

# Optimization of California bearing ratio of tropical black clay soil treated with cement kiln dust and metakaolin blend

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## Abstract

The study showcase the optimization of California bearing ratio (CBR) values of expansive soil treated with cement kiln dust (CKD) and metakaolin (MTK) blend based on Scheffe optimization method. The CBR values utilized in the design of road infrastructure is an important parameter because it provides the rating of soil material for use as subgrade, sub-base and/or base course of road pavement. Therefore, applying Scheffe optimization technique will eliminate the random selection of design mix ratios and other associated disadvantages during CBR tests. Based on the optimization exercise and its results, the maximum CBR (unsoaked and soaked) values of 69 and 50 % were achieved with a corresponding mix ratio of 1.0:0.30:0.35:0.50 for black cotton soil, water, cement kiln dust and metakaolin respectively. During the course of this study, the laboratory results were used to develop two CBR models. The scheffe models developed are  $\hat{Y} = 34X_1 + 46X_2 + 40X_3 + 69X_4 - 8X_1X_2 - 4X_1X_3 + 34X_1X_4 + 8X_2X_3 + 34X_2X_4 + 30X_3X_4$  and  $\hat{Y} = 17X_1 + 28X_2 + 22X_3 + 50X_4 - 10X_1X_2 - 6X_1X_3 + 42X_1X_4 + 8X_2X_3 + 40X_2X_4 + 40X_3X_4$  for CBR unsoaked and soaked, respectively. In addition, the mathematical models were statistically scrutinized, confirmed for the adequacy and validity based on the outcomes of student t-test and analysis of variance (ANOVA). Also, the scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) techniques were used to explore the morphological and composition variations of the natural soil in contrast with the typically optimized soil-CKD-MTK blend. However, the SEM of the unaltered soil sample showed a smooth like surface, whereas the soil mixture optimally treated does not show same but rather demonstrated a rough like surfaced morphology. Thus, the observed variations might be due to the alterations of the soil fabrics possibly enhanced by the development of cementitious compounds (calcium silicate hydrate and calcium aluminate hydrate) as a result of pozzolanic reaction.

**Keywords:** Scheffe optimization; Soil stabilization; Black clay; Cement kiln Dust; Metakaolin; California bearing ratio

## 1. Introduction

The California bearing ratio (CBR) test is an important property used to express the strength of soil materials. Nevertheless, CBR test provides the engineer with information on quality and ratings of construction material. This test is generally an empirical method used in design of flexible pavement, also known as penetration test for evaluating the bearing capacity of subgrade soil. Prior to the use of any material for pavement subgrade, sub-base and base course, its suitability needs to be evaluated.

Tropical black clay soil rich in minerals like montmorillonite and illite are characterized by swell and shrink behaviour under changing moisture content and they are known as expansive soils. As a result of the swell-shrink behaviour, the expansive soils are considered as problematic because they create both construction and performance

problems when used as construction material in buildings and road pavements. Expansive soils are worldwide problems that have caused numerous challenges to civil engineers in the construction industry. It is imperative to reduce the damages posed by expansive soil which undermines the integrity of foundations and this have encouraged several researchers in making attempt to stabilize this soil so as to improve the geotechnical properties [1-2]. Hence, the conventional stabilizers (i.e. cement and lime) have been used to improve the strength and reduce the swell-shrink behaviour of black cotton soil [3]. Also, researchers [4-6] reported that the stabilization of this soil with cement or lime admixture is effective but very expensive. The utilization of these conventional stabilizers alone in soil stabilization have kept the cost of construction of stabilized roads very expensive and it will deprive some countries assess to infrastructural development. As a result of high cost, unfriendly environment and process involved in producing cement and lime, various sources of ash [7-10] and by-products like iron ore tailing, marble dust, limestone dust, granulated blast furnace slag and waste glass etc. have been used for stabilization of expansive soils [1,11-15]. However, the utilization of these wastes are gaining prominence and have thereby shown great influence on the geotechnical properties of these deficient soils.

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Cement kiln dust (CKD) is an industrial by-product of Portland cement produced in large quantities [16-17]. Million tons of CKD are generated and accumulated annually over the world [16] and can spread across large land mass, thereby having negative effect in the environment and human health. In addition, reusing this waste product will help in improving the properties of the soil for construction purpose, reduce cost of road construction and promote friendly environment. Metakaolin is a highly pozzolanic material obtained through the process of calcination of kaolinitic clay at temperatures range between 650 to 900°C depending on the nature and source of the kaolin [18-19].

An attempt has been made in this study to combine both cement kiln dust and metakaolin for the treatment of tropical black clay as a cost effective stabilisers and sustainability purpose. Incorporation of metakaolin will ascertain that there will be enough pozzolana in the soil matrix to react with both silica and alumina components of the black cotton soil and calcium oxide in cement kiln dust. Consequently, previous researchers have demonstrated positive results of metakaolin in concrete works [20-27] whereas scanty and far too little attention has been made towards the use of metakaolin in soil treatment [28-30].

Recently, research works on the use of optimization techniques such as scheffe's theory, response surface methodology (RSM), classical optimisation, genetic algorithm, fuzzy logic and artificial neural network in solving both geotechnical and concrete structural problems has gained prominence and useful results have been obtained [31-38]. Therefore, the utilization of statistical approach called Scheffe's simplex second order in optimizing construction materials for civil infrastructures have been widely studied and positive outcomes have been corroborated by [34-36, 39-41]. Interestingly, in the field of soil re-engineering they exist infinitesimal application of Scheffe's technique in optimizing the strength performance (California bearing ratio) of a tropical black clayey soil using CKD combine with other additive. In addition, far too little attention has been paid to the incorporation of micro morphological examination such as scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) of both unaltered and optimally ameliorated soil material. This indicates a need to understand the essence of optimizing additives content in ameliorating soil properties and the morphological changes in the treated soil matrix.

However, optimization in soil stabilization is the process of obtaining the best and most economical under any given circumstance. The essence of optimization is to either minimize the effort required or to maximize the anticipated benefit, since the effort required or the benefit preferred in any practical situation can be expressed as a function of certain decision variables [42]. Remarkably, the traditional trial method is not sufficient due to its cost implication and time-consuming nature. Therefore, the need arises for better approaches towards attaining optimal performance using a combination of stabilisers. The major goal of optimization is to ascertain that the mixture satisfies optimum requirements with respect to both engineering and economic standards.

In this work, a four component mixture (black cotton soil, water, cement kiln dust and metakaolin), thus the second degree (4, 2) simplex model which is a three dimensional factor space (tetrahedron) was studied. The number of terms in the response equation of simplex design depends on the number components of the mixture and the degree of polynomial of the simplex. The value could be obtained using the following equation:

$$N = \frac{(p+m-1)!}{m!(p-1)!} \tag{1}$$

Where N equals number of observations required, p equals degree of the polynomial and m equals number of components in the mixture

$$N = \frac{(4+2-1)!}{2!(4-1)!} = N = \frac{5!}{2!3!} = 10$$

This determines the number of runs in the experiment and the same number would be repeated for the control points.

## 2. Scheffe's optimization factor space

The performance of treated soils can be influenced by the adequate proportioning of its ingredients or test materials. Scheffe [43] developed an optimization theory that is used to optimize the behaviour of treated soils and considered experiments with mixtures of which the property studied depends on the proportions or percentages by weight of the components but not their quantities in the mixture. He introduced polynomial regression to model the response called "q, n-polynomials". These polynomials have to be of low degree (n), otherwise the polynomial contains a large number of coefficients, making interpretation difficult and requiring a large number of design points.

$$\text{if } n = 1; f(x) = \sum_{i=1}^q \beta_i x_i \tag{2}$$

$$\text{if } n = 2; f(x) = \sum_{i=1}^q \beta_i x_i + \sum_{1 \leq i \leq j \leq q} \beta_{ij} x_i x_j \tag{3}$$

$$\text{if } n = 3; f(x) = \sum_{i=1}^q \beta_i x_i + \sum_{1 \leq i \leq j \leq q} \beta_{ij} x_i x_j + \sum_{1 \leq i \leq j \leq k \leq q} (\beta_{ijk} x_i^2 x_j + \beta_{ijj} x_i x_j x_k) \tag{4}$$

The first four pseudo component located at the vertices of the tetrahedron simplex are: A1 [1:0:0:0], A2 [0:1:0:0], A3 [0:0:1:0], A4 [0:0:0:1]. Whereas, the six other pseudo mix ratios located at mid points of the lines joining the vertices of the simplex are A12 [0.5:0.5:0:0], A13 [0.5:0:0.5:0], A14 [0.5:0:0:0.5], A23 [0:0.5:0.5:0], A24 [0:0.5:0:0.5], A34 [0:0:0.5:0.5].

The response of the mixture is assumed to be a real value function on a simplex, to which an appropriate form of polynomial regression model is introduced. The polynomial function of degree n in q variable has  $C_{q+n}^n$  coefficients. If a mixture has a total of q components and  $x_i$  be the proportion of the *i*th component in the mixture such that  $x_i \geq 0$  ( $i = 1, 2, \dots, q$ ), then the sum of the component proportion is a whole unity, i.e.:

$$X_1 + X_2 + X_3 + X_4 = 1 \text{ or } \sum X_i - 1 = 0 \tag{5}$$

$$n = b_0 + \sum_{1 \leq i \leq q} b_i X_i + \sum_{1 \leq i \leq j \leq q} b_{ij} X_i X_j + \sum_{1 \leq i \leq k \leq q} b_{ijk} X_i X_j X_k + \dots + \sum b_{i_1 i_2} \dots i_n X_{i_1} X_{i_2} X_{i_n} \tag{6}$$

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_4 X_4 + b_{11} X_1^2 + b_{12} X_1 X_2 + b_{13} X_1 X_3 + b_{14} X_1 X_4 + b_{22} X_2^2 + b_{23} X_2 X_3 + b_{24} X_2 X_4 + b_{33} X_3^2 + b_{34} X_3 X_4 + b_{44} X_4^2 \tag{7}$$

Sum to one constant:  $X_1 + X_2 + X_3 + X_4 = 1$

The relationship obtained from Eq. (7) is subjected to normalization condition of Eq. (5) for a sum of independent variables. For a ternary mixture, the reduced 2<sup>nd</sup> degree polynomial can be obtained by multiplying Eq. (5) by  $b_0$ :

$$b_0 = b_0 X_1 + b_0 X_2 + b_0 X_3 + b_0 X_4 \tag{8}$$

$$b_0 = b_0 (X_1 + X_2 + X_3 + X_4)$$

Therefore, multiplying Eq. (5) by  $X_1, X_2, X_3$  and  $X_4$  in succession gives

$$X_1^2 + X_1 X_2 + X_1 X_3 + X_1 X_4 = X_1$$

$$X_1 X_2 + X_2^2 + X_2 X_3 + X_2 X_4 = X_2$$

$$X_1 X_3 + X_2 X_3 + X_3^2 + X_3 X_4 = X_3$$

$$X_1 X_4 + X_2 X_4 + X_3 X_4 + X_4^2 = X_4$$

$$\left. \begin{aligned} X_1^2 &= X_1 - X_1X_2 - X_1X_3 - X_1X_4 \\ X_2^2 &= X_2 - X_1X_2 - X_2X_3 - X_2X_4 \\ X_3^2 &= X_3 - X_1X_3 - X_2X_3 - X_3X_4 \\ X_4^2 &= X_4 - X_1X_4 - X_2X_4 - X_3X_4 \end{aligned} \right\} \quad (9)$$

Substitute Eq. (8) and (9) into (7):

$$\hat{Y} = (b_0 + b_1 + b_{11})X_1 + (b_0 + b_2 + b_{22})X_2 + (b_0 + b_3 + b_{33})X_3 + (b_0 + b_4 + b_{44})X_4 + (b_{12} - b_{11} - b_{22})X_1X_2 + (b_{13} - b_{11} - b_{33})X_1X_3 + (b_{14} - b_{11} - b_{44})X_1X_4 + (b_{23} - b_{22} - b_{33})X_2X_3 + (b_{24} - b_{22} - b_{44})X_2X_4 + (b_{34} - b_{33} - b_{44})X_3X_4 \quad (10)$$

$$\text{If we denote } \beta_i = b_0 + b_i + b_{ii} \text{ and } \beta_{ij} = b_{ij} - b_{ii} - b_{jj} \quad (11)$$

Then we arrive at the reduced second-degree polynomial:

$$\hat{Y} = \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_4X_4 + \beta_{12}X_1X_2 + \beta_{13}X_1X_3 + \beta_{23}X_2X_3 + \beta_{24}X_2X_4 + \beta_{34}X_3X_4 \quad (12)$$

Calculating the coefficients of the regression equation;

$$\begin{aligned} Y_1 &= \beta_1, Y_2 = \beta_2, Y_3 = \beta_3, Y_4 = \beta_4, \beta_{12} = 4Y_{12} - 2Y_1 - 2Y_2, \beta_{13} = \\ &4Y_{13} - 2Y_1 - 2Y_3, \beta_{14} = 4Y_{14} - 2Y_1 - 2Y_4, \beta_{23} = 4Y_{23} - 2Y_2 - \\ &2Y_3, \beta_{24} = 4Y_{24} - 2Y_2 - 2Y_4, \beta_{34} = 4Y_{34} - 2Y_3 - 2Y_4 \end{aligned} \quad (13)$$

These are the coefficients of the second degree polynomial for a q component mixture.

### 3. Materials and methods

#### 3.1. Materials

The soil sample used in this study was black cotton soil (BCS) collected from a single deposit in Deba, Gombe State, Nigeria using the method of disturbed sampling technique. The samples were taken at an average depth of 0.50 m from the natural earth surface, sealed in plastic bags and put in sack to avoid loss of moisture during transportation. The obtained samples were in hardened state because they were collected during the dry season and the soil samples were air dried before subjecting it to the pulverisation process so as to obtain particles passing sieve BS No. 4 (4.75mm aperture). The cement kiln dust (CKD) used for the study was obtained from United Cement Company of Nigeria (Unicem), Calabar, Cross River State. The raw material used in the production of metakaolin is kaolin also known as China clay, which is found in abundance in Nigeria. Kaolin clay was obtained from one of the kaolin deposit sites in Ohiya, Umuahia South Local Government Area, Abia State in the southern part of Nigeria. The metakaolin used in this study was produced through the calcination of kaolinite clay in a muffle furnace at temperature set of 850°C.

#### 3.2. Methods

The research methodology involves experimental design, formulation of models and laboratory tests. The number of components is 4 and a second degree polynomial was used, implying that 5 = q and 2 = m. The sampling and proportioning of the test materials were determined by a mix ratio model using the Scheffe's mathematical modelling approach. The proportions of the test materials: black cotton soil, water, cement kiln dust and metakaolin were obtained from iterations of the 4, 2 Scheffe's polynomial presented in Table 1. The values served as the percentage by weight of the dry solid added to the stabilization exercise.

#### 3.2.1. Experimental design

The response functions to be optimized is a function of the components X<sub>1</sub>, X<sub>2</sub>, X<sub>3</sub> and X<sub>4</sub>. The California bearing ratio (unsoaked and soaked) being the response are all dependent on the component proportions. The component materials used are black cotton soil, water, cement kiln dust and metakaolin. For each response, there are twenty (20) runs of experiment, the first ten (10) were obtained to formulate the model and are called trial mixes. Additional ten (10) mix ratios were also generated for each response and were used to validate the models.

#### 3.2.2. Components transformation

$$AZ = AX \quad (14)$$

Z represents the actual components, X represents the pseudo components and A is the constant; a four by four matrix for the present work under study. The value of matrix A will be derived from the first four mix ratios. The mix ratios are as follows:

$$\begin{aligned} Z_1 &[1.0: 0.1: 0.15: 0.2], \\ Z_2 &[1.0: 0.16: 0.2: 0.25], \\ Z_3 &[1.0: 0.25: 0.23: 0.4] \\ \text{and } Z_4 &[1.0: 0.3: 0.35: 0.5] \end{aligned}$$

The corresponding pseudo mix ratios are of an identity matrix form thus:

$$X_1 = [1: 0: 0: 0], X_2 [0: 1: 0: 0], X_3 [0: 0: 1: 0] \text{ and } X_4 [0: 0: 0: 1]$$

Substitution of X<sub>i</sub> and Z<sub>i</sub> into Eq. (13) use the corresponding pseudo components to determine the corresponding actual mixture components. However, X<sub>1</sub> equals proportion of soil, X<sub>2</sub> equals proportion of water, X<sub>3</sub> equals proportion of cement kiln dust and X<sub>4</sub> equals proportion of metakaolin.

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} a_{11} & a_{12} & a_{13} & a_{14} \\ a_{21} & a_{22} & a_{23} & a_{24} \\ a_{31} & a_{32} & a_{33} & a_{34} \\ a_{41} & a_{42} & a_{43} & a_{44} \end{pmatrix} \begin{pmatrix} X_1 \\ X_2 \\ X_3 \\ X_4 \end{pmatrix} \quad (15)$$

Therefore, the values of the actual mix ratios are substituted into Eq. (15) at each point or different run on the factor space and the resulting equation is solved.

For the first run;

$$\begin{pmatrix} 1.0 \\ 0.1 \\ 0.15 \\ 0.2 \end{pmatrix} = \begin{pmatrix} a_{11} & a_{12} & a_{13} & a_{14} \\ a_{21} & a_{22} & a_{23} & a_{24} \\ a_{31} & a_{32} & a_{33} & a_{34} \\ a_{41} & a_{42} & a_{43} & a_{44} \end{pmatrix} \begin{pmatrix} 1 \\ 0 \\ 0 \\ 0 \end{pmatrix} \quad (16)$$

Solving Eq. (16) yields the following coefficients of matrix A.

$$\begin{aligned} a_{11} &= 1.0 \\ a_{21} &= 0.10 \\ a_{31} &= 0.15 \\ a_{41} &= 0.20 \end{aligned}$$

For the second run;

$$\begin{pmatrix} 1.0 \\ 0.16 \\ 0.20 \\ 0.25 \end{pmatrix} = \begin{pmatrix} a_{11} & a_{12} & a_{13} & a_{14} \\ a_{21} & a_{22} & a_{23} & a_{24} \\ a_{31} & a_{32} & a_{33} & a_{34} \\ a_{41} & a_{42} & a_{43} & a_{44} \end{pmatrix} \begin{pmatrix} 0 \\ 1 \\ 0 \\ 0 \end{pmatrix} \quad (17)$$

Solving Eq. (17) yields the following coefficients of matrix A.

$$\begin{aligned} a_{12} &= 1.0 \\ a_{22} &= 0.16 \\ a_{32} &= 0.20 \\ a_{42} &= 0.25 \end{aligned}$$

For the third run;

$$\begin{pmatrix} 1.0 \\ 0.25 \\ 0.23 \\ 0.40 \end{pmatrix} = \begin{pmatrix} a_{11} & a_{12} & a_{13} & a_{14} \\ a_{21} & a_{22} & a_{23} & a_{24} \\ a_{31} & a_{32} & a_{33} & a_{34} \\ a_{41} & a_{42} & a_{43} & a_{44} \end{pmatrix} \begin{pmatrix} 0 \\ 0 \\ 1 \\ 0 \end{pmatrix} \tag{18}$$

Solving Eq. (18) yields the following coefficients of matrix A.

$$\begin{aligned} a_{13} &= 1.0 \\ a_{23} &= 0.25 \\ a_{33} &= 0.23 \\ a_{43} &= 0.40 \end{aligned}$$

For the fourth run;

$$\begin{pmatrix} 1.0 \\ 0.30 \\ 0.35 \\ 0.50 \end{pmatrix} = \begin{pmatrix} a_{11} & a_{12} & a_{13} & a_{14} \\ a_{21} & a_{22} & a_{23} & a_{24} \\ a_{31} & a_{32} & a_{33} & a_{34} \\ a_{41} & a_{42} & a_{43} & a_{44} \end{pmatrix} \begin{pmatrix} 0 \\ 0 \\ 0 \\ 1 \end{pmatrix} \tag{19}$$

Solving Eq. (19) yields the following coefficients of matrix A.

$$\begin{aligned} a_{14} &= 1.0 \\ a_{24} &= 0.30 \\ a_{34} &= 0.35 \\ a_{44} &= 0.50 \end{aligned}$$

Assembling the coefficients obtained from Eqs. (16) to (19) yields the following coefficient matrix, A.

$$A = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} \tag{20}$$

### 3.2.3. Determination of values of the actual components

The values of actual components of the mixture are obtained by multiplying the values of matrix A with values of matrix X.

For A<sub>12</sub>, substituting the values of X<sub>i</sub> gives Eq. (21)

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} \begin{pmatrix} 0.5 \\ 0.5 \\ 0 \\ 0 \end{pmatrix} \tag{21}$$

Solving Eq. (21) yields the values of actual components:

$$\begin{aligned} Z_1 &= 1.0 \\ Z_2 &= 0.13 \\ Z_3 &= 0.175 \\ Z_4 &= 0.225 \end{aligned}$$

For A<sub>13</sub>, substituting the values of X<sub>i</sub> gives Eq. (22)

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} \begin{pmatrix} 0.5 \\ 0 \\ 0.5 \\ 0 \end{pmatrix} \tag{22}$$

Solving Eq. (22) yields the values of actual components:

$$\begin{aligned} Z_1 &= 1.0 \\ Z_2 &= 0.175 \\ Z_3 &= 0.19 \\ Z_4 &= 0.30 \end{aligned}$$

For A<sub>14</sub>, substituting the values of X<sub>i</sub> gives Eq. (23)

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} \begin{pmatrix} 0.5 \\ 0 \\ 0 \\ 0.5 \end{pmatrix} \tag{23}$$

Solving Eq. (23) yields the values of actual components:

$$\begin{aligned} Z_1 &= 1.0 \\ Z_2 &= 0.20 \\ Z_3 &= 0.25 \\ Z_4 &= 0.35 \end{aligned}$$

For A<sub>23</sub>, substituting the values of X<sub>i</sub> gives Eq. (24)

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} \begin{pmatrix} 0 \\ 0.5 \\ 0.5 \\ 0 \end{pmatrix} \tag{24}$$

Solving Eq. (24) yields the values of actual components:

$$\begin{aligned} Z_1 &= 1.0 \\ Z_2 &= 0.205 \\ Z_3 &= 0.215 \\ Z_4 &= 0.325 \end{aligned}$$

For A<sub>24</sub>, substituting the values of X<sub>i</sub> gives Eq. (25)

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} \begin{pmatrix} 0 \\ 0.5 \\ 0 \\ 0.5 \end{pmatrix} \tag{25}$$

Solving Eq. (25) yields the values of actual components:

$$\begin{aligned} Z_1 &= 1.0 \\ Z_2 &= 0.23 \\ Z_3 &= 0.275 \\ Z_4 &= 0.375 \end{aligned}$$

For A<sub>34</sub>, substituting the values of X<sub>i</sub> gives Eq. (26)

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0 \\ 0 \\ 0.5 \\ 0.5 \end{pmatrix} \tag{26}$$

Solving Eq. (26) yields the values of actual components:

$$\begin{aligned} Z_1 &= 1.0 \\ Z_2 &= 0.275 \\ Z_3 &= 0.29 \\ Z_4 &= 0.375 \end{aligned}$$

The values of actual and pseudo components at different experimental points determined above are shown in Table 1.

### 3.2.4. Generation of actual components for control points

The mixture proportion of control points presenting actual and pseudo components are as follows;

For control point C<sub>1</sub>,

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.4 \\ 0.2 \\ 0.2 \\ 0.2 \end{pmatrix} \tag{27}$$

Solving Eq. (27) yields point C<sub>1</sub>:

$$\begin{aligned} Z_1 &= 1.0 \\ Z_2 &= 0.182 \\ Z_3 &= 0.216 \\ Z_4 &= 0.31 \end{aligned}$$

For control point C<sub>2</sub>,

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.2 \\ 0.4 \\ 0.2 \\ 0.2 \end{pmatrix} \tag{28}$$

Table 1  
Design matrix table based on Scheffe's (4, 2) - lattice polynomial.

Runs	Actual components				Response	Pseudo components			
	Z <sub>1</sub>	Z <sub>2</sub>	Z <sub>3</sub>	Z <sub>4</sub>		X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>
1	1.0	0.100	0.150	0.200	Y <sub>1</sub>	1	0	0	0
2	1.0	0.160	0.200	0.250	Y <sub>2</sub>	0	1	0	0
3	1.0	0.250	0.230	0.400	Y <sub>3</sub>	0	0	1	0
4	1.0	0.300	0.350	0.500	Y <sub>4</sub>	0	0	0	1
5	1.0	0.130	0.175	0.225	Y <sub>12</sub>	0.5	0.5	0	0
6	1.0	0.175	0.190	0.300	Y <sub>13</sub>	0.5	0	0.5	0
7	1.0	0.20	0.250	0.35	Y <sub>14</sub>	0.5	0	0	0.5
8	1.0	0.205	0.215	0.325	Y <sub>23</sub>	0	0.5	0.5	0
9	1.0	0.230	0.275	0.375	Y <sub>24</sub>	0	0.5	0	0.5
10	1.0	0.275	0.290	0.375	Y <sub>34</sub>	0	0	0.5	0.5

Solving Eq. (28) yields point C<sub>2</sub>:

Z<sub>1</sub> = 1.0  
Z<sub>2</sub> = 0.194  
Z<sub>3</sub> = 0.226  
Z<sub>4</sub> = 0.32

For control point C<sub>3</sub>;

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.2 \\ 0.2 \\ 0.4 \\ 0.2 \end{pmatrix} \quad (29)$$

Solving Eq. (29) yields point C<sub>3</sub>:

Z<sub>1</sub> = 1.0  
Z<sub>2</sub> = 0.212  
Z<sub>3</sub> = 0.232  
Z<sub>4</sub> = 0.35

For control point C<sub>4</sub>;

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.2 \\ 0.2 \\ 0.2 \\ 0.4 \end{pmatrix} \quad (30)$$

Solving Eq. (30) yields point C<sub>4</sub>:

Z<sub>1</sub> = 1.0  
Z<sub>2</sub> = 0.222  
Z<sub>3</sub> = 0.256  
Z<sub>4</sub> = 0.37

For control point C<sub>12</sub>;

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.25 \\ 0.25 \\ 0.25 \\ 0.25 \end{pmatrix} \quad (31)$$

Solving Eq. (31) yields point C<sub>12</sub>:

Z<sub>1</sub> = 1.0  
Z<sub>2</sub> = 0.2025  
Z<sub>3</sub> = 0.2325  
Z<sub>4</sub> = 0.3375

For control point C<sub>13</sub>;

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.10 \\ 0.30 \\ 0.30 \\ 0.30 \end{pmatrix} \quad (32)$$

Solving Eq. (32) yields point C<sub>13</sub>:

Z<sub>1</sub> = 1.0  
Z<sub>2</sub> = 0.223

Z<sub>3</sub> = 0.249  
Z<sub>4</sub> = 0.365

For control point C<sub>14</sub>;

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.30 \\ 0.10 \\ 0.30 \\ 0.30 \end{pmatrix} \quad (33)$$

Solving Eq. (33) yields point C<sub>14</sub>:

Z<sub>1</sub> = 1.0  
Z<sub>2</sub> = 0.211  
Z<sub>3</sub> = 0.239  
Z<sub>4</sub> = 0.355

For control points C<sub>23</sub>;

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.30 \\ 0.30 \\ 0.10 \\ 0.30 \end{pmatrix} \quad (34)$$

Solving Eq. (34) yields point C<sub>23</sub>:

Z<sub>1</sub> = 1.0  
Z<sub>2</sub> = 0.193  
Z<sub>3</sub> = 0.233  
Z<sub>4</sub> = 0.325

For control point C<sub>24</sub>;

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.30 \\ 0.30 \\ 0.30 \\ 0.10 \end{pmatrix} \quad (35)$$

Solving Eq. (35) yields point C<sub>24</sub>:

Z<sub>1</sub> = 1.0  
Z<sub>2</sub> = 0.183  
Z<sub>3</sub> = 0.209  
Z<sub>4</sub> = 0.305

For control point C<sub>34</sub>;

$$\begin{pmatrix} Z_1 \\ Z_2 \\ Z_3 \\ Z_4 \end{pmatrix} = \begin{pmatrix} 1.0 & 1.0 & 1.0 & 1.0 \\ 0.10 & 0.16 & 0.25 & 0.30 \\ 0.15 & 0.20 & 0.23 & 0.35 \\ 0.20 & 0.25 & 0.40 & 0.50 \end{pmatrix} * \begin{pmatrix} 0.30 \\ 0.20 \\ 0.30 \\ 0.20 \end{pmatrix} \quad (36)$$

Solving Eq. (36) yields point C<sub>34</sub>:

Z<sub>1</sub> = 1.0  
Z<sub>2</sub> = 0.197  
Z<sub>3</sub> = 0.224  
Z<sub>4</sub> = 0.33

Table 2  
Design matrix table for control points based on Scheffe’s (4, 2) factor space.

Runs	Actual Components				Response	Pseudo Components			
	Z <sub>1</sub>	Z <sub>2</sub>	Z <sub>3</sub>	Z <sub>4</sub>		X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>
1	1.0	0.182	0.216	0.31	C <sub>1</sub>	0.4	0.2	0.2	0.2
2	1.0	0.194	0.226	0.32	C <sub>2</sub>	0.2	0.4	0.2	0.2
3	1.0	0.212	0.232	0.35	C <sub>3</sub>	0.2	0.2	0.4	0.2
4	1.0	0.222	0.256	0.37	C <sub>4</sub>	0.2	0.2	0.2	0.4
5	1.0	0.2025	0.2325	0.3375	C <sub>12</sub>	0.25	0.25	0.25	0.25
6	1.0	0.223	0.249	0.365	C <sub>13</sub>	0.1	0.3	0.3	0.3
7	1.0	0.211	0.239	0.355	C <sub>14</sub>	0.3	0.1	0.3	0.3
8	1.0	0.193	0.233	0.325	C <sub>23</sub>	0.3	0.3	0.1	0.3
9	1.0	0.183	0.209	0.305	C <sub>24</sub>	0.3	0.3	0.3	0.1
10	1.0	0.197	0.224	0.33	C <sub>34</sub>	0.3	0.2	0.3	0.2

Both the actual and corresponding pseudo components obtained for the control points are presented in Table 2. These values are used to validate the formulated models.

3.3. California bearing ratio test

California bearing ratio (CBR) value is widely used in the design of materials for use as base and sub-base in pavement design which is an indicator for gauging the compacted soil strength and bearing capacity [44]. The test was done in accordance with BS 1377 and 1924 [45-46] for the natural and treated soils. Both soaked and unsoaked CBR were carried out in this study using BSL compaction. Curing of the specimens used for the CBR test was done for a period of 6 days and after the sixth day the specimens were submerged in water for 48 hours before testing. The curing period used was in accordance with the requirements of the NGS [47].

3.4. Microstructural analysis

Experiments were carried out to determine the effect of combining cement kiln dust and metakaolin on the strength behaviour of black cotton soil. Scanning electron microscopy (SEM) was applied to evaluate the micro morphological change of the stabilized samples and shed lights on the stabilization mechanisms. SEM analysis was performed on selected soil samples using Phenom world electron microscope. The electron diffraction spectroscopy (EDS) spectra were collected within the selected areas of SEM samples in order to verify the elemental energy spectra.

4. Results and discussion

4.1. Material characterization

Results of the preliminary investigation carried out on the natural black cotton soil are presented in Table 3. Fig. 1 shows the particle size distribution curve of the soil. The test soil was subjected to preliminary investigations and the results portrayed that the soil falls under A-7-6 (14) group using the AASHTO soil classification system [48] and CH in the Unified Soil Classification System [49]. The grain-size distribution curve of the unaltered BCS depicts that approximately 72 % of the soil material passed BS No. 200 sieve, which exceeds the limit of 35 % as documented in Nigeria General Specification [47]. Based on the compaction test, it shows that the soil has a maximum dry density of 1.61 g/cm<sup>3</sup> with a corresponding optimum moisture content of 18 %. The oxide composition of the cement kiln dust, metakaolin and black cotton soil used was measured through X-ray florescent test (XRF) the result is shown in Table 4. Fig. 2 shows the X-ray diffraction patterns of untreated black cotton soil. The X-ray

patterns revealed that clay minerals such as quartz, montmorillonite, kaolinite and rutile are the predominant (peak) minerals occurring in the soil sample under investigation.

4.2. California bearing ratio

The variations of the CBR (unsoaked) values of soil - cement kiln dust mixtures with metakaolin contents is shown in Table 5. For the unsoaked condition, the maximum increase in strength of 69 % was observed at a mix ratio of 1.0:0.30:0.35:0.50 for black cotton soil, water, cement kiln dust and metakaolin respectively beyond which there was a decrease in strength. The outcomes showed that the addition of cement kiln dust increases the strength values which could

Table 3  
Geotechnical properties of the test soil.

Property	Quantity
Natural Moisture Content, %	20.20
Percentage Passing BS No. 200 Sieve (75 µm aperture)	71.99
Liquid Limit, %	56.30
Plastic Limit, %	27.60
Plasticity Index, %	28.70
Linear Shrinkage, %	18
Free Swell, %	53.50
Specific Gravity	2.40
AASHTO Classification	A-7-6 (14)
USCS	CH
Maximum Dry Density (Mg/m <sup>3</sup> )	1.61
Optimum Moisture Content (%)	18
California Bearing Ratio (%)	3
Colour	Greyish black
Dominant clay mineral	Montmorillonite

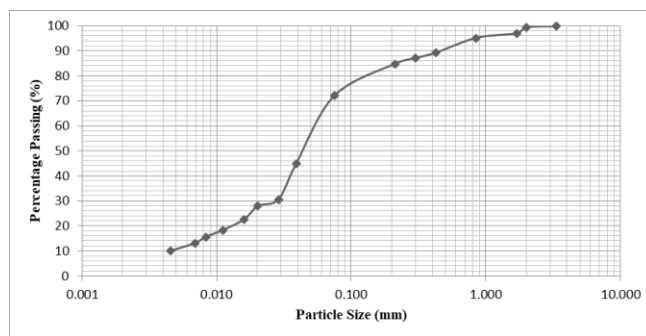


Fig. 1. Particle size distribution curve of the natural black cotton soil.

Table 4  
Chemical composition of cement kiln dust, metakaolin and black cotton soil.

Oxide	Composition by weight (%)		
	Cement kiln dust	Metakaolin	Black cotton soil
Silica (SiO <sub>2</sub> )	18.82	52.72	48.50
Lime (CaO)	66.82	0.18	0.90
Sulphur oxide (SO <sub>3</sub> )	2.01	0.99	-
Magnesium oxide (MgO)	0.01	0.09	2.22
Tin oxide (TiO <sub>2</sub> )	0.40	-	-
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> )	2.05	1.72	2.20
Alumina (Al <sub>2</sub> O <sub>3</sub> )	6.34	42.20	18.60
Alkali (Na <sub>2</sub> O)	0.20	-	1.55
K <sub>2</sub> O (Alkali)	1.0	-	0.70
Loss on ignition (LOI)	1.03	0.25	10.10

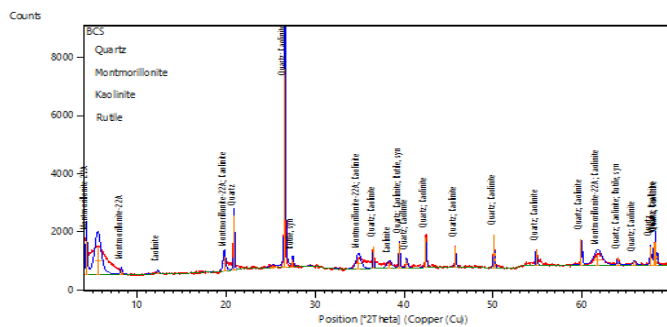


Fig. 2. X-ray Diffractometer for black cotton soil.

be connected with high silica content provided by metakaolin reacting with excess amounts of calcium hydroxide produced after hydration of cement compounds to further produce additional calcium silicate hydrates which are the major compounds responsible for strength. Thus, the boundary conditions for the acceptance of construction

Table 5  
Laboratory response of CBR unsoaked of soil-cement kiln dust mixtures with metakaolin.

Runs	Symbol of runs	Real components				Lab. response	Pseudo components			
		Z <sub>1</sub>	Z <sub>2</sub>	Z <sub>3</sub>	Z <sub>4</sub>		X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>
1	Y <sub>1</sub>	1	0.1	0.15	0.20	34	1	0	0	0
2	Y <sub>2</sub>	1	0.16	0.20	0.25	46	0	1	0	0
3	Y <sub>3</sub>	1	0.25	0.23	0.40	40	0	0	1	0
4	Y <sub>4</sub>	1	0.30	0.35	0.5	69	0	0	0	1
5	Y <sub>12</sub>	1	0.13	0.175	0.225	38	0.50	0.50	0	0
6	Y <sub>13</sub>	1	0.175	0.19	0.30	36	0.50	0	0.50	0
7	Y <sub>14</sub>	1	0.20	0.25	0.35	60	0.50	0	0	0.50
8	Y <sub>23</sub>	1	0.205	0.215	0.325	45	0	0.50	0.50	0
9	Y <sub>24</sub>	1	0.23	0.275	0.375	66	0	0.50	0	0.50
10	Y <sub>34</sub>	1	0.275	0.29	0.45	62	0	0	0.50	0.50
11	C <sub>1</sub>	1	0.182	0.216	0.31	49	0.40	0.20	0.20	0.20
12	C <sub>2</sub>	1	0.194	0.226	0.32	52	0.20	0.40	0.20	0.20
13	C <sub>3</sub>	1	0.212	0.232	0.35	51	0.20	0.20	0.40	0.20
14	C <sub>4</sub>	1	0.222	0.256	0.37	59	0.20	0.20	0.20	0.40
15	C <sub>12</sub>	1	0.2025	0.2325	0.3375	53	0.25	0.25	0.25	0.25
16	C <sub>13</sub>	1	0.223	0.249	0.365	58	0.10	0.30	0.30	0.30
17	C <sub>14</sub>	1	0.211	0.239	0.355	54	0.30	0.10	0.30	0.30
18	C <sub>23</sub>	1	0.193	0.233	0.325	55	0.30	0.30	0.10	0.30
19	C <sub>24</sub>	1	0.183	0.209	0.305	43	0.30	0.30	0.30	0.10
20	C <sub>34</sub>	1	0.197	0.224	0.33	50	0.30	0.20	0.30	0.20

materials for usage as either sub-base or base material is stipulated in [50]. However, a minimum CBR value of 60 - 80 % is required for base and from 20-30 % for sub-base both when compacted at optimum moisture and 100 % West African Standard [50]. Based on the criteria, the peak value for the unsoaked specimen of 69 % met the 60-80 % requirement for base courses [50].

On the other hand, the variations of the CBR (soaked) values of soil-cement kiln dust mixtures with metakaolin contents is shown in Table 6. For the soaked condition, the CBR values increased with higher additive contents, with a peak value of 50 % corresponding to a mix ratio of 1.0:0.30:0.35:0.50 for black cotton soil, water, cement kiln dust and metakaolin respectively. The trend of the unsoaked CBR values was similar to the soaked although with lower values and this is as a result of the ingress of water into the specimen thereby sapping and reducing its strength performance. However, based on the earlier adopted criteria, the peak CBR value of 50 % for the soaked specimen also met the Nigerian General Specification [47] requirement of 30 % for sub-base materials, for materials compacted at the optimum moisture content.

Finally, both the laboratory and model outcome of CBR (see Tables 5 and 6) shows that there was a remarkable rate of enhancement in CBR values for all soil material treated with the optimization mix ratios. The enhancement in the CBR values of BCS could be as a result of the interplay mechanism between the soil clay mineral and the additives which is due to the forming of cementitious effect by pozzolanic reactions. Similar verdicts were documented by previous investigators [1,51-52].

Thus the coefficients of the Scheffe's second degree polynomial are given as:

$$\beta_1 = 34, \beta_2 = 46, \beta_3 = 40, \beta_4 = 69, \beta_{12} = -8, \beta_{13} = -4, \beta_{14} = 34, \beta_{23} = 8, \beta_{24} = 34, \beta_{34} = 30$$

Substituting the obtained coefficients into Eq. (10) yields;

$$Y_{cbr(us)} = 34X_1 + 46X_2 + 40X_3 + 69X_4 - 8X_1X_2 - 4X_1X_3 + 34X_1X_4 + 8X_2X_3 + 34X_2X_4 + 30X_3X_4 \quad (37)$$

Table 6  
The CBR soaked of soil-cement kiln dust mixtures with metakaolin laboratory response.

Runs	Symbol of runs	Real components				Lab. response	Pseudo components			
		Z <sub>1</sub>	Z <sub>2</sub>	Z <sub>3</sub>	Z <sub>4</sub>		X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>
1	Y <sub>1</sub>	1	0.1	0.15	0.20	17	1	0	0	0
2	Y <sub>2</sub>	1	0.16	0.20	0.25	28	0	1	0	0
3	Y <sub>3</sub>	1	0.25	0.23	0.40	22	0	0	1	0
4	Y <sub>4</sub>	1	0.30	0.35	0.5	50	0	0	0	1
5	Y <sub>12</sub>	1	0.13	0.175	0.225	20	0.50	0.50	0	0
6	Y <sub>13</sub>	1	0.175	0.19	0.30	18	0.50	0	0.50	0
7	Y <sub>14</sub>	1	0.20	0.25	0.35	44	0.50	0	0	0.50
8	Y <sub>23</sub>	1	0.205	0.215	0.325	27	0	0.50	0.50	0
9	Y <sub>24</sub>	1	0.23	0.275	0.375	49	0	0.50	0	0.50
10	Y <sub>34</sub>	1	0.275	0.29	0.45	46	0	0	0.50	0.50
11	C <sub>1</sub>	1	0.182	0.216	0.31	30	0.40	0.20	0.20	0.20
12	C <sub>2</sub>	1	0.194	0.226	0.32	35	0.20	0.40	0.20	0.20
13	C <sub>3</sub>	1	0.212	0.232	0.35	33	0.20	0.20	0.40	0.20
14	C <sub>4</sub>	1	0.222	0.256	0.37	43	0.20	0.20	0.20	0.40
15	C <sub>12</sub>	1	0.2025	0.2325	0.3375	37	0.25	0.25	0.25	0.25
16	C <sub>13</sub>	1	0.223	0.249	0.365	42	0.10	0.30	0.30	0.30
17	C <sub>14</sub>	1	0.211	0.239	0.355	38	0.30	0.10	0.30	0.30
18	C <sub>23</sub>	1	0.193	0.233	0.325	40	0.30	0.30	0.10	0.30
19	C <sub>24</sub>	1	0.183	0.209	0.305	23	0.30	0.30	0.30	0.10
20	C <sub>34</sub>	1	0.197	0.224	0.33	32	0.30	0.20	0.30	0.20

Eq. (37) is the Scheffe’s model for the optimization of California bearing ratio (unsoaked) of black cotton soil treated with cement kiln dust and metakaolin blend.

Thus the coefficients of the Scheffe’s second degree polynomial are given as:

$$\beta_1 = 17, \beta_2 = 28, \beta_3 = 22, \beta_4 = 50, \beta_{12} = -10, \beta_{13} = -6, \beta_{14} = 42, \beta_{23} = 8, \beta_{24} = 40, \beta_{34} = 40$$

Substituting the obtained coefficients into Eq. (10) yields;

$$Y_{\text{cbr(s)}} = 17X_1 + 28X_2 + 22X_3 + 50X_4 - 10X_1X_2 - 6X_1X_3 + 42X_1X_4 + 8X_2X_3 + 40X_2X_4 + 40X_3X_4 \tag{38}$$

Eq. (38) is the Scheffe’s model for the optimization of California bearing ratio (soaked) of black cotton soil treated with cement kiln dust and metakaolin blend.

4.3. Testing of adequacy of prediction models

The student’s t-test and analysis of variance were used to evaluate the adequacy of the proposed models. In order to test the validity and adequacy of the models, additional ten points were used by comparing the experimental response of the control points with the predicted results of California bearing ratio test (unsoaked and soaked). In this test, the following hypotheses are examined:

There is no significant difference between the experimental values of the California bearing ratio test and the predicted values, this is the null hypothesis.

There is a significant difference between the experimental values of the California bearing ratio test and the model predicted values, this is the alternate hypothesis.

4.3.1. Student’s t-test of California bearing ratio (unsoaked)

In order to assess the validity of predicted models, a two-tailed student t-test was also conducted by comparing the two groups in this case and if t stat is greater than t critical two-tail, we reject the null hypothesis. Presented in Table 7 is the experimental and model

response of California bearing ratio (unsoaked) while Table 8 presents the result of the t-test for the control points. In this current research,

Table 7  
Experimental result of California bearing ratio (unsoaked) test and model test results.

Symbol of Response	Response	
	Model	Laboratory
C <sub>1</sub>	49.24	49
C <sub>2</sub>	52.12	52
C <sub>3</sub>	50.92	51
C <sub>4</sub>	59.28	59
C <sub>12</sub>	53.125	53
C <sub>13</sub>	57.04	58
C <sub>14</sub>	53.92	54
C <sub>23</sub>	55.12	55
C <sub>24</sub>	45.48	43
C <sub>34</sub>	50.04	50

Table 8  
T-Test: paired two sample for means.

Description	Model	Laboratory
Mean	52.6285	52.4
Variance	16.03185583	21.37777778
Observations	10	10
Pearson Correlation	0.990129315	
Hypothesized Mean Difference	0	
Df	9	
t Stat	0.834691633	
P(T<=t) one-tail	0.212746082	
t Critical one-tail	1.833112933	
P(T<=t) two-tail	0.425492165	
t Critical two-tail	2.262157163	



Table 9  
Analysis of variance for California bearing ratio (Unsoaked).

Anova: Single Factor						
Groups	Count	Sum	Average	Variance		
Model	10	526.285	52.6285	16.03186		
Laboratory	10	524	52.4	21.37778		
Source of Variation	Sum of Square	Degree of Freedom	Mean of Square	F-value	P-value	F crit
Between Groups	0.26106125	1	0.261061	0.013957	0.907266	4.413873
Within Groups	336.6867025	18	18.70482			
Total	336.9477638	19				

the t stat value of 0.834691633 was less than the t critical two-tail value of 2.262157163, thus, it implies that t critical is greater than t stat and this certifies that we accept the null hypothesis.

#### 4.3.2. Analysis of variance

During the analysis of variance (ANOVA) exercise, if F is greater than F crit, we reject the null hypothesis. Table 9 presents the result of the analysis, F equals 0.013957 whereas F crit equals 4.413873 so F crit is greater than F. Also, the source variation having P value less than 0.05 is identified as a significant parameter. Therefore, we do not reject null hypothesis. However, this infers that there exist no significant difference between the experiment and model results. Henceforth, the model is satisfactory for use in optimization exercise of California bearing ratio (unsoaked) of black cotton soil treated with cement kiln dust and metakaolin blend.

#### 4.3.3. Student's t-test of California bearing ratio (soaked)

In order to assess the validity of predicted models, a two-tailed student t-test was also conducted by comparing the two groups in this case and if t stat is greater than t critical two-tail, we reject the null hypothesis. Presented in Table 10 is the experimental and model response of California bearing ratio (soaked) while Table 11 presents the result of the t-test for the control points. In this current research, the t stat value of 0.825020482 was less than the t critical two-tail value of 2.262157163, thus, it implies that t critical is greater than t stat and this certifies that we accept the null hypothesis.

#### 4.3.4. Analysis of variance

During the analysis of variance (ANOVA) exercise, if F is greater than F crit, we reject the null hypothesis. Table 12 presents the result of the analysis, F equals 0.050179 whereas F crit equals 4.413873 so F crit is greater than F. Also, the source variation having P value less than 0.05 is identified as a significant parameter. Therefore, we do not reject null hypothesis. However, this infers that there exist no significant difference between the experimental and model results. Henceforth, the model is satisfactory for use in optimization exercise of California bearing ratio (soaked) of black cotton soil treated with cement kiln dust and metakaolin blend.

Table 12  
Analysis of variance for California bearing ratio (soaked).

Anova: Single Factor						
Groups	Count	Sum	Average	Variance		
Model	10	358.255	35.8255	18.13344		
Laboratory	10	353	35.3	36.9		
Source of Variation	Sum of Square	Degree of Freedom	Mean of Square	F-value	P-value	F crit
Between Groups	1.38075125	1	1.380751	0.050179	0.825276	4.413873
Within Groups	495.3009225	18	27.51672			
Total	496.6816738	19				

#### 4.4. Analysis of optimization models

The geotechnical properties tested for was California bearing ratio (unsoaked and soaked) and two models were formulated based on Scheffe's optimization technique. They were tested for adequacy

Table 10  
Experimental result of California bearing ratio (soaked) and model test results.

Symbol of Response	Response	
	Model	Laboratory
C <sub>1</sub>	32.40	30
C <sub>2</sub>	35.08	35
C <sub>3</sub>	34.04	33
C <sub>4</sub>	42.84	43
C <sub>12</sub>	36.375	37
C <sub>13</sub>	40.40	42
C <sub>14</sub>	37.48	38
C <sub>23</sub>	38.44	40
C <sub>24</sub>	28.04	23
C <sub>34</sub>	33.16	32

Table 11  
T-Test: paired two sample for means.

Description	Model	Laboratory
Mean	35.8255	35.3
Variance	18.13343583	36.9
Observations	10	10
Pearson Correlation	0.985338303	
Hypothesized Mean Difference	0	
Df	9	
t Stat	0.825020482	
P(T<=t) one-tail	0.21534171	
t Critical one-tail	1.833112933	
P(T<=t) two-tail	0.430683421	
t Critical two-tail	2.262157163	

using Student’s t-test and analysis of variance. The null hypothesis is accepted for the tested property based on the Student’s t-test showing that the models are adequate. The modelled values of CBR were close to the laboratory values and these show that the models are all adequate for predicting the various responses. With the aid of Scheffe’s optimization technique, a peak CBR (unsoaked and soaked) values of 69 and 50 % were achieved corresponding to a mix ratio of 1.0:0.30:0.35:0.50 for black cotton soil, water, cement kiln dust and metakaolin respectively.

4.5. Microstructural characterization

4.5.1. Scanning electron microscope (SEM)

Quite a number of researchers have documented the use of scanning electron microscope (SEM) in exploring the microstructural performance of soil materials [1, 53-56]. The examination of the micro morphological behaviour of both the unaltered and optimized soil material was investigated. This test was executed in order to shed lights and as well validate the stabilization mechanisms as a result of the optimization exercise. Figs. 3 and 4 represents the morphology of the untreated and treated soil specimens. The micrographs revealed that the natural soil has a smooth surface while the treated soil developed a rough surfaced morphology. This behaviour could be as a result of the change in orientation and fabric of the treated soil as a result of the reduction in the cohesiveness of the soil due to the cation exchange reaction. Similar behaviour was reported by [57]. Also, large voids evident in the untreated soil lessened in the optimally treated soil specimen. This closed voids (dense soil) micrograph illustrates the possible form precipitate of new cementitious compounds (calcium silicate hydrate CSH and calcium aluminate hydrate CAH) due to pozzolanic reaction which were shown within the pore spaces resulting in a reduction in the radius of pore spaces. These results agree with the findings of other studies, in which the microstructural changes eventually contribute to strength development with time [1,53-54].

4.5.2. Energy Dispersive Spectroscopy (EDS)

Figs. 5 and 6 depicts the EDS spectra of the untreated black cotton soil and optimally treated black cotton soil. The EDS elemental analysis works together with the SEM to provide the chemical composition of the natural black cotton soil and the optimally treated soil. Consequently, the essence of the EDS system is to give an understanding about the chemical analysis of features being witnessed in the SEM investigation. For the natural black cotton soil, the EDS analysis portrays major peak composition of the alumina silicate

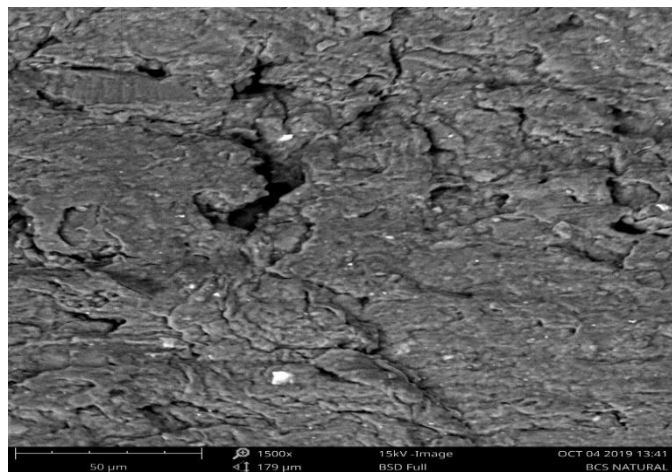


Fig. 3. Micrograph of natural black cotton soil at 50µm (7 day curing).

minerals [58] consisting of Si, Al and Fe with traces of the presence of Ca, Ag, Ti, Mg, Nb, P and Cl as shown in Fig. 5. The Si:Al peak height ratio of the soil is approximately 2:1, which confirms the presence of montmorillonite mineral in the soil [59]. The untreated soil has a higher iron content than the modified soil. For the optimally treated black cotton soil, the EDS analysis portrays an increase in silicon and calcium oxide as shown in Fig. 6. The higher content of silicon could not be unconnected with the high content of metakaolin compared to cement kiln dust. However, the increase in silicon and calcium oxide contents could be due to pozzolanic reaction in which the calcium from cement kiln dust and metakaolin reacted with alumina and silica from clay and metakaolin in the presence of water to produce stable calcium silicate hydrate and calcium aluminate hydrate which generates long term strength gain and improve the geotechnical properties of the soil [60].

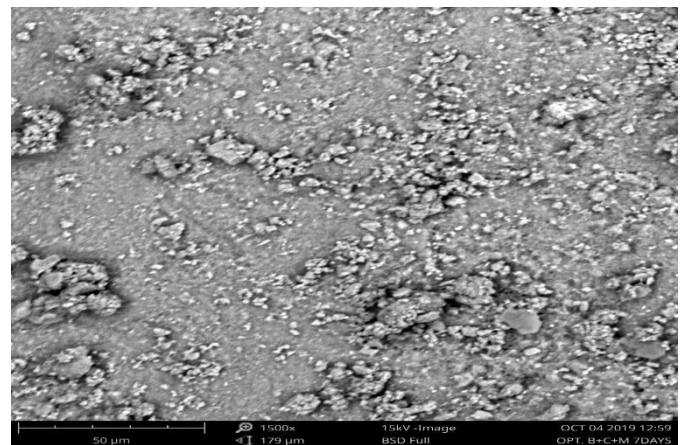


Fig. 4. Micrograph of black cotton soil optimally treated with 0.35 cement kiln dust 0.50 metakaolin blend at 50µm (7 day curing).

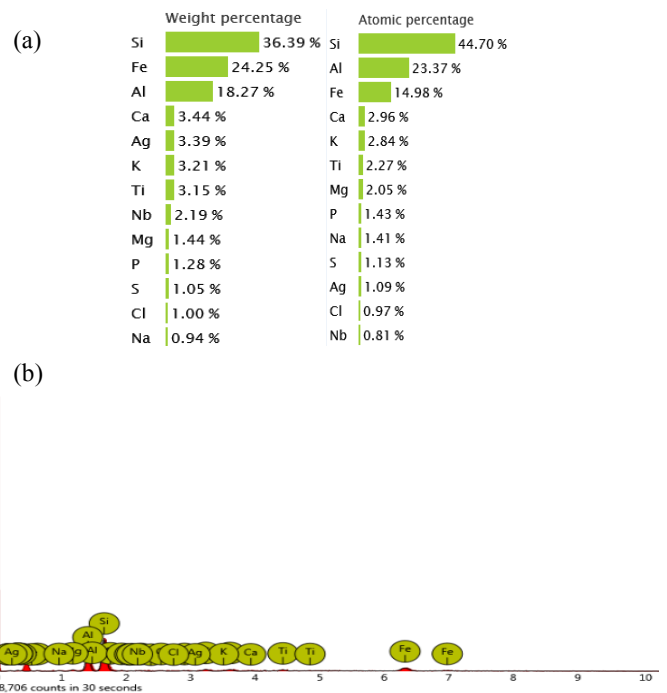


Fig. 5. (a) Bar charts representation of weight and atomic quantifications of element detected from EDS spectra of natural black cotton soil after 7 days curing period and (b) Energy-dispersive x-ray spectroscopy of natural black cotton soil after 7 days curing period.

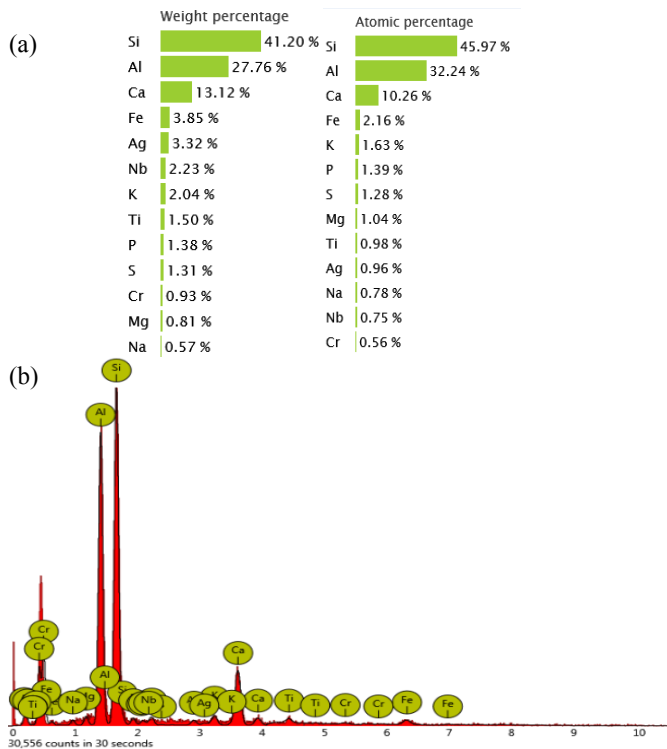


Fig. 6. (a) Bar charts representation of weight and atomic quantifications of element detected from EDS spectra of black cotton soil optimally treated with 0.35 cement kiln dust 0.50 metakaolin blend after 7 days curing period and (b) Energy-dispersive x-ray spectroscopy of black cotton soil optimally treated with 0.35 cement kiln dust 0.50 metakaolin blend after 7 days curing period.

## 5. Conclusion

From the above-mentioned optimization exercise by Scheffe method, the California bearing ratio (CBR) of tropical black clay soil treated with cement kiln dust and metakaolin was investigated. Based on the findings and results drawn from the present research work, the conclusions are as follows. The test soil falls within the class of A-7-6 (14) soil material based on the AASHTO soil classification system and CH in the Unified Soil Classification System, respectively. Generally, Scheffe's second degree polynomial was applied to formulate models for predicting California bearing ratio (CBR) (unsoaked and soaked). Based on the models developed, the highest CBR values of 69 and 50 % were achieved at a mix ratio of 1.0:0.30:0.35:0.50 with a corresponding mass conversion (percentage) values of 1.93:0.579:0.676:0.965 for black cotton soil, water, cement kiln dust and metakaolin respectively. The models developed from this research work provided a very good prediction of the response and as such the models can be utilized for good decision making on CBR values of black cotton soils having similar geotechnical properties. The student t-test and the analysis of variance (ANOVA) tests were used to check the adequacy of the models and the models were found to be adequate at 95 % confidence level. Also, with the aid of scanning electron microscopy and electron diffraction spectroscopy the combined effect of cement kiln dust (CKD) and metakaolin (MTK) in treating tropical black clay soil was qualitatively confirmed. Hence, combining CKD and MTK assisted the soil particles to form more compact micro structures and improved its geotechnical properties. Moreover, the utilization of waste materials (CKD and MTK) of this percentage could be beneficial for use as sustainable engineering materials for construction purposes, thus eliminating the nuisance associated with poor waste management.

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## Conflict of interest

The work is part of a Ph.D research work undertaken by Engr. Imoh Christopher Attah under the supervision of Engr. Prof. F. O. Okafor and Engr. Prof. O. O. Ugwu. No potential conflict of interest was reported by the Authors.

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