

OPTIMIZING RICE HUSK ASH FOR USE AS A POZZOLANIC CEMENT REPLACEMENT MATERIAL

Suhail Zaffar^{*1}, Aneel Kumar², Abdullah Saand³, Naeem Aziz⁴

^{*1}Council for Works and Housing Research Ministry of Science and Technology government of Pakistan, Karachi 75730, Sindh, Pakistan

^{2,4}Department of Civil Engineering, Mehran University of Engineering and Technology, Jamshoro 76062, Sindh, Pakistan

³Department of Civil Engineering, Quaid-e-Awam University of Engineering, Science & Technology, Nawabshah 67480, Sindh, Pakistan

^{*1}drsuhailzaffar@gmail.com

DOI: <https://doi.org/10.5281/zenodo.21257274>

Keywords

rice husk ash, strength activity index, weight loss, calcination, XRD

Article History

Received: 24 April 2025

Accepted: 06 June 2025

Published: 21 June 2025

Copyright @Author

Corresponding Author: *

Suhail Zaffar

Abstract

This study was conducted to determine the optimal conditions for producing pozzolanic ashes from Rice husk for use as cement substitute materials. When the Rice husk turned into ash, it develops a pozzolanic character due to the presence of silica. However, the transformation of silica from amorphous to crystalline and its associated pozzolanic reactivity depends on the calcination temperature, duration, and grinding. In this research the Rice husk were calcined at three different calcination temperatures (300°C, 400°C, and 800°C) for 2-, 4-, 6- and 8-hours duration. The obtained ashes were ground for 30 minutes and replaced by 20% with cement. The samples containing ashes were tested for compressive strength, X-ray diffractometry (XRD), weight loss, and strength activity index (SAI). It was observed that the RHA calcinated at 600°C for 2 hours showed better strength. The materials ground for the higher duration is recommended for higher SAI.

INTRODUCTION

The construction sector relies heavily on concrete, arguably its most indispensable material, and consumption levels continue climbing year after year [1]. Cement forms the backbone of concrete production, yet the process of manufacturing clinker cement's core component generates between 8% and 10% of the world's CO₂ emissions [2], [3]. Taken together, construction related activities are responsible for more than one-fifth of total atmospheric CO₂. Since producing cement demands substantial energy input, researchers have increasingly turned to supplementary cementitious materials (SCMs) as a means of partially replacing cement while lowering the overall carbon footprint of binder production

[4], [5]. In addition, the disposal of untreated waste materials from industries and agricultural lands in open-air landfills has always been an environmental concern [6]. Further, the availability of land for dumping waste is getting limited with time. This leads to the discharge of waste materials into rivers and lakes or burnt in the open air which causes air pollution as well as health hazards [2]. Utilizing such waste materials rich in silica extend the range of building materials to be used as cement's substitute and can solve the ecological problems [6], [7].

In past, several studies have been conducted to investigate the use of industrial by-products and agricultural wastes as a partial alternative to cement [5], [8], [9]. The most used industrial by-

products as an alternative to cement are reported to be fly ash, slag, and silica fume [10], [11]. In addition, different agricultural wastes in the form of ash are also widely used in concrete as CRM [12]–[14]. Generally, the common practice for the disposal of agricultural wastes is open-air burning [2]. This open-air burning process not only emits harmful gases but also fades cementitious properties which restrict its use as CRM in the production of cementitious material [6]. In this regard, incineration of agro-wastes in a controlled condition is a promising method to reduce environmental pollution and retain the material properties.

Sahan and Aydin [9] reported the highest strength up to 10 % substitution of RHA cement. Chandra et al., [5] also observed the highest strength development rate at 10% replacement. Overall, the optimum replacement of RHA is selected in the range of 10-20% [15]. The enhanced strength is due to the availability of amorphous silica which increase pozzolanic performance [16], [17].

Methodology

Materials

Type I ordinary Portland cement (OPC) conforming ASTM C150 [25] specifications were utilized in the research. The cement was obtained from a locally available materials supplier in the vicinity of Jamshoro. Natural hill sand passing from the #30 sieve and retained at the #60 sieve was utilized for the preparation of mortar samples. Rice husk was acquired from nearby rice mills, located in the nearby vicinity of Jamshoro district.

Calcination of Ashes

Rice husk used in the research were subjected to heat treatment, to convert into ashes. The heating was given in an electric muffle furnace. To obtain rice husk ash (RHA), the heating was performed at 300°C, 600°C, and 800°C for 2-, 4-, 6- and 8-hours durations. After heating required temperature and duration the sample was allowed to cool in the open air. All the obtained ashes were ground in the ball mill for 30 minutes and sieved from the #200 sieve.

Sample Preparation

The mortar samples of size 50 mm³ were prepared by substituting 20% of the activated ashes by weight of the cement. The sample was prepared using a cement to a sand ratio of 1:2.75 and the water-to-cement ratio was kept constant at 0.49 (ASTM C 311) [26]. The detail of mortar mixes is presented in Table 1. The nomenclature for mixes is adopted in such a way that it signifies the type of ash used in a mortar mix. For example, the control mix denotes a mix containing only cement without ashes. RHA300-2H, RHA300-4H, RHA300-6H, and RHA300-8H signify rice husk ash calcinated at 300°C for 2, 4, 6, and 8 hours. RHA600-2H, RHA600-4H, RHA600-6H, and RHA600-8H signify rice husk ash calcinated at 600°C for 2, 4, 6, and 8 hours. RHA800-2H, RHA800-4H, RHA800-6H, and RHA800-8H signify rice husk ash calcinated at 800°C for 2, 4, 6, and 8 hours. The mixing, compaction, and molding of mortar specimens were performed following ASTM C305 [27] and ASTM C109 [28] guidelines. The samples were demolded after 24 hours and left for 28 days of water curing.

Table 1: Mix Details

Mix No.	Mix ID	Cement	Sand	RHA	w/b
A	Control (Cement)	1	2.75	-	0.49
B	RHA300-2H	0.80	2.75	0.20	0.49
C	RHA300-4H	0.80	2.75	0.20	0.49
D	RHA300-6H	0.80	2.75	0.20	0.49
E	RHA300-8H	0.80	2.75	0.20	0.49
F	RHA600-2H	0.80	2.75	0.20	0.49
G	RHA600-4H	0.80	2.75	0.20	0.49
H	RHA600-6H	0.80	2.75	0.20	0.49

I	RHA600-8H	0.80	2.75	0.20	0.49
J	RHA800-2H	0.80	2.75	0.20	0.49
K	RHA800-4H	0.80	2.75	0.20	0.49
L	RHA800-6H	0.80	2.75	0.20	0.49
M	RHA800-8H	0.80	2.75	0.20	0.49

Testing

X-ray diffraction analysis

Mineralogical analysis on RHA samples calcined at 300, 600, and 800 °C was performed by X-ray diffractometry with a copper tube and a nickel filter.

The compressive strength of the 50 mm mortar cube at 28 days of moist curing was determined following ASTM C109-21 standard [28]. The pozzolanic activity of the ash samples at different calcination temperatures was examined as per ASTM C618 specification [29].

The strength activity Index (SAI) was determined on cubes at 28 days of curing by replacing 20% cement with a weight of the ash. SAI was then calculated as per ASTM C311 [26] using the following formula: $SAI = (A/B) \times 100$, where A = average compressive strength of ash blended cubes, MPa (psi), and B = average compressive strength of control mix cubes, MPa (psi).

Results and Discussion

X-ray diffraction

X-ray diffraction analysis of RHA at different duration and calcination temperatures

Highly reactive RHA contains a large amount of amorphous silica with the presence of silanol groups [30]. Combustion temperature and time are important factors in determining whether

silica remains amorphous or becomes crystalline in RHA. The XRD analyses were performed to identify the changes in the development of amorphous and crystalline phases at different combustion temperatures (300, 600, and 800 °C) and durations (2, 4, 6, and 8 hours). The XRD patterns of RHA depicting the crystallographic structure of silica are shown in Figure 1. The intense broad peak observed for the RHA samples at 300, 600 and 800 °C indicated that silica remained essentially amorphous showing the crystalline reflections at $2\theta = 44^\circ$ and 22° respectively. Incineration at 300°C showed the amorphous silica with mild peaks of crystalline quartz. The intensity of peaks at 600°C and 800°C remained unchanged at 4, 6, and 8 hours of incineration. However, a slight drop in the peak was observed at $2\theta = 44^\circ$ but the formation of mild silica peak at $2\theta = 38^\circ$ maintained a similar crystallinity. An increase in the incineration temperatures i.e., 600 °C and 800°C, strong silica peaks developed depicting the formation of crystalline silica. This shows that the transformation of amorphous silica to crystalline form occurs at higher temperatures. This is also evident from Figures 1b and 1c, the intensity of the peaks at 600°C and 800°C remains sharp at all calcination durations. The results are also consistent with the Patel's study [30].

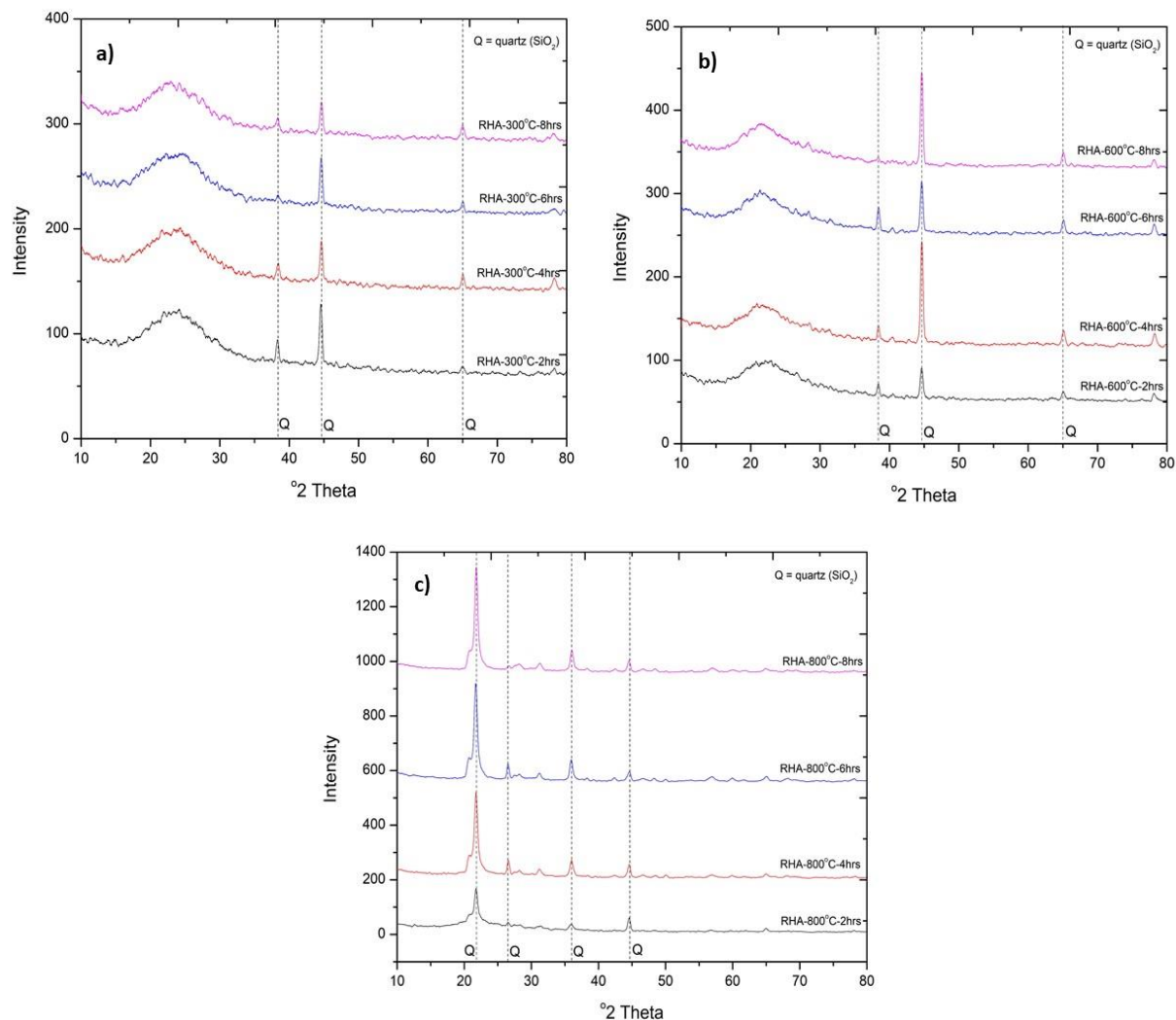


Figure 1: XRD pattern of rice RHA calcinated at a)300°C b) 600°C and c) 800°C

Compressive Strength and Strength Activity Index

Compressive Strength of RHA

The behavior of compressive strength of mortar samples containing RHA at different calcination temperatures and duration is present in Figure 2. It can be observed that the compressive strength of mortar samples containing RHA treated at higher durations during 300°C calcination temperature is increasing. The increase in strength was observed as 7%, 9%, and 11% compared to RHA300-2H. However, a significant drop in strength was observed compared to the control mix containing cement. The drop in strength was between 32%-

40%. A lower drop in strength was recorded when RHA was treated for higher durations. The highest strength gain was recorded for the RHA sample treated at 600°C for 2 hours (RHA600-2H). The increase in strength is due to an increase in pozzolanic activity of RHA- cement mortar [32], [33]. The improved pozzolanic performance is attributed to the presence of high reactive silica present in a material (Figure 1b). The strength for the remaining mixes calcinated at this temperature for a higher duration was observed to be identical but 4% and 31% lower than the respective mix (RHA600-2H) and control mix respectively. the utilization of an RHA calcined at 800°C for a

higher duration caused reduction in compressive strength up to 12% compared to sampled containing RHA calcined at 600°C. While a drop of 38% in compressive strength was recorded compared control mix. The decline in compressive strength is attributed to the poor pozzolanic

activity due to the formation of crystalline silica at 800°C calcination. This can be referred from the XRD graph shown in Figure 1c. from the experimental results, this can be concluded that the high reactive RHA can be achieved at 600°C calcination temperature for two hours.

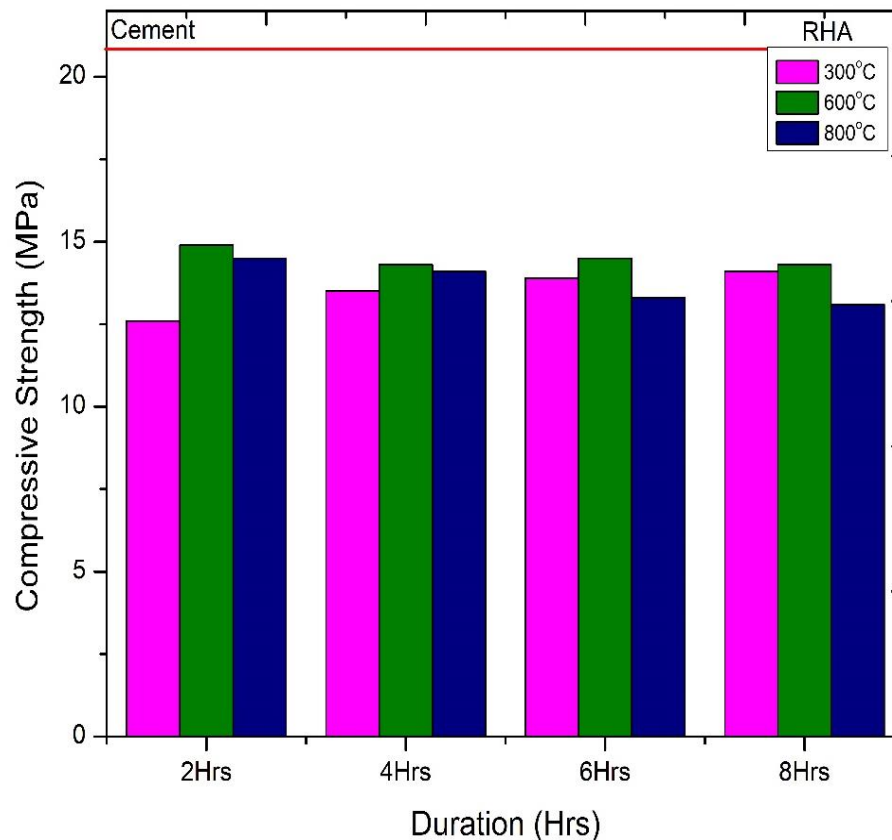


Figure 2: Compressive Strength behavior of RHA-cement mortar at different calcination temperatures

Strength Activity Index

Strength activity index (SAI) or pozzolanic activity index (PAI) is one of the most important tests which measures the compressive strength development rate of mixtures containing pozzolans. Figure 3 shows the SAI of RHA calcined at different temperatures for different

duration when used as a 20% substitute by weight of the cement. According to ASTM C618, the material can classify as pozzolan when its SAI is equal to or greater than 75%. This means that the mortar mix containing pozzolan should have the compressive strength equal to or greater than 75% of the control mix at 28 days.

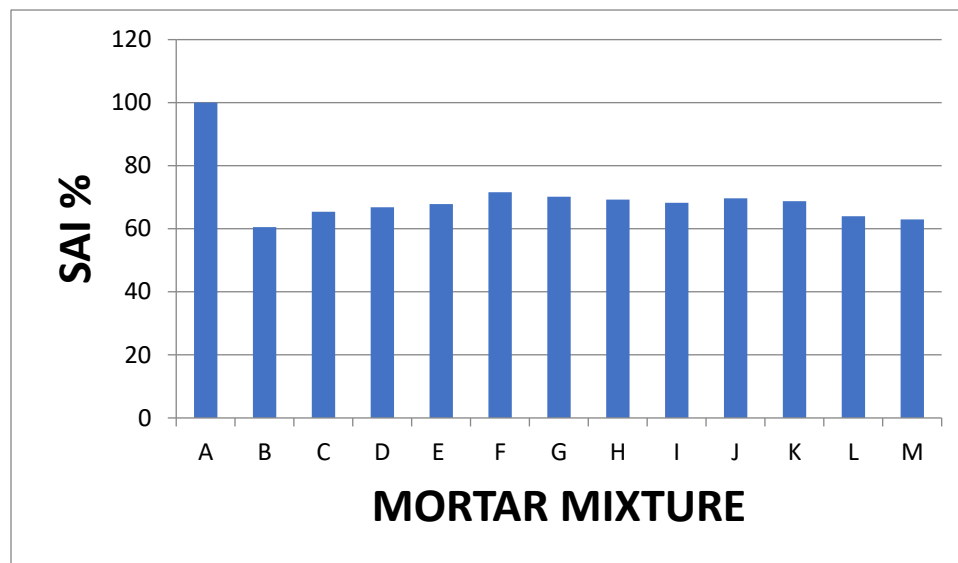


Figure 3: SAI of RHA at different incineration temperatures

Weight Loss

Weight loss is an important parameter in terms of obtained mass to be used as a substitute for cement after calcination. The weight loss of RHA subjected to different calcination temperatures

and durations are shown in Figure 4. The increase in weight loss was observed with the increase in calcination temperature. The highest weight loss of 85% was obtained during eight hours at 800°C calcination temperature for RHA.

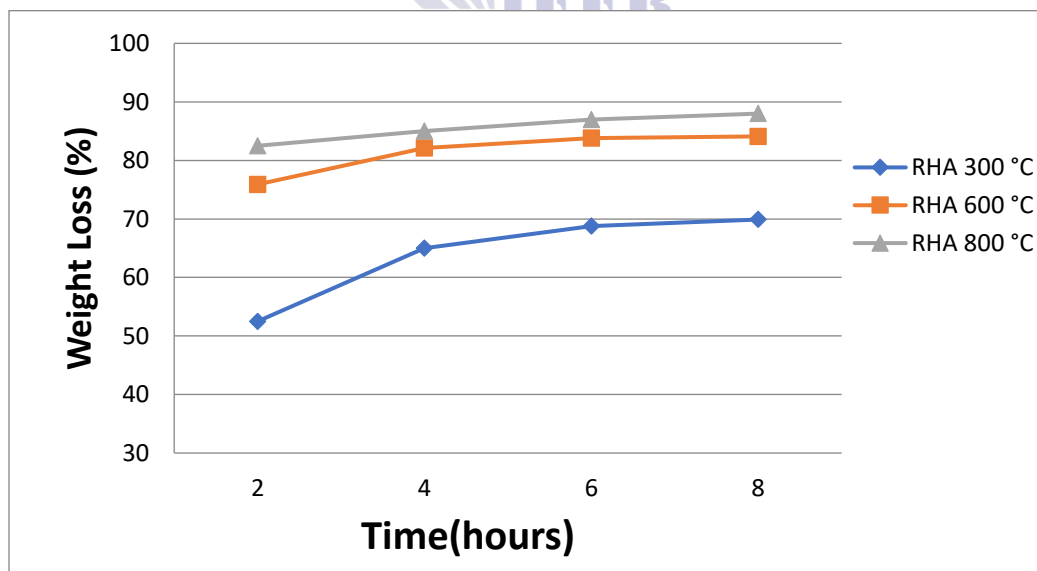


Figure 8: Weight loss (%) of RHA at different incineration temperatures

Conclusion

This research was conducted to evaluate the optimum conditions for obtaining different ashes that can be used as a partial substitute to cement. In this research, RHA were considered as potential

partial cement replacing materials. The prime focus was to find the optimum calcination temperature and duration to obtain highly reactive RHA to be used in cement-based materials. The ashes were prepared at controlled burning

temperatures of 300°C, 600°C, and 800°C for 2, 4, 6, and 8-hours duration. Based on the experimental results, the following findings are drawn.

- Based on the XRD patterns the silica was presented in the amorphous state when RHA were calcinated at 600°C. The optimum calcination duration for producing good quality RHA as 6 hours. While the duration for obtaining highly reactive RHA was evaluated as 2 hours.
- The highest compressive strength was obtained for mix (RHA600-2H)
- The highest SAI was achieved for the mortar samples containing RHA calcinated at 600°C for 2 hours duration (RHA600-2H).
- The increase in weight loss was observed with the increase in a calcination temperature. The highest weight loss of 85% was obtained during eight hours at 800°C calcination temperature for RHA.

References

- [1] M. Manjunatha, D. Seth, K. Balaji, and S. Chilukoti, "Influence of PVC waste powder and silica fume on strength and microstructure properties of concrete: an experimental study," *Case Studies in Construction Materials*, vol. 15, p. e00610, 2021.
- [2] R. Kumar, N. Shafiq, A. Kumar, and A. A. Jhatial, "Investigating embodied carbon, mechanical properties, and durability of high-performance concrete using ternary and quaternary blends of metakaolin, nano-silica, and fly ash," *Environmental Science and Pollution Research*, pp. 1-15, 2021.
- [3] R. Kumar, M. Yaseen, N. Shafiq, A. Jalal, and others, "Influence of metakaolin, fly ash and nano silica on mechanical and durability properties of concrete," in *Key Engineering Materials*, 2017, vol. 744, pp. 8-14.
- [4] D. B. Viet, W.-P. Chan, Z.-H. Phua, A. Ebrahimi, A. Abbas, and G. Lisak, "The use of fly ashes from waste-to-energy processes as mineral CO₂ sequestrers and supplementary cementitious materials," *Journal of Hazardous Materials*, vol. 398, p. 122906, 2020.
- [5] S. Chandra Paul, P. B. K. Mbewe, S. Y. Kong, and B. Šavija, "Agricultural solid waste as source of supplementary cementitious materials in developing countries," *Materials*, vol. 12, no. 7, p. 1112, 2019.
- [6] A. Kumar, R. Kumar, V. Das, A. A. Jhatial, and T. H. Ali, "Assessing the structural efficiency and durability of burnt clay bricks incorporating fly ash and silica fume as additives," *Construction and Building Materials*, vol. 310, p. 125233, 2021.
- [7] W. Xu, T. Y. Lo, and S. A. Memon, "Microstructure and reactivity of rich husk ash," *Construction and Building Materials*, vol. 29, pp. 541-547, 2012.
- [8] M. Ahad, M. Ashraf, R. Kumar, and M. Ullah, "Thermal, Physico-Chemical, and Mechanical Behaviour of Mass Concrete with Hybrid Blends of Bentonite and Fly Ash," *Materials*, vol. 12, no. 1, p. 60, 2019.
- [9] H. \cSahan Arel and E. Aydin, "Use of industrial and agricultural wastes in construction concrete," *ACI Materials Journal*, vol. 115, no. 1, pp. 55-64, 2018.
- [10] T. Nochaiya, W. Wongkeo, and A. Chaipanich, "Utilization of fly ash with silica fume and properties of Portland cement-fly ash-silica fume concrete," *Fuel*, vol. 89, no. 3, pp. 768-774, 2010.
- [11] M. D. A. Thomas, M. H. Shehata, S. G. Shashiprakash, D. S. Hopkins, and K. Cail, "Use of ternary cementitious systems containing silica fume and fly ash in concrete," *Cement and concrete research*, vol. 29, no. 8, pp. 1207-1214, 1999.
- [12] B. S. Thomas, J. Yang, K. H. Mo, J. A. Abdalla, R. A. Hawileh, and E. Ariyachandra, "Biomass ashes from agricultural wastes as supplementary cementitious materials or aggregate replacement in cement/geopolymer concrete: A comprehensive review," *Journal of Building Engineering*, p. 102332, 2021.

- [13] M. Amin, A. M. Zeyad, B. A. Tayeh, and I. S. Agwa, "Effects of nano cotton stalk and palm leaf ashes on ultrahigh-performance concrete properties incorporating recycled concrete aggregates," *Construction and Building Materials*, vol. 302, p. 124196, 2021.
- [14] F. Martirena and J. Monzó, "Vegetable ashes as supplementary cementitious materials," *Cement and Concrete Research*, vol. 114, pp. 57–64, 2018.
- [15] V. Charitha, V. S. Athira, V. Jittin, A. Bahurudeen, and P. Nanthagopalan, "Use of different agro-waste ashes in concrete for effective upcycling of locally available resources," *Construction and Building Materials*, vol. 285, p. 122851, 2021.
- [16] R. Embong, N. Shafiq, A. Kusbiantoro, and M. F. Nuruddin, "Effectiveness of low-concentration acid and solar drying as pre-treatment features for producing pozzolanic sugarcane bagasse ash," *Journal of Cleaner Production*, vol. 112, pp. 953–962, 2016.
- [17] E. Arif, M. W. Clark, and N. Lake, "Sugar cane bagasse ash from a high efficiency co-generation boiler: Applications in cement and mortar production," *Construction and Building Materials*, vol. 128, pp. 287–297, 2016.
- [18] F. F. Ataie and K. A. Riding, "Thermochemical pretreatments for agricultural residue ash production for concrete," *Journal of Materials in Civil Engineering*, vol. 25, no. 11, pp. 1703–1711, 2013.
- [19] D. Ramachandran, V. Vishwakarma, and K. Viswanathan, "Detailed studies of cow dung ash modified concrete exposed in fresh water," *Journal of Building Engineering*, vol. 20, pp. 173–178, 2018.
- [20] S. Sahin, B. Kocaman, I. Orung, and S. Memis, "Replacing cattle manure ash as cement into concrete," *Journal of applied sciences*, vol. 6, no. 13, pp. 2840–2842, 2006.
- [21] ASTM C150 / C150M - 20, "Standard Specification for Portland Cement," ASTM International, West Conshohocken, PA, 2020.
- [22] "ASTM C311/C311M – 18 Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency."
- [23] "ASTM C305-20 Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency."
- [24] "ASTM C109 / C109M-21, Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50 mm] Cube Specimens), ASTM International, West Conshohocken, PA, 2021."
- [25] ASTM C618-19, "Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete," ASTM International, West Conshohocken, PA, 2019, www.astm.org, 2019.
- [26] D. G. Nair, A. Fraaij, A. A. K. Klaassen, and A. P. M. Kentgens, "A structural investigation relating to the pozzolanic activity of rice husk ashes," *Cement and Concrete Research*, vol. 38, no. 6, pp. 861–869, 2008.
- [27] M. Patel, A. Karera, and P. Prasanna, "Effect of thermal and chemical treatments on carbon and silica contents in rice husk," *Journal of Materials Science*, vol. 22, no. 7, pp. 2457–2464, 1987.
- [28] K. Ganesan, K. Rajagopal, and K. Thangavel, "Rice husk ash blended cement: Assessment of optimal level of replacement for strength and permeability properties of concrete," *Construction and building materials*, vol. 22, no. 8, pp. 1675–1683, 2008.
- [29] D. D. Bui, J. Hu, and P. Stroeve, "Particle size effect on the strength of rice husk ash blended gap-graded Portland cement concrete," *Cement and concrete composites*, vol. 27, no. 3, pp. 357–366, 2005.