Strength Properties of Nano Silica Concrete Produced with Millet Husk Ash as Partial Replacement of Cement

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In the construction industry, concrete is one of the most commonly utilized materials, with production reaching approximately 27.3 billion tons annually as of 2021. When compared to other building materials such as steel, wood, and plastic, its global usage is significantly higher. Nevertheless, its low compressive-to-tensile strength, pronounced brittleness, and limited flowability, coupled with the impact of harsh environmental conditions, raise concerns about its longevity. This study, provide results on the compressive and flexural strength of concrete mixtures at intervals of 7, 21, and 28 days, focusing on 100 mm concrete cubes and 100 mm x 500 mm concrete beams with 0, 1.5, 3, and 4.5% Nano silica added to 5, 10, and 15% MHA, at a water-binder ratio of 0.5. The specimens were cured in water for 7, 21, and 28 days. The concrete mix containing 5% MHA and 1% Nano silica exhibited optimal performance at 28 days, achieving compressive and flexural strengths of 27.20 and 16.95 N/mm2, respectively. Thus, this mix is recommended for concrete production.
Keywords: Concrete; Millet husk ash (MHA); Nano-Silica; Compressive strength, Flexural strength

Introduction

In today's world, industrial growth and technological progress necessitate large-scale infrastructure development. Unfortunately, as infrastructural advancements continue, the demand for cement production escalates, leading to increased greenhouse gas emissions (Praveenkumar & Sankarasubramanian, 2021). Research indicates that cement clinker production is a primary contributor to greenhouse gas emissions (Wang *et al.,* 2020). A significant consequence of cement production is the release of greenhouse gases (Said *et al.,* 2012), which can be greatly mitigated through methods such as carbon dioxide sequestration (Liu *et al.,* 2021), catalytic conversion (Osazuwa & Cheng, 2020), amine scrubbing (Wang J *et al.,* 2020), and integrating supplementary materials or alkali-activated cementitious materials (Shi *et al.,* 2021), which possess pozzolanic properties and thereby decrease the dependence on cement (Said *et al.,* 2012).

According to Neville (2012), pozzolan refers to siliceous or aluminous substances which react with CAOH in the presence of water to form compounds with cementitious properties. Pozzolan must be in a finely divided form in order to react with calcium hydroxide in the presence of water, resulting in the creation of stable calcium silicates with cementitious properties. Neville (2012) also emphasized that the silica needs to be in an amorphous (or glossy) condition, as crystalline silica possesses very low reactivity. These pozzolanic materials can enhance the strength and durability of concrete and mortar (Neville, 2012), thereby decreasing the rate of heat release, which is advantageous for mass concrete and mortar applications (Onogwu *et al.,* 2024).

Pozzolans derived from agricultural waste (e.g., Bagasse ash, Rice husk ash, Millet husk ash, Fonio husk ash) helps to alleviate disposal problems for the environment (Praveenkumar & Sankarasubramanian, 2021).

Millet husk ash (MHA) is a by-product generated from the combustion of millet husk. Millets are small-seeded crops or grains that are widely cultivated around the globe for food and fodder, particularly in the semi-arid regions of Asia and Africa (Onogwu *et al.,* 2024). The presence of agro-waste like millet husks rice which can lead to environmental harm when left unused. By converting these materials into useful components for mortar and concrete production, the environmental issues associated with waste disposal can be mitigated. Also using MHA as a partial replacement of cement in concrete production will help to reduce CO2 emission in to the atmosphere during the production process of cement.

Nano silica has gained the interest among the various Nano materials due to its early age reactivity. It has earned more benefits in concrete and glass industries (Nandhini, 2021). Around 46% of the researchers have used the powdered form. Moreover, Nano Silica (NS) might be pyrogenic NS, precipitated NS or NS gel (Kumar *et al*., 2020). In aqueous suspension, colloidal NS is comprised of amorphous hydroxylated Silica particles of size ranging between 1–500 nm (Garg *et al*., 2020). To improve the mechanical and durability performance of the cement concrete structures, NS can be used as an additive (Singh *et al*., 2019).Due to its early age reactivity; NS in cement concrete reacts with calcium hydroxide to form C-S–H gel which fills up the pores and thereby improving the early strength. Moreover, with the better filling effect and particle size distribution, NS reduces the porosity of the concrete when compared with other mineral admixtures (Abhilash *et al*., 2021).

Therefore, new techniques must be developed to improve control and reduce both greenhouse gas

emissions and energy consumption in cement manufacturing. This study, therefore, reports on the use of Millet husk ash as partial replacement of cement and Nano silica as additive in the production of concrete.

Materials and Methods

Materials

The materials utilized for this study include: Portland cement (PC), millet husk ash, fine aggregate, coarse aggregates, and water. The PC (CEM I 42.5 N) used in this study is the Dangote (3X) brand, sourced from local cement suppliers in Minna.

For this study, millet husk ash was sourced from Kotangora town in Niger State. The husk was incinerated using a locally made incinerator available at the Concrete Laboratory of the Department of Building at the Federal University of Technology, Minna, Niger State. The husk was burned in the open air and uncontrolled temperature for approximately 24 hours and allowed to cool before being harvested and ground with a grinding machine in Gidan-Kwano village, Minna, Niger State. The processed millet husk ash was then sieved through a 75µm mesh in accordance with ASTM C430-2014 before being stored in a sealed polythene bag. A hydrothermal method as shown in plate 1-3 below was used to produce the Nano silica.

Plate 1: Equipment used for the preparation of Nano-SIO_2 from MHA

Plate 2: Preparation process of Nano-SIO_2 from MHA

Plate 3: Balling process

The fine aggregate for this study is a river sand obtained from Gidan-Kawnu river, which conforms to BS EN 196-1:2016 standards. The coarse aggregate used was crushed granite sourced from Minna, characterized by its strength, density, durability, and cleanliness, with a size of 10mm, in line with BS EN 196-1:2016.

The water used for both the production and curing of the concrete samples in this research was clean, potable water sourced from the Building Laboratory at the Federal University of Technology, Minna.

Methods

Chemical and physical of constituent materials

X-ray fluorescence (XRF) was utilized to assess the chemical compositions of the binders (PC and MHA) at the National Geoscience Research Laboratory located in Kaduna State. After the processes of calcination, grinding, and sieving, approximately 100g of these binders were sealed in polythene bags and sent for analysis of the oxide compositions in accordance with BS EN 197-1: 2016. Wet sieving was employed to determine the particle size distribution of the aggregate samples. Additionally, the specific gravities and moisture content of both the aggregates and binders were evaluated in accordance with ASTM C430 (2014).

Production of concrete specimen

The concrete was prepared using 100mm cube specimen for assessing compressive strength and 100 x 500mm beams for evaluating flexural strength. The varying percentages of MHA utilized were 5%, 10%, and 15%, with each percentage incorporating 0%,

1.5%, 3%, and 4.5% of Nano silica derived from MHA, while CEM I acted as the control. Three cube and beam specimens were created from each mixture, resulting in 117 cubes and 117 beams altogether, which were cured for 7, 21, and 28 days. The concrete samples were proportioned and mixed in a ratio of 1:1.5:3, with a water/cement (w/c) ratio of 0.5, in accordance with BS EN 196-1:2016 standards.

Table 1: Mix Details for Concrete Samples

Mix ID	MHA kg/m²	Nano silica kg/m²	CEM 1 kg/m²	F/A kg/m²	C/A kg/m²	Water kg/m²	SP kg/m²
M_0			383.20	800.94	1087.75	191.60	5.75
M_{10}	19.16		364.04	800.94	1087.75	191.60	5.75
M_{11}		5.46	358.58	800.94	1087.75	191.60	5.75
M_{12}		10.92	353.12	800.94	1087.75	191.60	5.75
M_{13}		21.84	342.20	800.94	1087.75	191.60	5.75
M_{20}			344.88	800.94	1087.75	191.60	5.75
M_{21}		5.17	339.71	800.94	1087.75	191.60	5.75
M_{22}	38.32	10.34	339.54	800.94	1087.75	191.60	5.75
M_{23}		20.68	324.20	800.94	1087.75	191.60	5.75
M_{30}			325.72	800.94	1087.75	191.60	5.75
M_{31}		4.89	320.83	800.94	1087.75	191.60	5.75
M_{32}	57.48	9.78	315.94	800.94	1087.75	191.60	5.75
M_{33}		19.56	306.16	800.94	1087.75	191.60	5.75

Results and Discussion

Physical properties of constituent materials

Particle size distribution (PSD) of aggregates

The particle size distribution (PSD) of the aggregates is displayed in Table 2.

Table.2: Summary of sieve analysis of aggregates

Item	Sand	Granite
D_{10}	650	10000
D_{30}	820	11000
D_{60}	870	13000
C_u	1.40	1.3
C_c	0.59	0.93
FM	2.87	

The analysis presented in table 2 above shows that the fine aggregate has a uniformity coefficient (Cu) of 1.40, a coefficient of curvature (Cc) of 0.59, and a fineness modulus (FM) of 2.87, which corresponds to the fine sand classification by Shetty (2009). The coarse aggregates utilized in this study exhibit a uniformity coefficient (Cu) of 1.32 and a coefficient of curvature (Cc) of 0.92, categorizing them as uniformly graded stone according to Shetty (2009).

Particle size distribution (PSD) and BET specific surface area of binders

Figures 1-4 illustrate the particle size distribution and specific surface area of CEM II of grade 42.5N and MHA utilized in this research. The outcomes of the BET analysis indicate that the particle sizes of the binders differ in terms of surface area and pore size diameter, as demonstrated in Figure 1. These variations in binder particle sizes might be attributed to the production methods employed (Onogwu *et al.*, 2023). The CEM II was produced using a ball milling method, whereas the MHA production involved a grinding process equipped with a 5 µm sieve in the machine's grinding compartment, allowing for the achievement of particle sizes close to 5 µm. According to Onogwu *et al.* (2023), both calcination and grinding methods influence the microstructure and pore characteristics of cementitious materials. As observed in Figures 1 and 2, 90% (D90) of the particles for CEM II and MHA measure smaller than 4000 and 3800 µm, respectively. The median particle sizes (D50) for CEM II and MHA are recorded at 48.8 and 32.0 µm, respectively. Furthermore, the particle sizes below 10% (D10) fall within the ranges of 4.90 and 158 µm for CEM II and MHA, respectively. Also, from the figures, the BET surface areas for CEM II and MHA are 256.84 m²/g and 98.72 m²/g, respectively. The BET analysis reveals that the pore diameters for the binders are 6.42 and 5.92 µm for CEM II and MHA, while the pore volumes are 0.188 cc/g and 0.128 cc/g for CEM II and MHA, respectively. A comparison of the DA BET analysis shows that the MHA sample possesses the smallest pore size diameter, classifying it as a macro-mesoporous material (Vieira *et al.*, 2020).

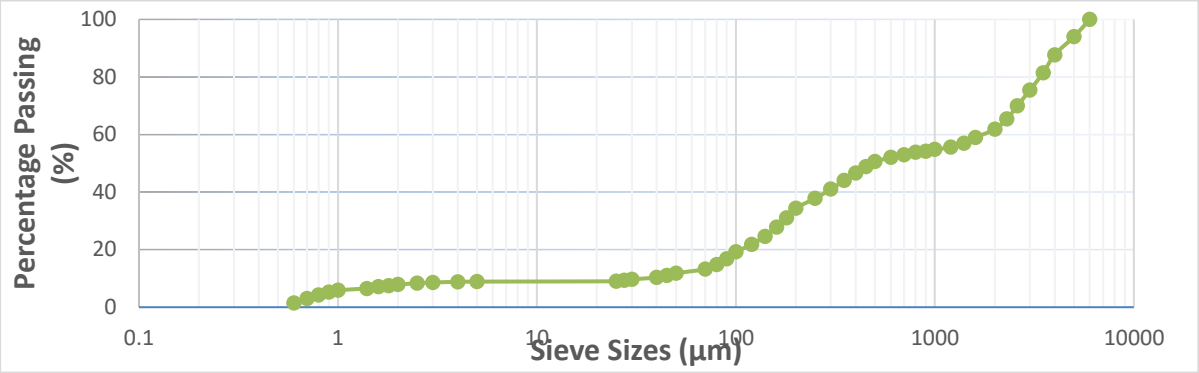

Figure 1: Particle size distribution of CEM I, 42.5N

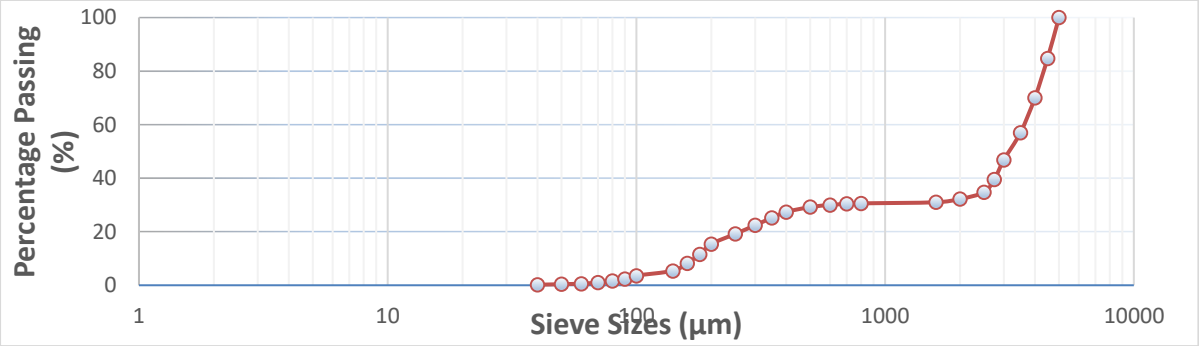

Figure 2: Particle size distribution of MHA

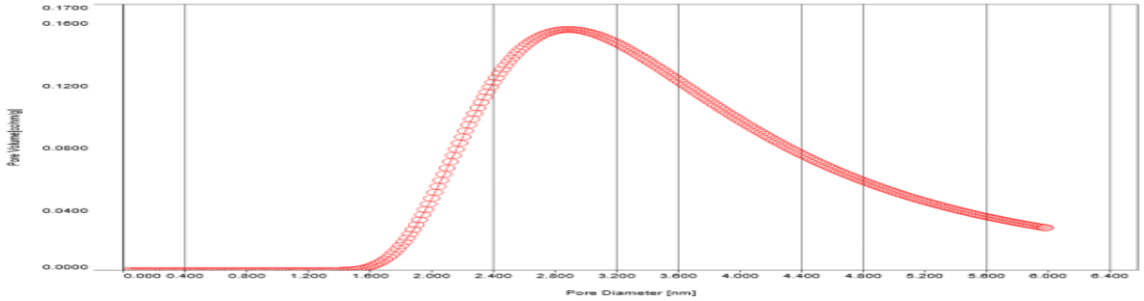

Figure 3: BET Distribution Analysis CEM I

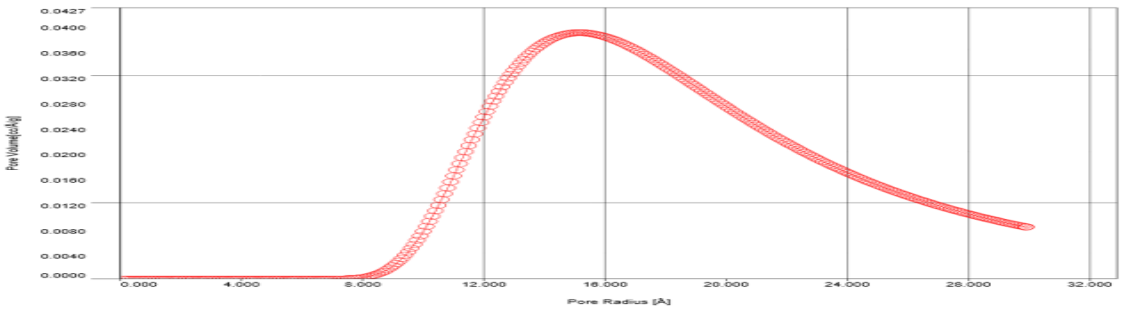

Figure 4: BET Distribution Analysis MHA

Specific gravity and bulk density of constituent materials

The specific gravities and bulk densities of the constituent materials are shown in table 3. The results in table 3 suggest that the values obtained are similar to those reported by Onogwu *et al.* (2023) regarding the specific gravity and bulk density of CEM I, MHA, fine aggregates, coarse aggregates, and superplasticizers used in concrete production (Neville, 2012).

Table 3: Specific Gravity and Bulk Density of Constituent Materials (kg/m3)

Materials	Specific gravity	Bulk density (kg/m3)
CEM 1	3.15	3150
MHA	2.63	2630
Fine Aggregate	2.79	2790
Coarse aggregate	2.85	2850
Superplasticizer	1.06	1060

Moisture Content of CEM 1, MHA and fine aggregates

Table 4 displays the moisture content results for CEM 1, MHA, and the fine aggregates utilized in this study. The findings in Table 5 indicate that the moisture content of the fine aggregates meets the ACI E1-99 specifications (which state that the moisture content for fine aggregates should range from 0 to 10%), while the moisture content for the binders (CEM 1 and MHA) complies with BS EN 196-1:2016 (which states that the moisture content for binders should range from 0 to 3%). Therefore, this suggests that the tests conducted on both the fine aggregates and binders adhered to the established standards.

Table 4: Moisture Content of the Material Samples

Materials	Weight of material before oven dry (kg)	Weight of material After oven dry (kg)	Moisture content (%)
CEM 1	0.5	0.491	0.9
MHA	0.5	0.498	0.2
fine aggregates	0.4	0.381	1.9

Loss of ignition of the binder

Table 5 displays the Loss of Ignition (LOI) for the binders (CEM1 and MHA) utilized in this research. The findings indicate that the binders exhibit LOI values of 0.0 for PC and 0.55 for MHA. This measurement was taken after the MHA was pre-heated in an oven at 105°C. The LOI values are in accordance with BS EN 196-1:2016, which requires that they do not exceed 10% of the total content .

Table 5: Loss of Ignition of the Binder

Binder	CEM 1	MHA
Wt. of Crucible (g)	52	51
Weight of Crucible + Binder (g)	73	65
Weight of Crucible + Ignited Binder (g)	73	64.45
L.O.I	0	0.55

Chemical properties of binders

The XRF analysis results for MHA and PC can be found in table 6. It is evident that MHA qualifies as a class N Pozzolan, as the combined total of the primary oxides (SiO_2 + Al_2O_3 + Fe_2O_3) reaches 86.46%, exceeding the specified minimum limit of 70% according to Neville (2012). In addition, the primary component of PC is calcium oxide (CaO – 60.35%). This aligns with the oxide composition for CEM I Portland cement documented in the literature (Neville, 2012; Mehta & Monteiro, 2014).

Table 6: Oxide Composition of Binder Constituents

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	M_2O_5	P_2O_5	LOI	SiO_2 + Al_2O_3 + Fe_2O_3
MHA (%)	78.92	2.69	4.85	1.55	0.73	0.66	5.21	1.13	2.06	1.17	2.20	86.46
CEM II (%)	25.64	5.24	7.15	60.35	0.41	0.11	0.05	0.31	0.04	0.03	0.67	38.03

Compressive strength

The compressive strength of the Nano silica MHA concrete at 7, 21, and 28 days of curing in ordinary water are illustrated in Figures 5-7.

At 7 days of curing, the compressive strength values for the different mixes are as follows: M0 (control) measures 21.75 N/mm^2, while the 5% MHA mixes with 0, 1.5, 3, and 4.5% Nano-Silica show results of M10 = 21.64 N/mm^2, M11 = 21.89 N/mm^2, M12 = 21.58 N/mm^2, and M13 = 21.56 N/mm^2. For the 10% MHA with 0, 1.5, 3, and 6% Nano-Silica, the values are M20 = 20.58 N/mm^2, M21 = 20.41 N/mm^2, M22 = 20.30 N/mm^2, and M23 = 19.72 N/mm^2. Lastly, the mixes with 15% MHA and varying Nano-Silica percentages (0, 1.5, 3, and 6%) yield M30 = 19.86 N/mm^2, M31 = 19.64 N/mm^2, M32 = 19.52 N/mm^2, and M33 = 19.10 N/mm^2.

At 21 days, the compressive strength values for the different mixes are as follows: M0 (control) at 24.82 N/mm², 5% MHA with 0, 1.5, 3, and 6% Nano-Silica yielding M10 = 23.12 N/mm², M11 = 25.01 N/mm², M12 = 22.82 N/mm², and M13 = 22.10 N/mm². For 10% MHA with 0, 1.5, 3, and 6% Nano-Silica, the results are M20 = 22.83 N/mm², M21 = 22.62 N/mm², M22 = 21.82 N/mm², and M23 = 20.68 N/mm². Lastly, for 15% MHA with 0, 1.5, 3, and 6% Nano-Silica, the compressive strengths are M30 = 21.03 N/mm², M31 = 20.92 N/mm², M32 = 20.42 N/mm², and M33 = 20.12 N/mm².

At 28 days, the compressive strength values for the different mixes are as follows: M0 = 26.84 N/mm² (control), 5% MHA with 0, 1.5, 3, and 6% Nano-Silica (M10 = 24.82 N/mm², M11 = 27.20 N/mm², M12 = 23.82 N/mm², and M13 = 23.60 N/mm²), 10% MHA with 0, 1.5, 3, and 6% Nano-Silica (M20 = 23.02 N/mm², M21 = 22.98 N/mm², M22 = 22.88 N/mm², and M23 = 21.90 N/mm²), and 15% MHA with 0, 1.5, 3, and 6% Nano-Silica (M30 = 22.96

N/mm², M31 = 21.84 N/mm², M32 = 21.03 N/mm², and M33 = 21.62 N/mm²).

The results indicate that, regardless of curing age, the proportion of MHA and the incorporation of Nano-MHA enhance the compressive strength (Wang *et al.*, 2020). Additionally, the data shows that an increase in Nano-MHA corresponds with a reduction in compressive strength, with M11 (5% MHA and 1.5% Nano) achieving the highest compressive strengths of 21.89, 25.01, and 27.20 N/mm² at 7, 21, and 28 days of age compared to the control and the other mixtures.

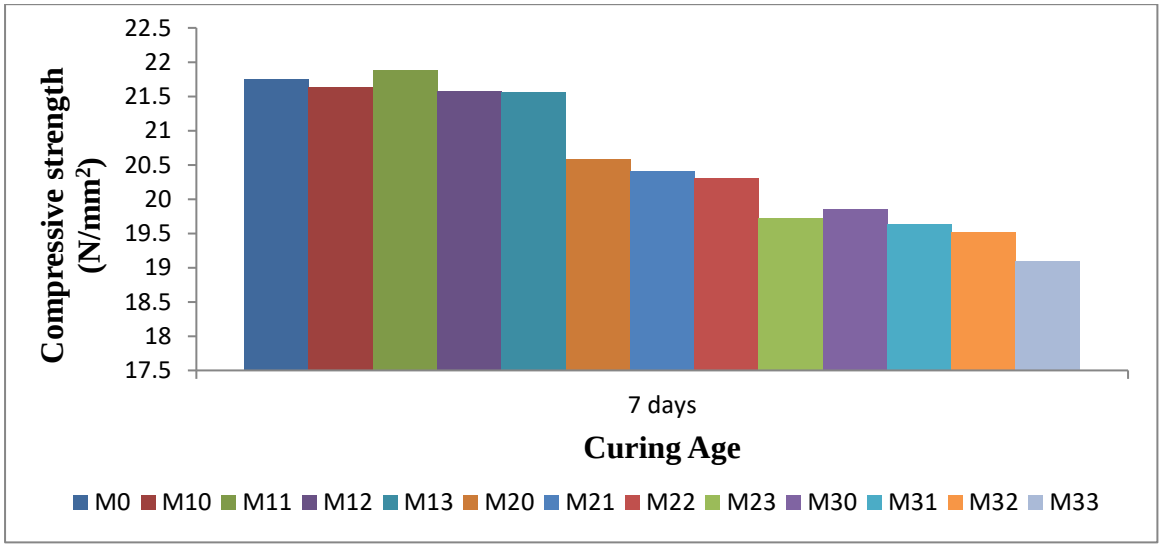

Figure 5: compressive strength of Nano silica MHA concrete at 7 days

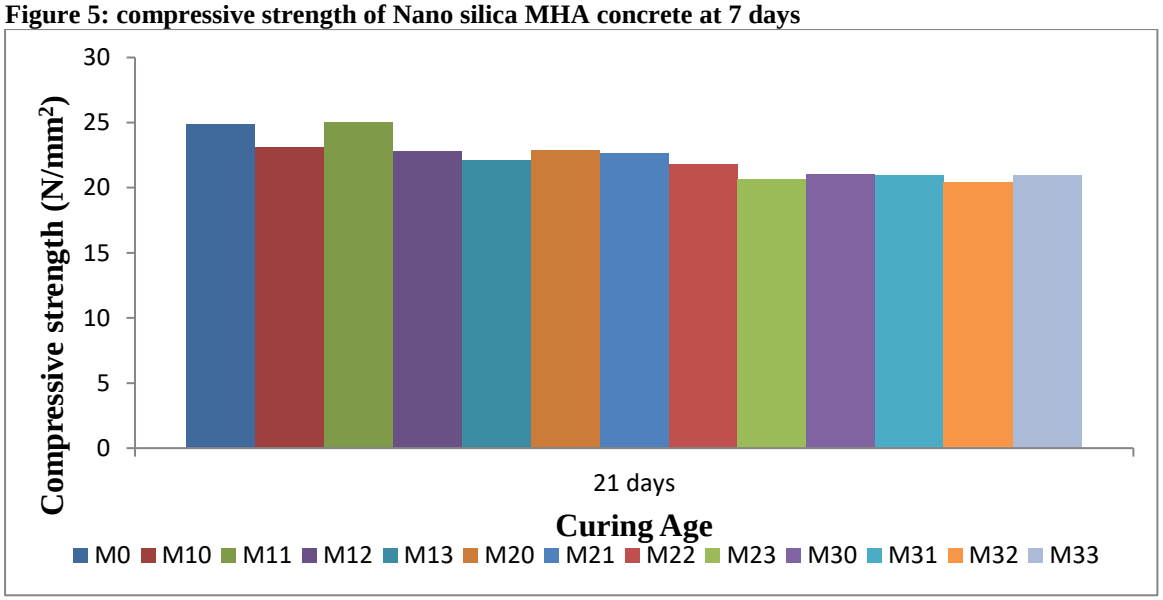

Figure 6: compressive strength of Nano silica MHA concrete at 21 days

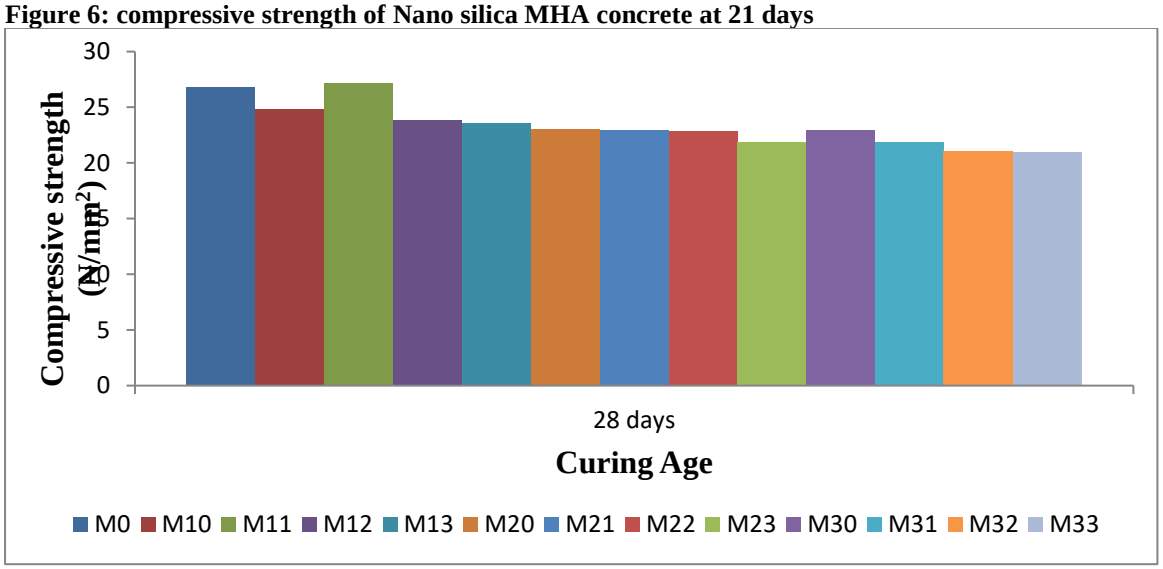

Figure 7: compressive strength of Nano silica MHA concrete at 28 days

Flexural strength

Figures 8-10 illustrate the flexural strength of the Nano silica MHA concrete A at 7, 21, and 28 days, respectively, when cured in ordinary water.

At 7 days old, the flexural strength values for the mixtures are as follows: M0 = 8.12 N/mm^2 (control), 5% MHA with 0, 1.5, 3, and 6% Nano-Silica (M10 = 6.42 N/mm2, M11 = 8.38 N/mm^2, M12 = 6.03 N/mm^2, and M13 = 5.89 N/mm^2), 10% MHA with 0, 1.5, 3, and 6% Nano-Silica (M20 = 6.12 N/mm^2, M21 = 5.83 N/mm^2, M22 = 5.40 N/mm^2, and M23 = 5.01 N/mm2), and 15% MHA with 0, 1.5, 3, and 6% Nano-Silica (M30 = 5.10 N/mm2, M31 = 4.92 N/mm^2, M32 = 3.89 N/mm^2, and M33 = 3.20 N/mm^2).

At 21 days, the flexural strength values for the various mixtures are: M0 = 13.03 N/mm^2 (control), 5% MHA with 0, 1.5, 3, and 6% Nano-Silica (M10 = 9.14 N/mm^2, M11 = 13.80 N/mm^2, M12 = 8.42 N/mm^2, and M13 = 8.12 N/mm^2), 10% MHA with 0, 1.5, 3, and 6% Nano-Silica (M20 = 8.15 N/mm^2, M21 = 7.82 N/mm^2, M22 = 7.10 N/mm^2, and M23 = 6.16 N/mm^2), and 15% MHA with 0, 1.5, 3, and 6% Nano-Silica (M30 = 7.12 N/mm^2, M31 = 6.80 N/mm^2, M32 = 6.60 N/mm^2, and M33 = 5.80 N/mm^2).

At 28 days, the flexural strength values are: M0 = 16.12 N/mm^2 (control), 5% MHA with 0, 1.5, 3, and 6% Nano-Silica (M10 = 14.80 N/mm^2, M11 = 16.95 N/mm^2, M12 = 12.89 N/mm^2, and M13 = 12.12 N/mm^2), 10% MHA with 0, 1.5, 3, and 6% Nano-Silica (M20 = 12.10 N/mm^2, M21 = 10.12 N/mm^2, M22 = 8.98 N/mm^2, and M23 = 8.12 N/mm2), and 15% MHA with 0, 1.5, 3, and 6% Nano-Silica (M30 = 10.12 N/mm2, M31 = 8.18 N/mm2, M32 = 7.26 N/mm^2, and M33 = 7.12 N/mm^2).

The results clearly demonstrate that the addition of nanoparticles enhances Flexural strength, regardless of the curing age and the percentage of MHA used (Wang *et al*., 2020). Furthermore, the data indicates that an increase in Nano-MHA contributes to a reduction in Flexural strength, with M11 (5% MHA and 1.5% Nano) showing the highest flexural strength of 8.38, 13.80, and 16.95 N/mm^2 at 7, 21, and 28 days, respectively, in comparison to the control and other mixtures.

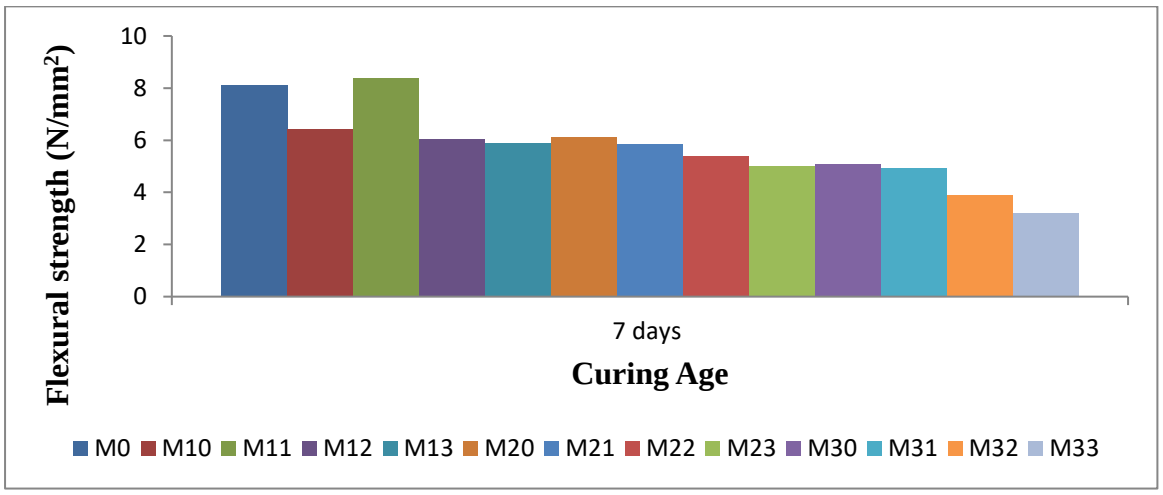

Figure 8: Flexural strength of Nano-MHA at 7days

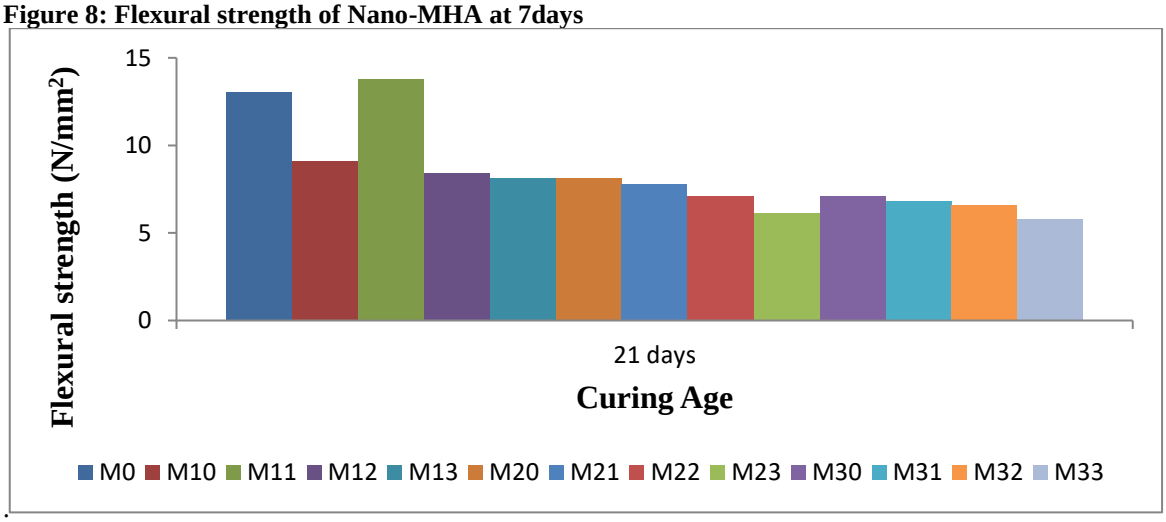

Figure 9: Flexural strength of Nano-MHA at 21days

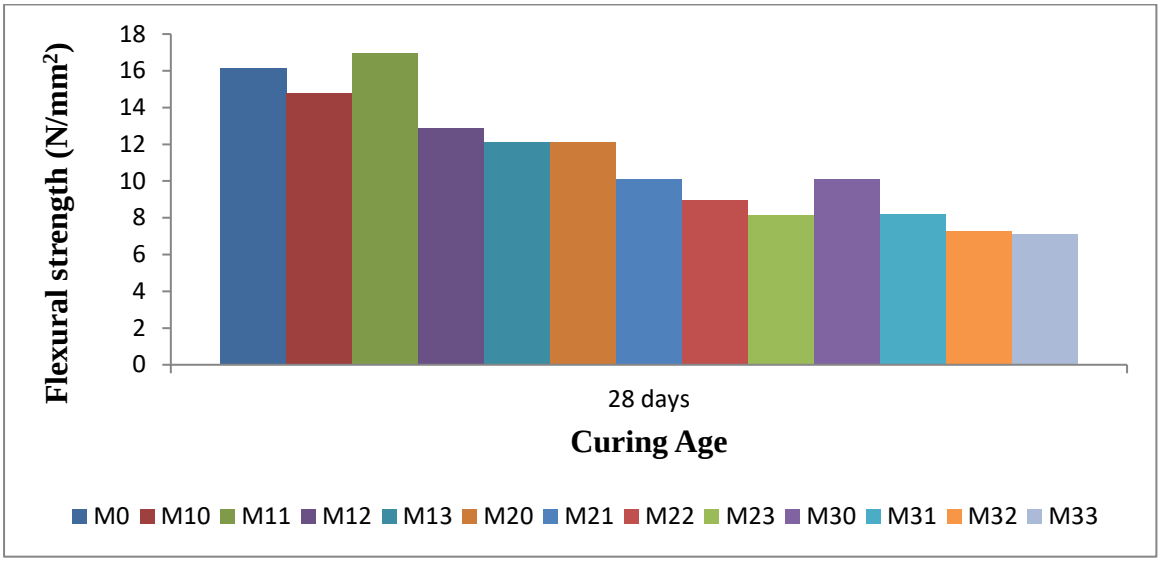

Figure 10: Flexural strength of Nano-MHA at 28days

Conclusion

The research examined the strength properties of nano silica concrete produced with Millet husk ash as partial replacement of cement**.** The findings indicated that the nano-silica derived from millet husk ash positively influenced the compressive and flexural strength, of the concrete. According to the study, the millet husk ash utilized met the minimum standard for classification as a pozzolan. The optimal modified concrete strength (compressive and flexural strengths) were observed in concrete mixtures containing 1.5% Nano silica and 5% millet husk ash, while higher amounts of nano-millet resulted in reduced compressive and flexural strengths. It is advised to use 5% millet husk ash along with 1.5% nano-silica. From the study, it was recommended that durability study of nano silica MHA concrete can be a potential research outcome to be sought after.

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