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APPLICABILITY OF SOME CALCINED CLAY AND CALCIUM CARBIDE WASTE IN CEMENT MIXES FOR DEVELOPMENT OF POZZOLANIC BINDER

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Abstract
This paper presents findings of an investigation on the applicability of Imotoyewa (IM), Ifonyintedo (IF), and Owode-Ketu (OK) clay from Nigeria and calcium carbide waste (CCW) as a partial substitute for Ordinary Portland Cement (OPC) in the development of a pozzolanic binder. The CEM II was partially replaced with calcined Ifonyintedo clay (C-IF), calcined Owode-ketu clay (C-OK), and calcined Imotoyewa clay (C-IM) at intervals of 10% up to 50%, and CCW was also used to replace C-IM, C-IF, and C-OK at the same interval in a separate mix. Physicochemical, mineralogical, microstructural, Zeta-sizer analysis, and Brunauer-Emmett-Teller (BET) methods were used for characterization. The Strength Activity Index (SAI) of the burnt clay (CC), setting times of CEMII-CC, CC-CCW, and compressive strength by partially replacing cement with CC and whole cement replacement using CC with CCW were investigated. Results from the SAI indicated a high pozzolanic effect. C-IF with about 40% kaolinite content gave the highest mortar strength than the control at a substitution level of 20%. Kaolinite on the XRD trace was 34-40 wt% in the clays, consistent with BET values. The BET and porosity of the CC were well above OPC, while C-IF clay, with the highest kaolin content, had the highest limit. ST tests revealed that in the CEMII-CC and CC-CCW pastes, CC and CCW inclusion have a linear relationship with the ST and water content of the paste, owing to the pozzolanic reaction and dilution impact. They inhibit cement hydration; thus, their ability to retard could be useful with concrete in hot weather. The C-IF's reactivity with $Ca(OH)_2$ from CCW and CEM: CIC mortar showed similar and noticeable trends on the FTIR. Results have demonstrated the development of pozzolanic binders from blended cement mortar, calcined clay, and CCW mortar.

1.0 INTRODUCTION
Cement mortar and concrete are well-known man-made materials. The high cost of cement makes it difficult for the Nigerian government to achieve its needs, which raises the price of mortar and concrete constructions and reduces housing affordability. More than 10% of Nigeria's yearly budget will be required to cover the 19.5 million tonnes of cement needed in the country in a year. The burning of fossil fuels, especially carbon dioxide, a precursor to global warming, and the release of greenhouse gases during the calcination of calcium carbonate (limestone) are two major climate issues associated with the making of concrete and OPC [1–8]. By 2050, construction would need about 18 billion tonnes of concrete, due to the growing need for infrastructure in developing nations like Nigeria. Many countries continue to rely

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on Ordinary Portland Cement (OPC) for manufacturing concrete due to the need for higher living standards [9, 10]. To produce mortar or concrete, it is therefore required to discover an appropriate substitute for cement, completely or partially. A pozzolanic binder is developed through the chemical reaction of a pozzolan, a siliceous or aluminous siliceous material in finely divided form, with calcium hydroxide ($Ca(OH)_2$) in the presence of water at ordinary temperature to form compounds possessing cementitious properties.

Pozzolanic binders haves improved durability, reduced environmental impact, and increased sustainability. [11] In their investigation on the pozzolanic behaviour of calcined clays, 700˚C was chosen as the ideal temperature for the calcination of clays, and these clays were rich in kaoline. This happened because the XRD test carried out could not determine kaolinite peaks. The samples were calcined at 700˚C for 28 days and showed a considerable increase in CH consumption, according to the results. Research on the impact of clays with a lower proportion of burnt kaolin on the compressive strength of OPC mortars conducted by [12] and [13] has shown that clays with approximately 40% kaolin are usable as supplementary cementitious material (SCM). It is used to substitute up to 30% of Portland cement since their results indicated that for hydration higher than seven days, blended mortars's compressive strength was greater than the reference value. Emphasis was placed on the phases of cement hydrate that provided strength and gave rise to the burnt clay pozzolanic reaction: CASH and CSH with long chains and other alumina hydrates. In similar research, it was also observed that hemicarboaluminate $(CaO)_4Al_2O_3(CO_2)_{0.5}(H_2O)_{12}$) and monocarboaluminate ($(CaO)_4Al_2O_3CO_2(H_2O)_{11}$) were formed from the developed hydration systems that included limestone [13]. After the pozzolanic reaction occurred between three and seven days of hydration, the developed strength of blended cement increased with increasing kaolin content in calcined low-grade kaolin [13].

[14] and [15], in their studies on using kaolinite clays from Turkey, Nigeria, and Ghana, reported that the minerals in them are cost-effective pozzolanic additives in cement and concrete. [16] investigated the importance of using a mixture of burnt kaolinite and bentonite as SCMs and revealed that the mortar strength of cement-SCM increased by 10% against 100 wt% cement at 90 days. Other efforts on the development of pozzolanic binder include the works of [17] on pozzolan and bagasse ash with waste of calcium carbide (CCW); [18] on calcium carbide waste and fly ash; and [5] on Powdered Calcined Clay and slack lime $Ca(OH)_2$ in a mortar test. Using calcium carbide waste (CCW) and clay that has been calcined from Imotoyewa, Ifonyintedo, and Owode-ketu clay deposits in Ogun State, Nigeria, in partial substitution of OPC in a mortar test, the current study explores the creation of a pozzolanic binder. The waste product known as calcium carbide waste (CCW) is created when calcium carbide and water react to form acetylene (C_2H_2). Acetylene is then used to produce polyvinyl chloride (PVC) and to weld steel, particularly in the automobile sector. About 274,000 tonnes would be produced in a year when other industrial sources are not included [19]. These wastes cause environmental danger due to their detrimental effects on disposal locations, diminished plant and animal life, potential reduction in arable land, and risk to human life. Thus, this study aims to find out the applicability of calcined Imotoyewa (C-IM), Ifonyintedo (C-IF), Owode-ketu (C-OK), and waste from spent calcium carbide (CCW) for the development of a pozzolanic binder in a mortar test. Calcium carbide waste (CCW) should serve as an additional $Ca(OH)_2$ source to aid the pozzolanic reactions. The following parameters were investigated to achieve this aim: physicochemical, microstructural properties, mortar strength, and setting times.

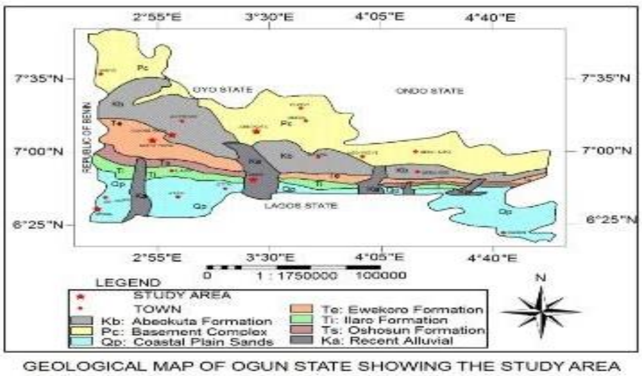

Figure 1: Geological setting of study area (Source: NBRRI Report, 2016)

2.0 MATERIALS AND METHOD

2.1 Materials

Unprocessed clays were mined from Imotoyewa (IM), Owode-ketu (OK), Ifonyintedo (IF), and Owode-Ketu (OK) in Ogun State, South-West Nigeria, on a global position system (GPS) Latitude N 7° 05.674' and Longitude E 2° 53.305', N 7° 07.520' and E 2° 53.635', N 6° 32.674' and E 2° 51.305' at a depth of 1 m below the surface, respectively. The study area is captured in Figure 1.

Below are some calcined samples of the clays (see Figure 2) at intervals of 50°C from 600˚C-700˚C respectively, as earlier reported in our previous study [20].

(a) Ifonyintedo-IF

(b) Owode-ketu-OK

(c) Imotoyewa-IM

Figure 2: Calcined (a) Ifonyintedo-IF (b) Owode-ketu-OK (c) Imotoyewa-IM clays

Cement manufactured by Dangote Cement Plc. (CEM II B-L, 42.5 N)-"3X" by [21]; water and fine aggregate were also used. Waste from processed calcium carbide was also used as a partial substitute for CEMII by weight. River sands from Abeokuta in Ogun State, Nigeria, are the source of fine aggregate. The sizes of the sand particles were defined as those that were kept on 63µm sieves but passed through sieves with an opening size of 2.00 mm. It was verified to be devoid of harmful chemicals and salt. The CCW was obtained from the dump site of a vehicles' maintenance workshop in Ogun State, Nigeria, where calcium carbide produces oxyacetylene flame for welding automobile parts. The waste was ground into a fine powder and kept in a cool location. Superplasticizer which was sourced from Baden Aniline and Soda Factory (BASF), and potable water were provided at the concrete laboratory of the Civil and Environmental Engineering Department, Covenant University, Ota. For this study, a mix ratio of 1:3 by weight of CEMII and sand was used in line with [22], and the water-to-cement ratio of 0.5 was chosen for optimal workability. CEM II in the mix was partially substituted with calcined Ifonyintedo clay (C-IF) and calcined Owode-ketu clay (C-OK), and calcined Imotoyewa clay (C-IM) at intervals of 10 wt% up to 50 wt%, and CCW was also used to replace C-IM, C-IF, and C-OK at the same interval in a separate mix. The mortar with 0% CCW and calcined clays served as the reference.

2.2 Laboratory Tests

2.2.1 Characterization

Tests performed on the materials included the distribution of the sizes of the particles, moisture contents, specific gravity, bulk and dry densities, and the distribution of the sizes of the particles. In line with [24], samples were taken and subjected to analysis using the combination of the PW-2404 X-ray Fluorescence Spectrometer (XRF) and X-ray diffraction (XRD)-MINIFLEX 600. The materials-specific surface area (SSA) was estimated through the Brunauer-Emmett-Teller (BET) multipoint approach [14]. The CARY 630 Fourier transform infrared (FTIR) spectrum was used to analyse samples in line with [25], covering a wave number of 4000–650 cm-1. The Barrett-Joyner-Halenda (BJH) method was used to evaluate sample porosity, and the desorption isotherm was employed to assess the mesoporosity at relative pressures ranging from 0.4 to 0.967 [20]. The calcined clay sample was analysed at the Chemistry Laboratory of Lafarge WAPCO Plc., Ewekoro, Ogun State, using X-ray diffraction-MINIFLEX 600 (XRD). Other analyses were done at the Department of Chemical Engineering, Ahmadu Bello University, Zaria (ABU, Zaria).

2.2.2 Determination of the standard consistency and setting times

In line with [26], the Vicat needle and probe were used to investigate the setting times and consistencies of the binder. A pulverised calcined portion of the clay (CC) was utilised to partially substitute the paste's CEMII content at different CEMII replacement levels: 10%, 20wt%, 30wt%, 40wt%, and 50wt%. In CC-CCW pastes, CC was also partly substituted with CCW in increments of 10% weight, as Table 2 illustrates. The control was the paste that was devoid of both CC and CCW. After blending the CC and CCW pastes, a consistent colour was obtained. The laboratory experiment was conducted at 25 ± 2°C and 70 ± 5% humidity to preserve stability and avoid fluctuations that could affect the experimental results.

2.3 Mortar Strength and Strength Activity Index (SAI)

In compliance with [21], a mortar strength test was performed with 40 x 40 x 40 cubes. A total of 125 specimens, each measuring 40 by 40 by 40, were made. Every single cube specimen was cured in a water tank by immersion, starting from the moment they were removed from the moulds, which are about 24 hours from the time of casting, and continuing until the testing day, when they were removed from the curing water and left to air dry before being tested for strength. The mortar strength test was conducted on the specimen at 7, 14, 21, 28, and 60 days of cure age. To conduct compressive strength testing, calcined clay (CC) was added up to 40 wt% as a substitute for the CEM II at 10 wt% intervals. Moreover, calcium carbide waste (CCW) was also used up to 40 wt% as a substitute for calcined clay (CC) at intervals of 10% in a binary mix (Table 1). The mortar strengths were evaluated 60 days from the 21st day of curing while applying optimum workability. The results of the setting times determined the maximum replacement of 40% CEM II with CC and CCW. A 1:3 binder-to-sand ratio was used. The needed quantity of water was added, and mixing was thorough. To remove the voids, the moulding required tamping the cement and vibrating the mould. After a day, the cube specimens were de-moulded and allowed to cure for 28 days in a tank with water that had an average temperature of 22°C plus or minus 1°C. The standard procedure outlined in [21] was used to establish the mortar strength. The strength figures provided are the means of five tests, expressed as a percentage of the reference mortar's strength

$$SAI = \frac{X}{Y} \times 100\% \quad (1)$$

Where, X is the mortar strength of the pozzolana blend specimen in MPa, and Y is the strength of the reference mortar in MPa.

Table 1: Mix Ratio for evaluation of the mortar strength

Sample (%)	Control	A1	A2	A3	A4	B1	B2	B3	B4	B5	B6	B7
CEM II	100	90	80	70	60	-	-	-	-	-	-	-
Calcined Clay(CC)	-	10	20	30	40	60	50	40	30	20	100	
CCW	-	-	-	-	-	40	50	60	70	80		100

Table 2: The material's physical properties

Properties	Sand	CEM II	CCW	C-IF	C-OK	C-IM
Moisture Content (%)	0.21	-	15.4	18.7	17.3	21.7
Specific Gravity	2.61	3.12	2.31	2.71	2.68	2.60
Bulk Density (kg/m^3)	1389.7	1255.3	647.6	893.50	843.50	796.50
Dry Density (kg/m^3)	1395.1	-	520.3	773.12	673.12	598.12
Fineness (passing through 90µm)	-	46.2	38	33.4	32.84	34
Coefficient of Uniformity (Cu)	2.29	-	-	-	-	-
Curvature (Cc)	1.07	-	-	-	-	-

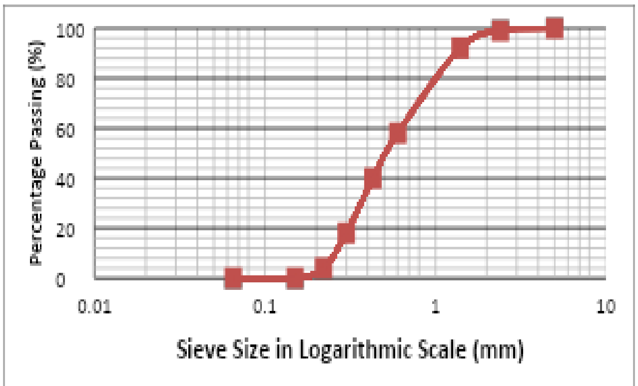

Figure 3: Particle size distribution of the fine aggregate

3.0 RESULT AND DISCUSSION

3.1 Physical Properties

The fineness of the calcined clays (CC) and calcium carbide waste (CCW) are comparable to CEM II (Table 2); nevertheless, based on specific gravities (SG) and bulk density, the CC and CCW were lighter than the CEM II. This implies that a higher volume of CCW and CC will be needed to replace a unit weight of cement, and compared to CEM II, CCW and CC had higher moisture content. This suggests that the water-binder ratio (w/b) may rise, which will probably cause a fall in the rate of development of mortar strength. The value of sand SG was within 2.5–3.0 similar to existing reports [1, 4] for average weight aggregate and can therefore be classified as normal weight aggregate. Additionally, 2.12 and 1.07 were coefficients of uniformity (Cu) and curvature, respectively (Figure 3). Similar to the outcomes found in [5, 7], this sand falls within the range advised [4, 21] and can be used to produce high-quality concrete.

3.1.2 BET and specific gravity of the samples

From Zetasizer analysis and Brunauer-Emmett-Teller (BET) technique, specific surface area (SSA) and other data on Table 3 were computed. 90% (D90) of CEM II, CCW, C-IF, C-IM, and C-OK particles are smaller than 300, 320, 743, 420, and 453 nm, respectively. The median particle size, D50, was within 49-353 nm, while D10 falls within 3.6-7.24 nm, which indicates that a wider particle size distribution will be pronounced for CIC/CCW blended cement.

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Similar data were reported in our previous study [20]. From Table 4, the specific surface area (SSA) measured via the Single-Point and Multi-Point BET models indicated 1.57–5.32 × 10^2, 3.19–8.52 × 10^2 m^2/g, respectively, which agree with values of 10–20 m^2/g reported for pure kaolinite [27, 28]. The samples examined using the DA BET mode had pore diameters ranging from 2.93 to 3.00 nm, consistent with mesoporous material [28, 29]. These values show that CEM II is the finest, which could be due to different production methods and grindability problems in the calcined clay and CCW due to the adherence of samples to grinding media caused by hygroscopic water [1]. The porosity of the calcined clays was in the range 0.1-0.18 cm^3/g and above CEMII, while IF had the lowest porosity among the clays. [13] reported a similar trend, which indicated that the clay with the highest content of kaolin had the lowest porosity.

Table 3: Samples' BJH mesoporous pore volume and BET SSA

Binder	BET SSA (m^2/g)		D_{90}	D_{50}	D_{10}	Pore diameter mode-DA (nm)	Pore Volume (cm^3/g)
	Single Point	MultiPoint					
CEMII	5.32 × 10^2	8.52 ×10^2	300	49.8	4.88	2.92	-
CCW	3.67 × 10^2	6.67 × 10^2	320	50	7.24	3.00	0.34
C-IF	1.81x10^2	3.47× 10^2	743	353	3.6	3.00	0.14
C-IM	1.57 × 10^2	3.22× 10^2	420	150	3.9	3.00	0.18
C-OK	1.87 × 10^2	3.19× 10^2	453	220	4.3	3.00	0.16

Table 4: Normal consistency and setting time value

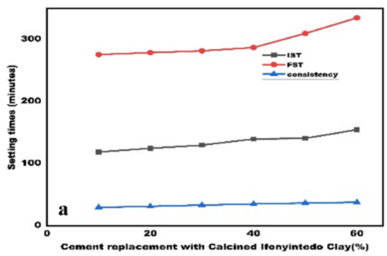
Paste replacement level	Weight of binder (g)	Normal Consistency (%)	Penetration (mm)	Initial Setting (min)	Final Setting Time (min)	Specific Gravity
Control	320	27	33	130	238	3.12
B1	320	32	34	394	516	2.65
B2	320	35.8	34.8	405	523	2.61
B3	320	36	34.7	445	548	2.59
B4	320	38	35	449	571	2.57
B5	320	41.6	33	465	603	2.52

Table 5: Chemical composition of the samples

SAMPLE (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	Mn_2O_5	P_2O_5	TiO_2	LOI
Ifonyintedo (IF)@6002H	65.42	23.2	2.63	1.91	0.26	0.00	0.1	0	0.11	0.21	2.98	3.28
Owode-ketu(OK) 700@2H	62.45	19.07	6.38	2.57	0.6	0.13	0.19	0.85	0.21	0.26	4.17	2.2
Imotoyewa (IM)@6502H	63.20	16.23	6.44	0.48	0.48	0.00	0.1	0.58	0.11	0.16	3.50	2.78
ASTM: C618-08 CLASS N	(SiO_2+Al_2O_3+Fe_2O_3) ≥ 70%			(CaO + MgO) ≤5%		4% Max						10% Max
CCW	4.32	1.25	0.27	80.45	0.00	0.06	0.00	0.00	0.02	0.07	0.1	12.46
CEMII	16.14	4.48	2.27	60.38	1.21	1.97	1.51	0.00	0.10	0.45	0.31`	11.35

3.1.3 Effect of calcined clay and calcium carbide waste on setting times

Normal consistency values of various A-B mixes were found to vary from 27 to 41% (Figure 4a-4c and Table 4), in the range recommended for OPC. Due to CC and CCW-specific surface area (SSA; 3.42–6.67 m^2/g), the mix containing a higher fraction of CC requires more water (greater consistency values) to lubricate the particle surface. The consistencies of B1-B5 (CC-CCW mixes) are directly correlated with the CCW content as it reduces consistencies. Except for mix B5, the mixes' initial setting (IS) and final setting (FS) times ranged from 115 to 465 minutes, while their FS times ranged from 270 to 571 minutes. The B6 (100% CC) sample displayed an IS time of 19 hrs, and the FS time exceeds 25 hrs (Table 4), attributable to a very slow chemical reaction at ambient conditions. A similar remark was also made by [30], and the observed trend conforms with existing works [5, 7, 8]. As the calcined clays (CC) increased (Figure 4a-4c), so did the paste's normal consistencies and initial and final setting times. CC delays cement setting times by preventing cement hydration. This retarding quality could be useful when pouring concrete in hot weather or while building roads and needing to move concrete over great distances. The strength development required for formwork removal is suitable for the FST (280–292 minutes), as seen in A1–A4 and CEMII–CC. This result is comparable to existing studies [4, 5, 20].



(a) Ifonyintedo

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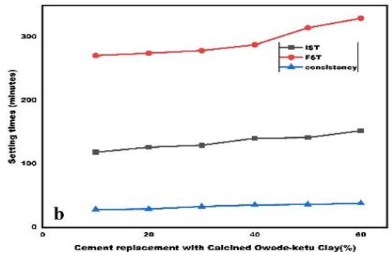
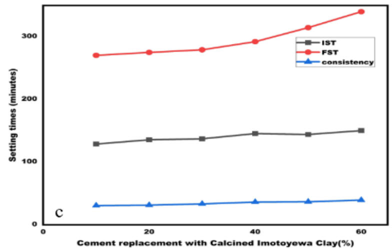


(b) Owode-Ketu



(c) Imotoyewa clays

Figure 4: Setting times of CEMII blended with calcined

3.3 Chemical Analysis of Samples

The results obtained from the XRF analyzer for all the samples tested (Table 5) are: Al_2O_3 (16–23%), SiO_2 (62–65%), and Fe_2O_3 (2–6%) make up the majority of the calcined Imotoyewa (IM), Ifonyintedo (IF), and Owode-ketu (OK) clays, which agrees with the kaolinite chemical constituents [13]. Due to air exposure to CEM II, the amount of carbonation and hydration of free magnesia was below the 10-weight percent upper limit [31]. Since the LOI of CCW was greater than 10 wt%, heat treatment may be necessary to improve the material's performance. The total of the principal oxides (SiO_2 + AI_2O_3 + Fe_2O_3) ≥70 wt%; $SO_3 \leq 4.0$ wt% in the calcined clays confirms it is class N pozzolan [32]. Table 6 shows that the principal oxides in the CEM II and CCW powders were similar in composition. However, the CaO in CCW was almost 20% greater than the value in CEM II. Furthermore, all the samples have alkalis K_2O and Na_2O with combined percentages of less than 4 wt%, which lowers the possibility of potentially harmful alkali-aggregate interactions [1]. None of the samples included cyanide, which corrodes reinforcement. Given the differences in SiO and CaO between the CCW and CC, substituting some of these materials for CEMII will help compensate for the SiO and CaO deficiencies caused by using just one of these materials to produce concrete.

3.4 Analytical Technique

3.4.1 Scanning electron microscopy (SEM)

The secondary electron mode of the SEM images is presented in Figures 5a-5c with the irregular shape of flakes of silicate from kaolinite, round platelets, and bright luminesce of quartz. This was in line with XRD and FTIR data in the sections below.

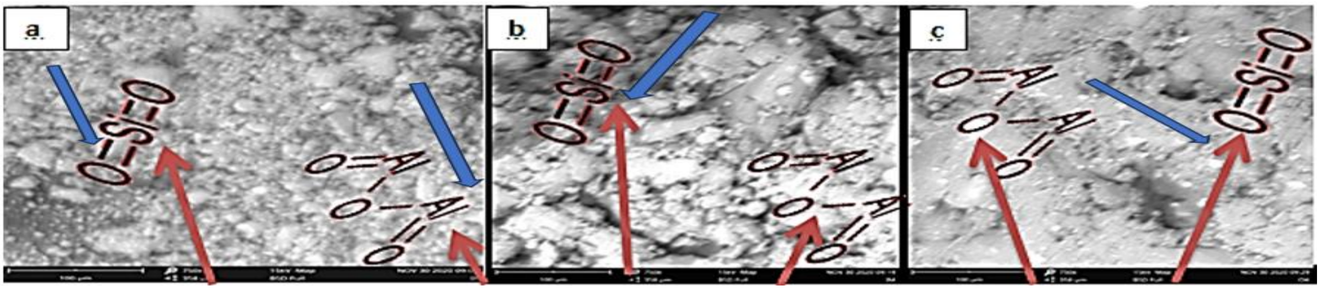


Figure 5: Images of SEM **(a)** Ifonyintedo (IF) clay **(b)** Imotoyewa (IM) clay sample **(c)** Owode-Ketu (OK)

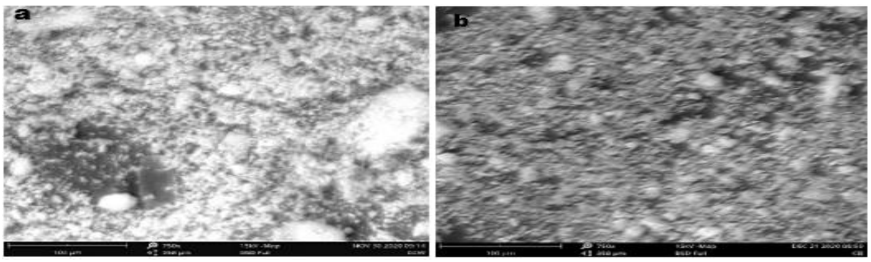


Figure 6: SEM micrographs of **(a)** calcium carbide waste (CCW) and **(b)** Ordinary Portland cement (CEMII)

CCW (Figure 6a) revealed spherical particles with layers of rod-like structures covering ≤200 nm and showing crystals of rhombohedral particles (50–100 nm) similar to calcite. The micrograph is similar to images obtained by [18], however, it is different from the pictures of [32]. The observation could result from the sources and processes that produced the CCW. The CCW shows a low-porous material made of O and Ca elements [32]. Figure 6b is a typical microstructure of plain CEMII showing bright grey areas of calcium-silicate compound (C_3S and C_2S).

3.4.2 X-ray diffraction (XRD)

The strongest reflections on Figure 7 were those of Quartz in the clay samples. CEMII had similar clay minerals except wollastonite (Ca_2SiO_4, Ca_3SiO_4); a product of the chemical reaction between CaO and SiO_2 during cement manufacturing processes. The peak of kaolinite minerals ($Na_4Al_3Si_3ClO_{16}$, $Al(SO_4)(OH)5H_2O$, and garnet) were also prominent in the clay samples covering 10°2θ to 70°2θ at an angle of 2θ; similar reflections were found in the works of [6, 29]. From Table 6, it was seen that values for kaolinite minerals in calcined Ifonyintedo clay were insignificant at 600°C, however, Imotoyewa and Owode-ketu values were greater than 8% at the same temperature. The clays had at least 34 wt% of kaolinites in them; [9] reported that clays with kaolinite constituents higher than 30 wt% are usable in the concrete blend. [33] revealed that the conversion of kaolinites into meta-kaolinite occurred during the dehydroxylation phase (400−700°C). These materials obtained during the dehydroxylation phase are usually pozzolanic and are usable in concrete [6, 9, 25, 29].

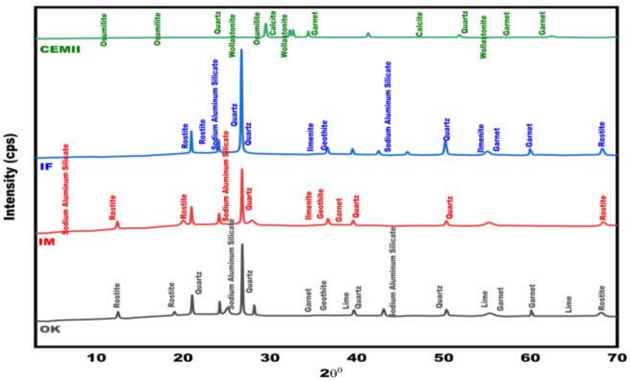


Figure 7: XRD trace of CEMII, Ifonyintedo, Imotoyewa, and Owode-ketu clay

The iron-bearing mineral goethite (FeO(OH)) was seen in the raw sample at (33.1 °2θ), however, at 24.3, 33.1, and 35.6 °2θ, the haematite reflections were only seen mostly in the calcined Owode-ketu and Imotoyewa samples (Table 6). This indicates that calcined Ifonyintedo clay (low iron content) would possess better architectural and marketing value. The CCW comprises CaO and calcite ($CaCO_3$) in line with the XRF data of 80.45%; similar compositions were recorded in studies on whole cement replacement with CCW and pulverised calcined clay [20].

Table 6: Mineralogy of the calcined clay

Sample (%)	Calcite	Mica/Illite	Kaolinite	Hematite	Quartz
Calcined Ifonyintedo clay (IF) @6002H	0.01	6.05	1.13	0.97	91.84
Calcined Owode-ketu clay (OK) @6002H	0.00	5.02	8.64	3.50	82.84
Calcined Imoto-Yewa clay (IM) @6002H	0.02	5.44	10.67	3.81	79.97

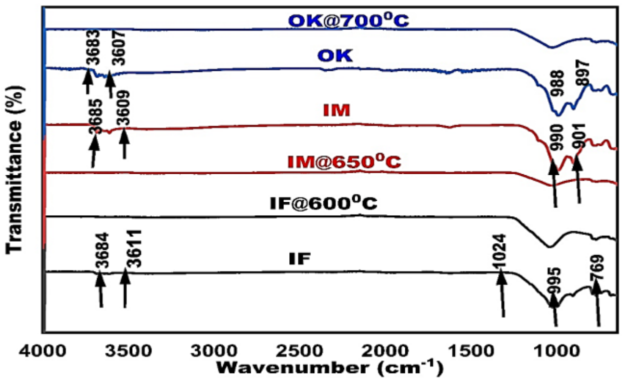


Figure 8: FTIR Spectra of the samples

3.4.3 Fourier Transform Infrared Spectroscopy (FTIR)

The absorption band of the raw samples (Figure 8) at 3683 - 3697cm^{-1} suggests that kaolinites ($Na_4Al_3Si_3ClO_{16}$, $Al(SO_4)(OH)5H_2O$ and garnet) are present owing to the vibration of the in-phase symmetric stretching of hydroxyl group found in the inner-surface [27, 34, 35, 36]. In the raw and calcined clay spectra presented in Figure 8, there were changes in the crystal structure as the calcination temperature increased. The kaolinite OH groups stretching at the inner surface (3696-3620cm^{-1}) were not seen after calcination of Ifonyintedo (IF) clay at 600 °C; however, in the case of Imotoyewa and Owode-ketu (OK) clay, the spectra disappeared at 650°C and 700°C, respectively.

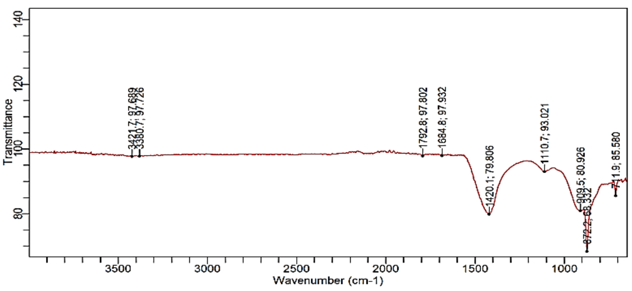


Figure 9: FTIR data of the CCW

According to [25], the absence of the bands at these temperatures indicates that the kaolinite was fully dehydroxylated at these temperatures. The stable bands of stretching of quartz at 796, 779, and 693 cm^{-1} mean quartz was unaltered at the test temperatures. The calcium carbide waste (CCW) spectra (in Figure

https://doi.org/10.4314/njt.v44i1.8

9) presented a narrow and intense band at 3421 and 3380cm^{-1} Caused by vibration νas O-H in the structure of $Ca(OH)_2$, and the bands of CO_3^{2-} at 1420 cm^{-1} (νasC-O) and 870 (δ C-O) in line [20, 27, 35]. The CEMII spectra (Figure 10) presented the characteristic Si-O (3462 cm^{-1}) with the signal at ~1397 cm^{-1} caused by monocarboaluminates and calcite νas C-O; similar bands were reported by [34, 37].

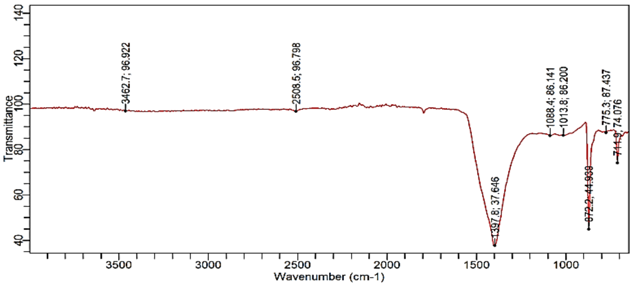


Figure 10: FTIR data of the CEMII

Furthermore, for broadband covering 800–1300 cm^{-1}, the shoulder at 1013 cm^{-1} was caused by the vibrations of νas S-O antisymmetric stretching from aluminates of SO_4^{2-} groups; also the vibration of Si-O antisymmetric stretching from wollastonite of SiO_4^{4-} groups and the 875 cm^{-1} shoulder was assigned to CO_3^{2-} (δ C-O) as shown in Figure 10. The structural vibrations aligned with those reported by [27, 34].

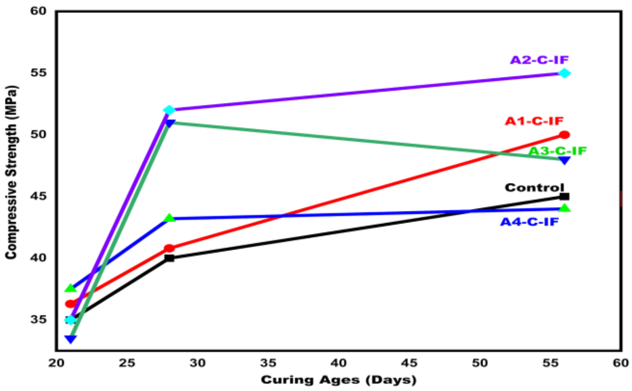


Figure 11: Mortar strength of calcined Ifonyintedo blended cement

3.5 Mortar Strength and the Strength Activity Index (SAI)

From Figure 11-13, results show that at all CEMII replacements with calcined clays (CC), whose mortar strength was above the control. The creation of the additional C-S-H gel was responsible for improved strength, resulting in the pozzolanic and hydration reactions. The SAI of C-OK, C-IM, and C-IF on the 28th day were 121%, 119%, and 123%, respectively, higher than the set value [23], but lower than the 129% obtained in similar studies [5, 7]. The behaviour of the clay minerals at 600°C, 650°C, and 700°C was very alike; hence, reactivity was expected to be close. However, for architectural and marketing purposes, it is beneficial that the mortar containing calcined Ifonyintedo clay (C-IF), which presents a similar colour to the conventional mortar, be selected above others.

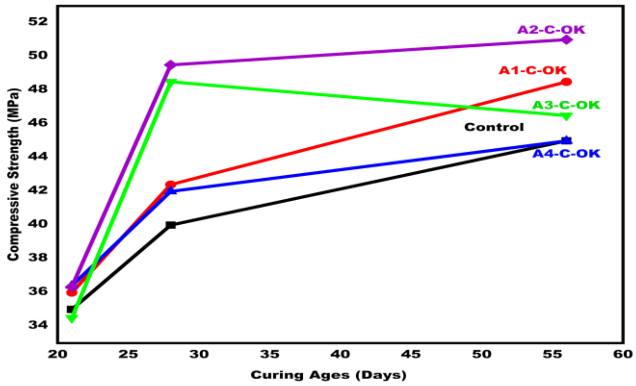


Figure 12: Mortar Strength of calcined Owode-Ketu blended cement

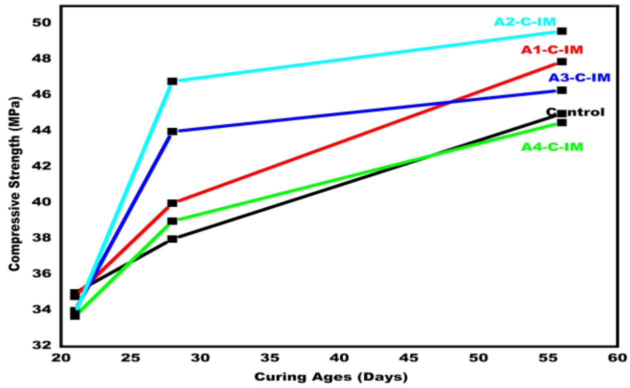


Figure 13: Mortar strength of calcined Imotoyewa blended cement

3.6 Calcined Clay/Calcium Carbide Waste Mortar Strength

A pozzolanic reaction was used to create the strength-forming C-S-H matrix after mortar was made without the addition of CEMII. From Figure 14, 10 MPa at 28 curing days was the maximum strength obtained at B; C-IF: CCW (60:40). This value is about 42% of the minimum standard [38] of 24.1 MPa set for concrete and general construction and about one-third of the 32.5N/R cement. A reduced strength was however observed from the 28th to the 56th curing day, contrary to pozzolanic reactions, and impurities such as zinc, nickel, manganese, copper, and lead in the carbide waste could be responsible [5, 7]. B6 and B7 did not do well because they were difficult to cure and demould. The fresh shrinkages of CEMII-calcined clay mortars were higher than the mortars of CCW-calcined clay, as the samples prepared under similar conditions by weight of CEMII-calcined clay mortars produced less volume of fresh mortars than the mortars of CCW-calcined clay.

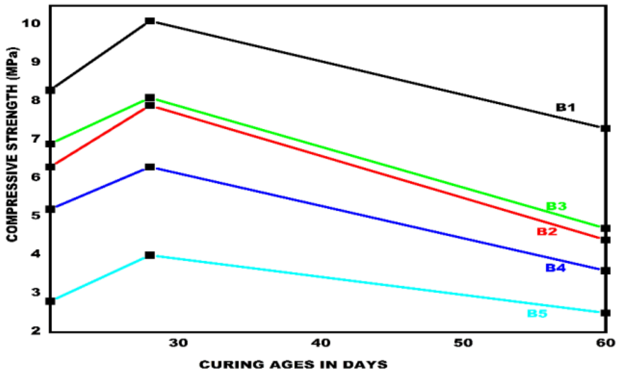


Figure 14: Mortar strength of calcined clay-carbide waste at different ages

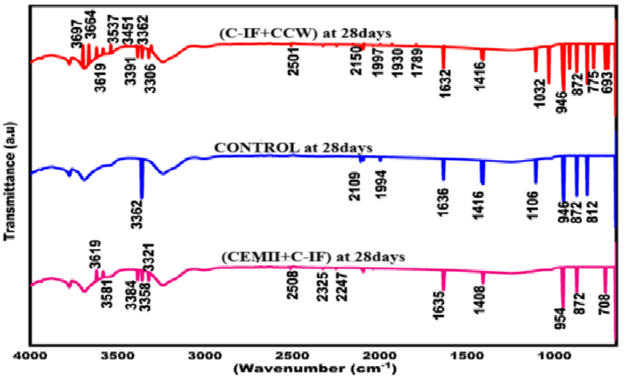


Figure 15: FTIR spectra at 60:40 **(a)** B4, i.e C-IF+CCW **(b)** A4, i.e CEM:C-IF

3.7 Validation Mortar Strength of Carbide Waste/Pozzolan by FTIR Analysis

The interaction that caused the formation of C-S-H gel (Figure 15) was investigated by performing a series of experiments with CCW**.** It can act as a source of Ca^{2+} that chelates with the mesoporous silicates that draw guest molecules, stimulating the conditions for silica to be involved in pozzolanic reactions [32]. First, the area ratio between the O-H band indicated that there is a higher fraction of $(Ca(OH))_2$ in the blended paste; according to [33, 37], a reaction at an early stage due to activation by CCW is possible. The spectra in Figure 15 suggested that there was a reaction between C-IF and CCW to form a C-S-H-like binder. The 3463 cm^{-1} could be a result of the vibration of ν_{as} O-H in the structure of CCW; bands of CO_3^{2-}at 872 (δ C-O) and 1397 cm^{-1} (ν_{as} C-O) reinforce the calcite reflections of the XRD trace. Similar and observable patterns were seen in Figure 15; obtained for B4 (C-IF with CCW) and A4 (CEM: C-IF) for 60:40 at 28 days for partial replacement with and without CCW. Also observed is a group of bands linked to silica at 954 and 946 cm^{-1}. The bands are in a position peculiar to vibrations of ν_{as} Si-O stretching of units of tetrahedral constituting a part of linear chains, found in the C-S-H structure [34, 37, 38]. The calcined clay with CCW can develop C-S-H gel when it reacts with Ca^{2+} ions existing in CCW.

4.0 CONCLUSION

A replacement of CEMII with calcined clay (CC) from 0–30 wt% produced higher mortar strength than the control. XRD trace supported by BET data established that at least kaolinite of 34 wt% was found in Ifonyintedo, Owode-Ketu, and Imotoyewa clays. These clays meet the strength activity index requirements and are Class N pozzolans with high reactivity. The data drawn from FTIR, XRD, and XRF analysis validated the findings in this study. A complete cement replacement with calcium carbide waste and calcined clay had a binding property that was moderate with close and noticeable trends of the strength-forming Calcium-Silicate-Hydrate on the FTIR. The ability to develop C-S-H gel from the reaction of calcined clay and calcium carbide waste as well as in blended cement is responsible for the development of a pozzolanic binder. The strength value was about 42% of the minimum standard of 24.1 MPa set for concrete and general construction and about one-third of the 32.5N/R cement.

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