SSRG International Journal of Civil Engineering
ISSN: 2348-8352/ https://doi.org/10.14445/23488352/IJCE-V11I8P107

Volume 11 Issue 8, 83-92, August 2024

Original Article

Utilization of Mesua Ferrea Seed Shell Ash (MFSSA) as Pozzolanic Material by Partial Replacement to Cement in Mortar

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Received: 08 June 2024 Revised: 16 July 2024 Accepted: 07 August 2024 Published: 29 August 2024

Abstract *- The sustainable and eco-friendly construction materials have sparked research into various additives that can enhance mortar properties without harming the environment. This study aims to explore the potential of Mesua Ferrea Seed Shell Ash (MFSSA) as a pozzolanic material by exhaustively characterizing its chemical, physical and mechanical properties. Studies were done to assess the influence of MFSSA content on the consistency and initial setting time of cement. MFSSA to regular Portland cement with proportions equal to 0%,2.5%,5%,7.5%, and 10% were by weight of the cement. The main question is what the influence on mortar was from the replacement of it by a new hydraulic and sand cement material. After 3, 7, and 28 days of curing, compressive strength tests were performed, thereby providing knowledge about its load-bearing ability during construction and life of the structure. The durability of the material was evaluated for periods of curing up to 56 days using a sodium sulphate resistance test. As the ratio of MFSSA was increased, consistency and setting times also improved. 3 days of curing: Conventional mortar with replacement 2.5% increased compressive strength from 23 kN/mm^2 to 25 kN/mm^2. It was also obtained that compressive strength and durability towards sodium sulphate also decreased as the level of MFSSA was raised. Sustainability in construction is being advocated in this study as a whole. This study not only explored the novel use of MFSSA as a pozzolanic substance but also contributed to understanding its potential impact on blended cement properties.*

Keywords *- Cement, Compressive Strength, Environment, Mesua Ferrea.*

1. Introduction

In recent years, the construction industries have been exploring the utilization of agriculture and industry waste as cementitious materials. The cement industry is a huge contribution of CO_2 that is produced during the manufacturing of cement. OPC production contributes 6% of worldwide CO_2 emissions (Imbabi et al., 2013). Ordinary Portland cement is the most commonly used type of cement, and it is a primary component for both mortar and concrete. In the process of combustion and calcination, greenhouse gases are emitted, accounting for roughly 40% and 60% of emissions, respectively.

The World Business Council for Sustainable Development (WBCSD) played an important role in establishing the Global Cement & Concrete Association (GCCA), a collaborative effort involving major cement manufacturers worldwide. The primary goal of this initiative is to conduct a thorough examination of the environmental impacts and resource depletion caused by global cement production (Danish et al., 2019).

The aim is to raise awareness and encourage proactive measures to mitigate the worst effects associated with cement production. The aim is to completely understand these problems before educating others about them in order to encourage action that can lessen the detrimental effects of cement manufacturing while raising awareness and encouraging actions that help the cement and concrete industry adopt more sustainable methods. Globally, 10 billion tons of concrete are used every year. The ability of concrete and mortar to withstand environmental challenges is constantly being improved through new techniques, methods, and construction materials, enhancing the feasibility of our surroundings. The growing need for cement worldwide has prompted researchers in both advanced and developing nations to explore innovative alternatives. Portland cement, which is an important component of concrete, is particularly carbon-intensive, contributing about 8% of global CO2 emissions annually. To mitigate these impacts, researchers are developing various methods, including the use of alternative materials and new manufacturing processes, to reduce the carbon footprint of concrete while maintaining its essential properties" (Hilburg, 2019; Crawford, 2023). This shift towards exploring alternative materials reflects a broader commitment to finding more eco-friendly solutions in the building industry. On the subject of supplementary cementation materials in order to reduce the CO_2 gas emissions. A large quantity of agricultural waste material, such as bamboo leaf ash, rice husk, sugarcane bagasse, etc. The properties that provide them are suitable for use as cementation materials in construction. These waste materials are partially replaced as a pozzolanic material with cement, enhancing both the durability and strength properties of mortar and concrete.

1.1. Pozzolanic Materials

Pozzolans are an amazing substance with siliceous and aluminous qualities that are abundant in nature. While pozzolana silicate cannot function as a binder on its own, it can go through a chemical transformation at room temperature with water and $Ca(OH)_2$ to generate compounds with cementitious qualities. One of Portland cement's hydration products is calcium hydroxide. The percolating water may leak off this chemical, which is soluble in water but lacks cementitious properties. Calcium silicate hydrate, also known as calcium aluminate hydrate, is created when the finely divided siliceous or aluminous substance interacts with calcium hydroxide. When pozzolanic material and this water-soluble Ca $(OH)_2$ combine, insoluble calcium silicate hydrate is created.

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Their actions can be shown as:

$$Ca(OH)_2 + SiO_2 \rightarrow C\text{-}S\text{-}H + H_2O$$

Calcium hydroxide + silica (pozzolana) → calcium silicate hydrate + water

This reaction is known as a pozzolanic reaction. The characteristic feature of the pozzolanic reaction is that the results are initially slow, which means that both the heat of hydration and the increase in strength will be slow. The paste becomes dense and impervious due to the reduction of Ca $(OH)_2$ and improves its durability. This characteristic is very important in the world of construction materials. Pozzolanas used in the construction sector are of interest, especially those made from agricultural leftovers and industrial byproducts. Silica fume, fly ashes, and the ash remaining after burning organic materials high in silica, like rice husks and sugarcane bagasse, are important examples. These materials are abundant and environmentally benign because they are essentially waste products from other businesses. A common byproduct of cement hydration, calcium hydroxide, combines with these elements to generate compounds that improve strength and durability.

Thus, it’s more than just a construction material; it is a sustainable solution that is improving the way we construct. Therefore, many studies have focused on the use of agro-industrial residues such as pozzolans. For example, CCA (Corn Cob Ash) was made by grinding dried corn cob ash until it was about 4.00 mm in diameter; this process was done to ensure proper combustion and to lower the carbon content, which can impact the pozzolanic properties of the CCA. Around 8 hours, CCA was burned at 650 degrees Celsius. During this time, the corn cobs underwent combustion and eventually turned into ashes (Adesanya and Raheem, 2008). Corn cobs were burnt under controlled conditions at a temperature of 600 °C for 2 hours to obtain corn cob ash (Akindahunsil et al., 2022). In both studies, it was observed that the percentage of silica content is different in CCA because of the method of burning the Corn cobs in an uncontrolled environment. Whereas other agro-waste used as pozzolanic material like bamboo ash, the sum of SiO_2, $Al2O_2$ and Fe_2O_3 is 88.75% more than 70%, which is required for a good pozzolana conforming to ASTMC 618 (Lucas et al., 2021). On comparing the chemical composition of CCA and OPC, it was observed that High silica content in SCBA (78.498%) and low silica in OPC (19.189%) indicate few aluminosilicates. SCBA's 89.618% combined $SiO_2+Al_2O_3+Fe_2O_3$ qualifies as Class F pozzolan (>70%) (Arif et al., 2016). This study centered on examining Mesua Ferrea Seed Shell Ash (MFSSA) chemical composition and physical properties and assessing the mechanical characteristics of cement blends produced by burning and grinding MFSSA with Portland Cement at various replacement levels. Significantly, there is no previous exploration of MFSSA as pozzolanic substances in construction materials, making this research a pioneering effort to understand its prospective effect on the characteristics of cement.

2. Materials and Methods

2.1. Materials

This chapter summarizes the study of materials and outlines the selected research approach that incorporates both qualitative and quantitative methods.

2.1.1. Mesua Ferrea

The material was collected from the NERIST (North Eastern Regional Institute of Science and Technology) campus, Nirjuli, Arunachal Pradesh, India. It is commonly known as Ceylon ironwood, a species of the family Calophyllaceae. It is native to tropical parts of India, southern Nepal, Sri Lanka, and more. It is an evergreen tree with a 20m-30m height. The various parts of the plant have been used for different purposes, such as medicine, fuel, perfume, cosmetic products, etc. In Assam, it is used to light a candle and cook fuel at home. It has a high calorific value of 36,970 Kj/kg (Kushwah et al., 2008). This tree takes 12 years or more to bear seeds. It bears flowers in the months of April to June and fruits from July to September (Sharma et al., 2017). Domestic industries in India have recognized the value of Mesua ferrea as a source of natural medicine. The plant plays an important role in the production of various Ayurvedic and herbal products that cater to the healthcare needs of both the Indian population and consumers worldwide. The demand for products derived from Mesua ferrea continues to grow as more people turn to natural and traditional remedies for their well-being.

Fig. 1 Mesua Ferrea trees in NERIST

2.1.2. Cement

The 43 grade of Ordinary Portland cement was used, conforming to IS 8112:1989. Ordinary cement is the most commonly used cement for all general concrete construction. Annual worldwide production of OPC is 3.8 million tonnes. The chemical composition of 43-grade OPC cement analysis in EDX is shown in Table 1, and a graphical representation is shown in Figure 1. The physical properties of cement by performing the respective test, which conforms to IS code 8112-1989, are illustrated in Table 2.

Table 1. Chemical composition of OPC in EDX analysis

Element	OPC	
	Weight %	Atomic %
O	28.81	57.35
Si	5.56	6.30
Ca	28.50	22.64
Al	1.60	1.89
Fe	1.73	0.98
Mg	0.75	0.98
S	1.25	0.99

2.1.3. Fine Aggregate

Fine aggregate is required to determine the strength of cement. It is categorized based on particle size; typically, the particles that are passing through a 4.75 mm sieve are used. The locally available river sand with particle sizes finer than 4.75 mm was used as fine aggregate for this study.

Based on the particle size distribution and fineness modulus, the IS 383-2016 divides the fine aggregate into four grading zones. Zone I and Zone II are generally used in concrete mixed, while Zone III and Zone IV fine aggregate are suitable for specialized applications where fine texture is required.

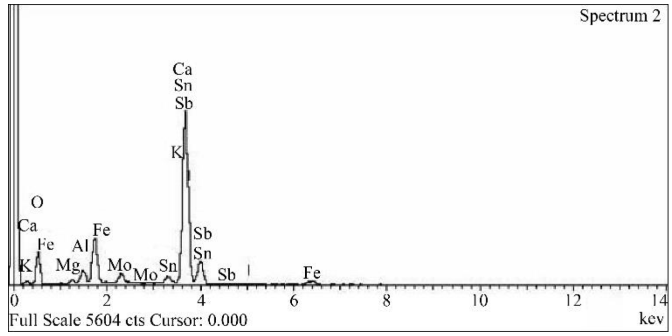

Fig. 2 Graphical representation of OPC in EDX analysis

Table 2. Physical properties of cement

Tests	Results	Procedure	Requirement as per IS 8112-1989
Fineness modulus	4.45 %	IS 4031 (Part 1)	Not more than 10 %
Consistency	33 %	IS 4031 (Part 4) 1988	No standard value
Initial setting time	1 hr 20 min	IS 4031 (Part 5) 1988	Minimum 30 min
Final setting time	4 hr 15 min	IS 4031 (Part 5) 1988	Maximum 10 hr

2.2. Methods

This research centers on investigating the practicality of integrating Mesua Ferrea Seed Shell Ash (MFSSA) into cement compositions. By introducing MFSSA in different percentages (0%, 2.5%, 5%, 7.5%, and 10%) relative to the weight of cement, the primary objective is to observe the physical properties, mechanical properties, and durability of cement. This endeavor seeks to elevate the overall durability and efficacy of the construction material.

2.2.1. Preparation of Materials

Preparation of MFSSA

Drying and thermal experiments were carried out in an open atmosphere in uncontrolled conditions to convert the outer cover part of Mesua ferrea into pozzolanic material. Around 3kg of ash was obtained from 22 kg of dry Mesua ferrea seed shell. The dark grey ash was obtained.

The collected sample was sieved in 90 µm. Sieving was done to segregate the unburnt carbon particles from the MFSSA particles. The specific gravity was found to be 2.43. The entire process of preparing pozzolan from raw materials to ash is depicted in Figures 3,4,5 and 6.

Fig. 3 Mesua ferrea seed shell.

Fig. 4 Burning in open atmosphere in Uncontrolled conditions

Fig. 5 Mesua ferrea seed shell ash

Fig. 6 90 µm sieve MFSSA

Chemical Analysis of MFSSA

In order to find the silica content in the Mesua ferrea seed shell ash sample, the EDX (Energy-Dispersive X-rays) analysis was conducted. EDX analysis is used to identify the elements in weight% and atomic%, as shown in Table 3 and Figure 8.

Table 3. Chemical composition of MFSSA

Elements	Weight %	Atomic%
O	51.98	68.76
Si	22.78	16.90
Ca	10.88	5.14
Al	6.22	5.26
Fe	5.85	2.41
Mg	1.53	1.22
S	0.76	0.31

Sieve Analysis

A sieve analysis test was applied to find the size distribution of fine aggregate. The sieve sizes were used as per IS code 386:2016 given in Table 4. The material retained on each of these sieves reveals the fraction of aggregate that is both finer and coarser than the size sieve above it.

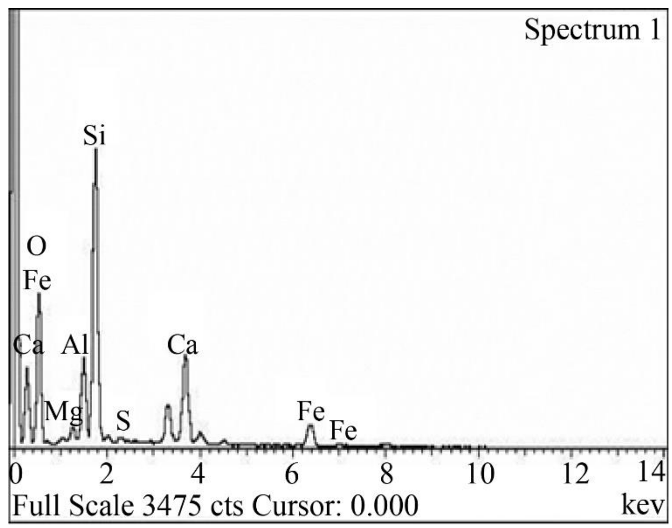

Fig. 7 Graphical representation of MFSSA in EDX analysis

2.2.2. Experimental Works

Consistency

The consistency is used to determine the amount of water necessary to make a perfect cement paste. In this study, the standard consistency (P) of all the samples was determined by using a Vicat apparatus with a plunger, as shown in Figure 9, conforming to IS 4031 (Part 4) 1988. In Figure 9, according to the Vicat test, the percentage of water added to cement so that the needle cannot penetrate 5-7 mm from the bottom of the mold. The depth indicates the standard consistency of the cement paste with the MFSSA replacement.

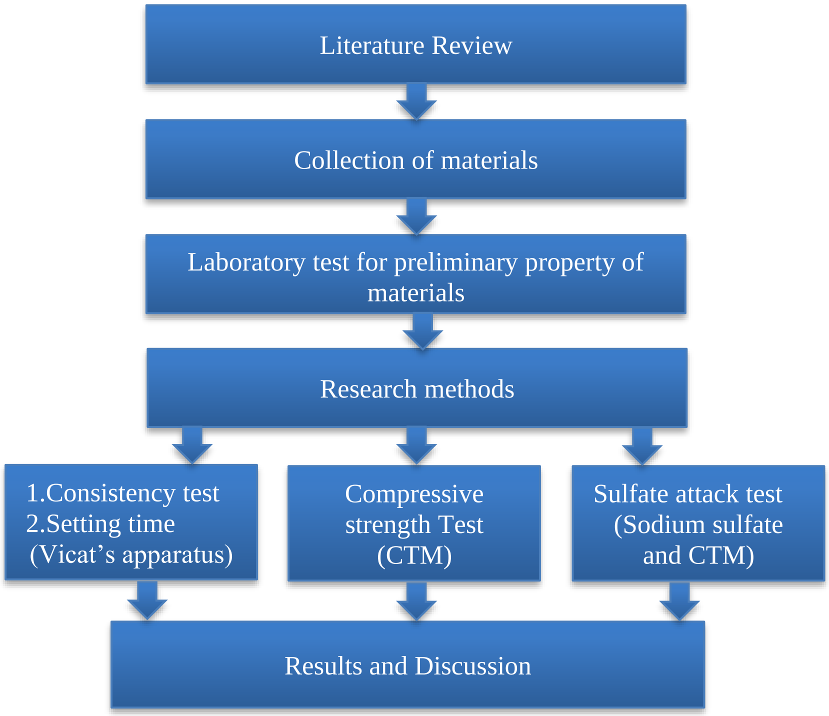

Fig. 8 Work flow chart

Table 4. Sieve analysis of fine aggregate

S.No.	Sieve size	Wt. of Retained (gm)	Cumulative wt. Retained (gm)	Cumulative wt. retained %	Cumulative wt. passing %
1	10 mm	0	0	0	100
2	4.75 mm	0.43	0.43	0.086	99.914
3	2.36 mm	2.5	2.93	0.586	99.414
4	1.18 mm	43.87	46.8	9.36	90.64
5	600 µ	106.4	153.2	30.64	69.36
6	300 µ	251	404.2	80.84	19.16
7	150 µ	81	485.2	97.04	2.96
8	pan	14.8	500	100	0

Setting Time

After conducting the consistency test, the different values of "P" were assessed to determine the initial and final setting of the cement and blended cement as per IS 4031 (Part 5) of 1988. A standard was employed utilizing the Vicat apparatus shown in Figure 10 above. The quantity of water added is (0.85P) % of the total weight of cement. The Vicat apparatus is specifically used to determine both the initial and final setting times.

Compressive Strength Test

The compressive strength test was performed on mortar cubes of size 70.6 mm x 70.6 mm x 70.6 mm. The cubes were tested after 3, 7 and 28 days of curing periods as per IS code 4031 (Part 6) of 1988. The mixed cement, mortar cube samples of a (1 cement : 3 sand) ratio were prepared by replacing cement with MFSSA at 0%, 2.5%, 5%, 7.5%, and 10% by weight of cement. The quantity of water [(P/4) + 3] % of the total weight was added, and a total of 45 cubes were cast. During the test, the machine applied compressive force to the mortar cubes and the corresponding resistance to this force was measured. This process allowed the determination of the compressive strength of the hardened mortar at each of the specified curing ages. In the above, the cubes were cast in a 70.6 mm mold of the cube and immersed in water for curing, which helps in the hydration of cement and gains strength. The CTM machine was used to determine the strength of the mortar cube.

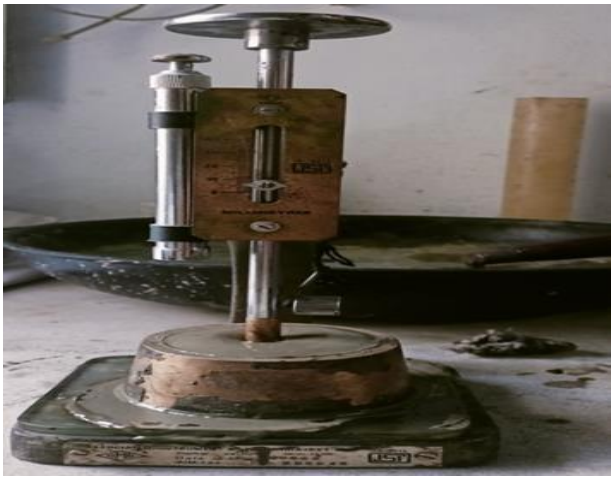

Fig. 9 Vicat's apparatus for consistency

Fig. 10 Vicat's apparatus for setting time

Sulphate Attack Test

Sodium sulphate was used for the sulphate attack analysis. It is an anhydrous white crystalline solid of the formula Na2SO4. In this study, a 5% sodium sulphate (Na_2SO_4) solution was created by blending the chemical with water, comprising 50 g of Na_2SO_4 in 950 ml of water, targeting a pH range of 6 to 8. This represents extremely severe sulphate exposure, as per ASTM C1012. Initially, 15 cubes of 70.6 mm were prepared with 3 cubes, allocated for each percentage of 0%, 2.5%, 5%, 7.5%, and 10%. After 24 hours, the mortar cubes were demolded and left to cure for 3 days and 7 days. It is required that the mortar acquire the strength of 20 N/mm^2 before being immersed in the sulphate solution. The following cubes were immersed in a sulphate solution for 56 days of curing. The sulphate resistance results were expressed in compressive strength loss compared with normal mortar.

3. Results and Discussion

3.1. General

The main objective of this study is to investigate the pozzolanic properties of Mesua ferrea seed shell ash (MFSSA), aiming to assess its impact on cement's physical and mechanical characteristics by conducting experiments focused on consistency, setting time, compressive strength, and durability. Sulphate attack analysis using a sodium sulphate solution demonstrated the durability of the blended cement. Preliminary tests, including sieve analysis and chemical composition analysis of MFSSA through EDX, were conducted.

3.2. Chemical Properties of MFSSA

Table 1 and Table 3 show that the Si percentage is higher MFSSA as compared with ordinary Portland cement, as we know that Si influences the strength of the cement matrix and setting time in cement. With an increase in the percentage of Si, both setting and strength will increase. The silicon content of MFSSA has been determined to be 22.78%. The amount of silica content is very low compared to other pozzolanas like sugarcane bagasse and rice husk ash. The process of burning mesua ferrea seed shells in an uncontrolled atmosphere may be caused by a low silica concentration in MFSSA.

3.3. Morphological Analysis of Cement and MFSSA

For the examination of microstructure properties, the SEM (scanning electron microscopy) test with a magnification of X1000 and an acceleration voltage of 20 kV was conducted. The distance between the microscope and the sample is 10 µm. The test was conducted to observe the surface images of cement and MFSSA shown in Figures 11 and 12, respectively. A high-clarity image is seen as the distance decreases between the microscope and sample, as shown in Figure 13, with the magnification of X10000, which gives a bolder picture than X1000.

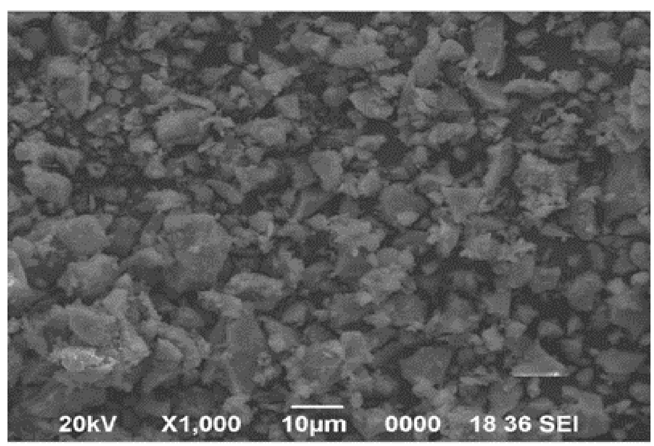

Fig. 11 SEM micrograph of OPC cement

Figure 12 displays that MFSSA has more fine particles than cement. The finer particles of MFSSA ensure active participation in pozzolanic activity. The particles are distributed in a wide range of sizes, with varying particles and irregular shapes. Irregularity enhances the bonding between the particles. The individual particles give a flaky morphology, and irregular voids contribute to a higher surface area, resulting in increased setting time. In the cement sample, Figure 11 shows the angularity and irregularity of the particles. Patil et al., 2018 examined the SEM of OPC and noted a similar microstructure in OPC.

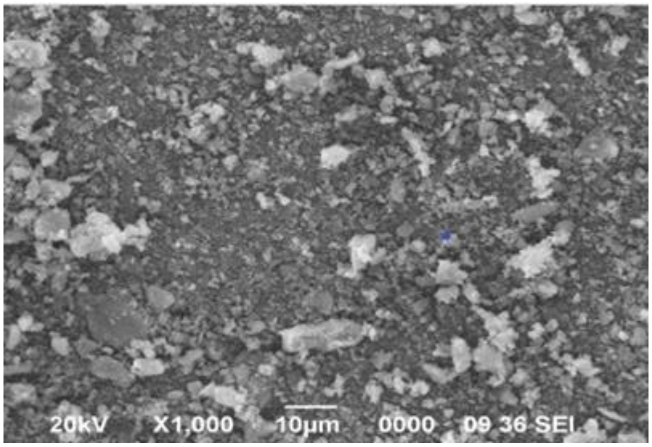

Fig. 12 SEM micrograph of MFSSA

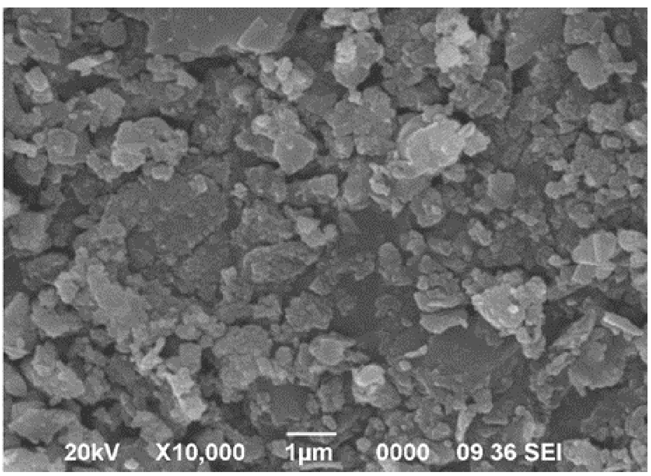

Fig. 13 SEM micrograph of MFSSA magnification of X1000

3.4. Physical Property of Fine Aggregate

Choosing the right type and quality of aggregates is absolutely crucial when it comes to the strength of mortar, particularly in masonry applications. In the composition of masonry mortar, a major portion is made up of fine aggregates, commonly referred to as sands. The choice of these fine aggregates has a profound impact on the characteristics of the mortar, influencing its properties in both the fresh and hardened states. The physical properties of the sand significantly affect the physical properties and compressive strength of the mortar, which is essential for the stability and durability of any masonry structure.

Table 5. Physical properties of fine aggregate

Tests	Results	As per IS 383: 2016
Fineness modulus	3.18	2.2–3.2
Specific gravity	2.51	2.1-3.2
Water absorption	0.72 %	< 5 %

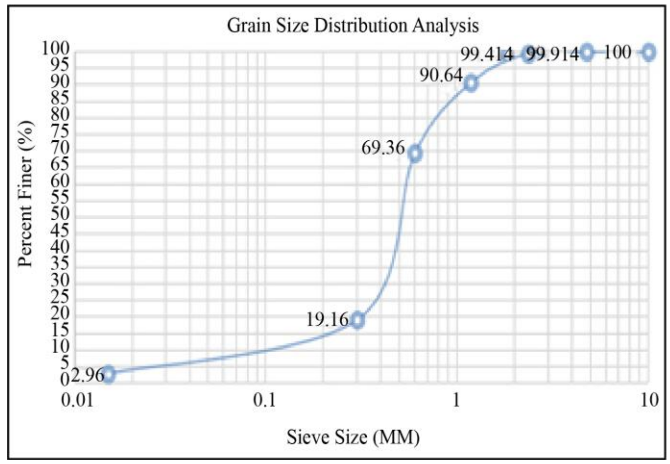

Fig. 14 Particle size distribution curve of sand

Figure 15 shows that the compressive strength of mortar gradually increases its strength with the age of curing. MFSSA shows a good increment in strength compared to the controlled specimen, with an optimum limit of 2.5 %, and beyond that limit, the strength of the mortar was decreased with an increase in the amount of MFSSA. As the mortar strength varies depending on curing age, it can be higher later on. The presence of a higher percentage of alumina and silica compared to controlled cement and when this ash is blended with cement and water, reactivity is very high and a high amount of Calcium Silicate Hydrate (CSH) gel is produced compared to controlled OPC, which is responsible for improving the cementitious properties.

Table 6. Physical properties of blended cement

	Percentage of replacement of MFSSA to cement				
	0 %	2.5%	5 %	7.5 %	10%
Consistency (%)	33 %	33.5%	35 %	36%	37.5%
Initial setting time	75 min	90 min	124 min	155 min	180 min
Final setting time	245 min	275 min	315 min	355 min	405 min

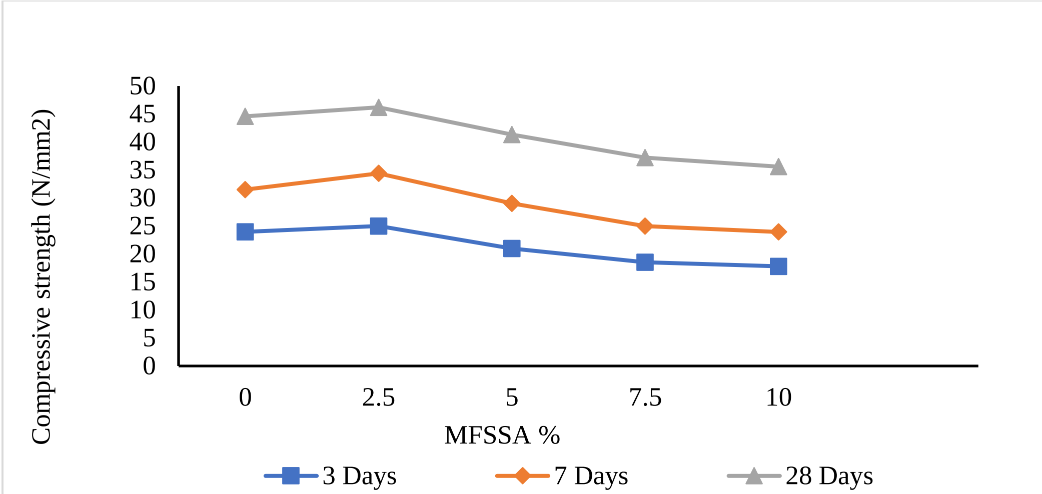

Fig. 15 Graphical presentation of compressive strength of mortar

Table 7. Compressive strength of mortar

Mix Proportion	Curing Period		
	3 Days	7 Days	28 Days
100 % cement	23.93	31.52	44.56
97.5 % cement + 2.5 % MFSSA	25	34.4	46.2
95 % cement + 5 % MFSSA	21	29.06	41.3
92.5 % cement + 7.5 % MFSSA	18.5	25	37.2
90 % cement + 10 % MFSSA	17.8	23.93	35.6

Table 8. Curing of mortar to reach 20 N/mm2 compressive strength

Mix proportion	Curing periods (Days)
100 % Cement	3
97.5 % Cement + 2.5 % MFSSA	3
95 % Cement + 5 % MFSSA	7
92.5 % Cement + 7.5 % MFSSA	7
90 % Cement + 10 % MFSSA	7

3.4.1. Sulphate Attack Test

It has been observed that the replacement level of MFSSA is directly proportional to the curing period because of the lower quantity of cement and the lower rate of hydration reaction, resulting in less strength development. The strength of mortar achieved in 3 and 7 days of curing was reduced after being stored in a sulphate solution for 56 days. The 3-day curing value of 23.93 N/mm2 was reduced to 20 N/mm2 in the controlled specimen. Meanwhile, in an earlier compressive strength test, 2.5% was the optimum strength value, and due to the sulphate attack, strength decreased, as shown in Figure 17. When mortars were exposed to a solution of sodium sulphate, the calcium hydroxide from the hydration reaction of cement reacted with sodium sulphate. It transformed into gypsum ($CaSO_4.2H_2O$), which led to the expansion of the outer surface of the specimen. The gypsum reacts with aluminum compounds to produce ettringite ($6CaO.Al_2O_3.(S0_4)_3.32H_2O$) which increases in instability and volume, which leads to further crack and loss of strength.

Fig. 16 Immersed cube in sodium sulphate

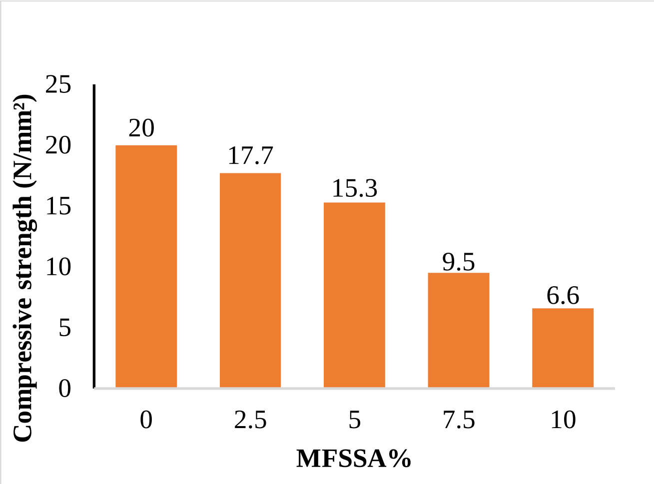

Fig. 17 Compressive strength of sulphate attack in mortar

Figure 17 shows that due to the sulphate attack, the compressive strength of mortar was reduced with the increment percentage of MFSSA in cement. It was found that the compressive strength loss increases with increasing the water-to-binder ratios. Due to the excess amount of water mixing in the mixture, the mortars get more porous. Because of this, there is a greater probability that sulphate ions will react with Ca $(OH)_2$, which lowers compressive strength. The strength had declined up to the optimum limit with the increase in MFSSA content, possibly due to insufficient time for reaction or inadequate particle size functioning effectively as filler material.

4. Conclusion

The following conclusions are taken from the results of an experiment conducted on MFSSA.

1. All the percentages of partial replacement of MFSSA in cement have a higher setting value as compared to control cement, so they are applicable for mass concreting, where a low development of heat is required. By precisely measuring initial and final setting times, this analysis ensures adherence to construction timelines, promotes optimal strength development and contributes to the overall durability and performance of concrete structures, making it an essential parameter for quality control in cement production and construction practices.
2. It was concluded that 2.5% is the optimum replacement to get higher compressive strength than control. In simpler terms, when more MFSSA was substituted for traditional cement in the mixture, mortar tubes became weaker, and their ability to withstand compression forces was reduced.
3. It was also concluded that the addition of MFSSA to cement was not suitable for sulphate resistance. The ratio of water to binder significantly impacts the deterioration of mortar from sulphate attacks.

4.1. Scope for Further Research

1) Explore additional tests and conduct comprehensive tests to assess the properties of mortar, like soundness, flow table analysis, water absorption, permeability, and resistance to chemical attacks. These examinations confirm the qualities that are necessary to preserve structural lifespan and integrity under the arrangement of environmental circumstances.

2) MFSSA in concrete
Investigate the use of MFSSA in concrete by partially replacing cement or sand. This approach seeks to enhance sustainability and performance by reducing the reliance on conventional materials. MFSSA's potential benefits include improved strength, reduced environmental impact, and reduced costs, making it a viable field for sustainable construction practices.

3) Combination research of MFSSA
Study the beneficial effects of combining MFSSA with other agro-waste and industrial byproducts to optimize concrete properties and promote eco-friendly construction materials.

4) MFSSA in soil stabilization
Investigate its use in soil stabilization for its beneficial properties that enhance the engineering performance of soils.

Acknowledgements

The authors acknowledge the North Eastern Regional Institute of Science and Technology for their help and encouragement during the investigation.

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