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Case study

The physico-mechanical influence of dehydroxylized activated local kaolin: A supplementary cementitious material for construction applications

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ABSTRACT

This work presents the effect of partially replacing metakaolin with Portland limestone cement to produce mortars for construction. Teleku Bokazo kaolin was explored as a SCM in the production of mortars. The hydration product between Portland limestone and metakaolin was studied. The kaolin was heat treated to form metakaolin and partially used to replace Portland limestone cement (PLC) in mortars to explore the optimum replacement and its mechanical and durability effect. The samples were characterized using X-ray diffraction (XRD) for phases and crystallinity of the kaolin. Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM) gave information on the functional groups formed during the hydration and structure and surface morphology respectively. The pastes and mortars produced were subjected to setting time, water absorption, flexure and compression strength test. The mechanical properties were observed to increase with increasing metakaolin replacements. Therefore, from the results obtained, it is suggested that ~20 % replacement of Portland limestone cement with Teleku Bokazzo metakaolin can be very suitable for construction applications.

1. Introduction

Portland cement is a binder accepted globally in the construction industry as a result of its properties and price [1]. As a binder the availability and abundance of the raw materials used in its production makes it difficult to be competed against or replaced. However, its production process poses serious environmental threats; the clinkering process leads to the emission of environmentally unfriendly carbon dioxide (CO_2) gas [2].

The construction industry noticing this effect, continually explores alternative materials to sustain the environment. Supplementary cementitious materials (SCMs) is an example of materials been explored to curb this problem. SCMs are materials when added to Portland cement contribute to the properties of concrete through hydraulic activity or pozzolanic activity or both [3]. SCMs such as zeolite, calcined clays, agricultural waste by-products and industrial waste have been used

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to reduce the demand for Portland cement [1,4–6]. The use of calcined clays as SCMs is a well suited alternative that require attention especially in countries with numerous deposits of clay. The large deposit of clays in Ghana, including kaolin can be exploited as SCM. Kaolin is a clay containing kaolinite mineral, which when heat treated forms a product called metakaolin ($Al_2Si_2O_7$) which exhibits strong pozzolanic reaction, thus active silica and alumina in the metakaolin react with portlandite ($Ca(OH)_2$) to form cementing compounds which improve the properties of mortars and concretes [7]. Metakaolin (MK) as a pozzolan in mortars and concretes improves mechanical properties at early ages, reduce hydration heat of cement, shrinkage, permeability, increase in the freezing and thaw resistance, resistance against chemical effects, decrease in alkali silica reactivity and many more [8]. The properties are possible due to the combination of pozzolanic reaction, accelerated cement hydration and the filler effects of the metakaolin [9]. Teleku Bokazo is one of Ghana's natural kaolin deposits. The kaolin from this site is mainly used in the paint industry. However, to the best of the authors knowledge it has not been used as a supplementary cementitious material for construction applications. Portland limestone cement is also one of the most used binders in Ghana's construction industry but its reaction products and mechanical effects with Teleku Bokazo kaolin is unknown. If Teleku Bokazo kaolin is well explored, it has the potential to be used in sustainable constructions where, metakaolin will substitute Portland limestone cement and provide equal or better mechanical properties without harming the environment.

2. Experimental

2.1. Materials and methods

In this study, limestone based Portland cement (32.5R) CEM II / B-L (GHACEM) which meets the Ghana standard GS 1118:2016 [10] was used as binder; river sand collected from the Bessblock company was sieved through a 1.70 mm mesh sieve and tap water was obtained from the Civil Engineering Laboratory of Ghana Standards Authority. The clay material (kaolin) that was used as the supplementary cementitious material was obtained from the Teleku Bokazzo deposit site in the Ellembelle District of the Western Region of Ghana (see Fig. 1). Typically, the raw mined kaolin samples were washed off their impurities by adding water to a constantly stirred kaolin blunger leading to a slurry with specific gravity of about 1.2. The slurry was then passed through a 350 μm sieve to eliminate coarse grained impurities; poured into a Plaster of Paris mould (POP) and the filtrate dried in an oven at a temperature of 120 °C for 7 h. A weighed amount (150 g) of the as-prepared kaolin was put into a ceramic crucible, placed into an electric furnace (Carbolite RWF 1200) and then calcined at 650 °C for two (2) hours at a heating rate of 10 °C/min to form metakaolin, which was followed by grinding and sieving through a 90 μm mesh sieve. The calcination temperature was chosen based on the kaolinite dehydroxylation temperature range.

2.1.1. Materials characterization

2.1.1.1. Characterization of kaolin clay. X-ray powder diffraction (XRD) data of the clay samples were collected on a Panalytical Empyrean diffractometer with a theta/theta geometry; and operating a Copper Kα radiation tube (λ =1.5418 Å) at 40 kV and 45 mA. The XRD profiles of all the randomly oriented powder specimens were recorded in the 5.0°–70° 2θ range with a step size of 0.017° and a counting time of 14 s per step. The diffraction patterns were then matched against the ICSD's PDF database and qualitative phase analysis was conducted using the X'Pert Highscore plus search match software (Panalytical, The Netherlands). The estimated mean crystallite size of the kaolinite sample was also calculated from the X-ray diffraction pattern based on the reflections located at the 001 plane at (12.4°, 2θ) and the basal 002 at (24.9°, 2θ) by using the Scherrer broadening technique [11]. The particle size distribution (PSD) analysis of the as-prepared kaolin samples were carried out on a Horiba Scientific, SZ-100 nanoparticle analyzer employing the low-angle laser light scattering (LALLS) technique within sensitivity regions of $< 1\ \mu m \geq 8\ \mu m$. Prior to the analysis, ≈15 mg of the kaolin sample was dispersed in 15 ml deionized water and subjected to continuous ultrasound treatment in a bath sonicator for 30 min to ensure dilution. The thermal stability of the various phases in the raw kaolin sample was studied on a standard NETZSCH TG 209F1 Libra TG/DTA instrument under nitrogen gas flow of 50 mL/min. Prior to analysis, a sapphire standard was used to calibrate the thermal response due to heat flow as well as the temperature. 16.3 mg of the raw kaolin specimen was placed in an alumina (Al_2O_3) crucible (100 mg capacity), subjected to a linear heating ramp between 25 °C and 800 °C at a rate of 10 °C/min and a cooling rate of 50 °C/min. The test measurements were made for the mass (loss) and enthalpy change of the sample as a function of the temperature. The mass losses were determined on the thermogravimetric (TG) curve by means of the tangent method [12]. The tangent method was also used to quantify the water loss during dehydroxylation of the kaolinite as well as for the determination of the percentage of the kaolinite minerals in the sample [13,14].

The surface morphology of the samples was carried out on an ultra-high vacuum and high resolution Philips FEI XL-30 FEG scanning electron microscopy equipped with an energy-dispersive x-ray spectrometer (EDS) and operated at 30 kV and resolution of 2 nm. Samples were rendered conductive by metalizing them with gold/platinum coating prior to the analysis. Images were acquired using a Gatan MiniCL imaging system at various magnifications.

Transmission FT-IR spectroscopic data were collected within a range of 4000-400 cm^{-1} and a resolution of 4 cm^{-1} using a Bruker ALPHA FT-IR Spectrometer equipped with a diamond ATR crystal (2 mm sample-crystal contact diameter) and an Opus acquisition software for analysis.

Fig. 1. Map of Teleku Bokazzo kaolin deposit site.

2.2. Metakaolin- cement composite mortar blocks batch fabrication

The preparation of the mortar blocks was performed according to British standard (BS EN 196-1:2016) [15] and cast using a 72 mm^3 mould. Metakaolin was used to replace ordinary Portland cement (PLC) by weight in percentages of 10 % to 35 % using a 5 % step size as shown in Table 1. In producing the mortar cubes, cement-to-sand and a water-to-cement ratio of 1:3 and 0.5 respectively were used. The samples were demolded after twenty-four (24) hours, the weight measured and cured by immersion in a curing pond at room temperature for three (3), seven (7), and twenty-eight (28) days. The same procedure was followed in casting the mortar beams for flexural analysis. The variation was the quantity of materials weighed and dimensions of the prismatic mould which was 40 mm x 40 mm x160 mm (see Table 2 for the batch formulation of mortar

Table 1
Batch formulations of kaolin-metakaolin-cement mortars.

Sample code	Compositions	PLC (%)	MK (%)	Remarks
0MK	CEM II 32.5R (PLC)	100	0	Control
10MK	CEM II 32.5R (PLC) + 10 %MK	90	10	10 %MK
15MK	CEM II 32.5R (PLC) + 15 %MK	85	15	15 %MK
20MK	CEM II 32.5R (PLC) + 20 %MK	80	20	20 %MK
25MK	CEM II 32.5R (PLC) + 25 %MK	75	25	25 %MK
30MK	CEM II 32.5R (PLC) + 30 %MK	70	30	30 %MK
35MK	CEM II 32.5R (PLC) + 35 %MK	65	35	35 %MK

preparation). The water consistency and setting time test was carried in accordance with British standard BS EN 196-3 [16] using Controls Vicat's apparatus. The water absorption by capillary action was done in accordance with BS EN 1015-18 [17]. Typically, masses were weighed and dried in the oven until constant masses were reached, then coated with wax leaving the rough side. They were then placed in a water bath to a depth of 10 mm for 24 h while measuring the masses after a period of time.

Compressive tests were carried out on a C-46L2 (CONTROLS, Italy) Universal Materials Testing Apparatus equipped with a maximum 3 kN cell at room temperature following the ASTM C918/C918M-13 Standard Test Method. The as-produced samples (72 mm x 72 mm x 72 mm) were compressed at a constant compression rate of 1 mm per min until the bodies were fragmented. The material's response (maximum stress or fracture) to the applied load was measured for three (3) samples of each preparation batch and the average values recorded as is in Eq. (1).

$$\text{Compressive Strength} = \frac{\text{Failure Load(F)}}{\text{Cross} - \text{sectional Area}(A_c)} \tag{1}$$

where ***F*** is the average compressive force exerted on the samples and ***Ac*** is the area of the concrete sample. The three-point bending (flexural) tests of the samples were carried out following ISO/ASTM (ISO4013:1978 ASTM C580-02(2012) standard procedures and recommendations. The flexural tests on the samples were performed on an Anderen MOR/5-TS/65 model Modulus of Rupture testing machine equipped with an automatic controller and a 5 kN load cell under ≈1 mm/min displacement cross-head feed rate. Descent velocity of the breaking blade is adjustable through a control panel, which is set up automatically with standardized increases of the applied load in accordance with ISO 10545-4, and with graphical display. Three (3) test bar specimens from each batch with dimensions of 40 mm x 40 mm x 160 mm were prepared for the flexural tests and the average values were determined according to Eq. (2),

$$\text{Flexural Strength}, \delta = \frac{3FL}{2wd^2} \tag{2}$$

where **F** is the failure load in Newton's, ***w*** is width, ***d*** is beam depth, ***L*** is distance between the supports used.

3. Results and discussion

3.1. Particle size distribution and XRD analysis

The particle size distribution analysis of the as prepare Teleku Bukazo kaolin as shown in Fig. 2 indicates a monodispersed distribution of average kaolin particle size of ≈1.87 μm.

Fig. 3 presents the X-ray diffraction (XRD) patterns of kaolin and metakaolin from Teleku Bokazo. The figure shows three main crystalline minerals present in the kaolin: kaolinite, quartz and illite. The crystallite size of the kaolinite mineral determined using the scherrer equation [11], indicated an average size of 17.8 nm The metakaolin was characterised by the total dehydroxylation of kaolinite causing an increase in the quartz intensity. The metakaolin XRD pattern looked similar to XRD patterns observed in [18–20] which had similar features after calcining kaolin at 750, 800 and 850 °C.

Studies by Moodi et al. [18], Badogiannis et al. [19] and Obada et al. [20–23], have shown that XRD peaks of kaolin contain illite, quartz and kaolinite. El-Diadamony et al. [9] also showed quartz as the main mineral in the XRD pattern of metakaolin after calcining at 800 °C for two hours. This studies have also been confirmed by Moodi et al. [18] and Obada et al. [20]. The main compounds identified in the control and different metakaolin replaced mortars after 14 days curing are presented in Fig. 4. The X-ray diffractions of MK replaced mortars were compared with the control mortar (Portland limestone cement mortar). Calcium silicate hydrate (C-S-H), calcium aluminium silicate hydrate (C-A-S-H), portlandite ($Ca(OH)_2$), ettringite (E) [$Ca_6Al_2(SO_4,SiO_4,CO_3)_3(OH)_{12}\cdot 26H_2O$], quartz ($SiO_2$), calcite ($CaCO_3$) and gypsum (G) ($Ca(SO_4)(H_2O)_2$) were the identified mineral phases in the mortars.

In the control mortar, the XRD revealed peaks of C-A-S-H, C-S-H, portlandite, quartz, gypsum, and calcite as the main phases. Although, it was noticed that 10–35 %MK mortars were also composed of C-A-S-H, C-S-H, portlandite, quartz, gypsum, and calcite. A new product known as ettringite was formed. Ettringite the distinguishing mineral phase formed in the MK mortars, is a product from the reaction of metakaolin, celite, portlandite and sulphate [9,24].

Table 2
Batch formulation for cubes mortar with metakaolin replacement.

Samples	Cement (g)	Sand (g)	Metakaolin (g)	Water (g)
0MK	600	1800	0	300
10MK	540	1800	60	300
15MK	510	1800	90	300
20MK	480	1800	120	300
25MK	450	1800	150	300
30MK	420	1800	180	300
35MK	390	1800	210	300

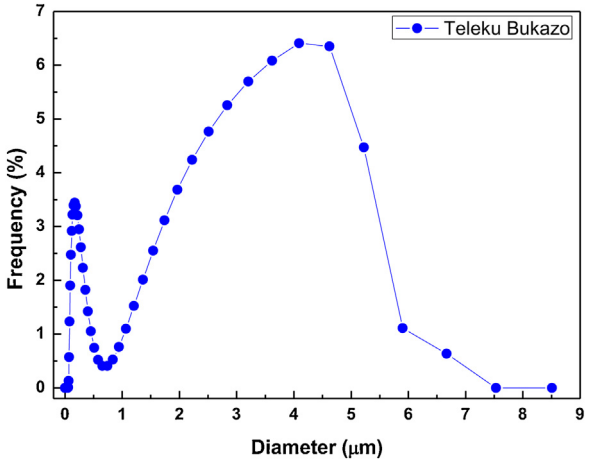

Fig. 2. Particle Size distribution of the s-prepared Teleku Bokazo kaolin.

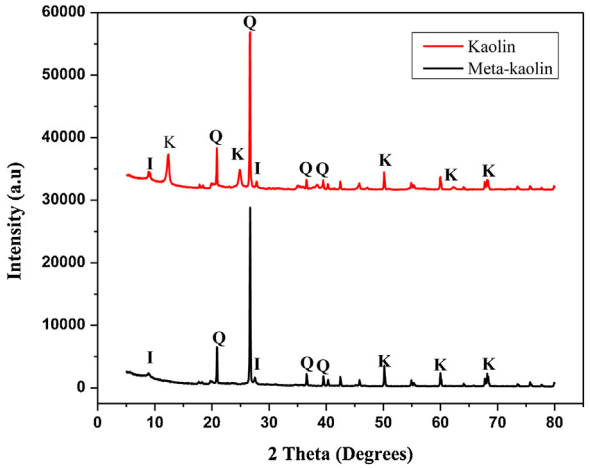

Fig. 3. XRD of Teleku Bokazo kaolin and metakaolin (I: illite, K: kaolinite, Q: quartz).

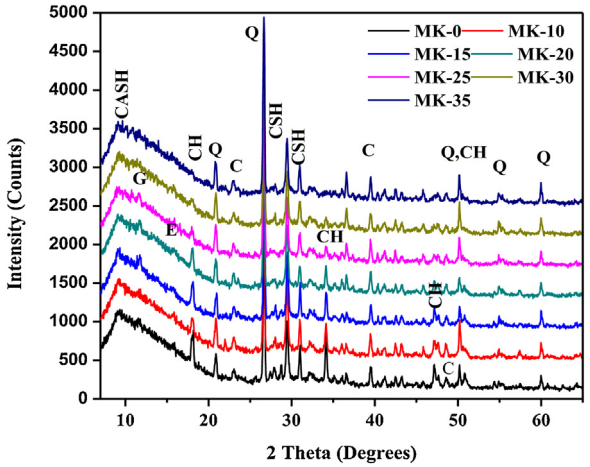

Fig. 4. XRD of mortar cubes with different metakaolin proportions after 14 days (C: calcite, CH: portlandite, Q: quartz).

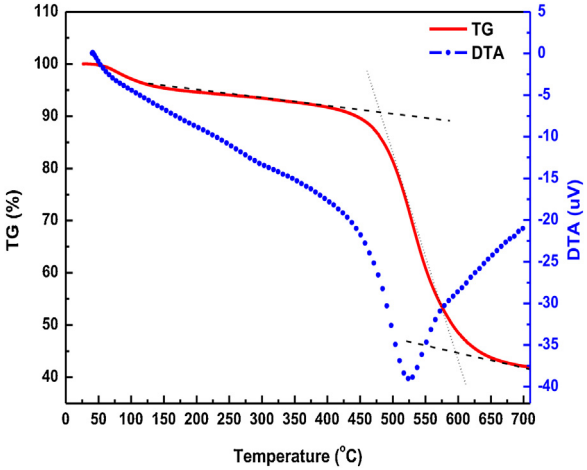

Fig. 5. TG-DTA of Teleku Bokazo Kaolin.

At diffraction angles 18.6° and 34° [2θ], it can be seen that, there was a gradual decrease in the intensity of portlandite peaks from control, 10–30 % MK and a disappearance of the peak at 35 % MK replacement. This observation could be attributed to the pozzolanic reaction between portlandite and metakaolin in the various MK mortars. Haturite, a hydrated calcium silicate is also a cementitious compound formed in the 15 % MK replacement. The quartz found in all the mixes was from the sand and metakaolin used in the mix whereas gypsum and calcite were from the Portland limestone cement.

The formation of ettringite in MK mortars agree with studies of Ramezanianpour et al. [25] and Cyr et al. [26]. The decrease in portlandite intensity in comparison to the control as the metakaolin content increases is as a result of the pozzolanic reaction between portlandite and metakaolin. Studies of Subasi et al. [8] and El-Diadamony et al. [9] also agree with the observation. The reference codes of the various mineral phases present in the mortars using X'Pert High score plus software is (C-A-S-H, card no. 76-0620), (C-S-H, card no.72–1907), (portlandite, card no.87-0673), (ettringite, card no.72-0646), (quartz, card no.46–1045), (gypsum, card no.70-0982), (calcite, card no.88–1807) and (hatrurite, card no. 98-006-4759).

3.2. Thermogravimetric analysis

Result of the TGA analysis of kaolin is shown in Fig. 5. The graph begins with a gradual loss in mass from 20−200 °C which can be attributed to the loss of moisture (dehydration) in kaolin as temperature is increased. A significant loss can be observed from 445−760 °C, within this range the kaolinite collapses from a structurally ordered form to a more disordered form known as metakaolin which is a more reactive form of kaolin [24,25,27].

The conversion of kaolin to metakaolin is further supported by the results from the XRD analysis by the disappearance of kaolinite peak. Dehydroxylation refers to the removal of the structural water molecules and can usually be observed in the 400 °C–800 °C range. The dehydroxylation peak observed within 400 °C and 650 °C is characteristic of the kaolinite structure and is often used to quantify the purity the percentage of kaolinite in the analysed kaolin sample. It is worth noting that, these peaks are indicative of the amount of hydroxyls molecules originally present in the kaolinite structure. The position of the dehydroxylation peak depends mainly on the type of structure and how the hydroxyls are bound to it, whereas its shape or range depend more on the crystallinity or particle size distribution: the more crystalline the material, the sharper the dehydroxylation peak for a given structural hydroxyl. Using the tangent technique on the thermogravimetric curve, the kaolinite content in the as-prepared Teleku Bokazo kaolin was estimated to be ≈45.64 %.

3.3. FTIR analysis

From Fig. 6, a characteristic peak for portlandite (*a by-product of the hydration of cement*) is observed at bands around 3640 cm^{-1} [28–30]. At 3403 to 3410 cm^{-1}, there is a broad OH stretching band which could be described as free water in the hydrated paste [29–31]. At 1740 cm^{-1}, vibrations of C=O represents a carbonyl group [32].

Infrared band at 1419 cm^{-1} represent C—O stretching vibration of CO_3^{2-} ion [29–31]. Si-O vibrations of calcium silicate hydrate, present simultaneously at 1087 cm^{-1} and from 956 to 963 cm^{-1} [28,29] is seen in all pastes.

3.4. Scanning electron microscopy analysis

Fig. 7 presents the scanning electron microscopy analysis of kaolin, metakaolin and the as-prepared mortars. The surface morphology of kaolin occurs as plate like structures stacked in layers to form a compact structure. Metakaolin occur as a

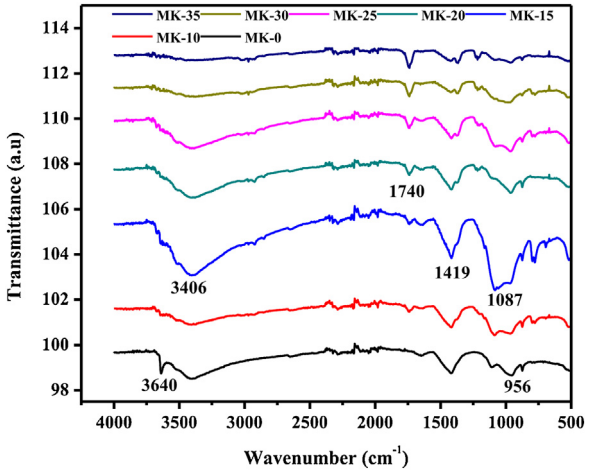

Fig. 6. FTIR of mortar paste with different metakaolin proportions after 14 days curing.

cluster of layered plates stacked over each other. The plate-like structures are indicative of kaolin and metakaolin. This observation is supported by Rashad [33] and Obada et al. [20]. From the images of the control mortar and metakaolin mortar, there was no clear difference in the surface morphologies of both mortars. There is a uniform mixture of the mortar ingredients, meaning there is good bonding between all particles.

3.5. Physical and mechanical properties

3.5.1. Water consistency and Initial setting time analysis

Fig. 8 presents the water consistency and initial setting time results of the cement mortar pastes containing 10–35 % replacement of metakaolin. From the results of water consistency, there was a progressive increase in the water consistency

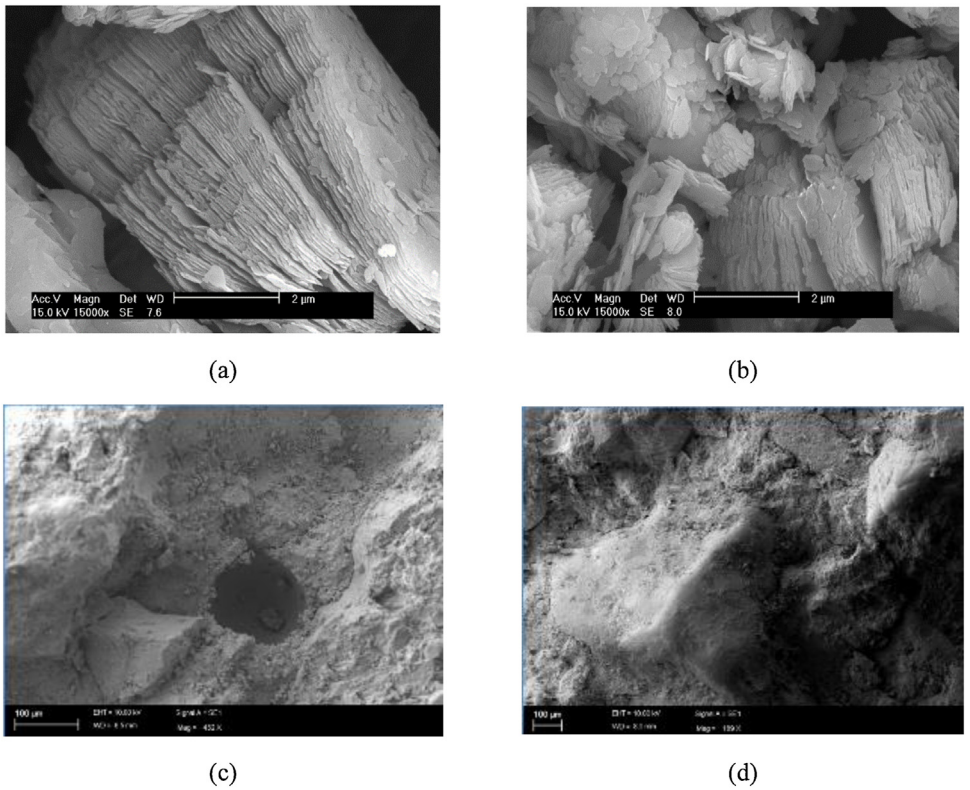

Fig. 7. SEM images of (a) kaolin and (b) metakaolin, (c) control mortar and (d) MK mortar.

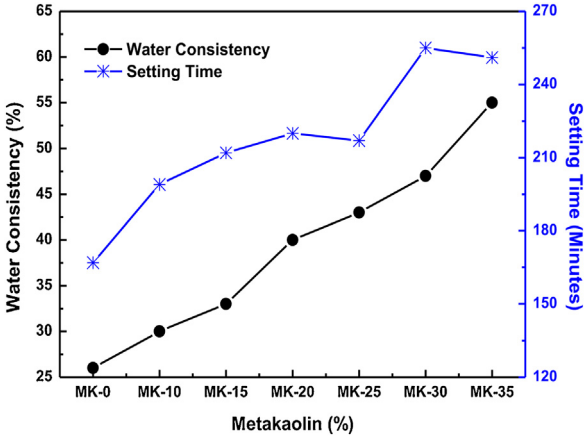

Fig. 8. Water consistency - Setting time relationship.

as the metakaolin content in the cement paste increased. The consistency value of the control paste was 26 % whereas that of 10 % and 15 % metakaolin content were 30 and ~33 % respectively. Mortar paste containing 20 % and 25 % metakaolin had consistency values of ~40 % and ~43 % respectively whereas that of 30 % and 35 % content of metakaolin were also ~47 % and ~55 % respectively. The addition of metakaolin increased the water demand from ~26 % to a maximum of ~55 %. The increase in water demand in PLC-MK is as a result of the large surface area, fine and porous nature of metakaolin. As the metakaolin was gradually increased, the amount of PLC decreased, increasing the effective surface area and affinity of the paste to absorb water, hence, the observation in an increase in water consistency. The results largely agree with the findings observed by El-Diadamony et al. [9], Bediako [34] and Atiemo et al. [35] that cement paste containing metakaolin clays increase the water demand for forming homogenous cement paste.

The initial setting time of the control paste was at 167 min. The addition of metakaolin from 10 to 35 % prolonged the initial setting time. Pastes containing 10–35 % metakaolin content took 199, 212, 220, 217, 255 and 251 min respectively to set. The control sample had the lowest initial setting time while 30 % metakaolin had the highest initial setting time. The reasons for the extended initial setting time of the pastes containing 10, 15, 20 and 30 % metakaolin is the coating of cement particles by metakaolin and dilution of PLC with metakaolin. For the 25 and 35 %MK that showed a lower setting time in comparison to 20 and 30 % MK respectively, the observation can be attributed to the formation of a dense binding phase as reported by brooks et al. [36]. The trend in the results agree with explanations showed in the studies by El-Diadamony et al. [9] Bediako [34], and Brooks et al. [36]. All pastes had their initial setting time greater than the minimum 75 min required for CEM II/ B-L 32.5R in GS 1118:2016 [10].

3.5.2. Water absorption due to capillary action of hardened mortar

The water absorption by the metakaolin mortar bars cured at 3, 7 and 28 days for 24 h are presented in Fig. 9; while the water absorption coefficient (WAC) values are presented in Table 3.

This absorption is mainly due to the capillary action on a cross-sectional area of ~ 1600 mm^2. The results showed a general decrease in the water absorption and water absorption coefficient from the 3 days to 28 days cured samples. The different metakaolin replacement proportions showed a high absorption of water compared to the control for 3 days cured mortar bars. The control after a day absorbed water to a depth of 6.25 mm with WAC of 1.22 $mm/hours^{0.5}$ while the metakaolin replacement proportions absorbed water to an average depth of ~8 mm per day. There was a gradual increase in water absorbed and WAC from 10 to 15 % MK and a decrease from 20 to 35 %MK. For mortar bars cured for 7 days there was a general decrease in absorbed water and WAC in comparison to the 3 days cured mortar. The water absorbed by control was less than the different metakaolin replacement proportions except for 10 % MK. The water absorbed by the control mortar was 6.2 mm per day and 7.2 mm per day for 10 % MK. The other mortars, 15–35 MK % absorbed ~ 6.18, ~ 4.57, ~ 5.49, ~ 4.6, and ~ 4.8 mm per day with their corresponding WAC presented in Table 3. WAC values for the 28 days aged mortars showed that all of the mortars exhibited a significant decrease in absorbed water in comparison to the 3 and 7 days cured prisms. The amount of water absorbed by the control after 28 days curing was less than half the water absorbed at 7 days, thus 3.0 mm per day. 10 %MK absorbed 2.1 mm per day, 15 %MK absorbed 2.5 mm per day, 20 %MK absorbed 3.26 mm per day, 25 %MK absorbed 2.5 mm per day, 30 %MK absorbed 2.6 mm per day and 35 %MK absorbed 1.6 mm per day. 35 % MK had the lowest WAC, with all the different MK mortars performing better than the control, except 20 % MK which had a WAC of 1.55 $mm/hours^{0.5}$. The overall improvement in water absorption by capillary action can probably be attributed to the filler effect of MK, and the formation of cementitious products by the hydration of PLC and MK, which grow and refine pores leading to a more compact mass with less pores to absorb water. The general decrease in water absorbed by capillary action agrees with Badogiannis et al. [15] that increasing MK content improves sorptivity.

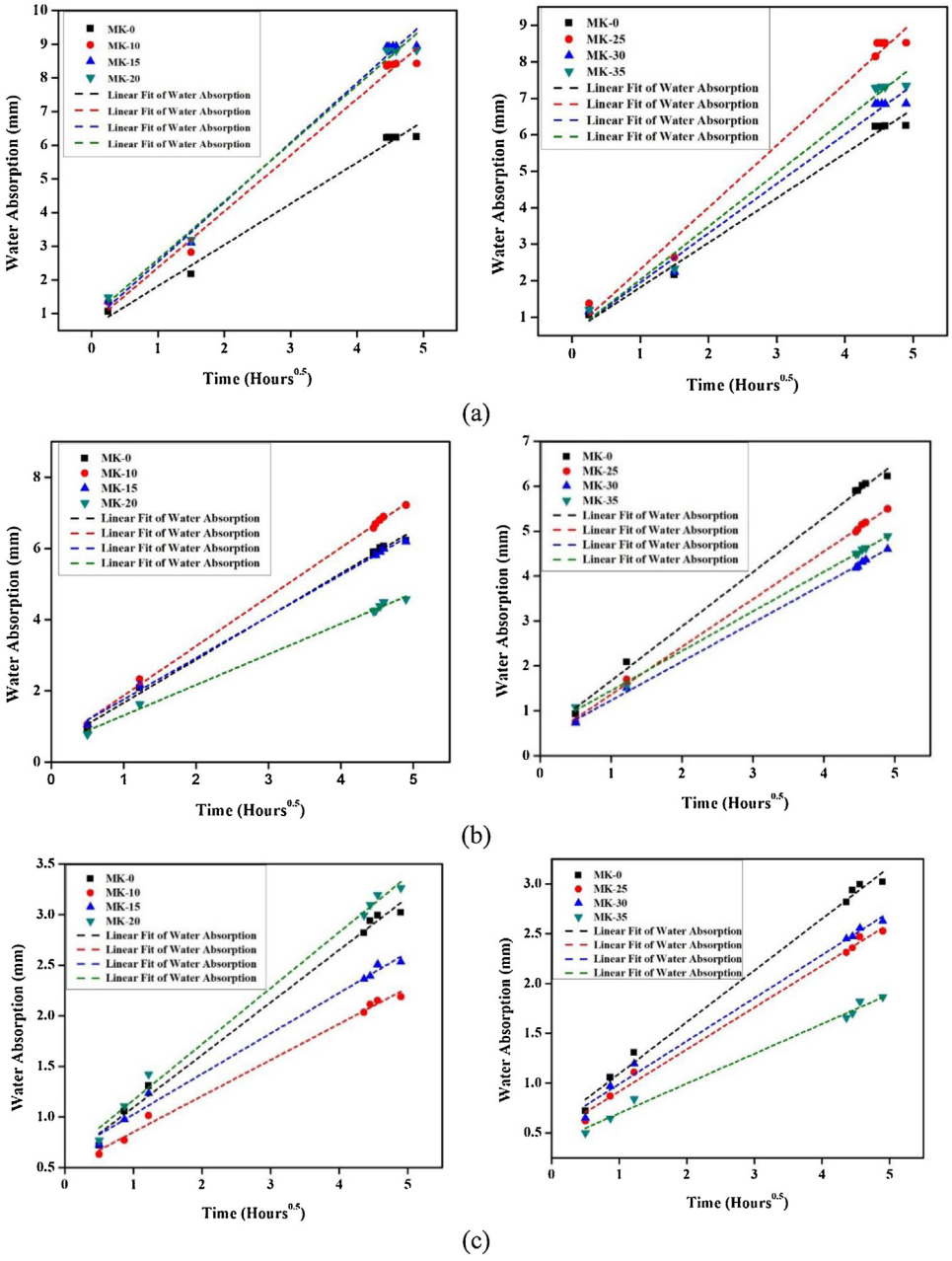

Fig. 9. Water absorption after (a) 3 days (b) 7 days (c) 28 days curing.

3.6. Flexural and compressive strength analysis

The effect of metakaolin on the bending characteristics of the mortar bars is shown in Fig. 10.

There is an appreciable increase in flexural strength of the mortar bars from 3 to 28 days. After three days of curing, the control mortar recorded a flexural strength of ~ 4.31 MPa with the 10–30 % metakaolin replacement showing a gradual increase from ~ 4.22, ~ 4.26, ~ 4.37, ~ 4.71 and ~ 5.23 MPa. At 35 % replacement, there was a decrease in strength to 4.42 MPa. However, the decrease in strength for mortar bar containing 35 % metakaolin is greater than that observed for the control.

The mortar bars cured for seven days showed a reasonable increase in strength in relation to the three days cured bars. The strength of the control was ~ 5.08 MPa. There was an increase in strength from 10 to 20 % metakaolin replacement, with a gradual decrease in strength from 25 to 35 % replacement. The modulus of rupture of mortar bars containing 10–20 % MK was 4.93, 6.30 and 6.46 MPa, while 25–30 % MK was 5.43, 5.29 and 4.56 MPa.

Table 3
Water absorption coefficient values after days of curing.

Sample	Water absorption coefficient(mm/hours$^{0.5}$)			Adj. R^2 Value		
	3 days curing	7 days curing	28 days curing	3 days curing	7 days curing	28 days curing
0MK	1.22252	1.20969	0.5186	0.98948	0.99698	0.99264
10MK	1.666945	1.38304	0.35631	0.98927	0.9985	0.99415
15MK	1.77369	1.16295	0.39975	0.99053	0.99771	0.99057
20MK	1.71948	0.85714	0.55315	0.99048	0.99612	0.99343
25MK	1.666945	1.06098	0.42113	0.98927	0.99889	0.99393
30MK	1.77369	0.86347	0.43165	0.99053	0.99877	0.99148
35MK	1.71948	0.88099	0.29896	0.99048	0.99903	0.99211

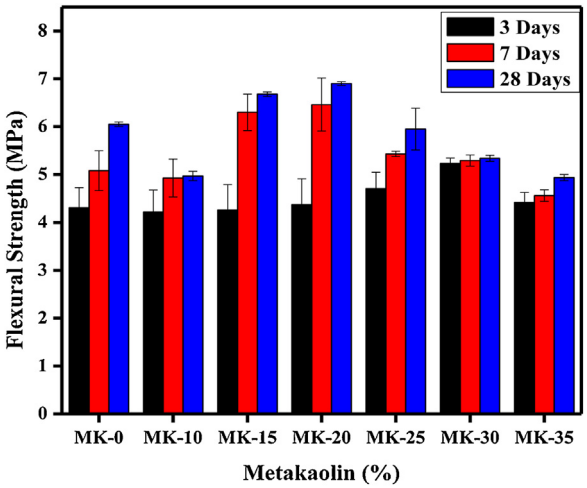

Fig. 10. Flexural strength of mortar prism.

Twenty-eight (28) days cured mortar followed the seven day cured mortar trend. There was an increase in flexural strength from 10 to 20 % and a decline in strength from 25 to 35 % metakaolin replacement. Only 15 and 20 % MK replacement had flexural strength greater than the control. The flexural strength of the 28 days aged mortar was 6.05 MPa for the control. Whereas 10–35 % MK was 4.97, 6.68, 6.90, 5.95, 5.34, and 4.94 MPa respectively.

The flexural strength of the mortar bars after 3 days can probably be attributed to the accelerated hydration and filler effect of the metakaolin [7]. Metakaolin particles serve as nucleating sites for formation of cementitious products while also reducing pores to form a compact and dense mass which enhances strength [9].

After 7 days aging, the pozzolanic effect adds to the 3 days attained strength [7]. The portlandite generated, which was produced from the hydration of cement reacts with metakaolin to produce C-S-H gels which fill, grow and add strength to mortars. At 20 % metakaolin replacement, this effect has probably reached the optimum and hence the strength is greatly enhanced. Beyond 30 % replacement, there was a reduction in strength which could be as a result of the dilution effect of metakaolin, where cement content is reduced and metakaolin increase hence reducing the overall strength.

For the 28 days cured mortar, the increase in strength is as a result of the continuous hydration of clinker minerals and pozzolanic reactivity of metakaolin. For 15 and 20 % metakaolin replacement, there is a balance for which enough clinker minerals have hydrated to produce portlandite for which metakaolin can react with. This balance gives 20 % metakaolin replacement the highest strength among the batch formulations. Generally, when an SCM partially replaces cement it is known as dilution and when the SCM causes a pozzolanic reaction it is known as compensation. The balance of the dilution and compensating effect is responsible for the overall strength of a mortar or concrete mix [37]. The decrease in strength observed in 25–35 %MK can be as a result of the dilution of cement with MK and slow hydration of remaining PLC [7,9]. The results obtained however contradicts with the observations by El-Diadamony et al. [9].

Fig. 11 presents the compressive strength results of the control and metakaolin replaced mortars. The strength values at 3 days of curing showed that aside 35 % metakaolin replacement, all the pozzolan mortars gained higher strength than the control. The recorded strength of the different metakaolin proportions from 10 to 35 % is 18.32, 20.58, 21.45, 19.77, 16.40 and 14.47 MPa. The control recorded a compressive strength of 15.91 MPa.

At 14 days of curing, the control strength was ~24.11 MPa. 10 and 35 % metakaolin mortars obtained lower strength values of 22.18 and 20.25 MPa respectively, in comparison to the control. However, the strength values of 15, 20, 25 and 30 % metakaolin content were 24.11, 26.34, 28.29 and 27.51 MPa respectively, which were all higher than the control mortar.

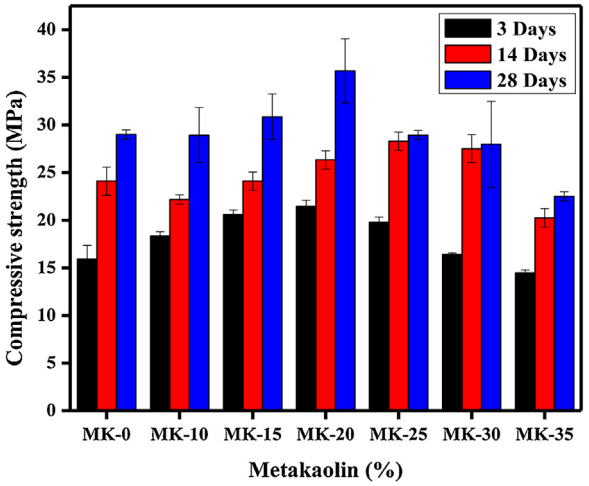

Fig. 11. Compressive strength of mortar cube.

For the 28 days curing strength, the control mortar, 10 and 25 % metakaolin mortars had strength values of ~29.0 MPa. Whereas 15 and 20 % metakaolin mortars had strength values of 30.86 and 35.69 MPa which were greater than the control strength value. Lower strength values were recorded for 30 and 35 % metakaolin replacement in comparison to the control, which was 27.97 and 22.51 MPa respectively.

The early strength observed after 3 days can be attributed to the accelerated hydration of PLC and filler effect of metakaolin. Metakaolin in the mortars served as nucleating sites to help speed up hydration, also the metakaolin filled the pores forming a dense mixture, with both effects causing an increase in strength [7,9]. As the metakaolin content increases, a decrease in compressive strength is expected. The decrease in 30 and 35 % MK mortar strength can be attributed to the dilution effect of metakaolin, where the metakaolin content increases and PLC content reduces causing a decrease in strength of the mortar [9].

The strength gained at 14 and 28 days can also be attributed to the pozzolanic effect of metakaolin [7,9]. At this stage, enough or all portlandite has been consumed by metakaolin in the mixture to produce hydrated products which grow and fill pores producing a more dense mortar with increased strength. The highest strength gained by the 20 % MK could be due to a balance between the accelerated hydration and pozzolanic effect of metakaolin. The decreased strength observed at 35 % MK is as a result of the dilution effect of MK. MK quantity had probably surpassed the minimum replacing threshold [7,9]. Again, the results showed that there is no significant increase in compressive strength after 14 days for 25–35 % metakaolin in comparison to 28-day strength. This results agrees partially with observations that after 14 days, there is no significant increase in compressive strength for samples that contain metakaolin up to 15 % [8], in contrast, 20 % MK continued to increase in strength.

4. Conclusion

The effects of partially replacing Portland Limestone Cement with metakaolin produced from Teleku Bokazzo Kaolin in the Western Region of Ghana has been reported in this study. The kaolin was calcined at ~650 °C into the reactive metakaolin with a dehydroxlation of the kaolinite. The metakaolin became reactive and hence its potential as a suitable supplementary cementitious material. Calcium silicate hydrate, calcium aluminium silicate hydrate, portlandite, calcite and ettringite were the major hydrated compounds formed in the hydrated pastes. The water demand increased with the addition of metakaolin to the cement paste in the water consistency test. The water demand increased from ~30 % to ~55 % in the 10 % MK – 35 %MK replacement in comparison to 26 % by 0 % MK. The addition of metakaolin decreased the amount of PLC and increased the effective surface area and potential of the paste to absorb water, hence, the observation in the reported increase in water consistency. The study again showed that, the addition of metakaolin generally increased the setting time of the cement pastes. For instance, supplementing PLC with metakaolin increased the setting time of the cement paste by ~35 %, i.e. from ~167 min in the reference sample to ~255 min in the 30 % MK replacement. It was also discovered that all the cement pastes met the ~75 min' minimum requirement for setting according to the Ghana standards for mortar pastes. The mechanical properties were observed to increase with increasing metakaolin replacements of up to 20 %. For instance, a 20 % replacement of Portland limestone cement recorded the highest bending strength of ~ 6.9 MPa after 28 days of curing whereas the compressive strength of the mortar cubes increased to ~36 MPa. According to Ghana Standards (GS 1118:2016), the minimum compressive strength is ~32.5 MPa after 28 days. Therefore, from the results obtained, it is suggested that ~20 % replacement of Portland limestone cement with Teleku Bokazzo metakaolin can be very suitable for construction applications.

Declaration of Competing Interest

Authors declare that there is no conflict of interest in this work.

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