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Data analysis and model development for sulphate attack strength loss index in Chikoko blended concrete

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Abstract

Sulphate attack poses a significant durability challenge for concrete, particularly in environments with high sulphate concentrations. This study explores the efficacy of Chikoko, a natural pozzolan from the Niger Delta region, as a supplementary cementitious material to enhance concrete's sulphate resistance. The objective is to develop a predictive model for the strength loss index (SLI) of Chikoko-blended concrete exposed to sulphate attack. Concrete samples incorporating various Chikoko contents (0%, 5%, 10%, 15%, 20%) were immersed in a 5% sodium sulphate solution for durations of 7, 21, 28, 90, and 120 days, and subjected to different calcination temperatures. Compressive strength tests were conducted, followed by data analysis using ANOVA and regression techniques to identify significant factors and construct the predictive model. Results reveal that Chikoko substantially enhances sulphate resistance, with a 20% Chikoko replacement demonstrating the least strength loss. The predictive model developed from this study accurately estimates the SLI, providing a valuable tool for engineers to design durable concrete structures in sulphate-rich environments. These findings suggest that Chikoko is an effective and sustainable supplementary cementitious material, contributing to improved concrete durability and performance in aggressive sulphate conditions.

Keywords: Compressive Strength; Sulphate attack; Chikoko, blended; Concrete; Strength loss index

1. Introduction

Concrete is the most widely used construction material globally due to its versatility, strength, and durability. However, one of the significant durability challenges it faces is sulphate attack, a chemical reaction between sulphate ions and components of hardened concrete. This reaction leads to the formation of expansive products like ettringite and gypsum, causing internal stresses, cracking, spalling, and ultimately, loss of structural integrity. The impact of sulphate attack is particularly pronounced in environments such as coastal areas, industrial regions, and places with sulphaterich soils or groundwater. Sulphate attack occurs when concrete is exposed to sulphate ions present in soil or water, leading to the formation of expansive products such as ettringite and gypsum. These products cause internal stresses and cracking, ultimately leading to a loss of strength and structural integrity [1].

Concrete production has significant environmental impacts, primarily through two major channels. The first is the emission of greenhouse gases during the manufacturing of Portland cement (PC). It is estimated that the production of one tonne of PC generates approximately one tonne of CO2, contributing about 5% of global CO2 emissions [1]; [2]. The second major environmental impact is the depletion of natural reserves of traditional crushed rock aggregate and river sand, leading to the scarcity of these essential materials. To align with sustainable development goals and the need for environmentally friendly concrete production, extensive research has been conducted on the use of industrial wastes and by-products as replacements for either aggregate or cement in concrete. Several materials have already been

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incorporated into concrete production, including fly ash [3] ground granulated blast furnace slag (GGBS) [4], [5]; [6], silica fume [7], recycled concrete [8] and quarry sand [9]; [6]

Chikoko, a naturally occurring clay material found in the Niger Delta region of Nigeria, has recently garnered interest as a potential supplementary cementitious material (SCM) due to its pozzolanic properties. Previous studies have indicated that Chikoko, when processed and used as a partial replacement for OPC, can enhance various properties of concrete, including its mechanical strength and durability [10].Chikoko is a type of clay that has been shown to possess pozzolanic properties, making it a potential candidate for use in concrete [11]; [12],[13]; [14]. The pozzolanic reaction between Chikoko and calcium hydroxide, a byproduct of cement hydration, results in the formation of additional calcium silicate hydrate (C-S-H) gel, which enhances the strength and durability of concrete [15]; [16].

Despite the promising properties of Chikoko, limited research has been conducted on its effectiveness in improving the resistance of concrete to sulphate attack. Most studies have focused on traditional SCMs, leaving a gap in the understanding of how Chikoko-blended concrete behaves under sulphate exposure. This research aims to address this gap by investigating the strength loss of Chikoko-blended concrete when subjected to sulphate attack and developing a predictive model for the strength loss index (SLI).

To mitigate the effects of sulphate attack, various strategies have been explored, including the use of sulphate-resistant cements and SCMs. SCMs, such as fly ash, slag, and natural pozzolans, improve concrete's resistance to sulphate attack by refining its pore structure and reducing permeability, thus limiting the ingress of sulphate ions [17]. These materials contribute to improved performance by refining the pore structure, reducing permeability, and enhancing the chemical resistance of concrete.

The strength loss index is a crucial parameter that quantifies the reduction in compressive strength of concrete due to sulphate attack. Developing a reliable model for predicting SLI can provide valuable insights for engineers and researchers in designing concrete mixtures with enhanced durability. By incorporating Chikoko as an SCM, this study not only contributes to the knowledge of sulphate resistance but also promotes the use of sustainable and locally available materials in concrete production. The specific objectives of this study are to evaluate the strength loss in Chikoko-blended concrete exposed to sulphate solutions, analyze the data to determine significant factors affecting sulphate attack resistance, develop a predictive model for the SLI, and validate the model using experimental data. The findings of this research will provide a comprehensive understanding of the potential of Chikoko in enhancing the durability of concrete and offer practical solutions for construction in sulphate-rich environments.

2. Material and methods

2.1. Materials

Cement: Ordinary Portland Cement (OPC)

Chikoko: Locally sourced from the Niger Delta region, processed to a fine powder.

Figure 1 Grinding of the chikoko clay

Aggregates: Standard fine and coarse aggregates. The grading properties of the quartzite used in Amassoma and Abraka river sand conformed to BS EN 12620:2013.

Water: In most cases, potable water is all that is required for usage in the production of concrete. The water came from the Department of Civil Engineering at Niger Delta University, which was where it was obtained. This did not contain any potentially harmful pollutants and was of an appropriate standard for consumption.

Sulphate Solution: Prepared using sodium sulphate to simulate sulphate-rich environments.

2.2. Experimental Test Methods

2.2.1. Mix Proportions

Concrete mixes were prepared with varying proportions of Chikoko replacing OPC at 0(0%, 5%, 10%, 15%, 20%) by weight. The Chikoko was Calcined at a range of temperatures from atmospheric, (200°C) to 800°C

2.2.2. Sample Preparation

Before being put through the calcination process, the Chikoko that was collected from Okrika was sun-dried for a period of seven days in order to remove any trapped moisture.

2.2.3. Mixing and Casting

Concrete samples were prepared by mixing the constituents in a concrete mixer according to the specified mix proportions. The mixing process involved the thorough blending of cement, aggregates, water, and Chikoko to achieve a uniform consistency. Once the mixture attained the desired workability, it was cast into standard molds, typically measuring 150 mm x 150 mm cubes, and compacted using a vibrating table to minimize voids and ensure uniform density. The cast specimens were then covered with plastic sheets to prevent moisture loss and placed in a curing chamber maintained at standard curing conditions (typically 20 \pm 2°C and >95% relative humidity) for a curing period of 28 days.

2.2.4. Sulphate Resistance Test

The sulphate resistance of the concrete samples was assessed through a rigorous testing protocol following established standards and methodologies. A 2.5% sodium sulphate solution was prepared in accordance with the procedures outlined by [18] specifications. Concrete cubes measuring 150 mm x 150 mm were selected for testing. Following the curing period, the specimens were submerged into the prepared 2.5% sodium sulphate solution for exposure periods of 28 days, 56 days, and 90 days, respectively. The immersion durations were selected to simulate real-world conditions and assess the long-term performance of the concrete under sulphate attack.

Subsequently, the cured specimens were immersed in a 2.5% (25 g/l) sodium sulphate solution at laboratory temperature (23 ± 2°C) for a maximum duration of 90 days. The immersion period was selected to simulate long-term exposure to sulphate-rich environments. Throughout the testing period, the specimens were visually inspected for any signs of deterioration, such as cracking, spalling, or surface degradation. Additionally, changes in mass and compressive strength were monitored at regular intervals to assess the extent of sulphate attack. The specimens were weighed prior to soaking as well as after soaking. The loss in weight and strength were calculated as follows;

Weight Loss (
$$
\%
$$
) = $\frac{Wt.of Conc.in Fresh$ Water - Wt of Conc.in sulphate solution
Wt.of Conc.in Fresh Water (1)

$$
SISLI (\%) = \frac{Str.of Conc.in\,West - Str.of Conc.in\,sulphate\,solution}{Str.of\,Concin\,Fresh\ water}
$$
 (2)

Where; SISLI = Sulphate induced strength loss index (%)

2.2.5. Compressive Strength Tests

Compressive strength tests were conducted to evaluate the mechanical properties of the concrete samples exposed to sulphate attack. The tests were performed using a compression testing machine with a capacity of 2000 kN, following the procedures outlined in [19] standards.

Each concrete cube was carefully positioned between the machine's two steel plates, ensuring proper alignment and contact. A progressive compressive load was then applied to the specimen at a constant rate until failure occurred. The load at failure, along with the corresponding deformation, was recorded to determine the compressive strength of the concrete.

The compressive strength of each sample was calculated by dividing the failure load by the cross-sectional area of the specimen. To ensure accuracy and reliability, three samples were tested for each mix at each testing age (28 days, 56 days,90 days and 120 days) to account for variations in material properties and testing conditions. These experimental test methods provided comprehensive insights into the sulphate resistance and mechanical performance of Chikokoblended concrete under aggressive environmental conditions, aiding in the development of predictive models and informed decision-making for concrete design and construction practices.

2.2.6. Data Analysis

The data obtained from the experimental tests were subjected to comprehensive statistical analysis to discern the significant factors influencing strength loss and to develop a predictive model for the Strength Loss Index (SLI).

3. Results and discussion

3.1. Compressive Strength of CPC-NS concrete cured in sulphate water

As can be seen in figure 1, the Compressive Strength **(**CS) findings for CPC-NS concrete that was cured in sulphate water were as expected. The findings point to a pattern that is analogous to what was seen in CPC-NS concretes cured with fresh water. In such a way that, temperature had a linear influence on CS, with higher CSs being produced at higher calcination temperatures; CPC content had a nearly quadratic effect on CS, with the maximum CS being achieved between 10 and 15%; and curing age had a linear effect on CS, with higher CSs being connected with longer curing durations.

Figure 1 Compressive Strength of CPC-NS concrete cured in sulphate solution

The results observed align with the established understanding that temperature, composition, and curing age are pivotal factors influencing the compressive strength of concrete. Specifically, the temperature at which materials are calcined significantly impacts the resulting CS, with higher calcination temperatures leading to increased strength due to enhanced pozzolanic reactions [1]

Furthermore, the content of CPC (Calcium Phosphate Cement) exhibits a nearly quadratic relationship with compressive strength. This suggests an optimal range for CPC content, where too little or too much can result in lower strength, but a balance between 10 and 15% maximizes the material's performance [17].

The findings for NS concrete control cured in a sulphate solution were: 21.19, 21.40, 25.23, 27.22, 29.17, and 30.07 $N/mm²$ for 7, 14, 28, 56, 90, and 120 days respectively. The strength loss indices for these durations compared to fresh water were 25.10%, 25.54%, 22.58%, 29.19%, 25.43%, and 28.01%. These results suggest that CS losses due to sulphate attack do not follow a linear trend but average around 26% over the curing period. The incorporation of CPC into NS concretes significantly reduced CS losses, especially in specimens with 10% and 15% CPC. This improvement aligns with research indicating that CPC enhances concrete durability and resistance in aggressive environments [17]. The data shows that NS concrete's compressive strength is notably affected by sulphate solutions, consistent with literature on sulphate attack's detrimental effects on concrete integrity, causing expansion and cracking [1].

The non-linear CS loss pattern suggests complex interactions between the concrete matrix and sulphate ions, potentially involving multiple degradation mechanisms [20]. However, CPC addition mitigates these effects by forming more stable and less permeable hydration products, enhancing resistance to sulphate attack ([21].

3.2. Strength Loss Analysis

The experimental results indicated that Chikoko blended concrete showed improved resistance to sulphate attack compared to the control. Samples with 20% Chikoko replacement exhibited the least strength loss.

Temp.		Curing Age (Days)						
(°C)	CPC(%)	$\overline{7}$	14	28	56	90	120	
	$\boldsymbol{0}$	25.10	25.54	22.58	29.19	25.43	28.01	
	5	12.32	10.77	18.48	20.91	19.30	16.58	
ATM	10	-24.40	-31.85	-34.77	-41.75	-42.12	-43.89	
	15	-46.02	-54.61	-43.50	-47.55	-45.62	-45.78	
	20	9.92	5.09	2.62	0.89	0.26	6.19	
	5	17.48	17.01	19.26	24.86	24.07	22.23	
	10	2.82	-2.59	-1.48	-6.18	-7.89	-9.75	
	15	-6.21	-6.38	-7.66	-15.37	-14.94	-15.47	
200 °C	20	15.07	9.15	7.34	16.92	15.91	11.90	
	5	26.10	26.18	26.60	29.53	26.73	25.40	
	10	7.48	5.70	4.04	0.24	1.08	-0.75	
	15	3.99	3.52	2.31	-4.99	-4.36	-5.71	
400 °C	20	-29.00	-32.34	-31.11	-29.13	-30.38	-30.97	
	5	32.14	32.63	34.70	36.74	35.89	35.96	
	10	14.88	12.78	11.19	8.94	7.95	6.16	
	15	10.98	8.75	7.22	3.69	3.48	2.58	
600 °C	20	27.09	25.53	24.71	25.84	24.40	23.25	
	5	32.79	33.12	31.77	33.64	32.08	31.34	
800 °C	10	8.02	10.58	9.82	8.35	7.79	6.02	

Table 1 Sulphate induced CS loss indices for NS concrete

\sim \sim ᅩ	7.80	5.25	212 J.LJ	2.21 4.41	<u>ິດ ຕ</u> ະ 2.35	ςQ ر سد
20	23.48	21.69	19.23 1 J.LJ	21.15	20.86	20.53

Table 2 Input Data for the Analysis, Model Development and Optimization of the SASLI of Chikoko Blended Concrete using Pulverized Fine Aggregate

Factor	Name	Units	Type	SubType	Min.	Maxi.	Coded Low	Coded High	Mean	Std. Dev.
A	Chik.	$\%$	Num.	Discrete	0.0000	20.00	$-1 \leftrightarrow 0.00$	$+1$ \leftrightarrow 20.00	9.46	6.85
B	Temp.	$\rm ^oC$	Num.	Discrete	30.00	800.00	-1 \leftrightarrow 30.00	$+1$ \leftrightarrow 800.00	419.64	285.04
C	Curing Age	Days	Categ.	Nominal	7	120			Levels:	5.00

Table 3 Design Summary for the SASLI of chikoko blended concrete

The overview of the design inputs for the data analysis of the SASLI of chikoko mixed concrete may be found in Table 3. Chikoko content and Temperature are both examples of discrete numerical factors, but curing age is an example of a nominal categorical component. In the following table (Table 3) you will find not only information regarding the minimum and maximum levels of the elements, but also information regarding other statistical variables.

Table 4 Model Fit Summary for the SASLI of chikoko blended concrete

It is common practice for the design to suggest a variety of potential models and then recommend the model that would be the most useful based on the confidence interval, which is quantified by the model's P-Value. A P-value of 0.05 or below is acceptable when analyzing data with a confidence interval of 95%. As a result, taking into consideration the suggested models that are displayed in Table 4, it can be concluded that the Quadratic model is acceptable as the study has suggested.

Table 5 Analysis of Variance (ANOVA) for the SASLI of chikoko blended concrete

The fact that the model has an F-value of 19.24 indicates that it is statistically significant, and there is only a 0.01% possibility that such a high F-value might be the result of random noise.

P-values that are lower than 0.0500 indicate that the model terms under consideration are significant and that the null hypothesis has been refuted. In this particular instance, each of the model terms is relevant.

Table 6 Coefficient of Regression and Fit Statistics for the SASLI of chikoko blended concrete

The model's R2, adjusted R2, and predicted R2 coefficients are 0.99703, 0.9199, and 0.3797, respectively, as shown in Table 6. These values represent the predicted R2 value. This is in relation to the index of the entire sum of squares and the sum of squares remaining after residualization. The fact that the model has an adjusted R2 of 0.9199 indicates that it can predict responses with an accuracy of 91.99% within the data set, but its predictive ability drops to 37.97% when it is applied to data that is not contained within the data set.

Given that 19.289 is more than 4, it may be deduced that the signal-to-noise ratio is satisfactory; hence, the model can be utilized to explore the design space. Adeq Precision was calculated as 19.289.

The Variance Inflated Factors (VIF) for the model coefficients of Sulphate attack strength loss index of chikoko-cement blended concrete, are observed to be less than 10, hence multi – collinearity severity is minimized.

The Models developed are therefore as shown below;

$$
Logit(SISLI) = LN\left(\frac{SISLI + 41.8}{18 - SISLI}\right) = Sn\tag{3}
$$

$$
S7 = 3.97 - 0.39C - 0.017T + 0.0003CT + 0.017C2 + 0.000014T2
$$
 (4)

$$
S^{28} = 2.82 - 0.43C - 0.014T + 0.0003T + 0.017C^2 + 0.000014 * T^2
$$
 (5)

$$
S^{56} = 2.87 - 0.41C - 0.015T + 0.0003T + 0.017C^2 + 0.000014 * T^2
$$
 (6)

$$
S^{90} = 4.51 - 0.51C - 0.016T + 0.0003T + 0.017C^2 + 0.000014 * T^2
$$
 (7)

$$
S^{120} = 0.69 - 0.85C - 0.0048T + 0.0003T + 0.017C^2 + 0.000014 * T^2
$$
 (8)

Where;

Figure 2 Model diagnostics for the SASLI of chikoko blended concrete

The link between the actual data sets and the anticipated data sets is illustrated in Figure 2. The term "plot" refers to a collection of points that fall between the range of -2 to $+2$. This provides an excellent explanation for why the model cannot accurately anticipate answers that are not included in the data set. Nevertheless, the unquestionably fits all of the other conditions, and it possesses a strong potential for predicting reactions within the scope of the data sets it possesses.

Figure 3 Model simulations for 7day SASLI of chikoko blended concrete

Figure 4 Model simulations for 28-day SASLI of chikoko blended concrete

Figure 5 Model simulations for 56day SASLI of chikoko blended concrete

Figure 6 Model simulations for 90day SASLI of chikoko blended concrete

Figure 7 Model simulations for 120-day SASLI of Chikoko blended concrete

The modeling of the effect of calcination temperature and additive content on the Sulphate Attack Strength Loss Index (SASLI) of Chikoko-blended cement concrete in relation to curing age is depicted in Figures 1-7. The influence of Chikoko concentration and calcination temperature exhibits a quadratic relationship with the SASLI after 7 days. However, due to the formation of wing-like structures at the extremes of Chikoko concentration, it is challenging to assert that the relationship is purely quadratic (refer to Figure 4.31 for further details). The optimal ranges of Chikoko concentration and calcination temperature to achieve minimal strength loss due to sulphate attack were found to be 2.5-10% and 360- 800°C, respectively.

At 28 days, the quadratic influence of calcination temperature and Chikoko concentration on SASLI is more evident, illustrated by an almost perfect bowl-shaped curve. The most favorable ranges for achieving minimal strength loss were identified as 7.5-12.5% for Chikoko concentration and 250-600°C for calcination temperature. This pattern persisted at 56 days, indicating the consistency of the model simulation (see Figures 2 and 5).

After 90 days, the optimal calcination temperature for minimizing SASLI remains within 250-600°C, while the effective range of Chikoko content shifts to 10-15%. The relationship continues to be quadratic for both SASLI response types (see Figure 4).

At 120 days, a wider range of Chikoko content is required to maintain reduced sulphate attack effects on the compressive strength (CS) of Chikoko-blended cement concrete. The optimal range for Chikoko content extends to 10- 20%. However, beyond 10% Chikoko concentration, no significant reduction in strength loss was observed. At this stage, the ideal calcination temperature range was found to be between room temperature and 360°C.For long-term durability, strength losses due to sulphate attack on concrete can be significantly minimized by incorporating approximately 10% Chikoko as an additive and calcining it at temperatures not exceeding 400°C (see Figure 7). These findings provide a practical guide for optimizing the use of Chikoko in concrete to enhance its resistance to sulphate attack over extended periods.

4. Conclusion

Chikoko, an indigenous supplementary cementitious material (SCM), has demonstrated significant potential in enhancing the resistance of concrete to sulphate attack. The input data demonstrates significant variability in compressive strength and SASLI across different levels of Chikoko content, temperature, and curing age. The integration of Chikoko into concrete mixtures not only improves the material's durability but also contributes to a sustainable approach by utilizing locally available resources. The resistance to sulphate attack is crucial for concrete structures exposed to environments with high sulphate concentrations, as it helps prevent deterioration and prolongs the lifespan of the structures.

The research has led to the development of a predictive model that accurately estimates the strength loss index of concrete subjected to sulphate attack. This model is a valuable tool for engineers and designers, allowing for the optimization of concrete mixtures to achieve better performance in sulphate-rich environments. By incorporating Chikoko, the model helps predict the expected degradation and guides the design of more durable and resilient concrete structures.

Recommendation

While the initial research provides promising results, several areas warrant further investigation to fully understand the capabilities and limitations of Chikoko as a supplementary cementitious material. The following recommendations are made

- Future research should focus on the long-term performance of Chikoko-blended concrete. This involves exposing the concrete to varying concentrations of sulphates over extended periods to assess its durability and resistance comprehensively. Long-term studies will provide more robust data on the effectiveness of Chikoko in real-world conditions.
- It is essential to evaluate the performance of Chikoko-blended concrete under different environmental conditions, including variations in temperature, humidity, and the presence of other aggressive agents. Such studies will help determine the adaptability and resilience of Chikoko-blended concrete in diverse settings.
- Conducting comparative studies between Chikoko and other commonly used SCMs will provide a clearer understanding of its relative advantages and potential drawbacks. Such comparisons will help position Chikoko within the broader context of sustainable and durable construction materials.

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare that they have no conflicts of interest related to this study.

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