

**DEVELOPMENT OF HIGH-PERFORMANCE CONCRETE FROM  
METASTABLE CALCINED CLAY AND RICE HUSK ASH WITH  
SUPERABSORBENT POLYMERS**

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**17PCB01679**

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SUPERABSORBENT POLYMERS**

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**A THESIS SUBMITTED TO THE SCHOOL OF POSTGRADUATE  
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AND TECHNOLOGY, COVENANT UNIVERSITY, OTA, OGUN STATE,  
NIGERIA**

**MARCH, 2022**

## **ACCEPTANCE**

This is to attest that this thesis is accepted in partial fulfilment of the requirements for the award of the degree of Doctor of Philosophy in Building Technology (Building Structures) in the Department of Building Technology, College of Science and Technology, Covenant University, Ota, Nigeria

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(Dean, School of Postgraduate Studies)

**Signature and Date**

## **DECLARATION**

I, **NDUKA, DAVID OBINNA (17PCB01679)**, declare that this research was carried out by me under the supervision of Prof. Olabosipo I. Fagbenle and Dr. Babatunde J. Olawuyi of the Department of Building Technology, College of Science and Technology, Covenant University, Ota, Nigeria and Department of Building, School of Environmental Technology, Federal University of Technology, Minna, Niger State, Nigeria, respectively. I attest that the thesis has not been presented either wholly or partially for the award of any degree elsewhere. All sources of data and scholarly information used in this thesis are duly acknowledged.

**NDUKA, DAVID OBINNA**

**Signature and Date**

## CERTIFICATION

We certify that this thesis titled "**DEVELOPMENT OF HIGH-PERFORMANCE CONCRETE FROM METASTABLE CALCINED CLAY AND RICE HUSK ASH WITH SUPERABSORBENT POLYMERS**" is an original research work carried out by **NDUKA, DAVID OBINNA (17PCB01679)** in the Department of Building Technology, College of Science and Technology, Covenant University, Ota, Ogun State, Nigeria under the supervision of Prof. Olabosipo I. Fagbenle and Dr. Babatunde J. Olawuyi. We have examined and found this work acceptable as part of the requirements for the award of Doctor of Philosophy (Ph.D) degree in Building Technology (Building Structures).

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## **DEDICATION**

I wholeheartedly dedicate this thesis to the Almighty Jehovah for his manifold wisdom, sound mind, guidance, strength, and healthy life bestowed on me throughout this study and beyond according to His eternal purpose.

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## LIST OF SYMBOLS

$\alpha_c$	Degree of hydration of binder
$\alpha_{max}$	Ultimate degree of hydration
$f_{c,cube}$	Cube compressive strength (N/mm <sup>2</sup> )
$f_{c,cube28}$	28th day cube compressive strength (also known as $f_{c28}$ )
$f_{c,cyl}$	Cylinder compressive strength (N/mm <sup>2</sup> )
$K_{Bolomey}$	Material constant given by the Bolomey's formula (A)
$RC_{28}$	Compressive strength of standard mortar after 28 days of curing (N/mm <sup>2</sup> )
$V_m$	Total surface area of the solid phases is related to the computed cement composition
$X_{pc}$	Gel-space ratio of the HPC mortar paste
$d_{max}$	Maximum size of aggregate
$f_c$	Characteristic strength obtained (also known as $f_{ck}$ ) - (N/mm <sup>2</sup> )
$f_{cm}$	Mean cylinder compressive strength (N/mm <sup>2</sup> )
$f_{cr}$	Target strength at maturity (often 28 days) - (N/mm <sup>2</sup> )
$f_{ct,fl}$	Flexural tensile strength
$f_{ct,sp}$	Splitting tensile strength
$f_{ctk}$	Characteristic tensile strength
$f_{ctm}$	Mean value of tensile strength (N/mm <sup>2</sup> )
$v_c$	Specific volume of anhydrous binder (i.e., binary or ternary)
$w_n$	Non-evaporable water
$\rho_c$	Relative density of the cementitious material
$B$	Second material constants by Bolomey
$\sigma$	Standard deviation
$n$	Porosity

## ABBREVIATIONS

ACI	American Concrete Institute
ACV	Aggregate crushing value
AEC	Architecture, Engineering, and Construction
AFS	Acetone formaldehyde sulphide
AGS	Autogenous shrinkage
AIV	Aggregate impact value
Al-MAS-NMR	Nuclear magnetic resonance spectroscopy
ASTM	American Society for Testing and Materials
BET	Brunauer–Emmett–Teller
$b_{wob}$	by weight of binder
$b_{woc}$	by weight of cement
BSI	British Standard Institute
$Ca(OH)_2$	Calcium hydroxide
$CaCl_2$	Calcium chloride
Cc	Coefficient of gradation
CEM I	Cement type I
CEM II	Cement type II
$CO_2$	Carbon dioxide
CS	Chemical shrinkage
C-S-H	Calcium-silicate-hydrate
Cu	Coefficient of uniformity
$D_{10}$	Cumulative 10% passing

D <sub>50</sub>	Cumulative 10% passing
D <sub>90</sub>	Cumulative 90% passing
EDX	Energy Dispersive X-ray
FA	Fly ash
FHWA	Federal Highway Administration of United States Department of Transport
FM	Fineness modulus
FTIR	Fourier Transmission Infra-red Spectroscopy
GGBS	Ground granulated blast-furnace slag
GHG	Greenhouse gases
HCL	Hydrochloric acid
HDC	High-durability concrete
HES	High early strength
HESC	High-early strength concrete
HPC	High-performance concrete
HPM	High performance mortar
HSC	High strength concrete
HVFA	High-volume fly ash concrete
IC	Internal curing
IC-agent	IC agent
ICP-MS	Inductive coupled plasma spectrometry
LOI	Loss on ignition
LWA	Lightweight aggregates
MCC	Metastable calcined clay

MIP	Mercury Intrusion porosimetry
MK	Metakaolin
Na <sub>2</sub> SO <sub>4</sub>	Sodium sulphate
NBRRI	Nigerian Building and Road Research Institute
NSC	Normal strength concrete
PC	Portland cement
PCE	Polycarboxylate Ether
pH	Power of hydrogen (it has a numeric value defined as a negative base 10 logarithm of the molar concentration of hydrogen ions). This is a measure of acidity (< 7) and alkalinity (> 7).
PMS	Melamine sulfonate
PNS	Poly (naphthalene sulfonate)
PR	penetration resistance
PSD	Particle size distribution
RH	Relative humidity
RH <sub>7</sub>	Factor Rate of hydration based on 7th-day degree of hydration for reference mixtures
RHA	Rice husk ash
RPC	Reactive powder concrete
SAP	Superabsorbent Polymers
SCC	Self-consolidating concrete
SCM	Supplementary cementitious materials
SCMs	Supplementary cementitious materials
SDGs	Sustainable Development Goals

SEM	Scanning Electron Microscopy
SF	Silica fume
SFRHPC	Self-compacting Fibre Reinforced High-Performance Concrete
SHRP	Strategic Highway Research Programme
SPSS	Statistical Package for Social Sciences
$t_1$	Early age at the time of one-day curing
$t_d$	Early age duration of curing after casting taken as greater than 1 and up to day 10
UHPC	Ultra-high-performance concrete
VES	Very early strength
VHS	Very high strength
W	Mixing water
W/B	Water: binder ratio
W/C	Water: cement ratio
WA	Water absorption capacity
XRD	X-ray diffraction
XRF	X-ray fluorescence

## ABSTRACT

The research was conducted to determine the potential use of metastable calcined clay (MCC) as a supplementary cementitious material (SCM) in a binary binder for high-performance concrete (HPC) production. The attractive properties of calcined clay based on literature have influenced an SCM choice in concrete production. Therefore, to improve the performance of the structural elements regarding increasing height, span length, and load, a thermally activated MCC of Nigerian origin gave a point of view to more investigation and compared with rice husk ash (RHA). Different HPC mixtures at 5-30% with MCC or RHA content of cement replacements of five steps intervals were produced with superabsorbent polymers (SAP) introduced as an internal curing agent. The water-binder ratio (W/B) of all the mixes was kept constant at 0.3 while Masterglenium Sky 504, a polycarboxylate ether-based superplasticiser (PCE), was used to improve the HPCs workability. To obtain the properties of the cementitious materials, the chemical composition, mineral phases, morphology, calcination efficiency, and physical properties were quantitatively analysed using the advanced techniques of X-ray fluorescence (XRF), scanning electron microscopy/energy dispersive x-ray (SEM/EDX), X-ray diffraction (XRD), Fourier transform infrared/Attenuated total reflection (FTIR/ATR), Thermogravimetric analysis (TGA), laser particle sizing and Brunauer–Emmett–Teller (BET) nitrogen absorption method. The MCC's effect on the workability, early-age degree of hydration, early-age compressive strength, hardened mechanical properties (compressive, splitting tensile and flexural strengths), durability (water absorption, sorptivity, and chemical attack), and microstructure (morphology and crystalline phases) of hardened MCC based HPCs samples were determined. The determined early-age compressive strength of HPC mortar was further curve-fitted into Powers' model to assess the relationship between compressive strength and gel/space ratio. All the properties of HPCs containing MCC were compared to those of PC mixes. The XRF result shows that the chemical oxide composition of MCC confirmed the pozzolanic material requirements with recorded high useful oxides content. At the same time, the SEM image presents particles of broad, solid masses with a wider surface area of irregular shape. The XRD results show that the MCC was a major illite-based clay mineral calcined at a maximum temperature of 650 °C, as revealed by the TGA. The MCC addition increases the slump flow of HPCs at certain cement replacement with a comparative early-age compressive strength with the control. The MCC incorporation at 10% cement replacement best improved the porosity of HPCs at a later age resulting in increased mechanical, durability, and microstructural properties of tested samples. A simulation of MCC and RHA contents into the Powers' model signalled compatibility for predicting strength development trends in the HPC with SAP. The study has shown that MCC and RHA, which are locally sourced, can be used to produce Class I (50 – 75 MPa) HPC with enhanced mechanical, durability and microstructural properties. Therefore, it is recommended that MCC addition within 10% binder content be adopted for low W/B Class I HPC at no deleterious results on mechanical and microstructural properties of the concrete.

***Keywords: High-performance concrete, metastable calcined clay, rice husk ash, superabsorbent polymers, supplementary cementitious materials, Sustainable Development Goals 9,12 and 13***