

Mechanical Properties of Dehydroxylated Kaolinitic Clay in Self-Compacting Concrete for Pavement Construction

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Abstract The high increase in the cost of cement has led to a reduction in concrete production in most developing and under-developed countries. Therefore, the need for a sustainable and cost-effective substitute for cement is necessary. This research focused on the application of dehydroxylated kaolinitic clay in the production of self-compacting concrete for pavement construction. The elemental and oxide composition of the cementitious material (cement and metakaolin) was assessed using atomic absorption spectrometry and a scanning electron microscope was used to determine the particle geometry. Six mixtures of SCC with 0%, 5%, 10%, 15%, 20% and 25% metakaolin replacement were incorporated into this concrete mix. The passing ability, segregation ability and the flowing ability of the fresh concrete were assessed. The strength properties of the various mixtures (compressive and flexural) of the samples were also assessed at 3, 7, 14, and 28 days. The rheological properties showed that the addition of dehydroxylated kaolinitic clay higher than 10% showed poor rheology. However, percentages greater than 15% gave a reduction in compressive strength and flexural strength. In a bid to encourage

sustainability in road construction and adopt the use of eco-friendly material, metakaolin is a viable material.

Keywords Pavement · Self-compacting concrete · Metakaolin · Mechanical strength · Rheology

1 Introduction

In line with the campaign for sustainability and the use of eco-friendly material in concrete road construction, the use of dehydroxylated kaolinitic clay in self-compacting concrete was espoused in this research. Dehydroxylated kaolinitic clay (DHKC) is obtained when kaolinitic clay is heated to a temperature of 650–900 °C. At this temperature, dehydroxylation takes place which is an endothermic process to form DHKC or dehydroxylated kaolin [1]. This material reacts chemically with hydrating cement due to its pozzolanic properties to form a modified paste microstructure with similar pozzolanic properties as silica fume, rice husk ash, fly ash, etc. [2]. In line with the use of other cementitious material, the use of DHKC has been found to have a good combination of properties [3]. Additionally, using this mineral material improved the workability, durability, strength, and cohesion of concrete structures [4].

Self-compacting concrete (SCC) is an innovation in the concrete construction industry. It is a quiet, cost-effective, energy saving, and low carbon footprint concrete due to the absence of vibrators [5]. According to the same author, SCC offers many advantages for the precast, prestressed concrete industry and cast-in-place construction such as low noise, less labor, faster construction, high strength etc. A lot of research has been done by [6–17] to mention a few on the

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application of self-compacting concrete. The use of DHKC in concrete production is enormous, and its application for structural application has proven to be satisfactory based on the strength and durability properties, but a dearth of literature exists on its use for pavement purposes. According to research by [18–21] strength and durability of concrete containing DHKC improved by assessing both chloride and sulfate attack. Accordingly, [1] worked on the combined effect of DHKC and silica fume on concrete strength. However, [22] and [23] developed statistical models for predicting the consistency of concrete incorporating Portland cement, fly ash and DHKC. The research of [24] experimentally studied a set of parameters of high-performance concrete (HPC) with DHKC. Researchers have reported [25] the synergistic effect of the use of fly ash, iron oxide, and DHKC as supplementary cementitious material in various proportions. They adopted the use of ordinary Portland cement (OPC), and the developed concrete was for structural application. However, this research focuses on the use of Portland limestone cement which conforms to [25] in the production of self-compacting concrete for road construction.

This is because of the dearth of literature on the application of self-compacting concrete in rigid pavement incorporating DHKC. The rigid pavement has numerous advantages over the flexible pavement but requires higher construction cost, and hence the replacement using a mineral with low-cost becomes paramount. This is required to make concrete affordable especially in developing countries with an abundance of kaolin.

This research assessed the mechanical properties of DHKC self-compacting concrete. Also, the use of Portland limestone cement was used instead of the conventional ordinary Portland cement (OPC). The goal of this research is to promote sustainability in pavement construction and reduce the cost of cement which is one of the most expensive constituents of concrete. Hence, this study assessed the mechanical properties of DHKC in SCC for pavement construction.

2 Methodology

2.1 Materials

Locally available Portland limestones cement type II conforming to [25] and [26] was used in the study. The DHKC used in this research was sourced from South Western Nigeria. Atomic absorption spectrometry was used in assessing the compound composition of the cement. SEM was used in assessing the elemental composition, particle geometry and the microstructure of the Portland limestone cement

and the DHKC. Aggregates play a key role in concreting as suggested by [26, 27], and [28].

In this research, the coarse and fine aggregates of size 4.5 and 19.5 mm were used throughout the research. A polycarboxylate-based high-range water-reducing admixture (HRWRA), super-plasticizer according to EFNARC, [29] was used in this research.

2.2 Dehydroxylation of the Kaolinitic Clay

The dehydration process of kaolinitic clay to a very large extent depends on the particle size and crystallinity. According to [30, 31] the use of crystal size from scanning electron microscopy (SEM) is paramount for the characterization and application of kaolin. Comparative assessment of the crystal size was done according to the result obtained from the particle geometry of the kaolinitic clay and dehydroxylated kaolinitic clay.

Additionally, the physical beneficiation and characterization of the kaolinitic clay was done to ascertain the basic features like structural area, pore-volume sizes, circularity, convexity, etc. as this to a very large extent affects the structural properties and the characteristic bonds. The outcome of [31] using the same source of kaolin showed that 750 °C was the appropriate dehydroxylation temperature which was adopted in this research.

2.3 Mix Design

Mixing of ingredients and proportions were done according to EFNARC, [32]. The design matrix of this research involved varying the quantity of cement and DHKC. Six concrete mixes were compounded with DHKC replacement at 0%, 5%, 10%, 15%, 20% and 25%. A polycarboxylate based high-range water-reducing admixture (HRWRA), CONPLAST super-plasticizer according to the EFNARC [26] specification was used in improving the workability. One unit of plain SCC mixture was designed at a w/c ratio of 0.45 with fine and coarse aggregate prepared according to the rational mix design method. The rheology of the concrete was assessed using the slump cone, V-funnel and the L-box apparatus according to the specification of [32] and [26]. This was used in assessing the passing ability, segregation ability and the flowability of the concrete mixtures with 0%, 5%, 10%, 15%, 20% and 25% replacement of DHKC. In a bid to attain the desired workability using the [31] approach several trials were made varying the water-cement ratio and superplasticizer dosage while the mass of fine and coarse aggregate was kept constant. Molds of dimension 150 mm × 150 mm × 150 mm and 100 mm × 400 mm × 100 mm were used for both compressive and flexural tests,

respectively with oil smeared on the inside of the mold to avoiding sticking after obtaining a uniform and consistent mixture. The concrete was mixed and cured by [33, 34].

2.4 Procedure

In this research, DHKC was heated to a temperature of 750 °C according to the study of [31] using the same source of kaolin and the same procedure. The DHKC was used as a replacement for the Portland limestone cement at 0%, 5%, 10%, 15%, 20% and 25% percentage of the dry weight of the cement. The proportions of the SCC mixtures are documented in Table 1. The total paste volume was kept constant for all SCC mixtures. This parameter is the sum of the volumes of the cementitious material, filler, water, and total entrained and entrapped air content. The total weight of the cementitious material (cement and DHKC) content was kept constant to provide a relatively high volume fraction of fine material that conforms to conventional SCC mixture design guidelines as specified by [32]. The sand to the total aggregate mass ratio (S/A) was also kept constant for all mixtures to ensure uniformity. The coarse and fine aggregates were first mixed with one-third of the water in the mixer, and then the cement and DHKC were added to the main mixer. At the final stage, the HRWRA was mixed with the remaining water in a separate container and added to the main mixer. The mix proportion of the constituents of the SCC is given in Table 1.

3 Result and Discussion

3.1 Chemical Composition of the Cement, Kaolin and DHKC

The chemical composition of the basic oxides of the cement used is shown in Table 2. Comparison of the result with the oxide composition of a similar brand of cement by

Table 2 Oxide composition of the Portland limestone cement

| Compounds | Yahaya 2009 | | Faleye 2009 |
|--------------------------------|-------------|-------|-------------|
| SiO ₂ | 19.07 | 18.02 | 20.62 |
| CaO | 64.22 | 51.67 | 59.6 |
| FeO ₂ | 8.72 | 10.5 | 3.22 |
| Al ₂ O ₃ | 2.96 | 1.25 | 6.01 |

[26, 35] and [36] was also done (Table 2). The result indicated that the oxide composition followed a similar trend. It also conformed to the standard specification for CEM II/AL according to [25].

3.2 Particle Geometry

DHKC is neither the by-product of an industrial process nor is it entirely natural. It is derived from a naturally occurring mineral and is manufactured especially for cementing applications. DHKC is produced under carefully controlled conditions to refine its color, remove inert impurities, and tailor particle size so that, a much higher degree of purity and pozzolanic reactivity can be obtained. The particle geometry is shown in Table 3. The particleometry of DHKC showed a similar trend as cement. The result showed that the particle size reduced after dehydroxylation.

3.3 Dehydroxylation of Kaolinitic Clay

After the dehydration process, the kaolin lost its crystal structure and became a mainly amorphous material. This also reduced the particle size (Table 3). The microstructure of the DHKC and the kaolin is as shown in Figs. 1 and 2.

3.4 Physical Properties of the Aggregates

The coarse and fine aggregate of size 4.5 and 19.5 mm were used all through the research to ensure a homogeneous mix

Table 1 Mix proportion

| Mixtures | Replacement % | Cement Kg | DHKC Kg | Fine aggregate Kg | Coarse aggregate Kg | Water Kg | SUPERLAST+VMA Kg | Total concrete Kg |
|----------|---------------|-----------|---------|-------------------|---------------------|----------|------------------|-------------------|
| Mix 1 | 0.0 | 394 | 0 | 670 | 591 | 158 | 5 | 1,838 |
| Mix 2 | 5.0 | 374 | 20 | 670 | 591 | 158 | 5 | 1,838 |
| Mix 3 | 10.0 | 355 | 39 | 394 | 591 | 158 | 5 | 1,838 |
| Mix 4 | 15.0 | 335 | 59 | 394 | 591 | 158 | 5 | 1,838 |
| Mix 5 | 20.0 | 315 | 79 | 670 | 591 | 158 | 5 | 1,838 |
| Mix 6 | 25.0 | 293 | 99 | 670 | 591 | 158 | 5 | 1,838 |

Table 3 Particle geometry of the cementitious material

| Property | Kaolin | | DHKC | | Cement | |
|-------------------------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
| | Median | Average | Median | Average | Median | Average |
| Circle diameter | 23.1 μm | 24.4 μm | 78.4 μm | 73.1 μm | 78.4 μm | 73.1 μm |
| Major axis | 29.9 μm | 32.5 μm | 88.6 μm | 94.2 μm | 86.6 μm | 92.2 μm |
| Minor axis | 17.3 μm | 18.6 μm | 54.8 μm | 60.7 μm | 53.3 μm | 59.8 μm |
| Circumference | 97 μm | 101 μm | 431 μm | 444 m | 424 μm | 441 μm |
| Convex hull | 82.2 μm | 91.3 μm | 315 μm | 299 μm | 314 μm | 297 μm |
| Circumscribed circle diameter | 32.9 μm | 36.9 μm | 120 μm | 119 μm | 119 μm | 116 μm |
| Area (μm^2) | 418 | 502 | 5.11E+03 | 4.88E+03 | 4.84E+03 | 4.68E+03 |
| Volume (μm^3) | 6.4E+03 | 9.43E+03 | 2.61E+05 | 2.71E+05 | 2.53E+05 | 2.69E+05 |
| Pixel count | 1493 | 1794 | 11391 | 11009 | 11374 | 11007 |
| Aspect ratio | 0.586 | 0.606 | 0.792 | 0.685 | 0.786 | 0.673 |
| Circularity | 0.586 | 0.598 | 0.293 | 0.362 | 0.283 | 0.326 |
| Convexity | 0.915 | 0.906 | 0.691 | 0.729 | 0.676 | 0.718 |
| Elongation | 0.403 | 0.394 | 0.218 | 0.332 | 0.208 | 0.327 |
| Grayscale | 178 | 178 | 138 | 136 | 136 | 135 |

following the empirical and rheological mix methodology for self-compacting concrete. Correspondingly, the water absorption and specific gravity test showed a satisfactory result as specified by [36] for rigid pavement construction (Table 4).

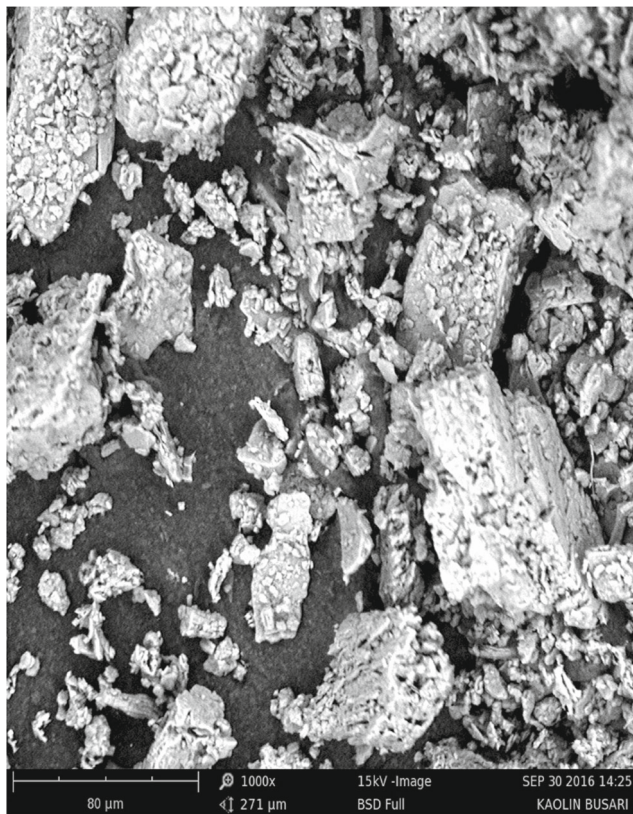
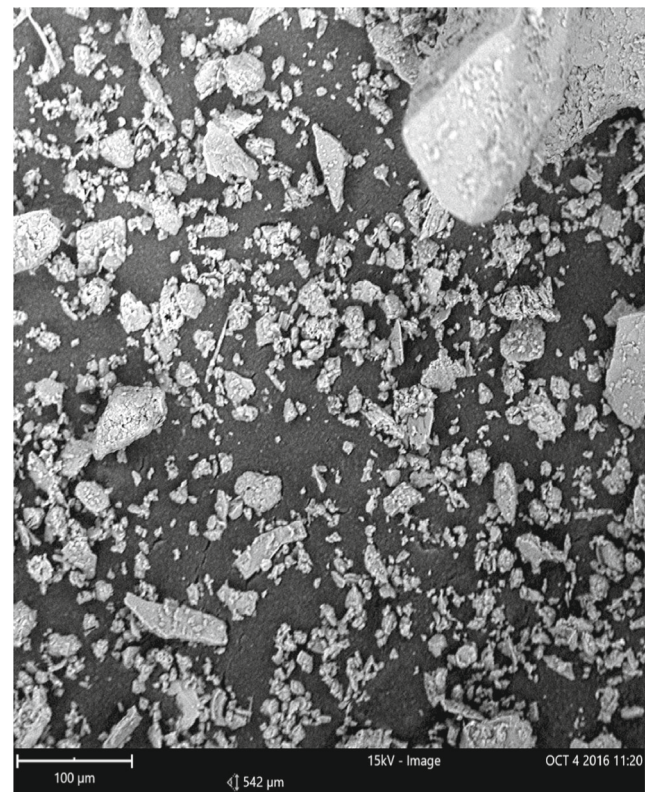
**Fig. 1** Scanning electron micrograph of DHKC**Fig. 2** Scanning electron micrograph of kaolin

Table 4 Physical properties of fine aggregate

| | Fine aggregate | | |
|---------------------------|----------------|----------|----------|
| | Sample 1 | Sample 2 | Sample 3 |
| Specific gravity | 2.55 | 2.5 | 2.54 |
| Apparent specific gravity | 2.71 | 2.61 | 2.63 |
| Water absorption average | 2.39 | 1.66 | 1.33 |

the acceptable criterion for the V-funnel result is between 6 and 12 seconds. Based on this criterion, the SCC mix with DHKC addition from 15% and above did not satisfy this criterion (Fig. 3). This may be because the incorporation of high DHKC content affects the viscosity of the mix.

3.5.2 Passing Ability Test L-Box

L-box height ratios were measured and the corresponding outcomes are presented in Fig. 4. As against the V-funnel and slump flow which were unsatisfactory at 15% DHKC addition, the result showed that at 10% and higher percentage replacement of DHKC the L-box result became unsatisfactory [32].

3.5.3 Filling Ability (Slump Flow)

The slump flow test was used to determine the ability of the SCC to flow in a non-restricted condition. Factors affecting the results of the T_{50} time are the amount, shape and size distribution of aggregates and also the viscosity and amount of paste. The mix with the lowest T_{50} time is the SCC with no DHKC addition (Fig. 5). With the replacement of DHKC above 10%, the T_{50} value became unsatisfactory based on the EFNARC [32] specification.

3.6 Strength Properties

3.6.1 Compressive Strength

The result of the analysis showed that the addition of DHKC to concrete showed an improved strength up to 15% replacement (Fig. 6). Upon further addition, the compressive

strength began to reduce. The improvement in the compressive strength may be attributed to the fact that DHKC reduces the size of pores in the cement paste and transforms many finer particles into discontinuous pores, therefore decreasing the permeability of the concrete. Moreover, DHKC possesses high pozzolanic reactivity, which can react with portlandite, $\text{Ca}(\text{OH})_2$, released during the hydration of Portland cement and the micro-filler effect which improved the packing of cement matrix [37]. The highest strength across all ages was recorded at 5% addition of DHKC. However, at 10% and 15% (Fig. 6), the strength was still higher than the control. However, a decrease in the compressive strength was noticed when compared with 0% DHKC at percentages higher than 15%. It is worth noting that the decrease in strength at 10% and 15% still satisfies the recommendation by [26] for rigid pavement construction.

Conversely, it was noticed that at a higher percentage of DHKC (20% and above) as seen in Fig. 6 with the equations governing the trend in Table 5. The compressive strength of the samples decreased across the board. This may be because the quantity of the DHKC was too much for the $\text{Ca}(\text{OH})_2$ released during hydration as suggested by [37] and this had an inverse effect on the packing of the cement matrix as asserted by the same author.

3.6.2 Equations

3.6.3 Flexural Strength

The use of DHKC becomes a suitable ingredient in the production of concrete of more than 4.0 MPa or where service environments, exposure conditions, and life cycle cost considerations dictate the use of high-performance concrete. The flexural property of the mixtures at the various replacements of DHKC showed a similar pattern with the compressive strength. At 5% addition of DHKC, the strength was also optimized (Fig. 7). At that percentage, the DHKC functions by converting an undesirable by-product of the cement hydration process, calcium hydroxide (free lime), to various forms of calcium aluminate [37].

The decrease in the flexural strength was noticed at 15% addition of DHKC. At percentages higher than this, the

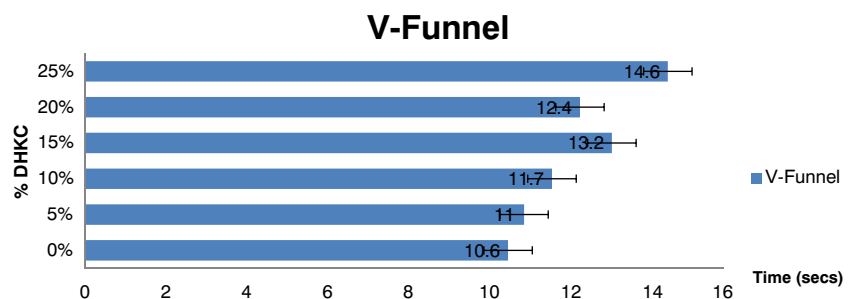
Fig. 3 V-funnel results

Fig. 4 L-box test result

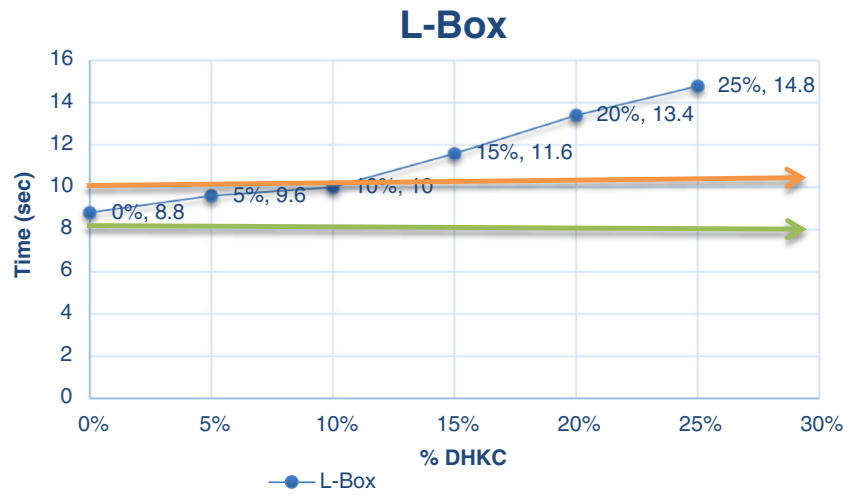


Fig. 5 Slump flow results

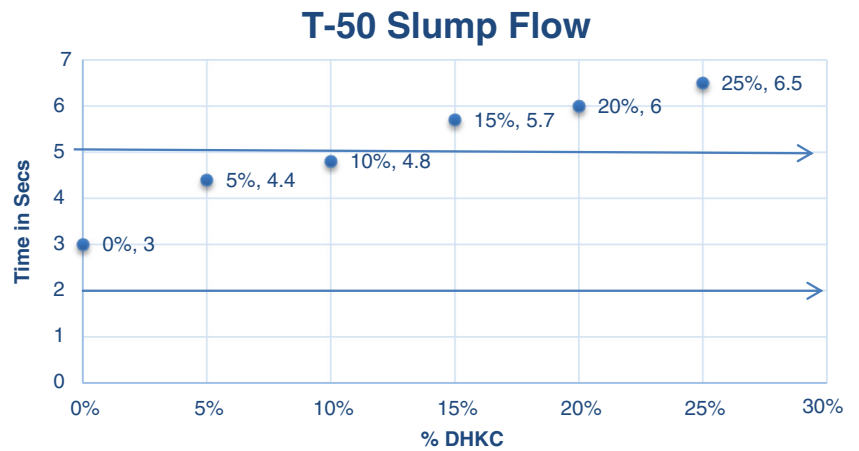


Fig. 6 Effect of age on compressive strength

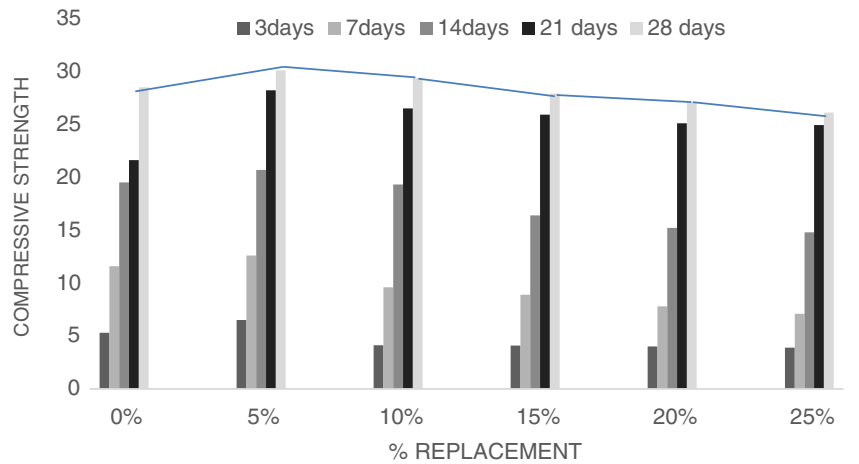


Table 5 Equations of the trend

| Age | Compressive strength | R ² |
|---------|--------------------------------|----------------|
| 3 Days | $0.0004x^2 + 0.0189x - 0.0286$ | 0.9959 |
| 7 Days | $0.013x^2 + 0.1111x + 2.54$ | 0.9907 |
| 14 Days | $0.0301x^2 + 0.3805x + 2.64$ | 0.5655 |
| 21 Days | $0.0075x^2 - 0.1491x + 5.264$ | 0.7822 |
| 28 Days | $0.0124x^2 + 0.1053x + 5.315$ | 0.8588 |

material does not contribute to the strength of the concrete. The flexural strength meets the criteria for road construction, according to [38].

3.6.4 Descriptive Statistical Analysis of the Input and Output Parameters

The mean and median values for the age of concrete, cement quantity, DHKC replacement and the compressive strength are in line with the criteria for random time series. A normality test was conducted for the variables in line with the normal (random time series) trend. Jarque-Bera chi-square values of the variables were greater than the probability values (Table 6). Hence the variables employed are normally distributed.

3.7 Descriptive Statistics

The standard deviations of 51.3, 34.68, 33.96 and 10.97 for age, cement, DHKC and compressive strength are far from the mean. This to a very large extent suggests a high degree of variability of the series around their mean value. The skewness indicator revealed that age and DHKC are positively skewed while compressive strength and cement

Table 6 Descriptive statistics

| | Age | Cement | DHKC | Compressive_strength |
|--------------|----------|-----------|----------|----------------------|
| Mean | 54.55556 | 344.2667 | 49.25000 | 24.58296 |
| Median | 28.00000 | 344.9500 | 49.25000 | 27.01000 |
| Maximum | 150.0000 | 394.0000 | 98.50000 | 42.10000 |
| Minimum | 3.000000 | 292.5000 | 0.000000 | 4.120000 |
| Std. Dev. | 51.30400 | 34.68610 | 33.96007 | 10.97528 |
| Skewness | 0.713715 | -0.055734 | 0.000000 | -0.439625 |
| Kurtosis | 2.012705 | 1.770820 | 1.731429 | 2.193046 |
| Jarque-Bera | 6.777697 | 3.427443 | 3.620865 | 3.204573 |
| Probability | 0.033748 | 0.180194 | 0.163583 | 0.201435 |
| Sum | 2946.000 | 18590.40 | 2659.500 | 1327.480 |
| Sum Sq. Dev. | 139501.3 | 63765.66 | 61124.17 | 6384.212 |
| Observations | 54 | 54 | 54 | 54 |

content are negatively skewed. Besides, the kurtosis value indicates that for all the F_{cu} and age values, cement and DHKC addition are platykurtic.

3.8 Mathematical Model

The least squares method was used in establishing the mathematical relationship between the estimated variables (predictive) and the effect on the compressive strength. The T-value showed that the variation under consideration is significant at 10%. The T statistics further revealed that cement, DHKC, and age are important factors to be considered in assessing the compressive strength of concrete. The coefficient of the cement was 0.495 which implies

Fig. 7 Flexural Strength at the Various DHKC replacements

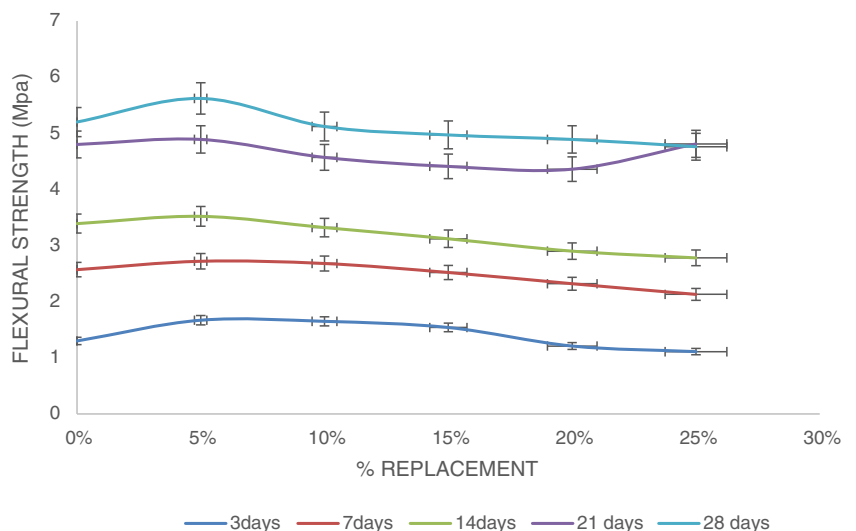


Table 7 Parameters

| Variable | Coefficient | Std. Error | t-Statistic | Prob. |
|----------|-------------|------------|-------------|--------|
| C | 15.5672 | 412.4028 | −0.425718 | 0.6721 |
| Age | 0.159617 | 0.018479 | 8.637692 | 0.0000 |
| Cement | 0.495775 | 1.045231 | 1.574321 | 0.6373 |
| DHKC | 0.421595 | 1.067577 | 1.794908 | 0.6946 |

that a unit increase in the cement content, will lead to a 0.495 increment in the compressive strength. Also, a unit increase in DHKC content and age will lead to a 0.421 and 0.15 increase in the compressive strength. The variables considered all showed a positive relationship with the compressive strength. This implies that the higher the cement, DHKC, and age the higher the compressive strength. R^2 of 0.686 was recorded for the relationship which showed that the explanatory power of the independent variable is satisfactory as seen in Table 7.

4 Conclusion

This research assessed the strength properties of self-compacting concrete using DHKC as an additive. The rheology and strength properties of six concrete mixes were examined at 3, 7, 14, 21, 28 and 56 days. The result of the analysis showed that:

1. The use of dehydroxylated kaolinitic clay in self-compacting concrete has been extended to rigid pavement construction.
2. The rheological properties of the mixtures were found not to be satisfactory at 15% and above addition of DHKC by dry weight of Portland limestone cement.
3. Also, at 5% addition of DHKC, the highest compressive and flexural strength was recorded at all ages.
4. Higher percentages of DHKC (10% and 15%) improved the strength when compared to the control mix.
5. However, reduction in the flexural strength of the samples was recorded at 20% DHKC addition.

It can be concluded that in a bid to optimize strength, 5% addition of DHKC would be required for construction that involves the use of Portland limestone cement. However, where the reduction in cement content has required the addition of DHKC, up to 15% is effective.

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