Influence of Superabsorbent Polymers on Properties of High-Performance Concrete with Active Supplementary Cementitious Materials of Nigeria

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Abstract. Concrete of strengths classes $\geq$ C55/67 referred to as high strength or high-performance concrete (HSC/HPC) are noted to be generally of low water/binder (W/B), made from binary or ternary cements with silica fume (SF) being a necessary constituent, and often requiring internal curing. Non-availability and high cost of SF in most sub-Saharan Africa like Nigeria however makes HSC/HPC production in this region very difficult and hence the continued search for alternative supplementary cementitious materials (SCM) with good performance properties as constituents of ternary/binary cements in HPC. This study thereby examines the strength properties of metastable calcined clay (MCC) based HPC cured internally with superabsorbent polymer (SAP) 0.2–0.3% (by weight of binder (b_{wob})). HPC mixtures of varied MCC and Rice husk ash (RHA) contents containing two SAP grain sizes labelled ($SP_1 < 300$ μm and $SP_2 < 600$ μm) were cast in 100 mm cubes and cured for varying ages (7, 14, 28 and 56 days) before testing. The hardened specimens were subjected to compressive strength and water absorption tests at the varied curing ages for the performance assessment of the binder types and SAP grain sizes in HPC with age. This study revealed the possibility of achieving Class 1 HPC (50–75 N/mm^2) utilizing industry manufactured calcined clay and locally produced RHA in Nigeria. The compressive strength of HPCs increased as the curing age increases for both SCM type, SAP contents and grain sizes. RHA based HPCs however showed better strength performance at the early ages than the MCC based. SAP addition in MCC based HPCs led to slight decrease in compressive strength as the SAP contents increased while the RHA based HPCs on the other hand, revealed slight increase in compressive strength with increase in SAP contents.

Keywords: Superabsorbent polymers · Metastable calcined clay · Rice husk ash · High-performance concrete · Strength properties

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1 Introduction

Within the past four decades, there is a growing acceptance of high-performance concrete (HPC) in construction facilities across the globe. The acceptance of HPC is due its better performance in mechanical strength, durability, flow-ability and economic cost amongst others when compared with normal strength concrete (Han et al. 2018; Nduka et al. 2018). ACI (1999) defined HPC "as concrete meeting special combinations of performance and uniformity requirements that cannot always be achieved routinely using traditional constituents and normal mixing, placing, and curing practice". The applications of this type of concrete have majorly been witnessed in the construction of tunnels, precast pylons, bridges, shotcrete repairs, tall buildings, parking garages and more (Aītcin 2004). Thus, the applicability of HPC in developing country like Nigeria would improve the future performances of infrastructure projects.

HPC is essentially a concrete with low water-binder ratio (W/B) ranging from 0.2–0.38. The substantial amount of cement and supplementary cementitious materials (SCMs) inherent in the mix results in increased temperature upon water addition and densification within the concrete area. Nduka et al. (2018) observed issues of concern like difficulty in curing vertical members, inaccessible locations in buildings and poor workmanship when external curing methods are used in HPC structures. Consequently, to mitigate these challenges in concrete production, internal curing (IC) with SAP has gained tremendous attention in literature and practice in producing HPC. SAP is largely polymers that has capacity to store and release large amounts of water. SAP's utilisation as an IC-agent in HPC is due to its ability for quick absorption of water and slow release of water during the hydration process of cement paste (Yang et al. 2019). It has been adjudged to be efficient in reducing autogenous shrinkage and penetration of deleterious ions in HPC mortars and concrete.

Among the various constituents of HPC, SCMs of pozzolanic nature play significant roles in meeting HPC requirements. SCMs are majorly siliceous/aluminous finely divided solid minerals that are used to substitute part of PC in concrete and mortar production and in the presence of water will react chemically with $Ca(OH)_2$ to form cementitious product (Zhou et al. 2017). Significances of SCMs include the refinement and improvement of pore size distribution, capillary pores and interfacial transition zone of hardened concrete, reduction of large pores, and improved density of cementitious products, improved workability in fresh state and lowering of W/B in HPC mix (Han et al. 2018; Busari et al. 2019). Silica fume (SF) is a prominent SCM used in HPC but its availability, economics and service considerations are pertinent issues of concern in HPC applications (Aītcin 2004). This thereby draws attention to abundant clay and some possible agricultural waste ashes like rice husk ash (RHA) available in this region towards filling the gap.

A number of studies have investigated the blend of industrial based pozzolans like SF, fly ash and slags (ground granulated blast furnace slag and corex slag) in HPC production. Studies on substitution of PC with metastable calcined clay (MCC) in achieving Class 1 (50–75 MPa) HPC is however scarce in literature. The present study thereby examines the possibility of incorporating commercially produced MCC as an alternative to SF in HPC production within Nigeria. It further compares the influence of SAP in MCC and RHA based HPCs.

2 Experimental Procedure

2.1 Materials

The materials used for the study are SAP, natural sand, crushed granite stone, PC (CEM II 42.5 N), MCC, RHA, water and superplasticizer. Two grain sizes of SAP tagged “FLOSET 27CS $\leq$ 300 μm (SP_1) and FLOSET 27CC $\leq$ 600 μm (SP_2)” as described in earlier publication (Olawuyi and Boshoff 2017) at varied SAP contents (0%, 0.2% and 0.3%) by weight of binder (b_{wob}) were used for the study. The absorption capacity determined by tea-bag test as reported in Olawuyi and Boshoff (2013) is 250 g/g in distilled water and 25 g/g in cement pore solution (CPS) for both the SP_1 and SP_2.

CEM II/B-LL, 42.5 N conforming to BS EN 197-1 (2011) and NIS 444-1 (2003) was used as the main binder (PC). MCC purchased from the Pozzolana Cement Plant in Ota, Ogun State and RHA obtained from Rice husk acquired from rice mill in Minna, Niger State, Nigeria served as the SCMs for this study. The rice husk was calcined to ash at 700 °C in a controlled incinerator and pulverised using a grinding mill at Building Laboratory, Federal University of Technology, Minna. Blends of the binders (as presented in Table 1 shows the two reference HPC mixtures while SAP at both 0.2% and 0.3% contents were incorporated with additional water of 12.5 g/g provided for SAP absorption for required specimen production.

The natural sand used at air dry condition had minimum particle size of 300 μm (i.e. all the particles smaller than 300 μm removed using the sieving method) in compliance with requirement for fine aggregate specification for HPC production (Neville 2012). The sand has the following physical characteristics: Fineness Modulus – FM = 2.87, Coefficient of uniformity – $C_u = 1.23$, Coefficient of curvature – $C_c = 3.28$ and dust content = 0.1%. This conforms to medium sand classification according to Shetty (2004). Crushed granite stone that passed through 13.50 mm sieve size and retained on 9.50 mm sieve size was used as course aggregate in compliance with typical HPC mixes found in literature (Aītcin 2004; Neville 2012; Olawuyi and Boshoff 2018). The crushed granite was used as saturated surface dry conditions after been washed to eliminate fine content that will likely increase water demand. Result of the physical properties tests on the constituent materials are found in Sect. 3.1 (Table 3).

A polycarboxylic ether (PCE) polymer-based superplasticizer supplied by BASF Limited was used as the superplasticizer and administered at constant concentration of 1.5% b_{wob}. Potable water as specified by BS EN 1008 (2002) available within the concrete laboratory of Department of Building Technology, Covenant University, Ota was used for mixing. Additional water for SAP based on 12.5 g/g of SAP (Olawuyi and Boshoff 2013) was also introduced.

2.2 Methods

2.2.1 Properties of Constituent Materials

The oxide composition of the cementitious materials (PC, MCC and RHA in powdered form) was examined at the Laboratory of the Ewekoro Factory of Lafarge Plc. About 100 g of the materials were packaged in a sealed polythene sheet and sent to the Laboratory for XRF analysis. The result is as presented in Sect. 3.3. The particle size distribution of the aggregate's samples (i.e. the sieved sand and granite stone) was determined by wet sieving while the specific gravity of the aggregate and binders were also determined in the Building laboratory of Covenant University. Section 3.3 further present and discuss the result of the physical properties of the constituent materials.

2.2.2 HPC Specimen Preparation

HPC target strength of C55/67 at 28 day with 0.3 W/B mix design utilizing British method was adopted as reference mix with the other mixes having different SAP contents (0.2% and 0.3%) for the two SAP grain sizes of SP_1 and SP_2. The total mass of the binders in the mixes include 485 kg/m^3 (90% b_{wob}) of PC, 55 kg/m^3 (10% b_{wob}) of MCC or RHA respectively. The details of the mix constituents for the reference HPC mixtures is presented in Table 1.

Table 1. Reference mixes composition

Constituents	Reference mixes (kg/m^3)	
	MCCC	RHAC
Water	156	156
PC (CEM II 42.5 N)	485	485
CA (13 mm maximum)	1050	1050
FA (retained on 300 µm sieve)	700	700
MCC	55	0
RHA	0	55
Superplasticizer (Masterglenium Sky 504 - 1.5% b_{wob})	8.1	8.1
Water/binder (W/B)^a	0.3	0.3

Legend

MCCC = 10% MCC based HPC
RHAC = 10% RHA based HPC
CA = Coarse Aggregate
FA = Fine Aggregate
^{a}W/B = (water + liquid content of superplasticizer)/(PC + MCC + RHA)

The varied SAP contents were incorporated for the two SAP grain sizes (SP_1 and SP_2) studied with additional water at 12.5 g/g provided for SAP absorption and the effect on the respective mixture's workability measured via slump flow table test in accordance with EN 12350:5, 2000. The result is presented in Sect. 3.3. The cast 100 mm cubes HPCs were de-moulded after 24 h and cured by full immersion in water with additional IC provided through the SAP incorporated for the respective ages before testing.

2.2.3 Water Absorption

The water absorption test was carried out on triplicate samples of 100 mm cube and the mean reported. The specimens were fully submerged in water for 56 days, then weighed and afterwards oven dried to a permanent mass at the temperature of 115 °C. The weight differences were then used in calculating the values of water absorption using Eq. 1. The terms w_1 and w_2 refers to dry and wet weight respectively.

$$\frac{W2 - W1}{W1} * 100 \tag{1}$$

2.2.4 Compressive Strength

HPC samples were made and cured in compliance with BS EN standards (BS EN 12350-1 & 5 2000; 12390-1 & 2 2000; 12390-3 2002) for compressive strength. The compressive strength tests were performed on 120 samples using 2000 kN loading capacity Eccles Compressive Testing Machine at 0.5 N/mm^2 loading rate. The concrete specimens were also weighted and dimension measured immediately after de-moulding and at the respective curing ages (7, 14, 28 & 56 days) after removal from curing tank for determination of the de-moulded density and final density of the hardened HPCs.

3 Results and Discussion

3.1 Physical and Chemical Properties of Constituent Materials

Result of the XRF analysis conducted on the PC, MCC and RHA powders are as presented in Table 2. The result reveal both MCC and RHA to be of good silica (SiO_2) content (55% and 94% respectively). The RHA is majorly SiO_2 (94%) with a silica-sesquioxide (S-S) ratio (SR) of 49.3 and aluminium-sesquioxide ratio (AR) of 1.1 is observed to be a very strong and reactive Class F Pozzolan in conformance to ASTM C618.

The SiO_2 content of the MCC is also far above the 35% minimum specified by the standard for a Class F Pozzolan. The sum of silica, alumina and ferric oxides (SiO_2+Al_2O_3+Fe_2O_3) for both the RHA (96%) and MCC (82%) is above the 70% specified for the Class of Pozzolan in ASTM C618. PC on the other hand is majorly calcium oxide (CaO - 64%). This is in agreement with oxides composition for CEM II Portland cement found in literature (Neville 2012).

Table 3 reveals the fine aggregate is observed to have a coefficient of uniformity (C_u) of 2.39, coefficient of curvature (C_c) of 0.94 and fineness modulus (FM) of 2.87 implying a medium sand of Shetty (2004) classification while the coarse aggregate used for the study is a uniformly graded stone. The details of physical properties as shown in Table 3 affirms that the fine and coarse aggregates are suitable for HPC production.

Table 2. Oxide composition of binder constituents

Oxides (%)	MCC	RHA	PC
SiO_2	55.04	93.60	21.50
Al_2O_3	24.49	1.00	5.20
Fe_2O_3	2.14	0.90	1.20
CaO	0.52	1.30	64.00
MgO	0.26	1.20	2.90
SO_3	0.02	0.10	4.50
Na_2O	0.05	1.70	0.60
K_2O	0.17	0.20	0.10
Minor Oxides	2.32	0.00	0.00
LOI	14.99	0.00	0.00
$SiO_2+Al_2O_3+Fe_2O_3$	81.67	95.50	27.90
SR	2.07	49.26	3.36
AR	11.44	1.11	4.33
Total	100.00	100.00	100.00

Table 3. Physical properties of materials

Item	Sand	Granite	RHA	MCC	PC
D_{10}	360	10000			
D_{30}	540	11000			
D_{60}	860	13000			
C_u	2.39	1.30			
C_c	0.94	0.93			
FM	2.87	–			
SG	2.65	2.7	2.3	2.5	3.4

3.2 Fresh Properties of HPC

Results of the flow table test conducted on the HPCs are shown in Table 4. The results revealed that all the mixtures are within the target limits of 600–700 mm slump flow. The variations as observed are very minor (665 to 675 mm) for all mixtures. This implies irrespective of the SAP grain sizes or contents, the mixtures have similar consistency and workability.

Table 4. Fresh properties of HPC mixtures

Specimen label	MCCC					RHAC				
	1	2	3	4	5	1	2	3	4	5
Constituents (kg/m3)	Ref	SP_1		SP_2		Ref	SP_1		SP_2	
SAP contents (%bwob)	0	0.2	0.3	0.2	0.3	0	0.2	0.3	0.2	0.3
Water	156	156	156	156	156	156	156	156	156	156
PC (CEM 42.5 N)	485	485	485	485	485	485	485	485	485	485
MC (10% bwob)	55	55	55	55	55	0	0	0	0	0
RHA (10% bwob)	0	0	0	0	0	55	55	55	55	55
CA (13 mm maximum)	1050	1050	1050	1050	1050	1050	1050	1050	1050	1050
FA (retained on 300 μm)	700	700	700	700	700	700	700	700	700	700
SAP	0	1.08	1.62	1.08	1.62	0	1.08	1.62	1.08	1.62
S/plasticizer	8.1	8.1	8.1	8.1	8.1	8.1	8.1	8.1	8.1	8.1
Additional Water (12.5 g/g of SAP)	0	13.5	20.3	13.5	20.3	0	13.5	20.3	13.5	20.3
Total W/B	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Slump Flow	670	665	665	670	665	675	675	670	675	675
Designed Density	2454	2469	2476	2469	2476	2454	2469	2476	2469	2476
De-moulded Density	2494	2473	2452	2457	2462	2436	2458	2487	2442	2443

Legend

SP_1 = SAP grain size type 1 ($\leq$ 600 μm)

SP_2 = SAP grain size type 1I ($\leq$ 300 μm)

3.3 Hardened Properties of HPCs

3.3.1 Compressive Strength of HPCs

Table 5 shows the compressive strength of various HPCs containing equal amount of CEM II 42.5 N, 10% MCC/RHA and SAP at varying contents and two grain sizes. All the HPCs revealed strength increases with increase in curing age with the RHACs showing better strength performance at the early ages. At 7-day curing, the reference MCC based HPC ($MCCC_1$) slightly outperformed reference RHA based HPC ($RHAC_1$) by 8.6% in compressive strength while SAP addition in MCCCs ($MCCC_2$ to $MCCC_5$) led to slight decrease in compressive strength as the SAP contents increased. On the other hand, SAP addition in RHACs ($RHAC_2$ to $RHAC_5$) resulted in slight increase in compressive strength with increase in SAP contents.

Thus, RHACs with SAP performed slightly better in strength than MCCCs at the early age. Similar trend was observed at the 14th day of curing. The 14th day strength of $RHAC_1$ is 4.37% above that of $MCCC_1$ while MCCCs containing SAP experienced slight strength decrease with SP_2 inclusion revealing better performance than SP_1. RHACs also had reduced compressive strengths at all SAP contents except $RHAC_3$ with 0.3% SP_1 content.

Both $MCCC_1$ and $RHAC_1$ at 28 days curing was noted to be at similar value of compressive strength (47.09 N/mm^2 and 47.10 N/mm^2 respectively). $MCCC_2$ to $MCCC_5$ mixes indicated increased in compressive strength over the control $MCCC_1$ by 3.8% while $RHAC_2$ to $RHAC_5$ also increased by 4.6% in compressive strengths over $RHAC_1$ control. The CS_{28} Factor (in Table 5) further revealed that SAP inclusion in all the HPC specimens resulted in slight compressive strength increases at the 28th day curing. Consequently, $RHAC_2$ to $RHAC_5$ showed better strength development with SAP inclusion than the $MCCC_2$ to $MCCC_5$. Thus, $RHAC_2$ to $RHAC_5$ slightly outperformed $MCCC_2$ to $MCCC_5$ by 0.86% with almost equal compressive strength for both SAP grain sizes at varying percentages.

Table 5. Compressive strength and CS_{28} factor

Specimen/age (days)	Compressive strength (N/mm^2)				CS_{28} factor			
	7	14	28	56	7	14	28	56
$MCCC_1$	42.97	43.15	47.09	51.51	0.91	0.92	1.00	1.09
$MCCC_2$	41.04	42.87	47.18	60.28	0.87	0.91	1.00	1.28
$MCCC_3$	40.46	41.32	47.61	60.53	0.86	0.88	1.01	1.29
$MCCC_4$	41.37	43.67	52.66	60.49	0.88	0.93	1.12	1.28
$MCCC_5$	40.67	43.81	48.28	62.72	0.86	0.93	1.03	1.33
$RHAC_1$	39.28	45.12	47.10	60.30	0.83	0.96	1.00	1.28
$RHAC_2$	42.49	44.44	49.88	61.60	0.90	0.94	1.06	1.31
$RHAC_3$	42.37	47.19	49.29	61.91	0.90	1.00	1.05	1.31
$RHAC_4$	40.47	42.12	49.09	61.63	0.86	0.89	1.04	1.31
$RHAC_5$	42.77	42.54	49.16	61.63	0.91	0.90	1.04	1.31

Legend

CS_{28} = the compressive strength value at respective curing ages as compared to the 28th day strength.

Similarly, 56 days curing period showed that RHAC_1 compressive strength outperformed MCCC_1 by 17.3%. There is no much significant difference in compressive strength value of the MCCCs at a round up value of 61 $\mathrm{N/mm}^2$ (for MCCC_2 to MCCC_5) that can be adduced to SAP contents nor grain size. MCCC_4 however exhibits the highest compressive strength value of 62 $\mathrm{N/mm}^2$. SAP addition was hence observed to about 20% strength value above the reference MCCC_1 strength value. In the same vein, the RHACs containing SAP (RHAC_2 to RHAC_5) gave a round up compressive strength value of 62 $\mathrm{N/mm}^2$ (1.63% above the reference RHAC_1) at all SAP content and grain size, implying no significance influence in compressive strength value attributable to SAP size nor contents.

It can therefore be inferred that the two SCMs (MCC and RHA) is effective in HPC production with similar compressive strength values achieved while SAP addition in the HPCs resulted in slight compressive strength increase at no significant influence by SAP grain size nor contents within the limits examined.

3.3.2 Water Absorption of HPCs

The water absorption of the reference HPC mixes (MCCC_1 and RHAC_1 respectively) as determined on the 56th day is low (Table 6). The absorbed water is 0.61 (MCCC1) and 0.48 (RHAC1) respectively. The RHACs containing SAP had same water absorption value (0.61%) except RHAC_4 with a slightly higher value (0.73). The water absorption values or MCCCs containing SAP (MCCC_2 to MCCC_5), was observed to be slightly higher with no particular SAP grain size nor contents. Both MCCCs and RHACs mixes are however observed to be generally of water absorption values below 5% irrespective of the SAP contents and grain sizes added, an indication of dense parking of the HPCs (i.e. low permeability). This implies the two SCMs when used to produce HPCs will give good resistance to aggressive chemical attacks in concrete and hence ensure good durability and long service life. SAP addition in the HPCs has little or no effect in their ability to resist aggressive chemicals.

Table 6. Water absorption of HPCs

Specimen		MCCC_1	MCCC_2	MCCC_3	MCCC_4	MCCC_5	RHAC_1	RHAC_2	RHAC_3	RHAC_4	RHAC_5
Mass (kg)	Wet	2.494	2.473	2.452	2.457	2.462	2.436	2.458	2.487	2.442	2.443
	Dry	2.479	2.436	2.436	2.442	2.439	2.424	2.443	2.472	2.424	2.428
Water absorption (%)		0.61	1.49	0.65	0.61	0.93	0.48	0.61	0.61	0.73	0.61

4 Conclusion

This study revealed the possibility of achieving Class 1 HPC (50–75 $\mathrm{N/mm}^2$) utilizing industry manufactured calcined clay and locally produced rice husk ash in Nigeria. The HPCs made from binary blends of CEM II/B-LL 43.5 N and MCC/ RHA had Masterglenium Sky 504 (a PCE) as superplasticizer with SAP of two grain sizes (SP_1 and SP_2) and contents (0.2% and 0.3%) incorporated as IC-agent. Inferences drawn from the study can thereby be highlighted as follows:

1. Both MCC and RHA sample used are good Class F Pozzolan having requisite physical and chemical properties as specified in the standards (ASTM C618)
2. All the HPCs (MCCCs and RHACs) studied are of similar range of workability (slump flow values of 665 to 675 mm) irrespective of the SCMs, SAP grain sizes and contents added.
3. The compressive strength of HPCs increased as the curing age increases for both SCM type, SAP contents and grain sizes. RHACs however showed better strength performance at the early ages than the MCCCs.
4. SAP addition in MCCCs led to slight decrease in compressive strength as the SAP contents increased while the RHACs on the other hand, revealed slight increase in compressive strength with increase in SAP contents.
5. SAP's influence on compressive strength of HPC at the later age is noted to be slightly positive. The increases in strength between 28th and 56th curing days depicts that the test for more than 28th days age is better seen as the actual properties of the tested concrete. The long term strength gain can be attributed to the latent reactions of pozzolanic materials (MCC and RHA) which is enhanced by internal curing provided by SAP.
6. The water absorption of the reference HPC mixes ($MCCC_1$ and $RHAC_1$ respectively) as determined on the 56th day is low (0.61 and 0.48). SAP addition resulted in higher water absorption with some inconsistencies. The values however being lower than 5% irrespective of the SAP contents and grain sizes added. This is an indication of dense parking of the HPCs (i.e. low permeability).

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