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POTENTIALS OF METAKAOLIN AND SAND AS LINER MATERIALS FOR WASTE CONTAINMENT APPLICATION

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ABSTRACT
The study investigated the suitability of Metakaolin (Mk) admixed with River sand in the stabilization of Black Cotton Soil (BCS). The suitability of material for constructing a liner or cover depends primarily on low hydraulic conductivity; however, most of the construction works involve the use of soil materials. Some of these soil materials, when encountered, may not be suitable or adequate for use due to their poor strength; therefore, there is a need for their stabilization. For this reason, there is a need for effective utilization of different stabilizers to improve the geotechnical properties of liner materials. Hence, the research uses Metakaolin and sand as liner materials for waste containment applications. The BCS was classified as A-7-6 and CH according to the American Association of State Highway and Transportation Officials (AASHTO) and the Unified Soil Classification System (USCS). The soil was stabilized with Metakaolin blended sand at various percentages. A series of tests, such as Compaction test (OMC, MDD), Unconfined Compressive Strength (UCS), Volumetric Shrinkage Strength (VSS) and Hydraulic Conductivity Test (K), were carried out. Optimization and models for estimation of the responses were done by central composite design (CCD). The result showed that the BCS was classified as A-7-6 and CH according to the AASHTO and USCS. The OMC was 9.8%, MDD was 1.69 Mg/m3. The soil has a specific gravity of 2.19, Natural moisture content of 0.71%, LL of 44.9% and PL of 25.8%. The replacement of black cotton soil with Metakaolin and sand reduces the hydraulic conductivity and Volumetric Shrinkage of the soil. The hydraulic conductivity of black cotton soil was 1.66397E^06 while its shrinkage was 8.98%. The best result recorded was achieved at 20% Mk and 20% sand, with hydraulic conductivity and volumetric shrinkage of 1.601E^06 and 4% respectively. Optimization was carried out and models were developed for the estimation of OMC, MDD, VSS, hydraulic conductivity and UCS of admixed black cotton soil. The numerical optimization was done using central composite design, and the ramp plot gave 20% Mk, 5% Sand, 12% OMC, 1.85% MDD, 345 kN/m2 for UCS at 28 days, VSS at 4.4% and 1.7 cm/s for hydraulic conductivity. Finally, 20% Mk and 5% sand were recommended for use as a replacement for BCS.

KEYWORDS: Optimization, Black Cotton Soil, Metakaolin, Hydraulic Conductivity, Unconfined Compressive Strength, Liner Material, Unified Soil Classification System.

1 INTRODUCTION

Proper and effective use of Liner materials is essential in preventing the infiltration of leachate into the ground and achieving sustainable impact for achieving the development goal. Liner is an important component of landfill in waste disposal, hence there is a need to use locally available material as liners and covers.

Nigeria is a developing country and may encounter some challenges related to the prompt execution of capital projects because of dwindling reserves of good-quality soils that will be required for construction works (Ibrahim *et al*., 2023).

To protect waste and groundwater from landfill contaminants, clay liners are widely used to impede the flow of leachate from the landfill. The suitability of a material for constructing a liner or cover depends primarily on low hydraulic conductivity, durability, resistance to weathering and compatibility (Chinade *et al*., 2017).

Clayey soils as traditional lining materials have been widely employed due to their low permeability characteristics and ability to absorb heavy metals from the leachate (Osinubi *et al.*, 2017; Abdulkarim *et. al.*, 2022). However, with the advancement in technology, various soil types, including sands, laterites, and lateritic soils, to provide cost-effective and sustainable liner materials (Ahmad *et al*., 2014).

Bottom and cover liners of landfills are often based on natural materials, including clays characterized by an approximate hydraulic conductivity coefficient usually below 1E-9 m/s. Most of the construction works

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involve the use of soil materials. Some of these soil materials, when encountered, may not be suitable or adequate for use due to their poor strength characteristics, and when such occurs, the engineer is left with the choice of borrowing suitable materials from other sites or improving the strength of the available ones (Agbede and Joel, 2011).

The engineer with the choice of adding stabilising agents to improve the properties of the available soil on site using various means of soil improvement techniques, and the soil itself is an essential component of the project's success. Some soil has a hard stratum to work on the other hand, some have very poor properties to withstand the loading. Additionally, the productivity of building businesses is affected by the mineralogical qualities that specific soils possess (Jangde and Khan, 2023).

Stabilisation involves the methods used for modifying the properties of soil to improve its engineering performance. In construction, the main objective of stabilisation is to increase the strength or stability of soil and to reduce construction costs by making the best use of locally available materials. For instance, one of the ways that soft soils may be stabilized so that construction can take place is by adding metakaolin to the soil. In the realm of soil stabilization, the use of metakaolin as a binding agent is something that is relatively new. The use of metakaolin as a cement substitute, aiming to create a pozzolanic effect and reduce costs, has been investigated and tested in the past (Jangde, 2023).

Metakaolin ash is a white material produced by the calcination of kaolin. Most kaolin retained its cellular structure through calcination at approximately 1000°C and then cooling, milling and sieving through the 425 mesh. To overcome this problem, treating the soils at the field level results in less manpower, less treatment area, and better results in performance. To determine the mix ratio that will yield blend and percentage replacements, Minitab Statistical Analysis Software was used to develop the illustrations. Despite the existence of several types of statistical analysis software, Minitab was selected because it is commonly used in industry, manufacturing and healthcare, is tailored for quality control applications, provides tools to check the assumptions associated with statistical tools, and is equipped with an Assistant tool that can aid students and practitioners in systematically selecting the proper technique to analyze data and interpret the results. Therefore, Minitab statistical software will be used in this study (Kenett and Zacks, 2021).

According to the researcher (Laigude*et al.,* 2018), they experimented by using sand and cement as a stabilizing material for black cotton soil; however, sand is a liquid or water impermeable material known for its strength, processing time and ability to bind other material substances.

In this study, a numerical optimization technique using the desirability function will be used with special emphasis on strength, hydraulic conductivity, water and suitability, in which the Minitab Statistical Software is incorporated. The Minitab uses statistical techniques for empirical model building; it comprises regression surface fitting to obtain approximate responses, design of experiments is conducted to obtain minimum variances of the responses and optimization using the approximated responses. The Minitab also aims to reduce the cost and numerical complexity of other expensive analysis methods (Kenett and Zacks, 2021)

2. METHODOLOGY

Mixing of the batched materials was done using a manual. The stabilised materials are black cotton clay, sand and metakaolin, which were thoroughly mixed for 15 minutes until the mix became homogenous, after which water was finally added at varying percentages of replacement. The percentage replacement of Metakaolin and black cotton clay with different blends of sand. The following laboratory experiments were carried out on the specimens: Natural moisture content, Particle size distribution, Specific gravity, Compaction, Volumetric shrinkage strain (VSS), Unconfined compressive strength (UCS), Hydraulic conductivity (k) and X-ray fluorescence (XRF)

3. RESULTS AND DISCUSSION

3.1 Soil Properties

The Table 1 presents the summary of the properties of the Natural Soil, while Table 2 gives the oxide composition of the natural soil and Kaolin powder, known as (Metakaolin). Table 3 shows the Compaction test, unconfined compressive strength test (UCS), Volumetric Shrinkage Strength test (VSS), and permeability test results for stabilized soil at varying percentages from the Experimental Design Module.

Table 1: Properties of the Natural Soil

Property	Value
Natural moisture content (%)	0.71
Liquid limit (%)	44.9
Plastic limit (%)	25.8
Plasticity index (%)	19.1
Volumetric shrinkage (%)	8.98
Activity	1.4
Specific gravity	2.19
PARTICLE SIZE DISTRIBUTION	
Percentage PASSING No. 200 sieve	87.7
Percentage (Fines) < 0.075 - 4.76mm (%)	87.7
Percentage Sand (0.075 - 4.76) (%)	12.3
Percentage gravel (< 4.76mm) (%)	0
COMPACTION PROPERTIES	
Maximum Dry Density (MDD) Mg/m³	1.69
Optimum Moisture Content (OMC) %	9.8
STRENGTH PROPERTIES	
7 days UCS kN/m^2	245
7 + 7 days UCS kN/m^3	95
14 days UCS kN/m^4	253
28 days UCS kN/m^5	362
Hydraulic conductivity	
Permeability	1.66397E^06
Additional information	
Colour	Black
Dominant clay mineral	Montmorillonite
AASHTO Classification	A - 7 – 6
Clay Soil (AASHTO)	Fair to poor
UCSC Classification	CH

Table 2: Oxide Composition of Sand and Metakaolin

Oxide Composition	Concentration (%)	
	Soil	Metakaolin
SiO_2	31.2	51.8
Al_2SO_3	20.2	37.2
Fe_2O_3	4.44	4.31
MgO	0.28	0.79
CaO	0.53	0.07
K_2	0	1.51

Preliminary test was conducted for the identification and determination of the Engineering properties of the Natural soil, as well as the Chemical Properties of Natural soil and Metakaolin. The results of the preliminary analysis on the natural soil are presented in Table 1, while those of the Chemical Properties of both Natural soil and Metakaolin are presented in Table 2. The liquid limit, plastic limit and plasticity index of the natural soil were observed to be 44.9, 25.8 and 19.1% respectively, which confirmed that the soil is highly plastic. The result of the Atterberg Limits is a very important parameter for determining soil behaviour and classification (Osinubi *et al.*, 2011).

The results obtained from the Atterberg limits and sieve analysis test classified the soil as A-7-6 (13) according to the American Association of State Highway and Transportation Officials (AASHTO, 2004) and Clay of high plasticity (CH) in accordance with the Unified Soil Classification System (ASTM, 1992). The percentage sand fraction of 65%, percentage silt fraction 23%, percentage clay fraction 28% and an Activity of 1.12, which indicated that Montmorillonite clay minerals are dominant in the soil. With these parameters, the

soil falls far below the standard recommended for most Engineering work based on the Nigerian General Specification (1997).

The Unconfined Compressive Strength (UCS) value of 362 at OMC 10.5 is relatively low, indicating that the material does not meet the minimum requirement of the Nigerian General Specification (1997) and other research findings by Moses and Osinubi (2013); Osinubi *et al.* (2011); Yusuf *et al*. (2011).

Physical observation of the natural soil showed that the soil is dark brown, while the various test results conducted are presented in Table 3 as a Summary of Experimental Design.

Table 3: Summary of Results

Run	Factors		Responses				
	Mk (%)	Sand (%)	OMC (%)	MDD (Mg/m^3)	UCS at 28days (kN/m^2)	VSS (%)	K (cm/s)
1	11	11	11.4	1.83	320	5	2.218E^09
2	11	20	11.9	1.76	310	4.9	1.912E^09
3	2	20	9.4	1.75	300	6.1	2.404E^09
4	11	11	11.4	1.83	320	5	2.218E^09
5	20	20	11.9	1.78	355	4	1.601E^09
6	11	11	11.4	1.83	320	5	2.218E^09
7	2	2	9.2	1.87	280	6.8	2.704E^09
8	11	11	11.4	1.83	320	5	2.218E^09
9	11	2	9.8	1.82	325	5.5	2.32 E^09
10	2	11	9.3	1.8	289	6.5	2.549E^09
11	11	11	11.4	1.83	320	5	2.218E^09
12	20	11	11.7	1.81	345	4.3	1.612E^09
13	20	2	12.4	1.88	340	4.5	1.704E^09

Note: MK=Metakaolin, OMC=Optimum Moisture Content, MDD=Maximum Dry Density, UCS=Unconfined Compressive Strength, VSS=Volumetric Shrinkage strength, K=Hydraulic Conductivity.

3.3 Performance Evaluation Test Results

3.3.1 Optimum moisture content (OMC)

The result of optimum moisture content is shown in Table 4, while the analysis of variance result is shown in Table 5

Table 4: OMC Result

Run	Factors		Responses
	Mk (%)	Sand (%)	OMC (%)
1	11	11	11.4
2	11	20	11.9
3	2	20	9.4
4	11	11	11.4
5	20	20	11.9
6	11	11	11.4
7	2	2	9.20
8	11	11	11.4
9	11	2	9.80
10	2	11	9.30
11	11	11	11.4
12	20	11	11.7
13	20	2	12.4

Table 5: ANOVA for OMC

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	11.48	2	5.74	16.33	0.0007
A-MK	10.93	1	10.93	31.13	0.0002
B-SAND	0.5400	1	0.5400	1.54	0.2433
Residual	3.51	10	0.3513		
Lack of Fit	3.51	6	0.5854		
Pure Error	0.0000	4	0.0000		
Cor Total	14.99	12			

Table 4 shows that values of OMC ranged between 9.3% to 12.4% compared to the control of 9.8%. Higher OMC values suggest the presence of fine-grained soils like clays, which require more water for effective compaction. Conversely, lower OMC values indicate coarser soils like sands, which compact well at lower moisture levels. Table 5 shows the ANOVA for OMC. The Model F-value of 16.33 implies the model is significant. P-values less than 0.05 indicate model terms are significant. In this case, A is a significant model term. Values greater than 0.1 indicate the model terms are not significant.

Table 6: Fit Statistics for OMC

Parameter	Value
R^2	0.7656
Adjusted R^2	0.7188
Predicted R^2	0.5547
Adeq Precision	11.5906

The Predicted R^2 of 0.5547 is in reasonable agreement with the Adjusted $R^2$of 0.7188; i.e. the difference is less than 0.2. Adeq Precision measures the signal-to-noise ratio. A ratio of 11.59 is greater than 4 and considered desirable. The model equation for OMC is shown in Equation 1and the 3-D response surface is shown in Figure1.

$$OMC = 8.953 + 0.15 * Mk + 0.0333 * sand \quad \ldots (1)$$

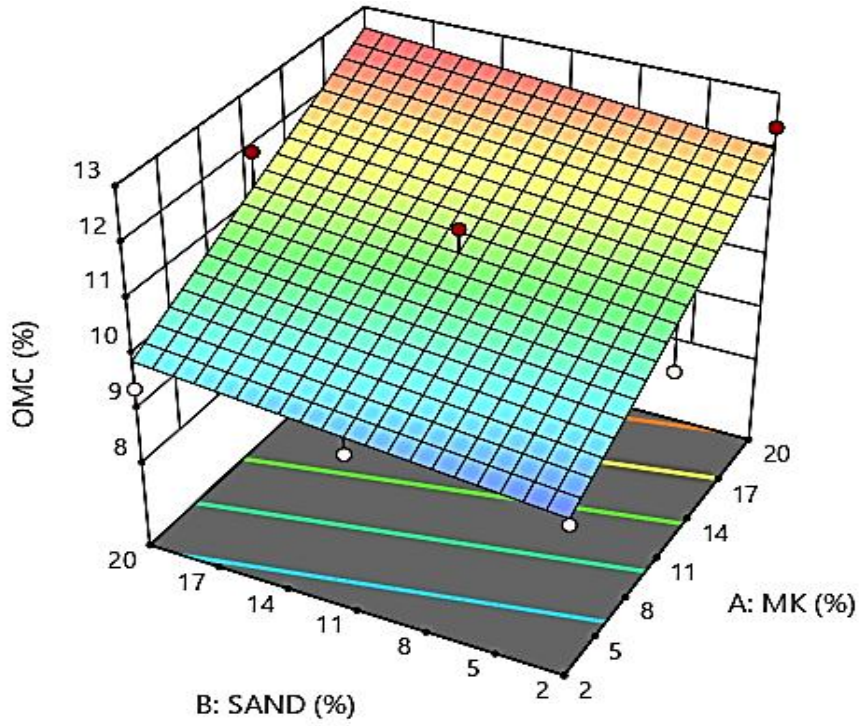


Figure 1: 3D Response Graph of the Effect of Sand and Metakaolin on OMC

Figure 1 shows a 3D surface graph of the interaction between sand and Mk and the influence on optimum moisture content. The 3-D surface elucidates the correlation between the dependent variables (responses) and the independent variables (factors). The graph shows that an increase in Mk and sand causes an increase in OMC.

4.3.2 Maximum dry density (MDD)

The ANOVA result of MDD is presented in Table 7, and the Fit statistics in Table 8.

Table 7: ANOVA forMDD

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	0.0135	2	0.0067	18.76	0.0004
A-MK	0.0004	1	0.0004	1.16	0.3069
B-SAND	0.0131	1	0.0131	36.36	0.0001
Residual	0.0036	10	0.0004		
Lack of Fit	0.0036	6	0.0006		
Pure Error	0.0000	4	0.0000		
Cor Total	0.0171	12			

The Model F-value of 18.76 implies the model is significant. The P-values less than 0.05 indicate model terms are significant. In this case, B is a significant model term. Values greater than 0.1 indicate the model terms are not significant.

Table 8: Fit Statistics for MDD

Parameters	Values
R^2	0.7896
Adjusted R^2	0.7475
Predicted R^2	0.6328
Adeq Precision	12.0792

The Predicted R^2 of 0.6328 is in reasonable agreement with the Adjusted R^2 of 0.7475; i.e. the difference is less than 0.2. Adeq Precision measures the signal-to-noise ratio. A ratio of 12.079 indicates an adequate signal. The model equation for MDD is shown in Equation 2

$$MDD = 1.864 + 0.0009 * Mk - 0.0052 * sand \quad \ldots (2)$$

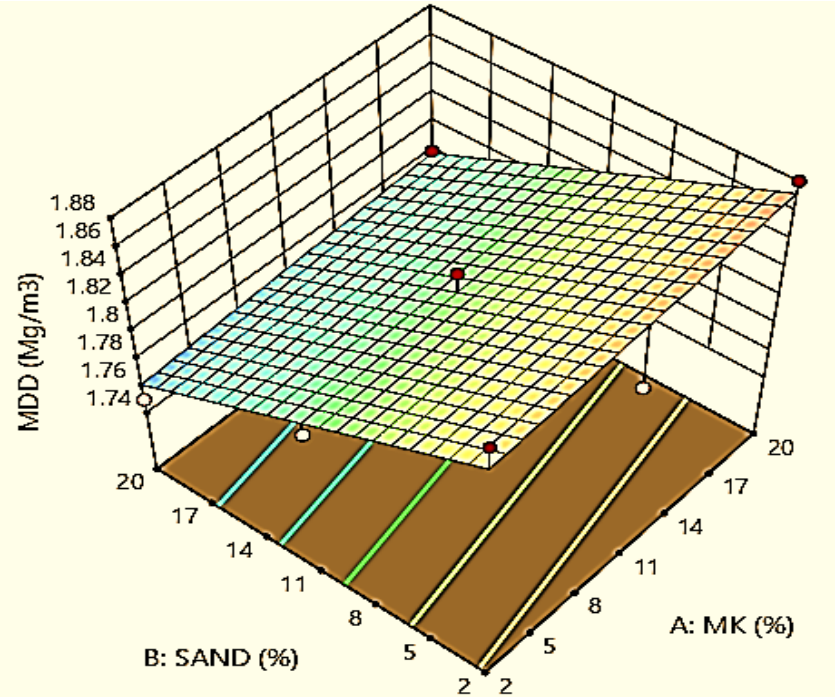


Figure 2: 3-D Response Graph of Effect of Sand and Metakaolin on MDD

Figure 2 shows the 3D surface graph of the interaction between sand and Mk and the influence on maximum dry density. The 3-D surface elucidates the correlation between the dependent variable (MDD) and the independent variables (sand and metakaolin). The graph confirms that increase in Mk shows a significant increase in MDD due to its pozzolanic reaction.

4.3.3 Result of unconfined compressive strength (UCS)

The 28 days UCS Analysis of variance is shown in Table 9 while the Fit statistics is presented in Table 10

Table 9: ANOVA for UCS

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	4940.17	2	2470.08	65.67	< 0.0001
A-MK	4873.50	1	4873.50	129.57	< 0.0001
B-SAND	66.67	1	66.67	1.77	0.2126
Residual	376.14	10	37.61		
Lack of Fit	376.14	6	62.69		
Pure Error	0.0000	4	0.0000		
Cor Total	5316.31	12			

The Model F-value of 65.67 implies the model is significant. P-values less than 0.0500 indicate model terms are significant. In this case, A is a significant model term. Values greater than 0.1 indicate the model terms are not significant.

Table 10: Fit Statistics for UCS

Parameter	Value
R^2	0.9292
Adjusted R^2	0.9151
Predicted R^2	0.8509
Adeq Precision	21.6097

The Predicted R^2 of 0.8509 is in reasonable agreement with the Adjusted R^2 of 0.9151; i.e. the difference is less than 0.2. Adeq Precision measures the signal-to-noise ratio. A ratio of 21.610 indicates an adequate signal. The model equation for UCS is shown in Equation 3

$$UCS\ at\ 28\ days\ =\ 279.862\ +\ 3.167 * Mk +\ 0.3704 *\ sand \quad \ldots (3)$$

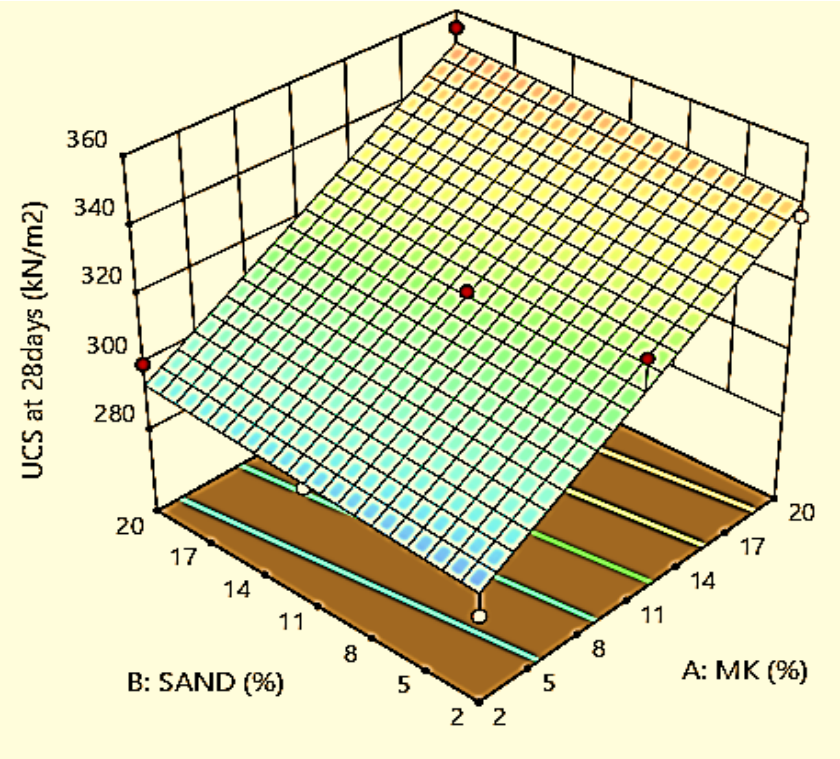


Figure 3: 3-D Response Graph of the Effect of Sand and Metakaolin on UCS

Figure 3 shows a 3D response surface graph of the interaction between sand and Mk and the influence on UCS. The 3-D surface elucidates the correlation between the dependent variables (responses) and the independent variables (factors). The graph shows that an increase in the percentage of sand causes a slight increase in UCS, while an increase in Mk causes a significant increase in UCS. The high increase by metakaolin is connected to the pozzolanic properties of Mk.

4.3.4 Volumetric shrinkage strength (VSS)

Analysis of Volumetric shrinkage is shown in Table 11 while the Fit statistics is shown in Table 12.

Table 11: ANOVA of the Quadratic model for V**SS**

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	8.14	5	1.63	264.29	< 0.0001
A-MK	7.26	1	7.26	1179.02	< 0.0001
B-SAND	0.5400	1	0.5400	87.70	< 0.0001
AB	0.0100	1	0.0100	1.62	0.2432
A^2	0.2155	1	0.2155	34.99	0.0006
B^2	0.0174	1	0.0174	2.82	0.1369
Residual	0.0431	7	0.0062		
Lack of Fit	0.0431	3	0.0144		
Pure Error	0.0000	4	0.0000		
Cor Total	8.18	12			

From Table 11, the Model F-value of 264.29 implies the model is significant. P-values less than 0.05 indicate model terms are significant. In this case, A, B, and A^2 are significant model terms. Values greater than 0.1 indicate the model terms are not significant.

Table 12: Fit Statistics for VSS

Parameters	Value
R^2	0.9947
Adjusted R^2	0.9910
Predicted R^2	0.9641
Adeq Precision	52.5227

The Predicted R^2 of 0.9641 is in reasonable agreement with the Adjusted R^2 of 0.9910; i.e. the difference is less than 0.2. Adeq Precision measures the signal-to-noise ratio. A ratio greater than 4 is desirable. The ratio of 52.523 indicates an adequate signal. This model can be used to navigate the design space. The model equation for VSS is shown in Equation 4. The 3-D response surface for sand, Mk and VSS is shown in Figure 4.

$$VSS = 7.4 - 0.21 * Mk - 0.062 * sand + 0.001Mk * sand + 0.0035 * Mk^2 + 0.001 * sand^2 \quad \ldots (4)$$

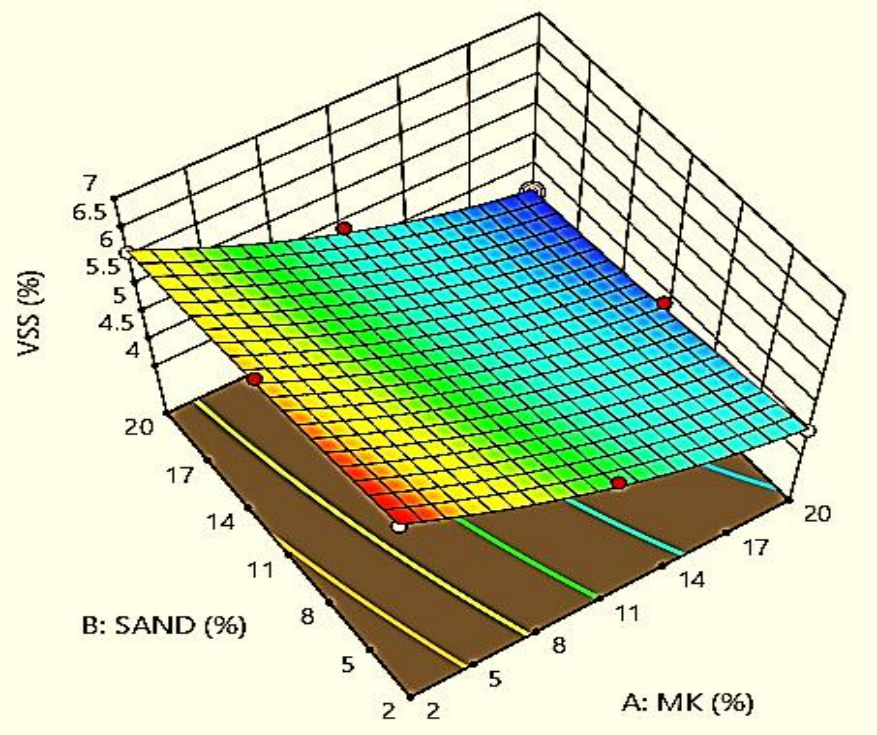


Figure 4: 3-D Response Graph of Effect of Sand and Metakaolin on VSS

Figure 4 shows a 3D surface graph of the interaction between sand and Mk and the influence on volumetric shrinkage strength. The 3-D surface elucidates the correlation between the dependent variable (VSS) and the independent variables (factors, e.g. sand and Mk). The graph shows that an increase in Mk and sand reduces VSS.

4.3.5 Hydraulic conductivity (K)

The analysis of variance and its fit statistics for hydraulic conductivity are presented in Tables 13 and 14 respectively.

Table 13: ANOVA for the Linear Model for K

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	1.36	2	0.6804	100.14	< 0.0001
A-MK	1.25	1	1.25	184.15	< 0.0001
B-SAND	0.1096	1	0.1096	16.13	0.0025
Residual	0.0679	1 0	0.0068		
Lack o Fit	0.0679	6	0.0113		
Pure Error	0.0000	4	0.0000		
Cor Total	1.43	2			

The Model F-value of 100.14 implies the model is significant. The. P-values less than 0.05 indicate model terms are significant. In this case, A, B are significant model terms. Values greater than 0.1 indicate the model terms are not significant.

Table.14: Fit Statistics for K

Parameters	Value
R^2	0.9524
Adjusted R^2	0.9429
Predicted R^2	0.9101
Adeq Precision	29.8921

The Predicted R^2 of 0.9101 is in reasonable agreement with the Adjusted R^2 of 0.9429; i.e. the difference is less than 0.2. Adeq Precision measures the signal-to-noise ratio. A ratio greater than 4 is desirable. The ratio of 29.892 indicates an adequate signal. The model equation for hydraulic conductivity is shown in Equation 5.The 3-D response surface for sand, Mk and hydraulic conductivity is shown in Figure 5.

$$K = 2.87 - 0.051 * Mk - 0.015 * sand \quad \ldots (5)$$

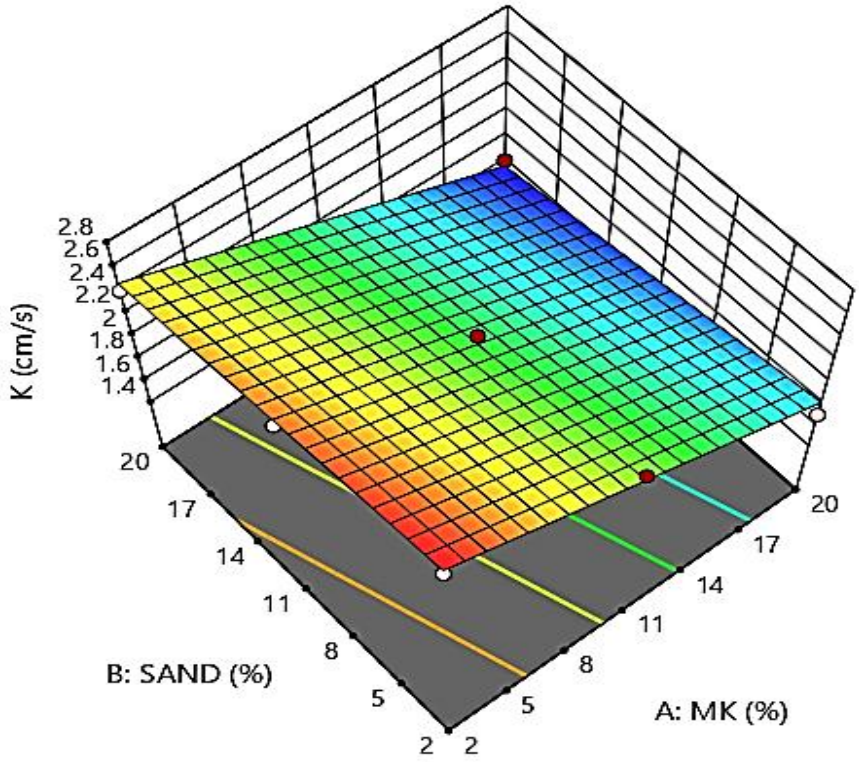


Figure 5: 3-D Response Graph of Effect of Sand and Metakaolin on Hydraulic Conductivity

Figure 5 shows a 3D surface graph of the interaction between sand and Mk and the influence on hydraulic conductivity. The 3-D surface elucidates the correlation between the dependent variable (K) and the independent variables (factors, e.g. sand and Mk). The graph shows that an increase in Mk and sand reduces the hydraulic conductivity of the black cotton soil.

4.4 Optimization of Mixtures by Numerical Method

The goals set for responses in numerical optimization are presented in Table 15.

Table 15: Goals for Numerical Optimisation

Name	Goal	Lower Limit	Upper Limit
A: MK	is in range	2	20
B: SAND	is in range	2	20
OMC	maximize	9.2	12.4
MDD	maximize	1.75	1.88
UCS at 28 days	maximize	280	355
VSS	minimize	4	6.8
K	minimize	1.601	2.704

The automatic optimisation function of Design-Expert software version 13 indicates that the optimal values of the factors for both factors and responses as shown in Figure 6.

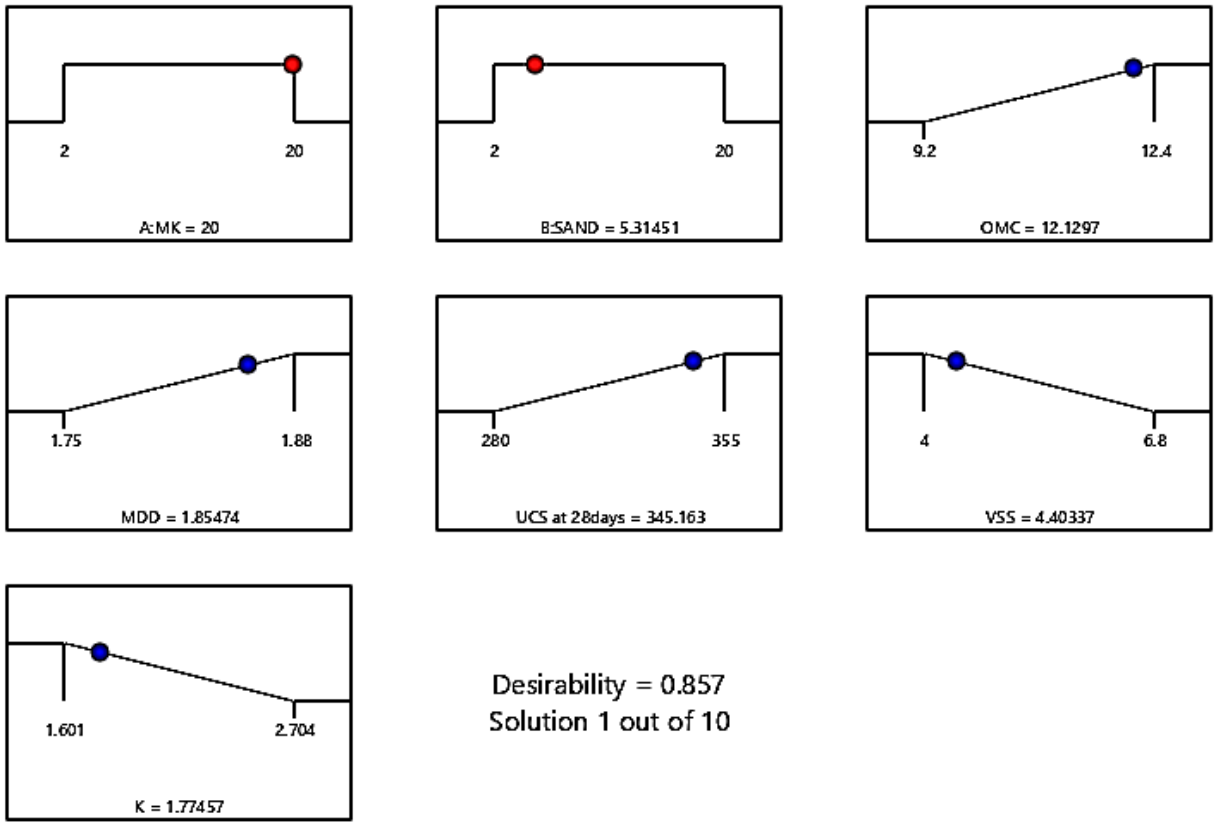


Figure 6: Ramp Plot Showing the Optimal Values for Factors and Responses

4.0. CONCLUSION

After the experimental work, the following conclusions were made:

i The suitability of black cotton soil stabilised with Metakaolin and sand as compacted liners in waste containment was investigated. The BCS was classified as A-7-6 and CH according to the AASHTO and the Unified Soil Classification System. The OMC was 9.8%, MDD was 1.69 Mg/m^3. The soil has a specific gravity of 2.19, Natural moisture content of 0.71%, LL of 44.9% and PL of 25.8%.

ii The replacement of black cotton soil with Metakaolin and sand reduces the hydraulic conductivity and Volumetric Shrinkage of the soil. The hydraulic conductivity of black cotton soil was 1.66397E^06 while its shrinkage was 8.98%. The best result recorded was achieved at 20% Mk and 5% sand, with hydraulic conductivity and volumetric shrinkage of 1.601E^06 and 4% respectively.

iii Optimisation was carried out and models were developed for the estimation of OMC, MDD, VSS, hydraulic conductivity, and UCS of admixed black cotton soil. The numerical optimisation was done using central composite design, and the ramp plot gave 20% Mk, 5% Sand, 12% OMC, 1.85% MDD, 345 kN/m^2 for UCS at 28 days, VSS at 4.4% and 1.7 cm/s for hydraulic conductivity.

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