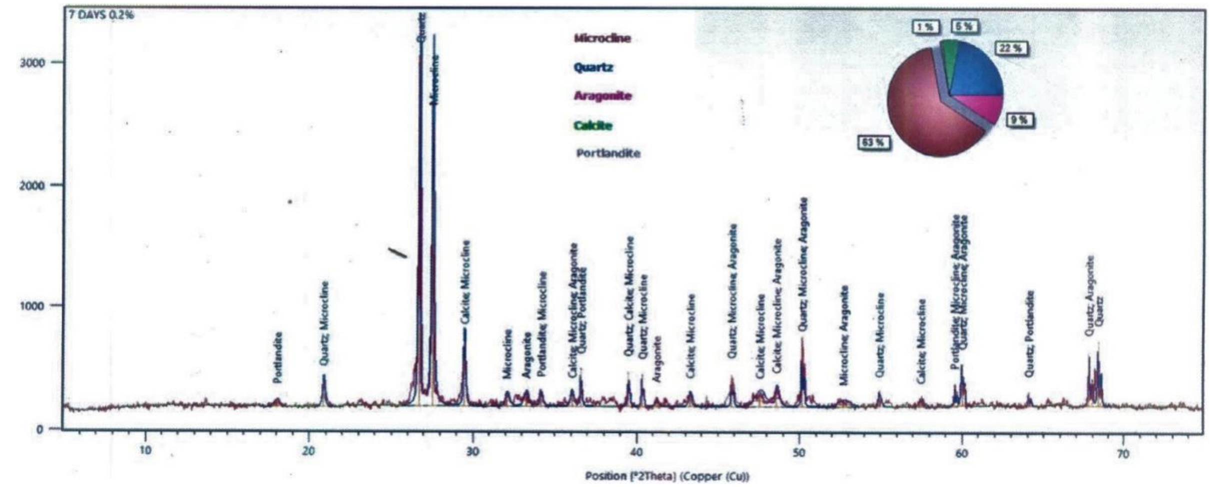
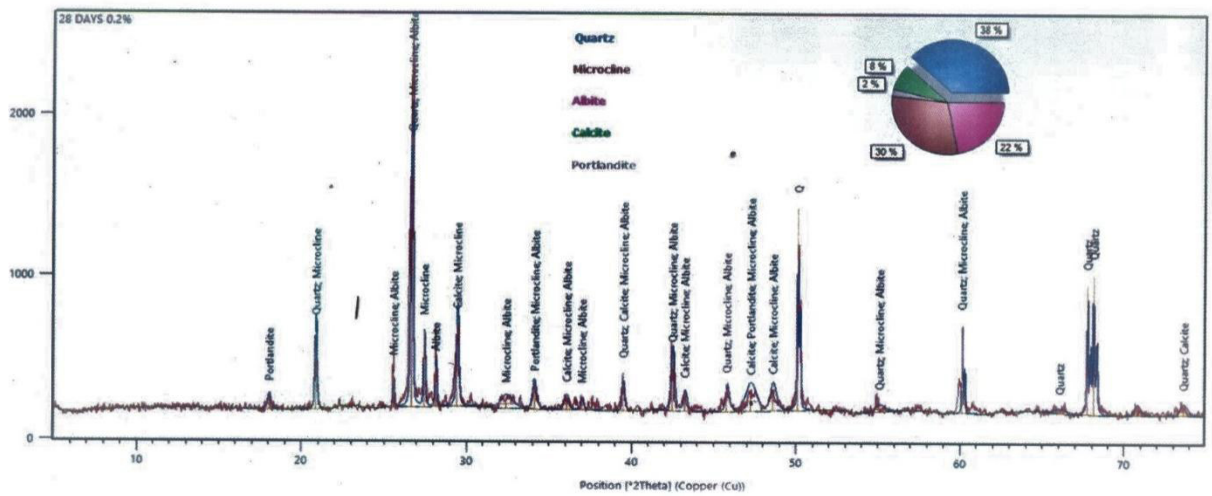
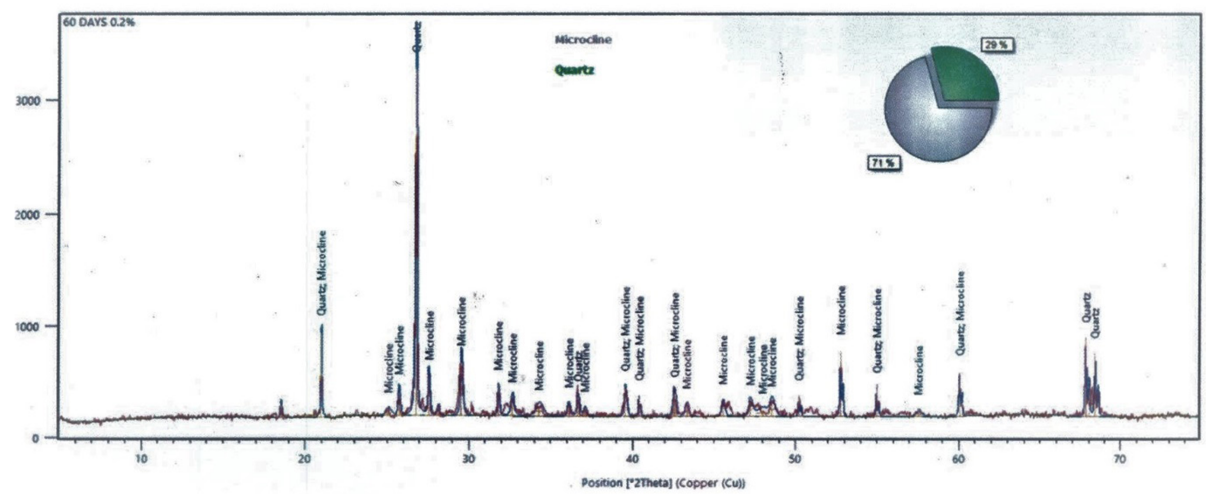


7 days



28 days



60 days

Figure 4. 0.2% Addition to Samples of XRD MOSPC

Microstructure and Acid-Base Resistance of MOSP-Concrete

The third phase was experimental set-up for the study on the microstructure and acid and sulphate resistance of MOSP-concrete. The samples for this phase were taken from the crushed samples on the cube compressive strengths at the optimum replacement of cement by wt. % which was at 0.2% MOSP for ages of 3 to 90 days.

The study on the microstructure was carried out using scanning electron microscope (SEM-Hitachi S4100) equipped with energy dispersion spectroscopy, EDS-Rontec), at 5kV and 25 kV. Thin sections mounted on glass slides were prepared from the crushed samples, and observed using the SEM. The micrographs are shown in Figure 5. For the study on the acid- sulphate resistance, the acids and sulphates were 20% dilutions. The media used were H_2SO_4, and HNO_3 for the acids, and Na_2SO_4 and $MgSO_4$, for the sulphates. The procedure was weighing a selected crushed sample, weighed and recorded as initial weight (W_1), then immersed in the selected medium for periods of 56 days at 7 days interval, weighed and recorded as the final weight (W_2). At the end of each curing regime, it was removed, rinsed by sprinkling a little water, and allowed for some seconds to dry. The sample was weighed and the weight recorded. An average of two measurements was recorded and the percentage wt. loss was calculated as:

$$Wt.\ loss\ (\%) = \frac{W_1 - W_2}{W_1}\ x\ 100 \qquad 1$$

Where: W_1 and W_2 are the initial and final weights of the sample. The results are shown Tables 8 and 9.

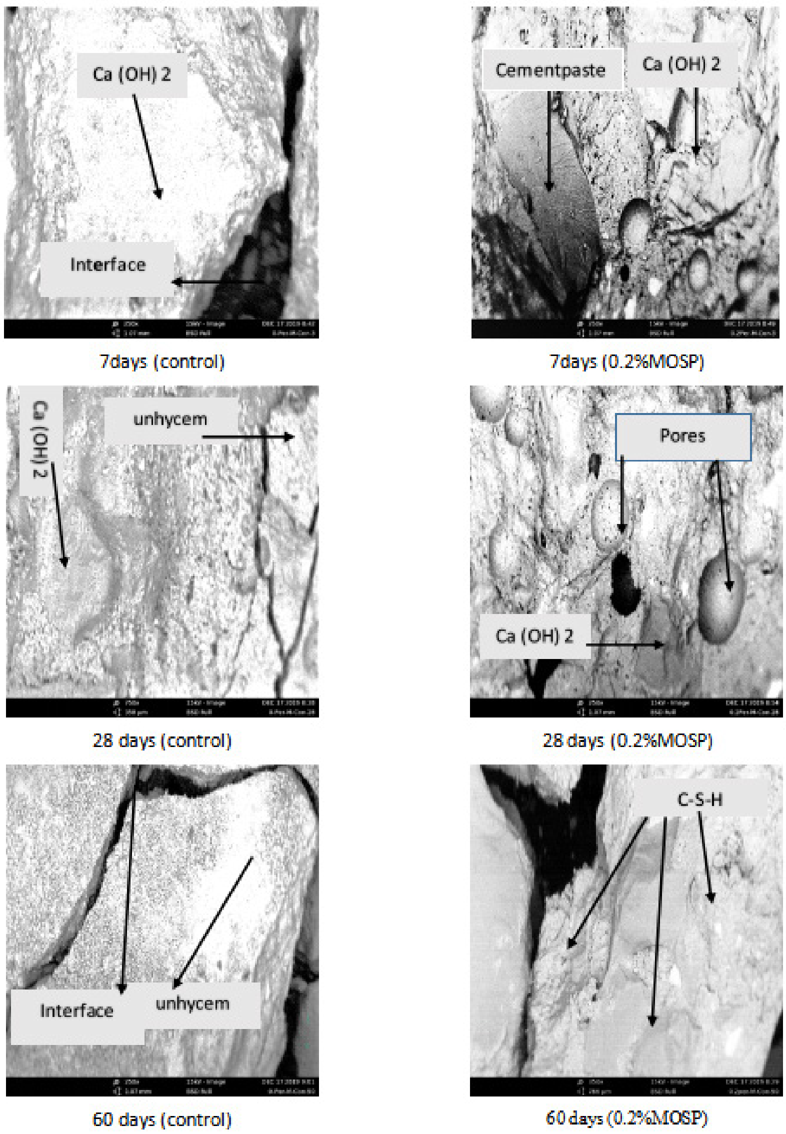


Figure 5. Micrograph of MOSP-Concrete

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Table 8. Acid Resistance of MOSP-Concrete

Age (Days)	Mix	Medium	Percentage Decay (%)							
			7 d	14 d	21 d	28 d	35 d	42 d	49 d	56 d
3	0.0% MSOP	20 % HNO_3	-8.3	-7.5	-26.2	-9.7	-21.6	-9.9	-8.5	-18.5
7			-14.5	-6.0	-6.6	-10.6	-6.0	-8.2	-10.2	-13.6
28			-20.1	-9.0	-15.5	-29.8	-16.7	-10.2	-18.1	-10.6
60			-17.3	-15.7	-4.8	-5.5	-10.3	-18.6	-16.8	-18.4
90			-13.7	-10.9	-24.0	-3.0	-15.2	-16.8	-11.8	-18.4
3	0.2% MOSP		-29.8	-14.3	-9.5	-13.6	-24.6	-15.9	-7.2	-15.8
7			-10.2	-4.8	-11.5	-10.4	-4.2	-10.7	-6.9	-13.9
28			-27.5	-12.7	-8.0	-6.8	-12.1	-8.8	-20.6	-16.0
60			-12.4	-8.2	-10.4	-4.9	-3.7	-20.5	-16.1	-26.4
90			-13.0	-12.5	-3.4	-4.9	-1.6	-17.1	-13.2	-21.6
3	0.0 % MOSP	20 % H_2SO_4	-66.5	-34.0	-28.9	-36.6	-49.5	-57.6	-44.0	-41.7
7			-26.6	-48.0	-50.0	-46.1	-36.1	-38.8	-26.5	-37.4
28			-50.5	-64.0	-35.0	-37.6	-21.4	-52.0	-55.3	-1.3
60			-49.5	-56.2	-46.9	-62.1	-28.7	-49.6	-49.0	-42.2
90			-32.4	-12.2	-19.6	-52.7	-36.8	-41.6	-40.8	50.0
3	0.2% MOSP		-60.3	-59.9	-37.8	-29.5	-23.3	-30.6	-27.8	-34.0
7			-39.8	-55.5	-52.7	-34.5	-35.4	-23.3	-27.1	25.2
28			-52.3	-48.7	-61.7	-26.6	-46.6	-29.5	-27.7	22.6
60			-61.5	-53.3	-68.2	-24.0	-38.8	-31.7	-32.1	-26.3
90			-67.2	-58.8	-63.9	-9.6	-28.6	27.6	-40.7	-20.1

Table 9. Base Resistance of MOSP-Concrete

Age (days)	Mix	Medium	Percentage Growth ((%)							
			7d	14d	21d	28d	35d	42d	49d	56d
3	0.0 % MOSP	20 % Na2SO_4	3.6	11.0	5.9	6.9	16.9	5.1	2.7	4.7
7			5.0	8.6	6.3	8.1	5.1	6.1	21.8	39.6
28			9.0	3.8	18.1	13.9	12.2	7.4	14.7	10.6
60			0.6	6.3	10.5	4.3	9.9	7.9	2.4	8.8
90			3.2	7.6	4.1	18.4	7.8	6.8	14.6	1.7
3	0.2 % MOSP		7.0	3.8	7.2	3.3	6.4	7.5	9.4	5.2
7			7.8	3.5	8.4	3.0	6.1	9.2	8.9	5.8
28			5.9	4.3	5.4	3.8	3.5	12.0	8.89	9.3
60			5.4	8.2	4.3	2.6	7.0	9.1	7.6	5.2
90			5.7	8.0	9.0	2.0	4.3	6.9	9.1	17.1
3	0.0 % MOSP	20 % $MgSO_4$	4.2	4.6	12.6	8.3	7.5	22.0	2.7	7.7
7			5.3	6.9	2.4	5.6	6.47	6.9	6.5	14.6
28			9.5	4.7	9.2	6.5	10.0	18.7	7.5	7.7
60			7.2	8.1	7.7	5.7	8.5	5.0	8.2	10.3
90			4.8	6.0	17.7	9.6	10.1	7.7	7.5	3.7
3	0.2 % MOSP		9.4	5.2	6.7	4.9	6.0	9.1	8.5	3.3
7			5.2	3.8	8.9	6.3	8.5	8.5	10.3	3.5
28			6.7	4.9	8.0	4.5	9.5	6.8	12.2	7.5
60			9.6	6.3	3.6	4.9	10.7	30.9	12.3	7.4
90			7.2	3.3	7.5	5.8	6.7	12.7	5.8	6.4

4. Discussion

Characteristics of MOSP

The major oxides in cement include CaO, SiO_2, Al_2O_3, and Fe_2O_3, while the minor oxides are SO_3, MgO, Na_2O and K_2O. Table 4 showed two outstanding and distinct chemical compounds in the chemical composition of MOSP. These are SO_3 (~28%) and Fe_2O_3 (~18%), respectively, which may primarily dictate the performance of MOSP as a composite material with concrete. The other relatively high elements are P_2O_5 (8%), CaO (8%), Al_2O_3 (6%), and SiO_2 (6%) respectively. SO_3 and TiO_2 with pH, approximately 7, are acidic oxides. They are therefore, corrosive to metals and also react with the alkalis [13]. The permissible level of SO_3 is in the range of 1-3% as against the 28% from Table 4. Horkoss et al studies were on the effects of SO_3 on the expansion of mortars, and showed that the cement produced with high sulphur clinker had lower expansion. He also observed that if the percentage surpassed the level of 3.5% it could increase the risk of DEF [14]. Therefore, the SO_3 content of 28.31% in MOSP, which exceeded the permitted range of (1-3)% must be of great interest as MOSP is proposed for use in concrete. Equally of concern are the percentage presence of K_2O (7.53% and Na_2O (1.40%), collectively called the alkalis, specified not to be greater than 0.4-1.3%. The alkalis react with active silica in aggregate to produce alkali-silica gel responsible for swelling under favorable conditions of moisture and temperature in voids and cracks [15]. With the works of Horkoss et al [16] SO_3 high content may not be a disadvantage but an advantage as it causes strength development to be slow (Retarder). Retarders are advised for hot weather concreting. High content of SO_3 from their works confirmed that it could form a resistance to sulphate attack. Again, the excess of phosphorous content of 2.0% will reduce C_3S content, and slow the hydration reaction, thereby, prolonging the induction [17].

Figures 1 and 2 show the diffractogramme and the microstructture of MOSP. Yhe XRD peaks and the pattern are shown below the diffractogramme. The peaks show that MOSP is not crystalline in nature but amorphous. The dominant minerals are cristobalite and quartz. Cristobalite (Si_8O_{16}) and quartz (Si_3O_6) are polymorphs of SiO_2. The solubility of silica minerals is a function of chemical composition, and the presence of a disrupted surface layer [18]. Quartz dictated in the sample is a hard, crystalline mineral composed as silica (silicon dioxide), whose atoms are linked in a continuous framework of SiO_4 silcon-oxygen tetrahedral, with each oxygen being shared between two tetrahedral. This gives an overall chemical formula of SiO_2. The four peaks are as shown.

Characteristics of MOSP-Paste and Slump (Fresh Concrete)

Figure 6 shows the plots of consistency of MOSP cement paste. Consistency is the water affinity of the material as it goes into reaction in the cement paste (the degree of wetness). The result showed that at replacement levels of 0% to 1.0%, the water requirement remained approximately constant at 34%. But beyond the replacement of 1.0% the water requirement continued to increase, and the increase recorded were 2.9%, 8.8%

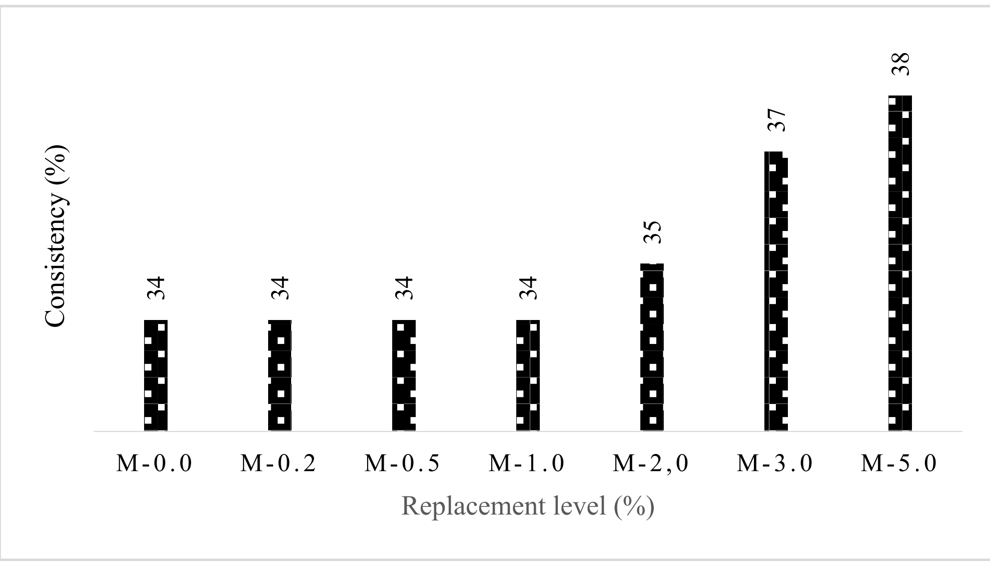

Figure 6. Consistency of MOSP-Paste

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and 11.8% to the control. Above 1.0% replacement the possible explanation meanwhile could be attributed to the harshness of the paste, and therefore, for nonporous concrete the water requirement should not be greater than 34% and cement replacement with MOSP by wt. % should not be > 1.0%.

Figure 7 is the setting times of the MOSP cement paste. Setting properties of concrete is the most important in the field of concrete works because it helps in the various concrete operations. The physical properties of MOSP morphologically are hollowed granule and armophous. It has been reported that such physical properties caused the powder become pasta if added with certain amount of water [19], and also tends to form agglomerate morphologically [20]. The main cause of cement setting is the onset of the formation of C-S-H, and defined as the mechanism that governs the transition from plastic phase to hardened one, characterized by an increase in the system viscosity [14]. Figure 6 showed a steady increase both for the initial and final setting times. The final setting time for replacements up to 3.0% of cement by MOSP did not exceed the 10 hours stipulated by the code. Above 3.0% the stipulated time of 10hrs was exceeded. We can therefore conclude that it may be safe to use MOSP for concrete to a level of replacement not exceeding 3.0% wt. % of cement. Although, the 3.0% by wt. % replacement may seem to have satisfied the setting time of 10 hrs stipulated. It is however safer to limit the replacements to between 0.2% and 0.5% because of the SO_3 content pending further works with the material. This

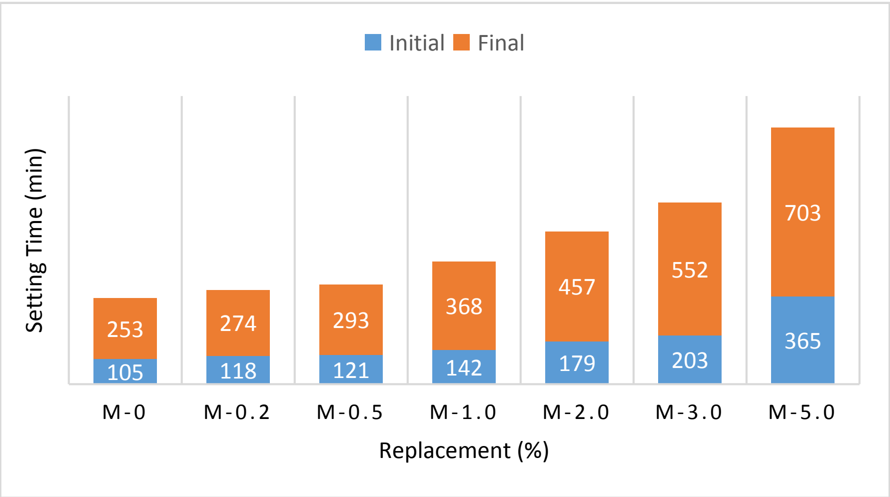

Figure 7. Setting Times of MOSP Cement Paste

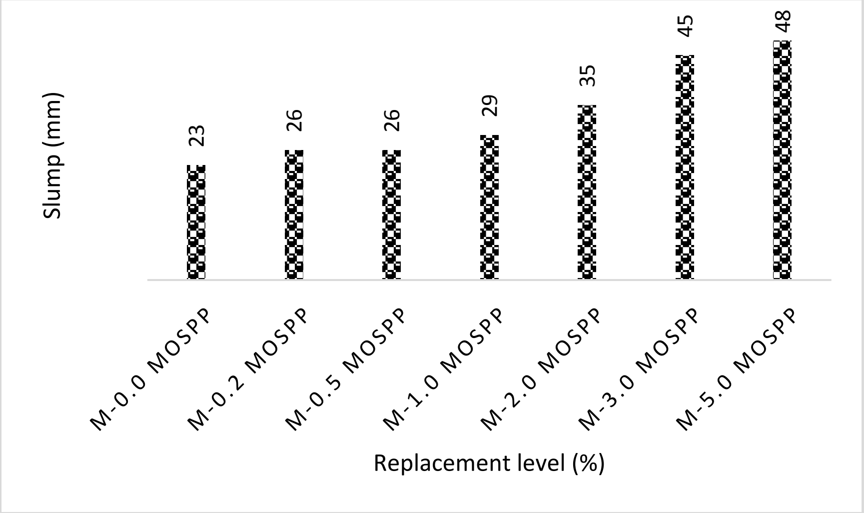

Figure 8. Slump of MOSP-Concrete

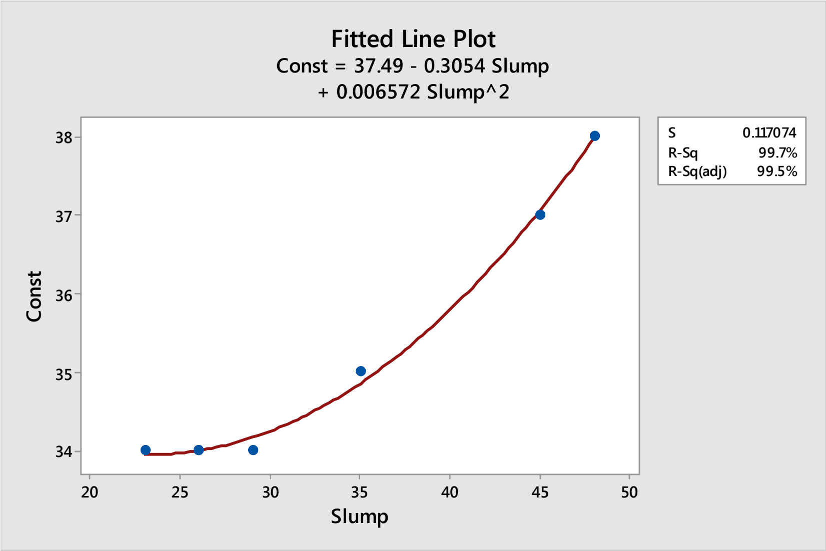

Figure 9. Consistency against the Slump

decision is based on suggestions of some researchers on the regulation of gypsum content in cementitious systems because of cement strength and its dimensional stability. They believed it may affect durability of cement concrete and thereby, cause serious damages [15]. High content of gypsum in cement was also considered as one of the main causes of DEF, and therefore limiting sulfate additions in cement is common to avoid DEF. This was also supported by the findings of Sayed Horkoss et al [16], who held a contrary view on cement produced with high sulfur clinker. They concluded that it gave a lower expansion to other types of gypsum. Therefore, in the applications of MOSP in cement concrete, we must be cautious because of the high SO_3 content. This work proposes to keep the replacement levels of MOSP at 0.2% and 0.5%, because of the SO_3 content, pending further works with this material. MOSP can also be used for hot weather concreting where enough time is needed to place the concrete due high temperature.

The slump of the MOSP concrete is shown in Figure 8 which is an expression of the workability of the concrete. The workability depends on the size and amount of the fine and coarse aggregates. The results showed that the use of MOSP in concrete will improve the workability of the concrete. An empirical relationship has been established between the consistency of MOSP-paste and the slump. This is given as Consistency = 37.49 +0.31slump + 0.001 slump2 with a standard deviation (s) of 0.1171, and a correlation factor of 99.7%. This is shown in Figure 9.

Mechanical strength of MOSP-concrete

The water absorption results showed increase as the replacement levels and age increased (Table 7). The replacement level of 0.2% gave the best behavior and thus could be said to be the optimum replacement level for best performance (Figure 10). This can also alley the fears raised on the setting times, limiting replacements from 0.2% to 0.3% by wt. % of cement. We can therefore conclude that the use of MOSP in concrete increased the water absorption. The differences in water absorption at 28 days and 90 days of curing were 2% to 41%, and 31% to 158%, respectively (Figure 11). The density of MOSP-concrete is a normal weight concrete as shown in Figure 12, and can achieve a value up to 2667 kg-m^3. The high value of the density could be attributed to the density of the MOSP which is approximately twice the density of cement.

From Figure 13, the compressive strength of MOSP-concrete increased as the replacements of cement by wt. % of MOSP were increased. The results showed that the compressive strength increased as MOSP by wt. % is increased for all ages of concrete specimen, up to 0.2% and 0.5%, with the best performance at 0.2%. This optimum was also achieved by Susilorini et al [2,3]. Although, it is observed that the compressive strength increased with age of curing above 0.5% replacement, however, increasing MOSP content decreased the compressive strength. The desired design strength of 20 kN-m^2 was satisfied by 0.2% and 0.5% replacements, respectively, that for 0.2% was achieved at the ages of 7 days (~20.0 kn-m^2), 28 days (25 kN-m^2), 60 days (27 kN-m^2), and 90 days (27 kN-m^2), while that for the 0.5 % was achieved at ages of 28 days (24 kN-m^2), 60 days (25 kN-m^2), and 90 days (~24 kN-m^2), respectively. These classes of concrete could be used for reinforced concrete members. For higher replacements of 1%, 2%, and 3%, they could not attain the desired strength of 20 kN-m^2,

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but could equally be used for non-load bearing members such as lintels, foundation concrete, etc. The percentage strength differences are shown in Figure 14. The SO_3 levels at 0.2% and 0.5% MOSP can be said to favor better production of hydrate generators of bonding properties which led to the increase in compressive strength. The decrease of resistance beyond the levels of 0.2% and 0.5% confirmed that there is an optimum content above which the strength gradually decreased.

X-Ray Diffraction Analysis of MOSP-Concrete

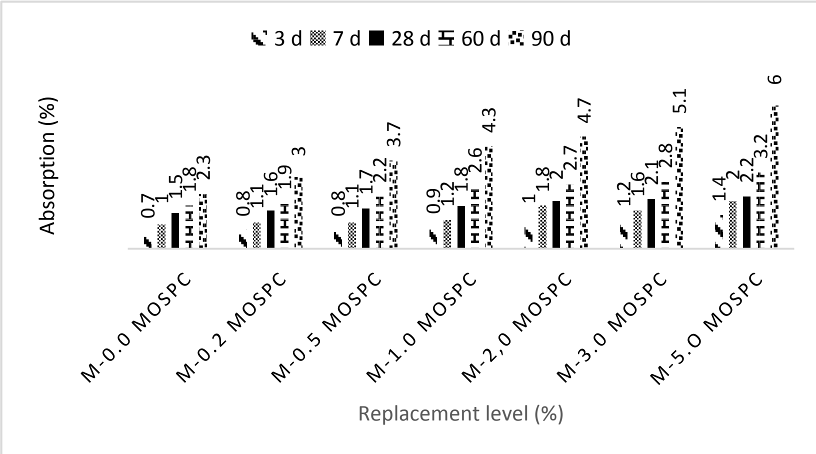

Figure 10. Water Absorption of MOSP-Concrete

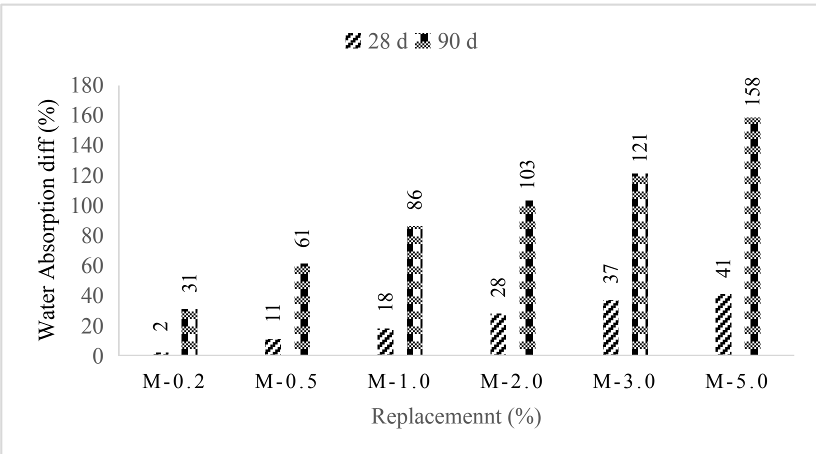

Figure 11. Water Absorption Difference (%)

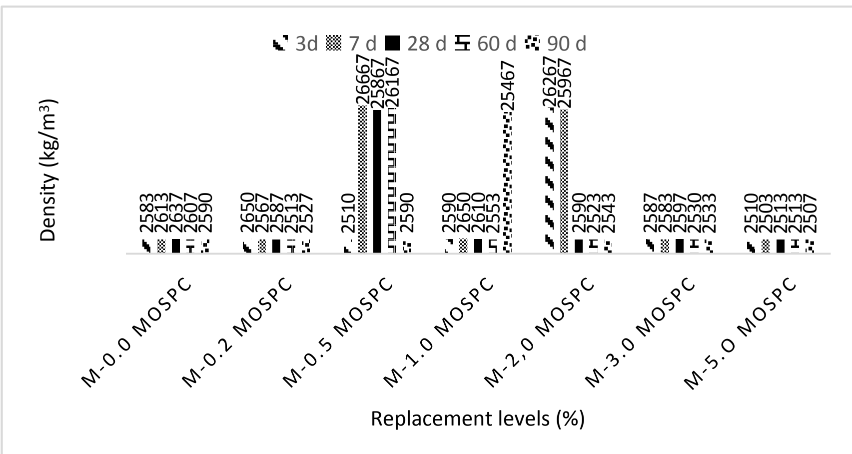

Figure 12. Density of MOSP-Concrete

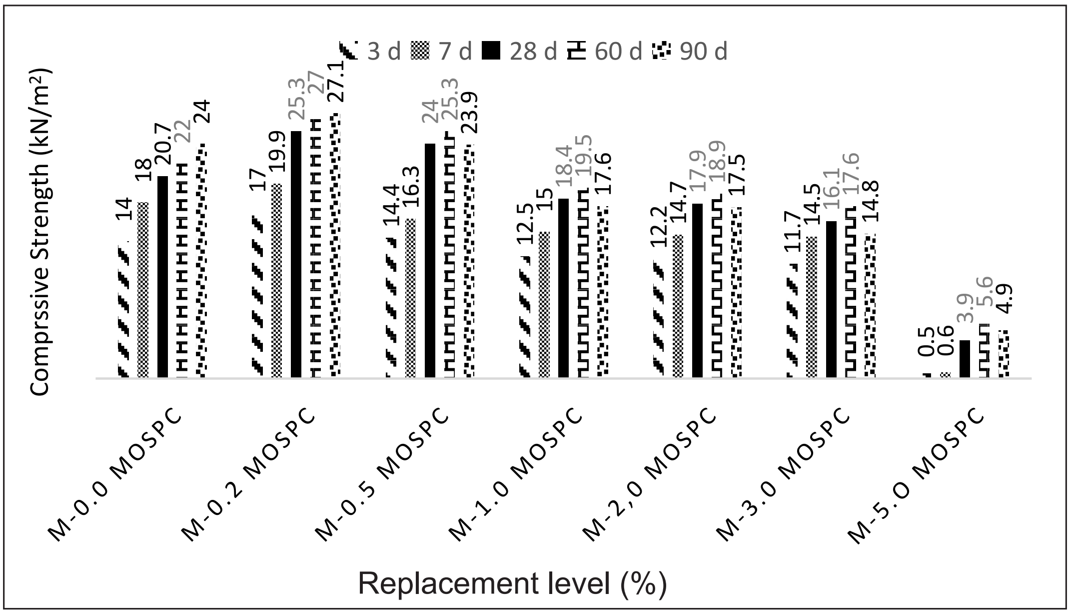

Figure 13. Compressive Strength of MOSP-Concrete

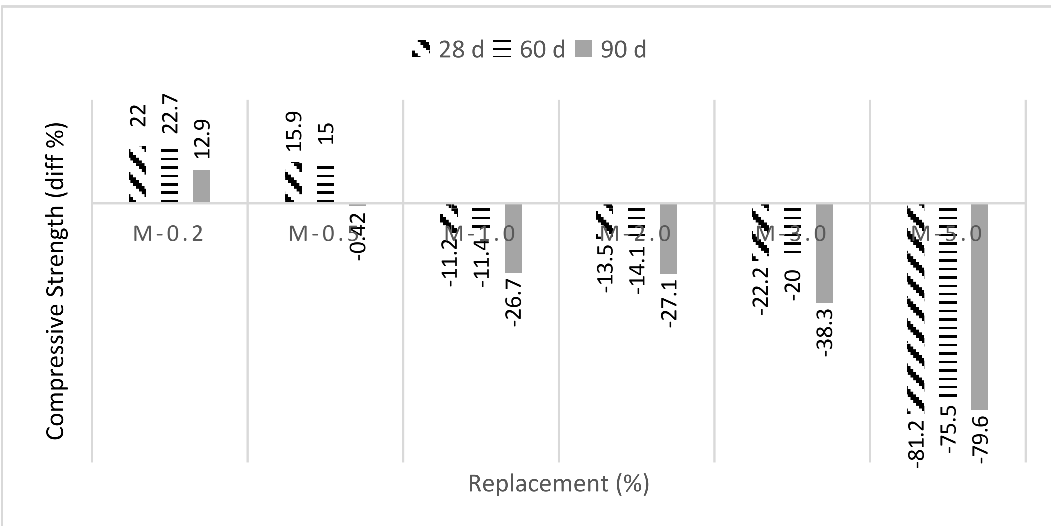

Figure 14. Compressive Strength Difference (%)

Figure 15 is information extracted from the x-ray diffractogrammes shown in Figures 3 and 4. It show that the quartz and microcline minerals are dictated at all ages of the samples (Control and 0.2% MOSP) but having various percentages. The two dominant minerals remaining at the end of the hydration process at the age of 60 days, for the 0.2% MOSP samples, were quartz and microcline. The percentage increase at 0.2% MOSP for quartz and microcline were approximately 37% and 43% respectively. While the percentage of quartz continued to increase, microcline remained constant above 28 days. Another dominant minerals are calcite and albite. Calcite is a polymorphy of calcium cabornate ($CaCO_3$). However, both disappeared at 28 days of curing. While albite was increasing, calcite decreased. The percentage increase and decrease with the control were 29% and 30% respectively. The presence of Portlandite at 7 and 28 days, and Araganite at 7 days, in the 0.2% MOSP has no explanation meanwhile. Enstaite and phlogophite dictated in the control samples were not found in the 0.2% MOSP samples.

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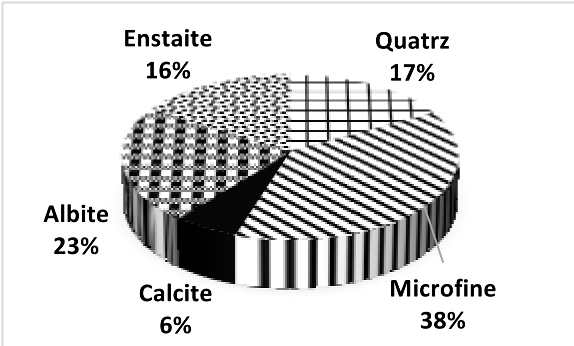
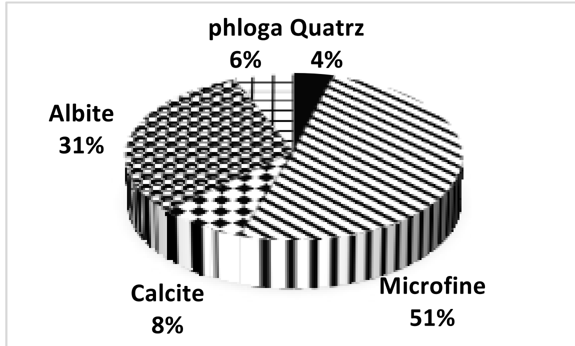
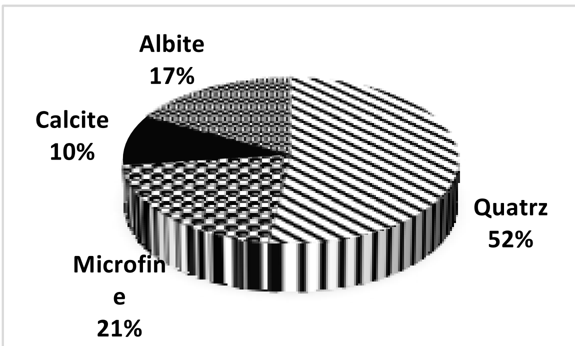


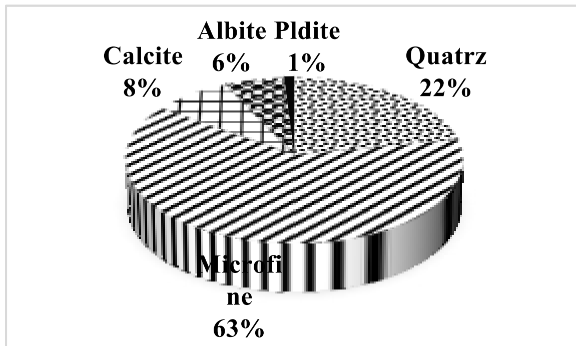
Seven Days-Control (0%MOSP)



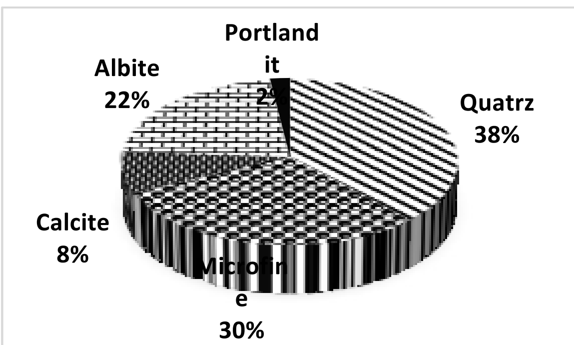
Twenty Eight-Day Control (0% MOSP)



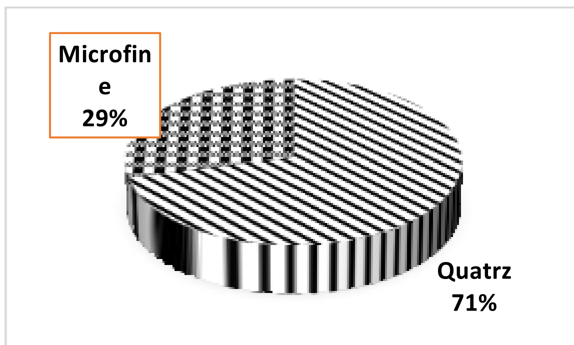
Sixty-Day Control (0% MOSP)



Seven-Day (0.2% MOSP)



Twenty Eight-Day (0.2% MOSP)



Sixty-Day (0.2 % MOSP)

Figure 15. Characteristics of x-ray diffraction peaks of MOSP-concrete

Microstructure Characteristics from the Micrograph of MOSP-Concrete

Figures 5 and 16 show the SEM of the MOSP-concrete and their mineral characteristics at the ages of 7, 28 and 60 days, respectively. Affecting factor for the effectivity of MOSP as an additive is the active compounds of the powder. When used as an additive or replacement material, the active compounds are dissolved in water and eventually go through hydration processes, modifying the end products (Figure 5). The size (212 μm) and physical properties of MOSP affect the amount of dissolved active compounds in water. Other studies about SEM result of MOSP indicated that the powder tends to form agglomerate morphologically [19].

At seven days of curing quartz, microfine, calcite and albite were dictated for both the control (0%) mix and the mix containing 0.2% MOSP but at various magnitude. However, enstaite and portlandite were peculiar as the case may be, to the control mix and the 0.2% mix, respectively. The view from Figure 5 showed agglomerate effects on the mix with 0.2%. The 28-day mineral characteristics showed that quartz, microfine, calcite, and

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albite were dictated. However, quartz and albite contents increased, while microfine decreased. These were opposite to the observed effects at 7-days. At 60 days only quartz and microfine were dictated, while calcite and albite had totally disappeared. The interpretation of this from Figure 5 showed that $Ca(OH)_2$ and the bubbles (porosity) noticed at 28 days have all disappeared and formed C-S-H responsible for strength. From understanding of hydration reactions of cement the predominant phases present in hardened concrete are monosulphate, Tobermorite (C-S-H) and portlandite, along with unreacted cement compounds and ettringite.

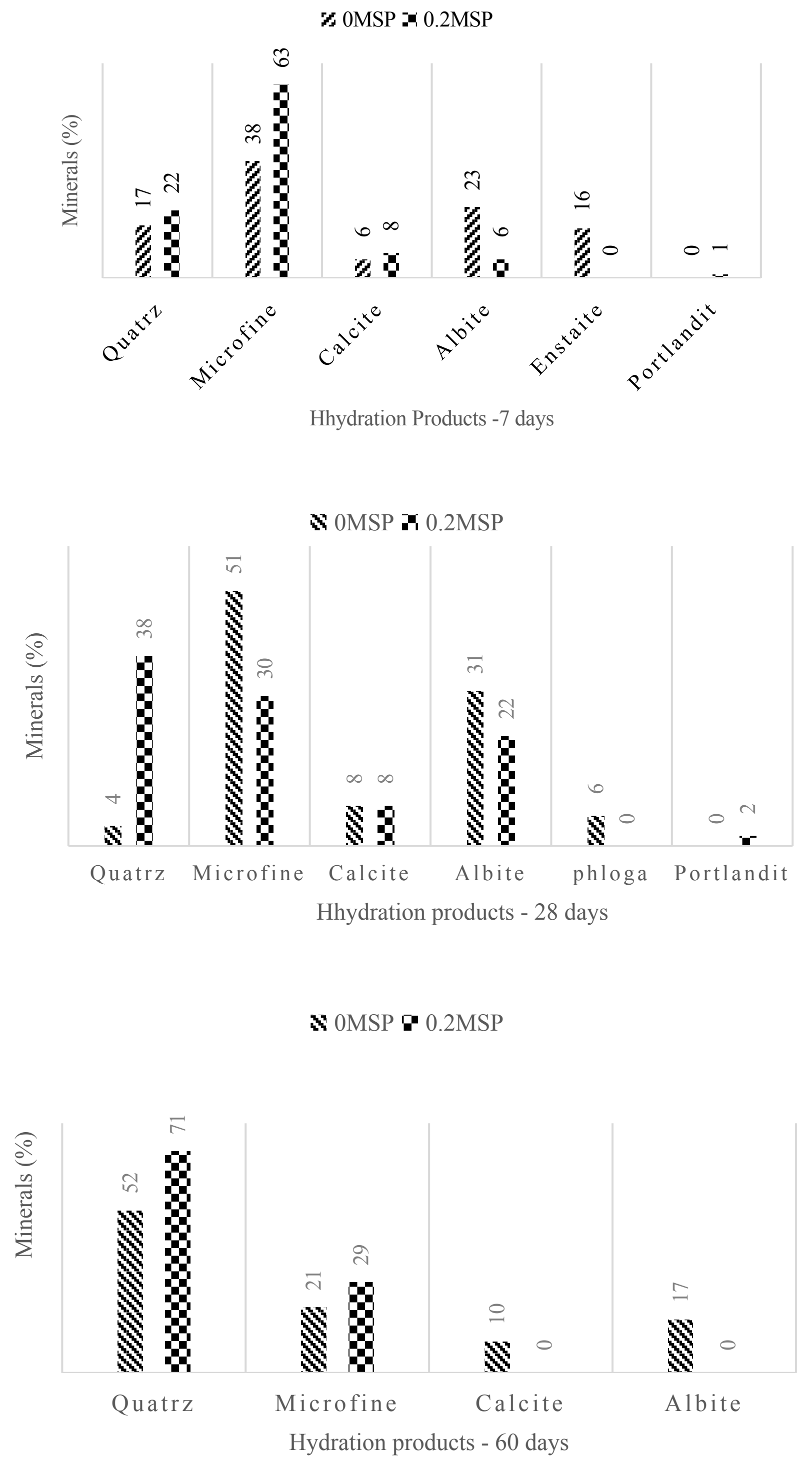

Figure 16. Hydration Products from the Micrograph

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Acid-Sulphate Characteristics of MOSP-Concrete

The presentations shown in Tables 10 and 11 are analysis performed on the crushed samples in the acid and sulphate media (Tables 8 and 9). Table 10 is the statistical characteristics of the performances in the various medium, while Table 11 is the summary. The Means of HNO_3 (-11.5%) and H_2SO_4 (-41.4%) showed deterioration effects (decrease) while, that of Na_2SO_4 (9.0%) and $MgSO_4$ (8.1%) exhibited some swelling (increase). The decay was more drastic on H_2SO_4 medium than in HNO_3 medium. The difference was approximately 260%. The growth between the Na_2SO_4 and $MgSO_4$ was marginal, and approximately 10%.

Table 10. Statistical Characteristics of the MOSP-Concrete Samples in Acid and Base Medium

Medium 20% Dilution	Mix	Age (Days)	Mean	SEMean	StDev	Variance	CoefVar
HNO_3	0%MOSP	3	-13.8	2.6	7.2	52.4	-52.6
		7	-9.5	1.2	3.3	11.2	-35.3
		28	-16.3	2.4	6.8	46.2	-41.9
		60	-13.4	2.0	5.7	32.9	-42.7
		90	-14.2	2.2	6.1	37.7	-43.2
		Average	-11.5				
	0.2%MOSP	3	-16.3	2.7	7.5	56.4	-46.0
		7	-9.1	1.2	3.4	11.7	-37.6
		28	-14.1	2.5	7.1	50.0	-50.3
		60	-12.8	2.8	7.8	61.1	-60.9
		90	-10.9	2.5	7.0	49.1	-64.2
		Average	-12.6				
H_2SO_4	0% MOSP	3	-44.9	4.4	12.6	157.7	-28.0
		7	-38.7	3.2	9.0	81.8	-23.4
		28	-39.4	7.2	20.3	411.2	-51.5
		60	-48.0	3.5	9.9	97.0	-20.5
		90	-35.8	5.0	14.0	196.8	-39.2
		Average	-41.4				
	0.2 % MOSP	3	-37.9	5.1	14.3	205.7	-37.8
		7	-36.7	4.3	12.1	147.1	-33.1
		28	-39.5	5.2	14.6	211.9	-36.9
		60	-42.0	5.9	16.8	282.7	-40.1
		90	-39.6	7.6	21.6	466.8	-54.6
		Average	-39.1				
Na_2SO_4	0%MOSP	3	7.1	1.7	4.7	22.0	66.1
		7	12.6	4.3	12.2	149.4	97.2
		28	11.2	1.6	4.5	20.4	40.3
		60	6.3	1.3	3.6	13.0	56.9
		90	8.0	2.0	5.8	33.0	71.6
		Average	9.0				
	0.2%MOSP	3	6.2	0.7	2.0	4.1	32.6
		7	6.6	0.8	2.4	5.7	36.4
		28	6.6	1.1	3.1	9.5	46.4
		60	6.2	0.8	2.2	4.7	35.3
		90	7.8	1.6	4.5	20.1	57.8
		Average	6.7				
$MgSO_4$	0%MOSP	3	8.7	2.2	6.2	38.3	71.1
		7	6.8	1.2	3.5	12.0	50.7
		28	9.2	1.5	4.2	17.7	45.6
		60	7.6	0.6	1.7	2.8	21.9
		90	8.4	1.5	4.4	19.0	52.0
		Average	8.1				
	0.2%MOSP	3	6.6	0.8	2.2	4.8	33.1
		7	6.9	0.9	2.5	6.4	36.9
		28	7.5	0.9	2.5	6.2	33.1
		60	10.7	3.1	8.7	75.2	80.9
		90	6.9	0.9	2.7	7.1	38.5
		Average	7.7				

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Table 11. Summary of Mean in Acid and Base Medium

Mix	Medium			
	Acid (%)		Base (%)	
	HNO_3	H_2SO_4	N_2SO_4	$MgSO_4$
0 % MOSP	-11.5	-41.4	9.0	8.1
0.2 % MOSP	-12.6	-39.1	6.7	7.7

In acid environment, sulphuric and nitric acids are strong acids. When an acid comes into contact with concrete, the first line of attack arises from the reaction of the acid with portlandite (calcium hydroxide – CH) leading to the formation of calcium sulphate (ie, gypsum) and water. In the case of sulphuric acid, the reaction may be written as:

$$H_2SO_4 + Ca\,(OH)_2 \rightarrow C_3A.3CS.H_{32} \quad (2)$$

Acid attack on concrete is generally exhibited at pH < 6.5 [Fattuhi and Hughes, 1983].

In sulphate environment, the two sulphate media used are 20% Na_2SO_4 and $MgSO_4$. The sulphate ions penetrate into the concrete, then react with free and abundantly available portlandite, which in turn reacts with monosulphate to form ettringite. The reaction is given as:

$$C_3A.CS.H_{12}\,(\text{monosulphate}) + 2CSH_2\,(\text{gypsum}) + 16H \rightarrow C_3A.3CS.H_{32}\,(\text{ettringite}) \quad (3)$$

The expansive nature suggests the formation of ettringite which is characterized by mass increase (Table 11). The chemical properties in the formation of iron (ii) hydroxide as shown in Equation 4:

$$Ca(OH)_2 + FeSO_4 + 2H_2O \rightarrow CaSO_4.2H_2O + Fe(OH)_2 \quad (4)$$

5. Conclusions

An evaluation on the use of Moringa oleifera seed powder (MOSP) as an additive for concrete production and the possible effects and transformations was undertaken in this work. The followings are conclusions on the work.

(1) The x-ray diffraction showed that the dominant minerals (cristobalite and quartz) in MOSP are polymorphs of silica, and the chemical properties show that MOSP has a high content of SO_3. From past works the high content was considered to show the strength development and provides resistance to sulphate attacks. Therefore, MOSP can be classified as a retarder, and good as a sulphate-resistant material for cement and concrete.

(2) The work recommends the limit of replacement of cement by wt. % of MOSP in concrete to be limited to 0.2% to 0.3%.

(3) The basic characteristics of the material as a paste showed that it could be used for hot weather concreting where enough time is allowed for concreting, and for non porous concrete, the water requirement should not be greater than 34%, and replacements not greater than 1.0%.

(4) In the fresh condition the workability was improved with MOSP added to concrete mix. However, the water absorption increased with increase in the quantity of MOSP. Therefore, in using MOSP for concreting, the need to use water-reducing agents maybe helpful.

(5) Addition of MOSP is beneficial in increasing the density of concretes. The compressive strength was increased but within the limits of replacement levels of 0.2% and 0.3%. Therefore, for optimum performance, replacement should not exceed 0.2% by wt. % of cement.

(6) MOSP-concrete in acid environment showed that acid attack appeared to be the most likely damage mechanism responsible for observed deterioration.

(7) From the study, in situations where acid and sulphate co-exist, acid mechanism maybe the overriding attack process responsible for observed degration in concrete.

Highlights

- The dominant minerals in MOSP are cristobalite and quartz, which are polymorphs of SiO_2.
- The high content of SO_3 (28 %) is not a disadvantage rather an advantage. This property makes MOSP a retarder by slowing down the strength development, and equally provides resistance to sulphate attack.
- The optimum replacement of cement by wt. % is 0.2 %, and improves workability, density, and compressive strength, but increases water absorption.
- MOSP-concrete in acid environment showed that acid attack appeared to be the most likely damage mechanism responsible for observed deterioration.

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