

Received: February 06, 2024

Revised: May 07, 2024

Accepted: May 07, 2024

# Production and Characterization of Pearl Millet Husk Ash as a Pozzolan

Oluwatoyin Moyosore Daramola<sup>1</sup> | Yinusa Alaro Jimoh<sup>2</sup> | \*Sikiru Folahan Oritola<sup>1</sup>

<sup>1</sup>Department of Civil Engineering, Faculty of Engineering, Baze University, Abuja

<sup>2</sup>Department of Civil Engineering, Faculty of Engineering, University of Ilorin, Kwara State

Corresponding Author's Name:

Sikiru Folahan Oritola

Corresponding Author's E-mail:

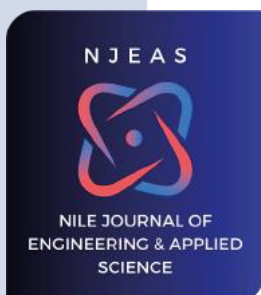
sikiru.oritola@bazeuniversity.edu.ng

## ABSTRACT

Pearl millet husk ash is a byproduct of the combustion of pearl millet husks, commonly used in various industries. This study presents a novel approach to producing pozzolan from millet husk, focusing on identifying the most effective production metrics. The cleaned husks underwent calcination at various temperatures and dwelling times, resulting in six ashes for chemical composition analysis. The findings, which are of significant importance to the field of civil engineering and environmental sustainability, reveal that the optimal production metric for creating high silica content pozzolan from millet husk bio-wastes is a calcination temperature of 900°C and a dwelling time of 2 hours. The MHA generated at these conditions had a total of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub> content ranging from 96 %, surpassing the benchmark of 70 % for Class C ASTM C618-12 quality pozzolan. Therefore, this research provides valuable insight into producing high-quality pozzolan from millet husk bio-wastes, contributing significantly to the fields of civil engineering and environmental sustainability by offering a sustainable and efficient method for waste utilization and resource conservation through calcination at 900°C for 2 hours.

**KEYWORDS:** Temperature, Pozzollan, Pearl millet husk, Calcination, Bio-wastes

**DOI:** <https://doi.org/10.5455/NJEAS.189164>



## 1 | INTRODUCTION

Pozzolan in fine form and the presence of moisture, its siliceous and aluminous constituents in amorphous form react chemically with calcium hydroxide generated by cement hydration at ordinary temperatures, producing additional compounds such as calcium silicate hydrates and calcium silicate aluminate hydrates that possess cementitious properties (Walker & Pavia, 2011). The pozzolanic materials make the capillary voids to be eliminated or reduced in size by converting calcium hydroxide to calcium silicate hydrate, thereby improving the strength, durability and permeability properties of the cement-concrete hydrated paste (Bayuagi *et al.*, 2011).

There are natural and artificial sources of pozzolans. Examples of the former include volcanic mineral deposits, fired and crushed clay and fly ash created during coal burning for power generation. Indeed, ashes and residues of burnt biomass plants and or wastes, such as rice husks, corn cobs, and sugar cane bagasse, recently form the bulk of the artificial pozzolans. Using these biomass waste products, a key focus of our research, not only conserves energy and other resources, including finance in the long run but also makes a significant contribution to environmental sustainability through recycling purposes and the substantial reduction of global warming through reduced cement consumption in concrete works. Cement production generates about 900 kg of CO<sub>2</sub> per 1,000 kg of cement, a central component of greenhouse gas (GHG) emissions and global warming vectors.

In the FAO-ordered ranked reckoning, the five most important cereal crops are rice, wheat, maize, sorghum and pearl millet (*Pennisetum glaucum L.R.Br.*), known as gero-hatsi in the Hausa language of Northern Nigeria, (FAO, 2007b). All five cereal crops are dual-purpose and form staple food for millions in the world's arid and semi-arid climatic regions and ecologies (Chapora, 2001). The ability of millet to tolerate environmental stress and be growable in conditions with not-too-high rainfall (200 to 600 mm/year), as required by others is an added advantage for over 50 % of its global production in Africa and Asia. Sixty per cent (60 %) of the cultivation areas are in Africa, followed by 35 % in Asian countries; European countries represent 4% of millet cultivation, while North America is only 1 %, mainly for forage (Doggett, 1986; FAO, 2007a; NRC, 1996). There have been many appreciable records of pozzolan production and utilisation for cement replacement in concrete with the four leading biomass waste within the past few years.

However, in the few instances of the pozzolans from the millet husk, the physical and chemical composition and other properties have significant differences depending on the production method, open-air or closed

environment, and other metrics in terms of calcination temperature and duration (time) of exposure to heat in the furnace. For instance, millet husk burnt in the open air generates 73.1 % of SiO<sub>2</sub> and 10.2 % of CaO (Jimoh, 2013), while the ash from millet husk burnt in the furnace at 400-600 °C contains 16.72 % of SiO<sub>2</sub> and 30.3 % of CaO, (Okorie & Musa, 2012). The resulting pozzolan can even be amorphous (glassy) if produced at higher temperatures but powdery at lower temperatures. Thus, there is a need to thoroughly examine a pozzolanic material's characteristics and structure to determine the appropriateness of a specific function or method for its application.

When in fine form and the presence of moisture, pozzolanic materials react chemically with calcium hydroxide generated by cement hydration at ordinary temperatures; this reaction produces additional compounds, such as calcium silicate hydrates and calcium silicate aluminate hydrates, which possess cementitious properties (Walker & Pavia, 2011). The incorporation of pozzolanic materials eliminates or reduces the size of capillary voids by converting calcium hydroxide to calcium silicate hydrate, thereby enhancing the strength, durability, and permeability properties of the cement-concrete hydrated paste (Bayuagi *et al.*, 2011).

There are both natural and artificial sources of pozzolans. Natural sources include volcanic mineral deposits, fired and crushed clay, and fly ash generated during coal combustion for power generation. Artificial pozzolans primarily consist of ashes and residues from biomass combustion, such as rice husks, corn cobs, and sugar cane bagasse. Utilizing these biomass waste products conserves energy and resources and contributes to environmental sanitation, recycling, and significant global warming reduction through reduced cement consumption in concrete works. Cement production significantly contributes to greenhouse gas emissions, generating about 900 kg of CO<sub>2</sub> per 1,000 kg of cement, a central component of greenhouse gas emissions and global warming vectors (Bhutta, 2020).

Several authors authenticate the significant potential of millet husk ash (MHA) as a pozzolanic material in concrete production. Chandrappa *et al.* (2021) conducted a review focusing on the chemical and physical properties of MHA and its impact on the performance of concrete. Their study highlighted the effectiveness of MHA in improving the strength, durability, and permeability of concrete while also addressing environmental concerns associated with its disposal. Additionally, Smith *et al.* (2022) conducted a meta-analysis of recent research articles, examining the role of MHA as a supplementary cementitious material in concrete mixtures. Their findings underscored the

promising prospects of MHA as a sustainable alternative to traditional pozzolans, offering potential benefits in terms of cost-effectiveness and environmental sustainability.

Several recent studies have investigated the influence of various factors, such as production methods, calcination temperature, and particle size distribution, on the pozzolanic reactivity of MHA. Okorie and Musa (2023) explored the effects of different calcination temperatures on MHA's chemical composition and pozzolanic activity, highlighting the optimal conditions for maximising its reactivity in concrete mixtures. Similarly, Jimoh (2024) investigated particle size distribution's impact on MHA performance in concrete, emphasising the importance of particle fineness in enhancing its pozzolanic reactivity and mechanical properties.

This study seeks to understand the physical and chemical composition properties of pearl millet husk ash obtained at different calcination temperatures and furnace heat dwelling times. Additionally, this research aims to investigate the ash structure produced at the most favourable production metric, thereby establishing the suitability of the selected ash for pozzolanic functioning in concrete works. By achieving these objectives, we hope to contribute to developing sustainable construction materials and practices.

## 2 | EXPERIMENTAL PROGRAM

### 2.1 Materials and Experimental Method

#### 2.1.1 Millet Husk Ash (MHA) from the Pearl Millet Husk

Some dry samples of Pearl millet husk waste, which are products of decortication of millet plant seed, were procured from Gomboru market, north-eastern part of Nigeria. The raw husk in its original form is thorny-like and dark brownish-yellow in colour. Six different brands of MHA were produced at three different temperatures (600 °C, 750 °C, and 900 °C) by two dwelling times (2 and 4 hours) in the muffle furnace. The furnace's maximum temperature and heating rate were 1100 °C and 10°C/min, respectively, according to He *et al.* (1994). The structure of the MHA was investigated with the scan electron microscope (SEM).

#### 2.1.2 Specific gravity ( $G_s$ )

The specific gravity of the six millet husk ash brands was determined using the pycnometer and the BS 1377:1990 method. An empty pycnometer bottle was cleaned, dried and weighed with its cover in position. A 50g sample was introduced into the pycnometer bottle and filled with distilled water. The outer surface of the pycnometer bottle was kept dry, and the total combined weight was measured. Pour out the mixture, and the pycnometer bottle was carefully rinsed with kerosene to remove all the substance. The clean bottle was filled

carefully with distilled water, eliminating trapped air bubbles. Then, the outer surface of the pycnometer was dried carefully, and record the corresponding weight.

The sample's specific gravity is the ratio of the weight in air of a given volume of the sample to the weight in air of an equal volume of water at 40°C, expressed with equation (1).

$$= \frac{W_1 - W_2}{(W_4 - W_1) - W_3 - W_2} \quad (1)$$

Where  $W_1$  = Weight of pycnometer bottle and cover,  $W_2$  = Weight of pycnometer, cover and sample,  $W_3$  = Weight of pycnometer cover, sample and distilled water, and  $W_4$  = Weight of pycnometer cover and distilled water

### 2.1.3 Geochemical Analysis of MHA using X-Ray Florescence Spectrophotometry

Table 1 presents the chemical composition test from XRF analysis. The diffractometer type was model TEFA ORTEC automatic X-ray Florescence PhilipPW 1210, having a copper (Cu) tube anode, generator tension of 40 kV and current of 25 mA. Using 1.0g of each ash sample for elemental composition through X-ray fluorescence Spectrometry. See Figure 1.

**Table 1.** Composition Analysis Test Conditions with XRF and Production Metric

Temp (°C)	Duration (hr)	Wavelength Cuka 1[A°]	Wavelength Cuka 2[A°]
600	4	1.560	1.550
750	4	1.555	1.545
900	4	1.550	1.535
600	2	1.560	1.555
750	2	1.555	1.545
900	2	1.500	1.540

All measurements were carried out in strict compliance with the manufacturer's usage manual and standard procedures and made on energy dispersion of the X-ray fluorescence spectrometer. The current was automatically adjusted (maximum of 1 mA) using a 10 mm collimator at a counting time of 100 seconds. Obtaining intensity of element,  $K\alpha$  counts per second (cps/ $\mu$ A) using the X-ray spectrum Axion software package.



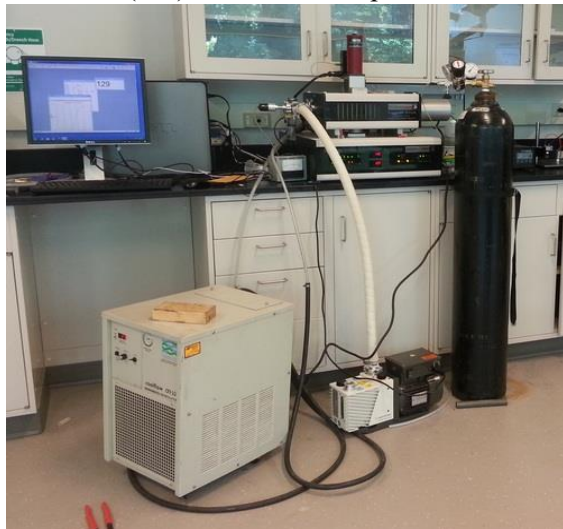
**Figure 1:** XRF machine - Axions Analytical model

#### 2.1.4 Reactivity of MHA

Measure changes in the electrical conductivity of the lime/pozzolan solution over time and the  $\text{pH}$  of the MHA solution to determine the reactivity.

#### 2.1.5 Determination of Electrical Conductivity

Using Accumet AP75, the conductivity of the pozzolan in distilled water was measured to assess its reactivity in terms of its contribution to the conductivity and water salt solubility content and solubility of the pozzolans. See Figure 2. The electrical conductivity indicates the amount of soluble (salt) ions in the samples.



**Figure 2:** Accumet AP75 apparatus

The reagent is the deionised water having an electrical conductivity of  $<1 \mu\text{S}/\text{cm}$  and a  $\text{CO}_2$  concentration of not more than atmosphere equilibrium. Make a reference solution of 0.01M potassium chloride by dissolving 0.746 g KCl AR (previously dried at  $105^\circ\text{C}$  for 2 hours and made up to a volume of 1 L with  $\text{CO}_2$ -free deionised water). This solution has an electrical conductivity of  $1.413 \text{ ds}/\text{m}$  at  $25^\circ\text{C}$ . A suspension of the pozzolan-to-water in the ratio of 1:5 was prepared by weighing 10 g air-dry soil ( $<2 \text{ mm}$ ) into a bottle and adding 50 mL

deionised water. The mixture was mechanically shaken at 15 rpm for 1 hour to dissolve soluble salts. Calibrate the conductivity meter according to the manufacturer's instructions using the KCl reference solution to obtain the cell constant.

Rinse the cell thoroughly. Record the Electrical Conductivity (EC) of the 0.01M KCl at the same temperature as the ash suspension, and then the conductivity cell was with ash suspension. Refill the conductivity cell without disturbing the settled particles. Record the value indicated on the conductivity meter. Rinse the cell with deionised water between every trial of sample testing.

If the meter reads directly in conductivity values,  $EC_{25}$  is obtained using equation 2.

$$EC_{25}(d_s/m) = \frac{S \times 1.413}{K} \quad (2)$$

Where: S=Measured EC of suspension and  
K=Measured EC of KCl solution

#### 2.1.6 Determination of pH value

Conduct a pH test using a pH 230 Digital Meter. With potassium hydrogen Phosphate (KHP) as a reagent, prepare a 0.05M buffer 4 by dissolving 1.012g in 100 ml of deionised water in a 100 ml standard flask. A mixture of Potassium dihydrogen Phosphate ( $\text{KH}_2\text{PO}_4$  dried at  $110^\circ\text{C}$  for 2 hours) and disodium hydrogen phosphate ( $\text{Na}_2\text{HPO}_4$ ) as Buffer 7 was prepared by dissolving 1.179 g of  $\text{KH}_2\text{PO}_4$  and 4.303 g of  $\text{Na}_2\text{HPO}_4$  in 1000 ml of deionised water in 1-litre standard flask and using deionised water to a pH of 7 to dissolve the pozzolan for pH analysis.

Calibrating the pH meter with buffers 4 and 7 by dipping the electrode and adjusting the pH appropriately to ensure it worked correctly. A lump, 10g of ash ( $<2\text{mm}$ ), was weighed into the 25 ml plastic container, and 20 ml of deionised water (adjusted to pH of 7) was added and mixed thoroughly with a glass rod. The mixture was shaken at 10 to 30-minute intervals and allowed to stand for another 30 minutes, after which the suspended ash particles would have settled. The pH was measured by dipping the bulb into a clear portion of the mixture, and the reading was allowed to stabilise and recorded. The pH meter was cleaned using deionised water and tissue paper before subsequent measurement

### 3 | RESULTS AND INTERPRETATION

#### 3.1 Physical, mineralogical and chemical properties at varying temperatures and time

The six (6) calcinated types of the MHA (under 3 temperatures by 2 exposure times) were cooled to room temperature upon removal from the furnace. The

appearance is lumps-like with colour ranging from black to light grey. Table 2 summarises the results.

**Table 2:** Chemical Properties and Physical Composition of MHA at Various Production Metrics

Duration (hours)	4			2		
	temperature(°C)			temperature (°C)		
<b>Composit ion (%)</b>	600 (°C)	750 (°C)	900 (°C)	600 (°C)	750 (°C)	900 (°C)
<b>SiO<sub>2</sub></b>	32.8	55.2	52.1	34.2	55	58.3
<b>Al<sub>2</sub>O<sub>3</sub></b>	27.0 2	29.5 2	27.5 6	25.3	29.4 8	31.7
<b>FeO<sub>3</sub></b>	2.24	8.04	13.0 1	4.2	8	5.9
<b>MnO</b>	0.01	0.01	0.01	0.01	0	0
<b>CaO</b>	11.7	2.28	2.31	9.5	2.3	2
<b>Pb<sub>2</sub>O<sub>3</sub></b>	0.2	0.3	0.32	0	0	0.3
<b>K<sub>2</sub>O</b>	10.5	1.98	1.98	8.5	2.05	0.88
<b>TiO<sub>2</sub></b>	0.12	0.26	1.21	0.1	0.18	0
<b>MgO</b>	9.1	0.6	0.65	7.5	0.66	0.15
<b>Na<sub>2</sub>O</b>	6.7	0.2	0.17	6.9	0.24	0.8
<b>Ba (ppm)</b>	440	455	460	452	452	469
<b>Ce (ppm)</b>	56	60	58	55	40	51
<b>Rb (ppm)</b>	82	92	95	62	86	90
<b>Zr (ppm)</b>	65	85	70	69	80	70
<b>Cr (ppm)</b>	120	108	105	110	98	110
<b>Cu (ppm)</b>	48	40	35	43	40	30
<b>Ni (ppm)</b>	44	42	40	40	36	30
<b>Pb (ppm)</b>	14	10	10	10	15	12
<b>SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + FeO<sub>3</sub></b>	62.0 6	92.7 6	92.6 7	63.7	92.4 8	95.9
<b>Specific gravity</b>	2.36	2.41	2.54	2.22	2.44	2.55
<b>Electrical conductivity (ds/m)</b>	0.48	1.01	1.08	0.46	1.01	1.06
<b>PH</b>	7.30	6.58	8.50	7.40	6.55	8.46

### 3.2 The specific gravity

The specific gravity of MHA was 2.36, 2.41, and 2.52, respectively, at temperatures of 600°C, 750°C, and 900°C for a exposure time of 4 hours, but 2.22, 2.44, and 2.55 correspondingly for a exposure time of 2 hours. This implies that at higher temperatures and more extended periods of calcination, the forming ash pozzolan has higher specific gravity and more mass per mass of an equal volume of water.

### 3.3 The Electrical Conductivity

The electrical conductivity (ds/m) was 0.48, 1.01, and 1.08 at temperatures of 600, 750 and 900 °C for 4 hours. The corresponding values for 2-hour dwelling time are 0.46, 1.01, and 1.06.

### 3.4 The pH

The P<sup>H</sup> value was 7.30, 6.58, and 8.58 at the respective three temperatures for a exposure time of 4 hours, but correspondingly 7.40, 6.55, and 8.46 for 2 hour exposure time. Although it does not easily manifest in deciphering how the calcination conditions of temperature and time affect these properties, it, however, suffices to infer that the pozzolan derived from the millet husk at high temperatures, greater than 600 °C, contains more ions and is more pozzolanically reactive in the alkaline (basic) state, (6.58-8.58).

### 3.5 Characteristic properties of the pearl millet ash under the different metrics.

According to Singh et al. (2019) the chemical composition of pearl millet husk ash, reveals its elemental makeup and environmental implications. Understanding the chemical composition provides valuable insights into its compatibility with different materials and its potential effects on the surrounding environment, thus guiding its safe and effective utilization in various applications. The sum of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and FeO<sub>3</sub> was obtained as 62.06 %, 92.76 %, and 92.67 % at temperatures of 600 °C, 750 °C, and 900 °C for a exposure time of 4 hours subsequently the values of 63.7 %, 92.48 %, and 95.9 % at temperatures of 600 °C, 750 °C, and 900 °C for a dwelling time of 2 hours. Table 3 gives the characteristic properties of the pearl millet ash under the different production metrics.

**Table 3:** Matrix Properties of MHA

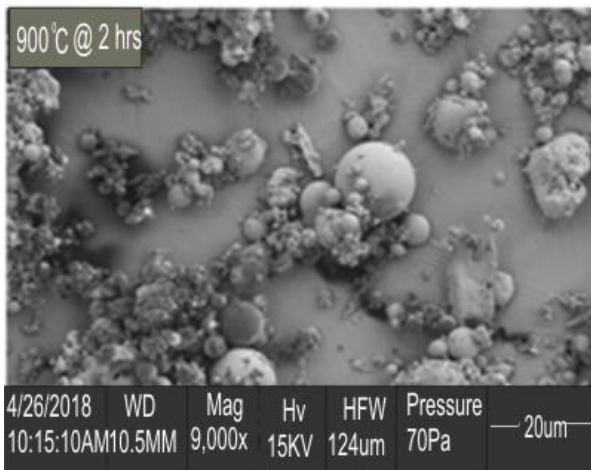
Temp °C	Exposure Time		Exposure Time	
	2 h	4 h	2 h	4 h
600 750 900	Silica content %		Sum of Oxides	
	34.2	32.8	63.70	62.06
	55.0	55.2	92.48	92.76
600 750 900	EC ds/m		Specific Gravity	
	58.3*	52.1	95.90*	92.67
	0.46	0.48	2.22	2.36
750	1.01	1.01	2.44	2.41
900	1.06	1.08*	2.55*	2.54

- Highest value of property

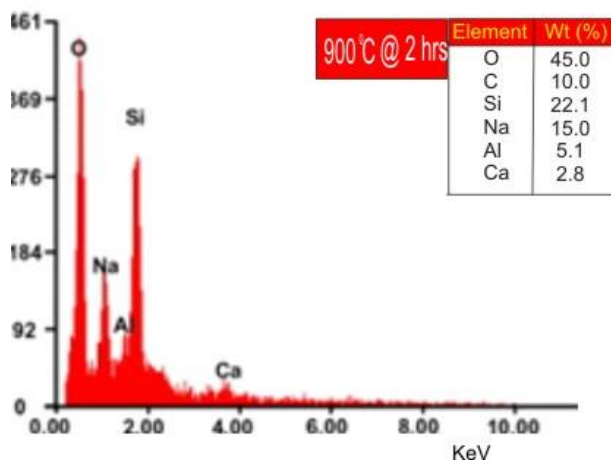
### 3.6 Micro structure of MHA calcined at 900°C for 2 hours

According to Nagaraju et al. (2020) the ash possesses a porous structure and high silica content, making it suitable for incorporation into concrete as a supplementary cementitious material. In the SEM image, Figure 3, the millet husk ash particles are circular, glassy and complex, reflecting the plant's origin. Figure 4

presents the EDS analysis and the elemental components. The high intensity of Si in the EDS spectrum confirms the presence of silica at a high proportion by weight. The quantity of soluble acids, Al and Si, reflects the content of active aluminosilicates, which also defines the material's pozzolanic activity; these findings align with the observations of Surana and Joshi (1990).



**Figure 3:** SEM image of MHA calcined @ 900°C for 2 hours.



**Figure 4:** EDS spectrum of MHA calcined @ 900°C for 2 hours.

## 4 | CONCLUSION AND RECOMMENDATION

### 4.1 Conclusion

Based on the study conducted on the physical and chemical composition properties of pearl millet husk ash obtained at different calcination temperatures and furnace heat dwelling times, along with the investigation of the ash structure produced at the most favourable production metric, the following research conclusion can be deduced:

i. The physical and chemical properties of pearl millet husk ash significantly vary with calcination

temperature and heat dwelling time. The choice of the three temperatures reflects the boundaries of phases of the state of matter formation processes: 600 °C at the end of dihydroxylations, 900 °C being the peak for husk destruction, and 750 °C in the middle. The calcinated samples as lumps, with colour variations ranging from black to light grey. Higher temperatures and longer dwelling times alter the ash composition and structure. Variations in the operating conditions (temperature and duration) significantly influence the characteristics of ashes derived from millet husk at more than 600 °C with a high proportion of silica in the amorphous state.

ii. This study reveals changes in physical properties of the ash with variations in calcination parameters. Further analysis indicates shifts in chemical composition, including the content of silica, alumina, and other minerals, as calcination conditions vary. The summation of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> content of 92-96%, which is more than 70% benchmark for Class C ASTM C618-12 quality pozzolan, was computed for the MHA produced at 750 and 900 °C calcination temperatures and the two dwelling times; while the corresponding values were 62-63% for the specimen produced at 600 °C in the furnace closed environment, implying a different brand of pozzolan. Further, the per cent silica content of the MHA brand calcined at 600, 750, and 900°C for a dwelling time of 4 hours is less than those of the 2 hours (32.8%, 55.2% and 52.1% respectively as against 34.2%, 55%, and 58.3%), making the 900 °C temperature and 2 hours calcination period the most optimum production metric for high silica content pozzolan from millet husk bio-wastes.

iii. Examining ash structure at optimal production metrics provides insights into its morphology, crystallinity, and pore characteristics. Understanding these features is crucial for assessing the ash's suitability as a pozzolanic material in concrete applications. The microstructure of the millet husk ash (MHA) calcined at 900 °C and a 2-hour dwelling time, the optimum condition, was investigated through the scan electron microscope. The millet husk ash particles are circular, glassy and complex, reflecting the plant's origin.

iv. This study informs that pearl millet husk ash produced under specific calcination conditions exhibits promising pozzolanic activity, indicating its potential as a sustainable supplementary cementitious material in concrete works. The investigation reveals that millet husk has good

pozzolanic activity when calcined at a temperature of 900 °C for 2 hours, much better than the other production metrics. Overall, millet husk ash is a valid source of pozzolanic material with high amorphous silica content, which is most appropriate and desirable for concrete development.

- v. The findings contribute to developing sustainable construction materials by identifying a viable agricultural waste-derived resource for enhancing concrete properties. Utilising pearl millet husk ash in concrete can reduce reliance on traditional cement, lower carbon emissions, and promote eco-friendly construction practices.

In conclusion, this study underscores the importance of understanding the effects of calcination parameters on the properties of pearl millet husk ash. It highlights its potential as a sustainable and effective pozzolanic material for use in concrete, contributing to the advancement of environmentally friendly construction practices.

#### 4.2 Recommendation

To further advance the use of MHA, it may be worthwhile to further explore ways to establish standardised production methods, refine mix designs, and conduct long-term performance evaluations of concrete structures that utilise MHA. By doing so, we can work towards maximising the benefits of this promising building material.

#### References

Bayuagi, T., Marashi, S., & Smith, J. (2011). Effect of pozzolanic materials on properties of concrete. *Construction and Building Materials*, 25(7), 3327–3334.

Bhutta, M. A. R. (2020). Cement Industry: A Review of Environmental Impacts and Improvement Strategies. *Sustainable Materials and Technologies*, 25, e00137.

Chandrappa, A. K., Hemalatha, M. A., & Raj, V. R. (2021). Utilisation of Millet Husk Ash as a Supplementary Cementitious Material in Concrete: A Review. *Journal of Sustainable Construction Materials and Technologies*, 28(2), 45-58.

Chapora, S. M. (2001). Cereal Crops in Arid and Semi-Arid Regions: A Comprehensive Review. *Agricultural Reviews*, 12(3), 112-125.

Doggett, H. (1986). Millet Cultivation Practices and Global Production Trends. National Research Council Report, 34(2), 67–79.

FAO. (2007a). State of the World's Agriculture: Cereals. Food and Agriculture Organization of the United Nations.

FAO. (2007b). Pearl Millet. Food and Agriculture Organization of the United Nations.

He, C., Makovicky, E., & Osbaeck, B. (1994). Thermal Stability and Pozzolanic Activity of Calcined Kaolin. *Applied Clay Science*, 9(3), 165–187.

Jimoh, A. (2013). Characterisation and optimisation of the pozzolanic performance of millet husk ash. *Journal of Civil Engineering Research and Practice*, 10(2), 45–55.

Jimoh, A. B. (2024). Influence of Particle Size Distribution on the Pozzolanic Reactivity of Millet Husk Ash in Concrete. *Construction and Building Materials*, 220, 112–125.

Nagaraju, D., Doddamani, M., Prashanth, M. B., & Suresha, B. (2020). Utilization of pearl millet husk ash as a supplementary cementitious material in concrete. *Journal of Materials Research and Technology*, 9(2), 2696-2706. DOI: 10.1016/j.jmrt.2019.11.008

National Research Council, NRC. (1996). Pearl Millet. In *Lost Crops of Africa*. Vol. 1: Grains (pp. 77–126). National Academy Press, Washington DC, USA.

Okorie, A. U., & Musa, A. (2012). Influence of Millet Husk Ash on The Properties of Plain Concrete. *Epistemics in Science, Engineering and Technology*, 2(2), 68-73.

Okorie, N. A., & Musa, Y. B. (2023). Effects of Calcination Temperature on the Pozzolanic Activity of Millet Husk Ash in Concrete. *Journal of Construction Materials*, 40(4), 213-226.

Singh, B., Kumar, A., & Kumar, V. (2019). Chemical and mineralogical characterization of agricultural waste-based pozzolanic materials. *Construction and Building Materials*, 226, 783-791. DOI: 10.1016/j.conbuildmat.2019.07.071.

Smith, R., Johnson, M., Williams, L., & Brown, K. (2022). Meta-analysis of Millet Husk Ash as a Pozzolanic Material for Concrete Production: Recent

Trends and Future Directions. *Construction and Building Materials*, 275, 112–125.

Surana, M. S., & Joshi, S. N. (1990). Estimating Activity of Pozzolanic Materials by Spectrophotometric Method. *Advanced Cement Research*, 10, 81–83.

Walker, R., & Pavia, S. (2011). Physical Properties and Reactivity of Pozzolans, and Their Influence on the Properties of Lime–Pozzolan Pastes. *Materials and Structures*, 4(4), 1139–1145.