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Performance Evaluation and Microstructural Analysis of Concrete Containing Metakaolin and Neem Seed Ash.

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Abstract

This experimental research assessed the performance of concrete containing Metakaolin (Mk) and Neem Seed Ash (NSA). Portland Limestone Cement was partially replaced by metakaolin and neem seed ash at different proportions in concrete. The performance of the admixtures in concrete mixtures was assessed in terms of mixture workability (Slump test) and strength (compressive and tensile strength) properties. The result of the slump test for the Mk/NSA concrete showed that they were workable. The result was 42 mm for control, Mk 2.5%, NSA 2.5% was 44 mm, Mk 5%, NSA 5% was 48 mm, 7.5% Mk, 7.5% NSA was 51 mm, and 10% Mk, 10% NSA was 55 mm. The higher the replacement of cement with Mk/NSA, the higher the slump value. The consistency of cement was 29% and increased with increasing NSA and Mk content. At replacement of cement by Mk/NSA at 5, 10, 15 and 20%, the consistency values were 31, 33, 35 and 38%. Optimization of compressive and tensile strength of the admixed concrete by Response Surface Methodology was done using Box-Behnken Design (BBD). The optimal result obtained was 5% Mk and 5% NSA at 80 days of curing gave maximum compressive strength of 34.2 N/mm^2 and tensile strength of 3.5N/mm^2. BBD was used for analysis of variance and for developing model equations for compressive and tensile strength. Microstructural analysis of the admixed concrete showed a denser and reduced pore structure compared to the control sample. It was concluded that Mk and NSA are useful materials in production of viable and cost-effective concrete. Further research should be carried out to evaluate the effects of NSA in ternary mixture with other waste agricultural or industrial materials in concrete production.

Keywords:

Neem Seed Ash, Metakaolin, Optimization, Strength, Microstructural Analysis

1.0 Introduction

Global infrastructural development has led to drastic surge in the usage of concrete, thereby retaining its spot as the most commonly used building material (Faraj *et al.*, 2022).

Cement, a major binder in concrete production is very harmful to the environment due to carbon dioxide emission from its production (Sani and Adetoye, 2024; Pam *et al.,* 2025). Reducing cement production while maintaining sustainable development has been an important issue in the development of construction materials. Efforts are geared towards providing alternative, cheap and affordable materials that can replace cement in concrete. Sarki-Shanu *et al.* (2025) said most researchers in construction industry are curtailing the price of

building materials by the use of locally available materials for concrete. Replacing Portland cement with percentages of pozzolana has been reported as a good alternative. Agricultural and industrial waste materials can be used as partial replacement for cementing material (Ajwad *et al*., 2022; Adanu *et al*., 2024).
Over the years, researchers have suggested different industrial by-products and agricultural residues as alternative binders in the production of sustainable concrete (Raheem *et al*., 2021).

Metakaolin is a pozzolanic material and obtained by calcinations of kaolinite clay at temperatures from 700°C to 800°C (Afolayan *et al*., 2022; Abdullahi *et al*., 2024). The chemical composition is basically aluminous silicates hydrates associated with Mn, Fe, Ca, K, Na. Its crystal has a lattice structure of tetrahedral and octahedral layers.
In Nigeria, neem tree (Azadirachtaindica) is locally known as “dogonyaro”, “darbejiya” in Hausa, “igi-oba” in Yoruba and “ogwu-akom” in Igbo. Neem trees are found generally in every state of the country growing wild. The production of neem products from neem tree generates large quantity of neem seed as waste annually. There is need to reduce environmental pollution resulting from neem seed and therefore necessitate the burning to produce Neem Seed Husk Ash (NSHA) which is a good pozzolan.
Neem Seed Ash (NSA) is a suitable material for use as a pozzolan, since it satisfies the requirement for such a material by having a combined SiO_2, Fe_2O_3 and Al_2O_3 of more than 70 (Aishwaryalakshmi *et. al,* 2018).
The Production of the admixed concrete containing Mk/NSA results to reduction in use of cement and emission of fewer greenhouse gasses compared to traditional concrete. Incorporating locally sourced cheap materials makes the admixed concrete to be cost effective, compared to conventional concrete. Finally, this study will stimulate local economic growth by creating new job opportunities for people in the production of this admixed concrete.

2.0 MATERIALS AND METHODS

2.1 Materials

The materials to be used for the study include cement, metakaolin, neem seed husk, fine and coarse aggregates and water.

2.1.1 Cement

Portland cement was used as binder material in concrete mix. In the present study, portland limestone cement of 42.5 grade conformed to Nigeria Industrial Standard 444-14 was used.

2.1.2 Metakaolin

Metakaolin, an innovative clay product produced by controlled thermal treatment of kaolin gotten from Alkaleri in Bauchi state, Nigeria. Metakaolin was obtained by calcination of pure or refined kaolinitic clay at a temperature of 650^{o}C using a kiln fuelled by kerosene, at the Industrial Design Laboratory, ATBU Bauchi. On completion of burning, metakaolin was allowed to cool before grinding using pestle and mortar to a very fine texture. The resulting ash was required to pass through sieve of size 150µm, to obtain the required fineness. The determination of chemical composition of the ash was determined by X-ray fluorescence in accordance with specifications of ASTM E1621-13, at Ashaka Cement factory located at Bajoga in Gombe State.

2.1.3 Neem Seed Husk Ash

The Neem seed pod was collected from Bauchi metropolis in Bauchi State Nigeria. After collection, the seed was sun dried for three (3) days to reduce its moisture content and burnt in a furnace at a temperature of 650^{o}C at the controlled kiln in ATBU Bauchi. The resulting fine powder residue was passed through sieve size I50µm, to obtain the final sample of NSA to be used for the study. The chemical composition of the ash was determined by X-ray fluorescence in accordance with specifications of ASTM E1621-13, at Ashaka Cement factory located at Bajoga in Gombe State.

2.1.4 Aggregates

The fine aggregate used was obtained from a stream after Bayara, along Bauchi-Dass Road. The coarse aggregate for the work was crushed granite stone of maximum 20mm size, and was purchased from local supplier in Yelwa, Bauchi.

2.1.5 Water

Potable water was obtained from local well within Engineering block of ATBU, Yelwa campus. The water was clean and free from any detrimental contaminant.

2.2 Methodology

2.2.1 Concrete Mix Design

The concrete mix design for the concrete of M20 grade were based on the principles of existing American Concrete Institute Method of Mix Design (ACI–211.2003). Detail of the quantities of the concrete materials based on the mix ratio of cement: fine aggregate: coarse aggregate: water /cement ratio: superplasticizer. 1: 2.81: 2.30: 0.52: 0.02 are shown in Table 1.

Table 1: Mix Design of Concrete

Material		Normal Concrete
1	Cement (kg/m^3)	355
2	Water (litre/m^3)	185
3	Fine aggregate (kg/m^3)	800
4	Coarse aggregate (kg/m^3)	1016
5	Water/cement ratio	0.52
6	Superplasticizer (%)	7.1

2.2.2 Experimental Design

The Design-Expert software 13 was used to optimize the results of compressive and split tensile strength of concrete. The software was used to analyse variance and generate mathematical models. Response surface methodology (RSM) was adopted in the design of experimental combinations and also be used to quantify the relationship between the controllable input parameters and the obtained response surfaces. Experimental runs were created by Design-Expert software 13 using BBD response surface methodology for an M20 grade concrete. The parameters used were Metakaolin from 5 - 10%, NSA 5 - 10% and curing age (CA) from 3 - 90days as shown in Table 2.

Table 2: Factor and Factor Levels of Mixtures

Name	Units	Low	Middle	High
Mk	%	5	7.5	10
NSA	%	5	7.5	10
C.A	Days	3	28	90

2.2.3 Batching, Casting and Curing of Concrete

The constituents were measured using a manual weighing machine before mixing. Concrete mixtures were batched based on mix design. Specimens were removed from the moulds a day after casting and cured in water by immersion prior to testing according to BS 1881:111- (1983).

2.2.4 Standard Consistency Test and Setting Time

ASTM 187 was followed to achieve standard consistency with cement and combination with admixtures. The principle of standard consistency of cement is that consistency at which the Vicat plunger penetrates to a point 5-7mm from the bottom of Vicat mould.

Setting times for Cement/NSA/Mk pastes were determined using the Vicat apparatus, in accordance with the procedure outlined in ASTM C 150. The procedure consists of using a round needle with a diameter of 1.13 ± 0.05mm to penetrate the paste.

2.2.5 Workability (Slump Test)

Slump test of the freshly prepared concrete were carried out to determine the effect of MK/NSA cement replacement on the workability of concrete. The slump test is the most well-known and widely used test method to characterize the workability of fresh concrete. The inexpensive test, which measures consistency, is used on job sites to determine rapidly whether a concrete batch should be accepted or rejected. The test method is widely standardized throughout the world, as shown in ASTM C143 in the United States. The only type of slump permissible under ASTM C143 is frequently referred to as the "true" slump, where the concrete remains intact and retains a symmetric shape. A zero slump and a collapsed slump are both outside the range of workability that can be measured with the slump test. If part of the concrete shears from the mass, the test must be repeated with a different sample of concrete. A concrete that exhibits a shear slump in a second test is not sufficiently cohesive and should be rejected. Different types of slump are shown in Figure 1.

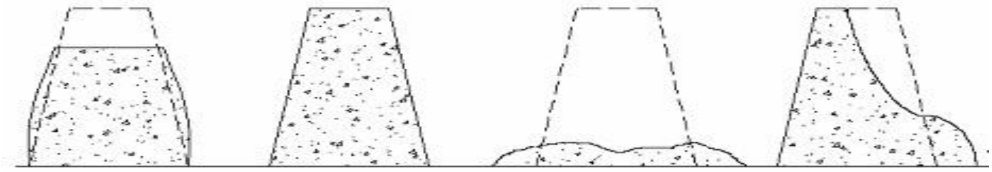


Figure 1: Four Types of Slumps.

2.2.6 Hardened Properties of Concrete

(a) Splitting Tensile Strength

The splitting tensile test was carried on cylindrical concrete specimens of 100 mm diameter and 200 mm height at ages of 3, 28 and 90 days, in accordance with provisions of BS EN 12390- 5:2000. The specimens were tested using a 3000kN capacity compression machine. The loading rate were kept constant at 0.3kN/min until the splitting tensile failure occurred. The split tensile stress was calculated using Equation 1 in accordance with ASTM C496.

$$f_t = \frac{2P}{\pi L D} \quad \text{(N/mm}^2\text{)} \qquad \text{... (1)}$$

Were

P = Maximum force applied to the specimen (N)
D = the diameter of the cylindrical specimen (mm)
L = length of the cylinder (mm).

After casting, the specimens were compacted on a mechanical vibrating table and left at room temperature for 24 hours. They were de-moulded and placed inside the curing tank until the testing day.

(b) Compressive Strength

The compressive strength test was carried out on cubic specimens of size (100 mm x 100 mm x 100 mm), in accordance to ASTM C109. The strength was recorded at 3, 28 and 90 days respectively. The average reading of three cubes was recorded as the strength at the respective age. The compressive strength was calculated using the Equation 2.

$$\text{Compressive strength} = \frac{Failure\ Load}{Area\ of\ Specimen} N/mm^2 \qquad \text{... (2)}$$

3.0 RESULTS AND DISCUSSION

3.1 Properties of Binders

The oxide composition of binders (Cement, metakaolin and NSA) is presented in Table 3.
Table 3: Oxide Composition of Cement, Mk and NSA

Oxide composition %	Cement	Mk	NSA
SiO_2	22.5	52.3	67.8
Al_2O_3	6.2	34.1	2.71
Fe_2O_3	4.1	3.5	3.29
CaO	61.8	0.44	2.71
MgO	1.4	0.07	0.45
SO_3	2.88	0.32	0.38
K_2O	0.72	0.48	10.9
Na_2O	0.13	0.12	0.17
LOI	3.2	7.5	12.20

In Table 3, the respective percentage contents of the major oxides of cement are SiO_2 as 22.5%, Al_2O_3 as 6.2%, Fe_2O_3 as 4.2% and CaO as 61.8% whereas the percentage contents of the minor oxides were: MgO as 1.4% SO_3 as 2.88 and the alkalis (Na_2O and K_2O) as 0.13% and 0.72 %. The loss of ignition as 3.2. This shows that the cement used for this research work conforms to the Nigeria Industrial Standard. The respective percentage contents of the major oxides of Mk are SiO_2 as 52.3%, Al_2O_3 as 34.1, Fe_2O_3 as 3.5% and CaO as 0.44% whereas the percentage contents of NSA are SiO_2 as 67.8%, Al_2O_3 as 2.71, Fe_2O_3 as 3.29% and the alkalis Na_2O as 0.17%. This shows that metakaolin and NSA have enough silica oxide and calcium oxide which enhance better strength development of concrete. The sum of SiO_2 +Al_2O_3 + Fe_2O_3 exceeds 70%. This demonstrated that the Mk and NSA are in the same category with the Class F fly ash (ASTM C618-05, 2005) with high pozzolanic properties.

3.2 Fresh Paste Properties of Mk and NSA

The consistency result, initial and final setting time of paste is shown in Table 4.Table
4: Consistency and Setting Time Results

Cement replaced by MK and NSA **(%)**	Consistency (%)	Initial setting time (mins)	Final setting time (mins)
Control	29	65	135
2.5 Mk, 2.5 NSA	31	105	181
5 Mk, 5 NSA	33	120	192
7.5 Mk, 7.5 NSA	35	135	210
10 Mk, 10 NSA	38	155	215

The consistency value of the cement is lower than the values of blended cement with NSA and Mk. The consistency of cement was 29% and increased with increasing NSA and Mk content. At replacement of cement by Mk/NSA at 5, 10, 15 and 20%, the consistency values were 31, 33, 35 and 38%. This trend may be due to the finer particles of NSA and Mk which are finer than those of cement and consequently increase the surface area available for contact with water thereby increasing the water demand of the OPC/NSA/Mk blended Paste. Results also show that as the percentage of NSA/Mk increase, the initial and final setting time also increase. These admixtures act as retarding agent for a concrete. The results were in accordance with the study conducted by (Egwuonwu *et al.,* 2019).

3.3 Workability of Concrete (Slump test result)

Workability of concrete was tested by slump test. The result of the slump test is presented in the Table 5.
Table 5: Result of Slump Test

Percentage Replacement of Cement	Slump Height (mm)
Control (0%)	42
Mk 2.5%, NSA 2.5%	44
Mk 5%, NSA 5%	48
Mk 7.5%, NSA 7.5%	51
Mk 10%, NSA 10%	55

The result of the slump test for the concrete with Mk and NSA replacement showed that at 0% (control), the result was 42 mm, 5% (Mk 2.5%, NSA 2.5%) was 44 mm, 10% (Mk 5%, NSA 5%) was 48 mm, 15% (7.5% Mk, 7.5% NSA) was 51 mm, and 20% (10% Mk, 10% NSA) was 55 mm. All replacements showed that concretes made with Mk and NSA were workable. The higher the replacement of cement with Mk and NSA, the higher the slump value. This shows that the Mk and NSA absorb more water. The slump result indicates a true slump which shows that the concrete is workable. The increase in the water consistencies could be attributed to the diminution of C_3S in cement, the unburnt carbon present in the ashes coupled with the porous nature of Mk and NSA and the narrower particle size distributions of the cement blends.

3.4 Results of Admixed Concrete with Mk and NSA

3.4.1Compressive strength properties

The result of the compressive strength test conducted on admixed concrete at 3 days, 28 days and 90 days are presented in Table 6.

Table 6: Compressive Strength Result

Run	Factor 1 A: NSA %	Factor 2 B: Mk %	Factor 3 C: C.A Days	Response 1 Compressive Strength N/mm^2
Control	0	0	90	26.2
1	5	7.5	90	28.0
2	5	5	28	34.0
3	7.5	10	3	10.0
4	7.5	7.5	28	26.0
5	7.5	7.5	28	26.0
6	10	10	28	23.1
7	5	10	28	27.0
8	10	7.5	90	27.0
9	7.5	7.5	28	26.0
10	7.5	5	3	15.0
11	10	7.5	3	12.0
12	7.5	10	90	27.0
13	10	5	28	25.7
14	5	7.5	3	15.0
15	7.5	7.5	28	26.0
16	7.5	7.5	28	26.0
17	7.5	5	90	28.4

The compressive strength of concrete containing metakaolin and Neem Seed Ash shows good performance than the control mix. The best result of 28.4 N/mm^2 was achieved at 7.5% of NSA and 5% of Mk at 90 days of curing. Concrete strength was enhanced in approximate proportion to the degree of addition of Mk and NSA. Compared to the findings from other studies, it appears that the results of this study adhere with some of the studies conducted by Ubojiekere *et al.* (2018). They reported the highest compressive strength when Metakaolin and NSA replaced cement in their research. For all mixtures, Metakaolin increased compressive strength appreciably. The analysis of variance and Fit statistics for compressive strength are shown in Table 7 and 8.

Table 7: ANOVA for Quadratic Model of Compressive Strength

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	678.85	9	75.43	73.02	< 0.0001
A-NSA	25.08	1	25.08	24.28	0.0017
B-Mk	23.70	1	23.70	22.95	0.0020
C-C. **A**	426.32	1	426.32	412.69	< 0.0001
AB	4.84	1	4.84	4.69	0.0672
AC	3.21	1	3.21	3.11	0.1212
BC	4.20	1	4.20	4.07	0.0836
A^2	3.60	1	3.60	3.49	0.1041
B^2	1.16	1	1.16	1.12	0.3244
C^2	349.14	1	349.14	337.98	< 0.0001
Residual	7.23	7	1.03		
Lack of Fit	7.23	3	2.41		
Pure Error	0.0000	4	0.0000		
Cor Total	686.08	16			

The Model F-value of 73.02 implies the model is significant. P-values less than 0.05 indicate model terms are significant. In this case A, B, C, C^2 are significant model terms. Values greater than 0.1 indicate the model terms are not significant. The model equation for compressive strength is shown in Equation 3.

$$Compressive\ strength = 16.4 - 0.36A - 1.76B + 0.91C - 0.012AB - 0.009AC + 0.012BC + 0.046A^2 + 0.062B^2 - 0.01C^2 \quad ...(3)$$

Table 8: Fit Statistics for Quadratic Model of Compressive Strength

Parameters	Values
Std. Dev.	1.02
Mean	23.66
C.V. %	4.30
R^2	0.9895
Adjusted R^2	0.9759
Predicted R^2	0.8317
Adeq Precision	29.4268

The Predicted R^2 of 0.8317 is in reasonable agreement with the Adjusted R^2 of 0.9759; i.e. the difference is less than 0.2. Adeq Precision ratio greater than 4 is desirable. The ratio of 29.43 indicates an adequate signal. The adjusted R^2 indicates that the model can explain response value change of 98%. Factors interactions and effect on compressive strength are shown in Figure 2 and 3.

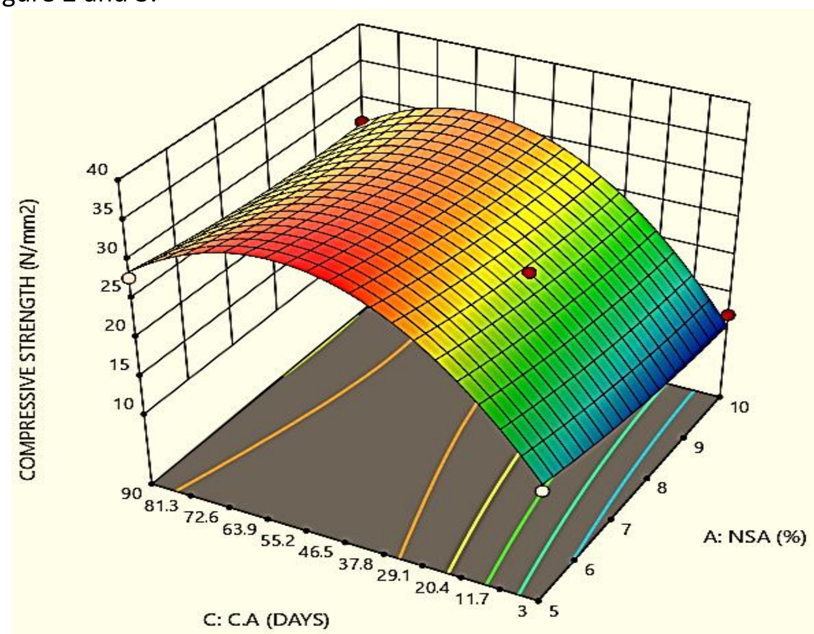


Figure 3: 3D Response Graph of Mk, Curing and Compressive Strength.

Figure 2 shows the influence of NSA and curing on compressive strength of admixed concrete. The higher the curing age, the higher the compressive strength while slight improvement was witnessed as the NSA increased from 5 to 10%. Figure 3 shows the influence of Mk and CA on compressive strength of concrete. Metakaolin significantly improved the compressive strength of concrete within the limits and the higher the curing age, the higher the compressive strength.

3.4.2 Tensile strength properties

The tensile strength property of the admixed concrete produced was determined after curing at 3, 28 and 90 days. The result of the test is shown in Table 9 and ANOVA in Table 10.

Table 9: Tensile Strength Result

Run	Factor 1 A: NSA %	Factor 2 B: Mk %	Factor 3 C: C.A Days	Response 2 Tensile Strength N/mm^2
1	5	7.5	90	3.5
2	5	5	28	3.0
3	7.5	10	3	2.0
4	7.5	7.5	28	2.3
5	7.5	7.5	28	2.3
6	10	10	28	2.4
7	5	10	28	2.7
8	10	7.5	90	3.2
9	7.5	7.5	28	2.3
10	7.5	5	3	2.5
11	10	7.5	3	2.2
12	7.5	10	90	3.3
13	10	5	28	2.6
14	5	7.5	3	2.2
15	7.5	7.5	28	2.3
16	7.5	7.5	28	2.3
17	7.5	5	90	3.4

The 3 days' tensile strength ranged from 2.0 to 2.2 N/mm^2. At latter ages (28, and 56) days, Mk and NSA addition increased the strength of admixed concrete possibly due to an improved transition zone. Mk and NSA slow down early age strength gain but boosts later age strength. This is because the pozzolanic reaction starts later than the hydration of the cement, and continues even after the cement stopped hydrating.

Table 10: ANOVA for Tensile Strength Model

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	3.60	9	0.4004	67.49	< 0.0001
A-NSA	0.1356	1	0.1356	22.86	0.0020
B-Mk	0.1047	1	0.1047	17.65	0.0040
C-C. **A**	2.53	1	2.53	426.73	< 0.0001
AB	0.0025	1	0.0025	0.4215	0.5369
AC	0.0106	1	0.0106	1.79	0.2233
BC	0.0329	1	0.0329	5.54	0.0507
A^2	0.1289	1	0.1289	21.74	0.0023
B^2	0.1684	1	0.1684	28.39	0.0011
C^2	0.0142	1	0.0142	2.39	0.1657
Residual	0.0415	7	0.0059		
Lack of Fit	0.0415	3	0.0138		
Pure Error	0.0000	4	0.0000		
Cor Total	3.64	16			

The **Model F-value** of 67.49 implies the model is significant. **P-values** less than 0.05 indicate model terms are significant. In this case A, B, C, A^2, B^2 are significant model terms. Values greater than 0.1 indicate the model terms are not significant.

Parameters	Values
Std. Dev.	0.077
Mean	2.62
C.V. %	2.94
R^2	0.9886
Adjusted R^2	0.9740
Predicted R^2	0.8241
Adeq Precision	19.0981

In Table 11, the Predicted R^2 of 0.8241 is in reasonable agreement with the Adjusted R^2 of 0.9740; i.e. the difference is less than 0.2. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. A ratio of 19.098 indicates an adequate signal. The model equation for Tensile Strength is shown in Equation 4.

$$T.S = 6.5 - 0.5A - 0.6B + 0.01C + 0.004AB - 0.001AC + 0.001BC + 0.03A^2 + 0.03B^2 + 3.9e - 05C^2 \ldots(4)$$

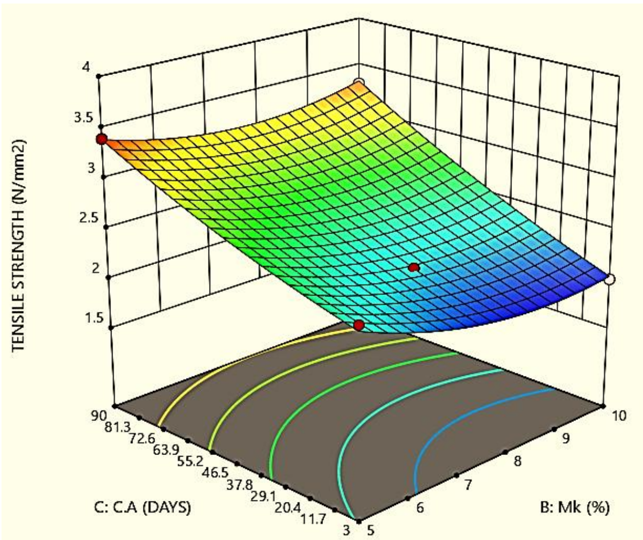


Figure 4: 3D Response Graph of NSA, Curing and Tensile Strength.

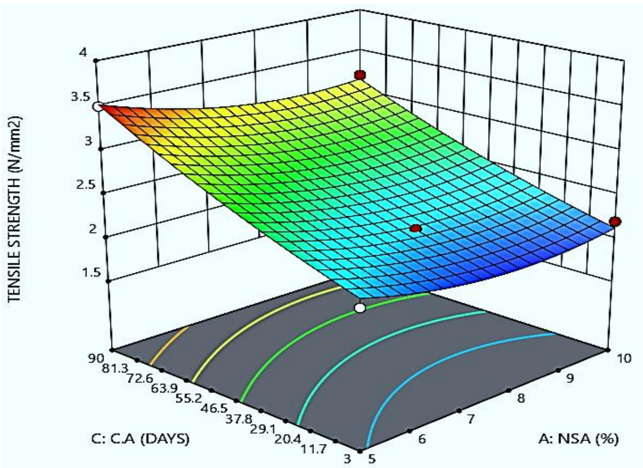


Figure 5: 3D Response Graph of NSA, Curing and Tensile Strength.

3.5 Optimization of Concrete

The goals set for responses in numerical optimization are presented in Table 12.

Table 12: Goals for Numerical Optimization.

Name	Goal	Lower Limit	Upper Limit
A: NSA	is in range	5	10
B: Mk	is in range	5	10
C: C.A	is in range	3	90
Compressive Strength	maximize	10	34
Tensile Strength	maximize	2	3.5

The automatic optimization function of Design-Expert software version 13 indicates that the optimal values of the factors as 7% Mk and 5.5% NSA at 46 days of curing with combined desirability of 1 as presented in Figure 6.

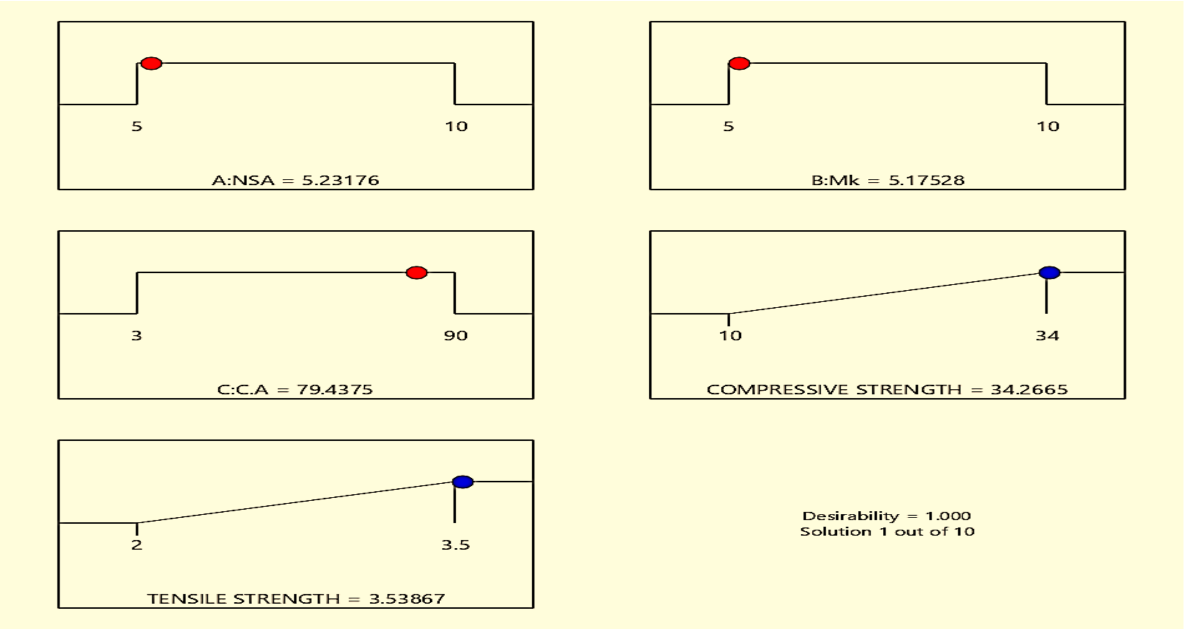


Figure 6: Ramp Plot Showing the Optimal Values for Responses

3.6 Microstructural Analysis of Concrete

Scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) analysis were used to examine the nature of the hydrated binder and the binder-aggregate interfacial zones. SEM and EDX analysis of concrete were conducted at 28 days of curing. SEM was used as an adjunct to optical microscopy to obtain high-magnification images of concrete fracture surfaces.

SEM Image of Pore Structure of Concrete (Control) and also admixed concrete at 28 days are shown in Plate I and II. Figure 7 and 8 show the EDX Spectrum of Control and admixed concrete at 28 Days of Curing

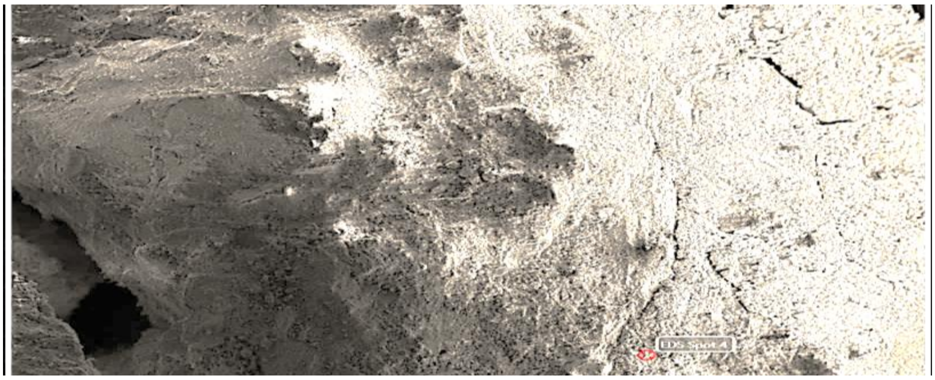


Plate I: SEM Image of Pore Structure of Concrete (Control) at 28 days.

Figure 7: EDX Spectrum of Control at 28 Days of Curing

Table 13: Quant Result - Analysis Uncertainty for Control

Element	Weight %	MDL	Atomic %
C K	7.14	1.51	12.43
O K	48.46	0.43	63.32
Al K	1.52	0.33	1.18
Si K	5.14	0.35	3.83
S K	0.90	0.44	0.59
K K	6.30	0.87	3.37
Ca K	27.34	2.16	14.26
Zn L	3.19	0.84	1.02

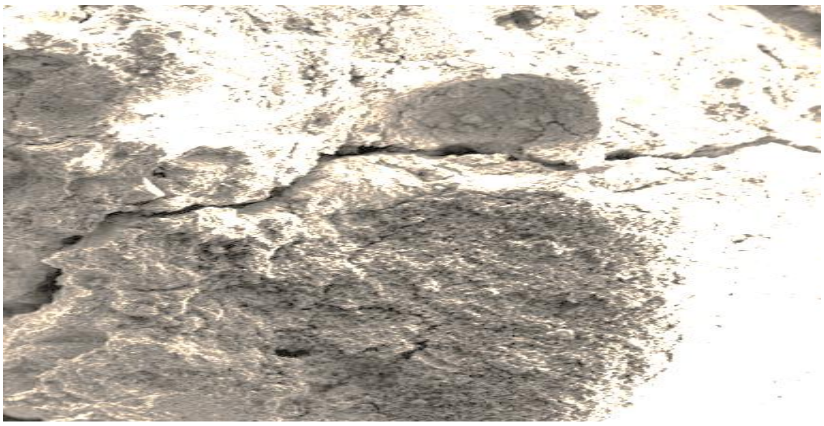
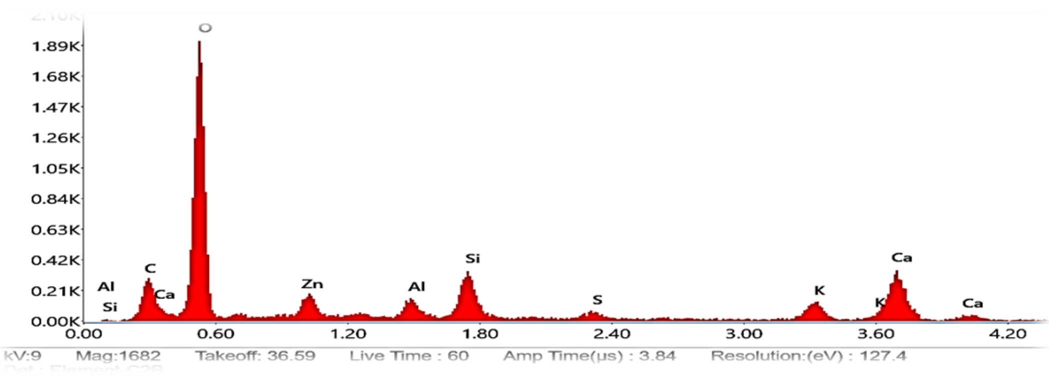


Plate II: SEM Image of Pore Structure of *Mk-NSA* Concrete (5% Mk - 5% NSA)



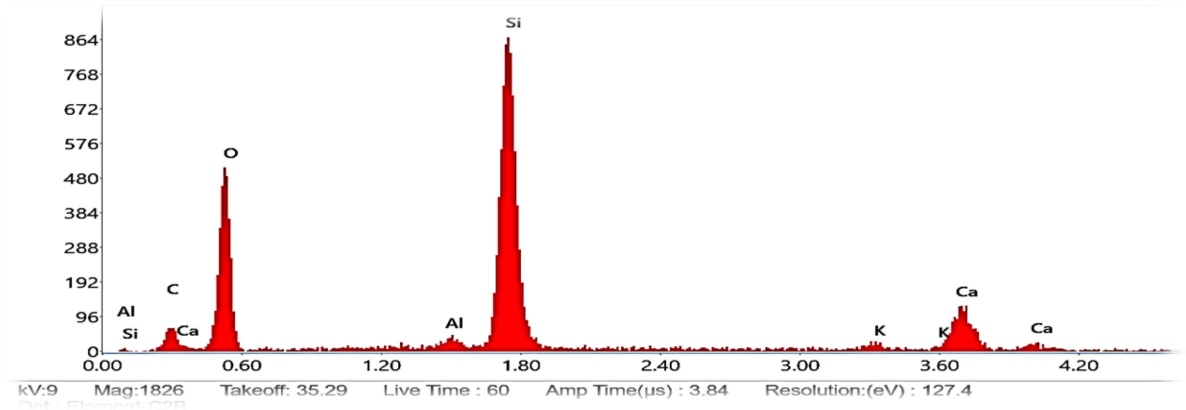


Figure 8: EDX Spectrum of Mk-NSA at 28 Days of Curing.

Table 14: Quant Result - Analysis Uncertainty for Admixed Concrete

Element	Weight %	MDL	Atomic %
CK	8.54	2.42	15.68
OK	31.34	0.41	43.18
Al K	0.99	0.35	0.81
Si K	33.24	0.39	26.08
K K	1.79	1.28	1.01
Ca K	24.10	2.36	13.25

The image in Plate I appears to be loosely packed and thus porous, on the other hand, the SEM images of Plate II containing 5% Mk and 5% NSA have few calcium hydroxide Ca $(OH)_2$ platelets and the pores are smaller and appear denser compared to the control. The pozzolanic effect involving the consumption of calcium hydroxide Ca $(OH)_2$ to produce the secondary C-S-H gel is seen in Plate II.

The high content of calcium (Ca) and Silica (SiO_2) was found in the EDX spectrum as presented in Figure 7 while the high content of silica oxide as shown in EDX spectrum in Figure 8 is attributed to the effect of pozzolanic reaction. Thus, it could be concluded that the improvement in the tensile and compressive strengths of admixed concrete with Mk and NSA was as a result of densification and pore refinement.

4.0 CONCLUSIONS AND RECOMMENDATIONS

4.1 Conclusions

Performance evaluation of concrete containing metakaolin and neem seed ash was studied, analysed and optimized using Box-Behnken Design. Based on the findings, the following conclusions were made:

the respective percentage contents of the major oxides of Mk are SiO_2 as 52.3%, Al_2O_3 as 34.1, Fe_2O_3 as 3.5% and CaO as 0.44% whereas the percentage contents of NSA are SiO_2 as 67.8%, Al_2O_3 as 2.71, Fe_2O_3 as 3.29% and the alkalis Na_2O as 0.17%. This shows that metakaolin and NSA have enough silica oxide and calcium oxide which enhance better strength development of concrete. The sum of SiO_2 +Al_2O_3 + Fe_2O_3 exceeds 70%. This demonstrated that the Mk and NSA are with high pozzolanic characteristics.

the consistency of cement was 29% and increased with increasing Mk and NSA content at equal quantities. At 5, 10, 15, and 20% cement replaced by Mk and NSA, the values were 34, 35, 37, and 41%. The result of the slump test for the concrete Mk and NSA replacement of the cement showed that at 0% (control), the result was 42 mm, 5% (Mk 2.5%, NSA 2.5%) was 44 mm, 10% (Mk 5%, NSA 5%) was 46 mm, 12.5% (5% Mk, 7.5% NSA) was 49 mm, 17.5% (10% Mk, 7.5% NSA) was 53 mm, and 20% (10% Mk, 10% NSA) was 55 mm. All replacements showed that concretes made with Mk and NSA were workable.

the compressive strength of concrete containing Mk/NSA show good performance than the control mix. The best result of 34 N/mm^2 was achieved at 5% of NSA and 5% of Mk at 28 days of curing. The 3 days' tensile

strength ranged from 2 to 2.2 N/mm^2. At latter ages (28, and 90) days, Mk and NSA addition increased the strength of admixed concrete possibly due to an improved transition zone.
the optimization function indicates that the optimal values of the factors as 5% Mk and 5% NSA at 80 days of curing, 34.2 N/mm^2 for compressive strength and 3.0 N/mm^2 for Tensile strength.
the microstructural analysis of the admixed concrete showed denser structure compared to normal concrete.

4.2 Recommendations

This study recommends the use of Mk at 5% and 5% NSA as cement replacement in M20 grade concrete.
The study also recommends the use of other waste materials in ternary mixture with NSA.

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