ENHANCING WATER ABSORPTION AND SHRINKAGE CHARACTERISTICS OF RICE-HUSK ASH BASED GEOPOLYMER MORTAR

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ABSTRACT

Ordinary Portland Cement (OPC) is a binding agent in concrete production. Despite the advantages OPC offers, its environmental impact cannot be overlooked. Cement production contributes to over 11% of global carbon emissions and plays a significant role in climate change. Geopolymer, which involves the alkali activation of alumino-silicate materials, is an alternative to OPC. Rice Husk Ash (RHA), obtained by burning rice husks, has gained attention as a sustainable material that can partially replace geopolymers. This study aims to create a geopolymer mortar mix incorporating Ground Granulated Blast Furnace Slag (GGBFS) and RHA as binders and optimise selected mixes' water absorption and shrinkage characteristics. The alkali activation of this binder leads to increased strength and durability characteristics. The study employs various tests to determine the optimum percentage of RHA to be used as a substitute. Shrinkage characteristics of selected mixes were studied and improved by incorporating Graphene Oxide (GO).

1.Introduction

Due to its advantages, cement is the most versatile binding agent widely used in most constructions. However, its production has a detrimental impact on the environment. Portland cement production emits high amounts of greenhouse gases into the atmosphere. CO2 is the most significant greenhouse gas contributing to the warming of the Earth's surface. The production of Portland cement is responsible for approximately 1.5 metric tonnes of CO2 emissions per year (equal to 11% of global CO2 emissions). Production of portland cement in the kiln also requires high temperatures which leads to extreme energy usage [1]. Geopolymers, as cement substitutes, have emerged as a promising solution to mitigate the environmental effects of cement manufacturing. When formulated and cured under the right conditions, this binder class offers excellent mechanical properties, good durability, and minimal environmental impact[2]. Geopolymers are produced when the solid aluminosilicate reacts with an alkali source, which is frequently supplied as a liquid.

Ground granulated blast furnace slag (GGBFS), fly ash, RHA, and calcined kaolinite-group clays are not just alternatives to cement; they are beacons of environmental optimism[3],[4]. With their significant pozzolanic activity, these materials offer a viable and sustainable alternative to cement. Their use in mortar production, improves mechanical properties and durability while minimising environmental impact. Using GGBFS and RHA has significant advantages, as they require less energy to produce than Portland cement and emit lower carbon.

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Understanding the engineering properties of geopolymer materials, particularly their shrinkage characteristics, is urgent in the quest for sustainable construction. Among the factors influencing these properties, alumino-silicates and alkaline activators play a significant role. The properties of geopolymer mortar depend primarily on the activator's type and concentration as well as the activation method. Shrinkage is a vital engineering property that influences concrete cracking and its durability[5]. The objective is to study the shrinkage characteristics of partial RHA-GGBFS geopolymer to understand the effect of different binder-alkali ratios on the properties of RHA-based geopolymer. The effectiveness of an admixture, Graphene oxide, in reducing shrinkage and water absorption is also studied.

2 Materials and Experimental Program

2.1 Materials

Ground Granulated Blast Furnace Slag (GGBFS), a derivative of the iron and steel manufacturing process, is provided by JSW Cements Ltd., Bilakalagudur. The properties of GGBFS used for the project are shown in Table 1. Rice Husk Ash (RHA), an eco-friendly precursor, was used in the project. The Nano research lab in Jamshedpur provides graphene oxide (GO). The properties of GO are shown in Table

2. Sodium hydroxide and sodium silicate are used to prepare the activation solution obtained from HCl Products Ltd., Kochuveli.

Table 1. Properties of GGBFS

Characteristics	Test results
Fineness (M^2/Kg)	441
Insoluble residue (%)	0.36
Magnesia content (%)	6.82
Sulphide content (%)	0.26
Sulphite content (%)	0.36
Manganese content (%)	0.16
Chloride content (%)	0.028
Glass content (%)	94.5
Moisture content (%)	0.09

Table 2. Properties of Graphene Oxide

Property	Value
Purity	98-99%
Diameter	<100nm
Thickness	0.5-2 Nm
Bulk Density	0.241g/Cc

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2.2 Mix Design

The objective was to create a sustainable geopolymer mix incorporating RHA as a substitute cementitious material for GGBFS. Five mixes were prepared with partial replacement percentages of RHA: 0%,20%,40%,60% and 80%. Table 3 summarises the mix design.

Table 3. Design Mix

Mix	GGBS(g)	RHA(g)	Sand(g)	GO (%)
RHA 0	230	0	690	-
RHA 10	207	23	690	-
RHA 20	184	46	690	-
RHA 40	138	92	690	-
RHA 60	92	138	690	-
RHA 80	46	184	690	-
GO 1	184	46	690	0.005
GO 2	184	46	690	0.01

The concentration and volume of the alkali activator solution were based on IS guidelines[6]. The amount of alkali activator used was 0.163 based on the weight of the mortar mix. The water-to-binder-to-water ratio was chosen as 0.66. After selecting RHA 20 as the optimum design mix, Graphene oxide is incorporated to improve the shrinkage characteristics of RHA geopolymer, thus preparing design mixes GO 1 and GO 2, activation Solution: NaOH, Na_2SiO_3 and Water in the ratio 1:2:3 by weight.

2.3 Experimental Methods

2.3.1 Compressive strength

The compressive strength of the specimen was conducted as per IS:4031(Part 6). For each mixture, three 70.6 ×70.6×70.6 mm cubic specimens were prepared. The test was performed using a compression testing machine, which applied a uniform load at 0f 35 $N/mm^{2.}$The test was done at seven days.

2.3.2 Water absorption

The test was conducted as per JC474-2008. The procedure involved preparing mortar cubes with dimensions 70.6 ×70.6×70.6 mm. The water absorption was then calculated as the percentage difference between the initial and final masses relative to the initial mass.

2.3.3 Shrinkage

The test was conducted as per IS 4031(Part 10). Specimens of size 141.2 ×70.6×70.6 mm were cast, conforming to the mix design. A length Comparer is used to measure the lengths of the specimen at 7 and 35 days. The average difference in length of three specimens to the nearest effective gauge length is taken as the drying shrinkage.

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2.3.4 Thermal conductivity

The Transient Line Source (TLS-100) equipment was utilised for the thermal investigation of cement mortar cubes. The 70.6x70.6x70.6 mm specimen dimensions were put into the sample holder, and the experiment started by progressively raising the temperature within a set range. The transient reaction was accurately assessed using the TLS-100

2.3.5 Scanning Electron Microscopy

Before Scanning Electron Microscopy (SEM) analysis, the samples were carefully prepared to provide the highest possible image quality. The samples for SEM analysis were 1cm*1cm*1cm cubes evenly shaped with flat surfaces. The samples were cured for seven days, dried and then sent to the Central Laboratory for Instrumentation and Facilitation (CLIF), University of Kerala, for analysis.

3 Results and Discussions

3.1 Compressive Strength

The results obtained from the compressive test are given in Tables 4.1 and 4.2. Figure 1 shows the variation of 7-day compressive strength with different design mixes of RHA and Graphene oxide.

Table 4.1. Seven-Day Compressive Strength of Specimens

Specimen	7 Day Compressive Strength (N/mm^2)
Cement Mortar	15
RHA 0	32
RHA 10	25
RHA 20	22
RHA 40	8
RHA 60	1
RHA 80	-

Table 4.2. Seven-Day Compressive Strength of Specimens

Specimen	7 Days Compressive Strength (N/mm2)
RHA 20	22
GO 1	32
GO 2	27

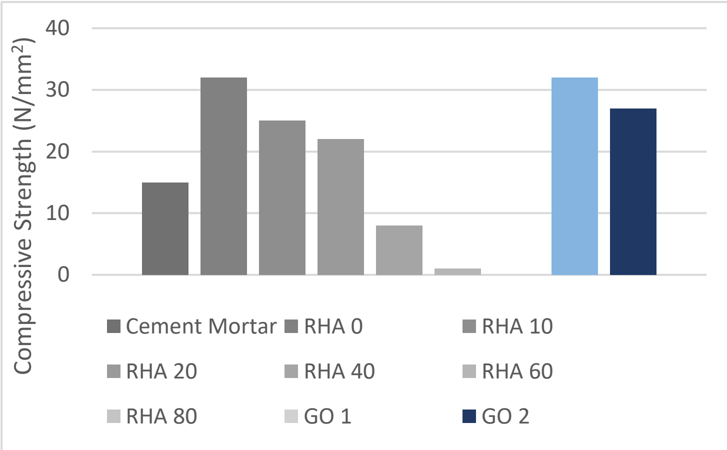

Figure 1. Compressive Strength of Different Mixes

The results and graph show that RHA0 has the highest compressive strength of 25N/mm2, followed by RHA20 with a strength of 22N/mm2. The formers show higher results than Ordinary Portland cement mortar, which showed a strength of 15N/mm^2. RHA40 and RHA60 were the least and showed a strength of 8 N/mm^2 and 1 N/mm^{2,} respectively, and RHA80, being the weakest mix, failed the test. Design mixes of graphene oxide incorporated RHA geopolymers also exhibited higher compressive strength than OPC mortar and RHA-20 mix.

3.2 Setting Time

The test was conducted as per ASTM-c807. Table 5 shows the initial setting time obtained for various mixes with different proportions of RHA replacement. Figure 2 shows the result obtained for the initial setting time. The results obtained were compared with that of ordinary Portland cement. The result indicates that RHA40 has the highest setting time of 28 minutes, and the lowest was for RHA0, showing 15 minutes. RHA20 has 21 minutes of initial setting time. Ordinary Portland cement mortar has an initial setting time of 30 minutes. In comparison, alkali-activated geopolymer mortars have a lower initial setting time than ordinary Portland cement mortars.

Table 5. Initial Setting Time of the Specimen

Specimen	Initial Setting Time (Min)
Cement Mortar	30
RHA 0	15
RHA 20	21
RHA 40	28

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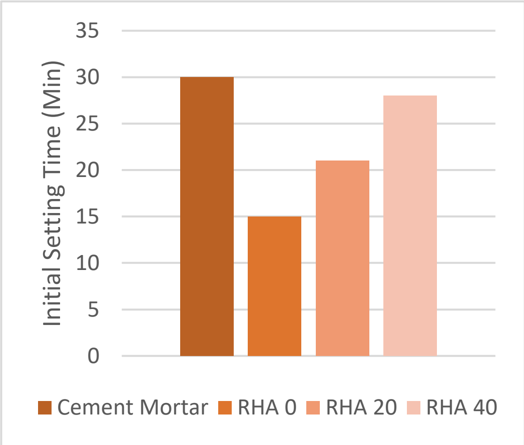


Figure 2. Initial Setting Time of Different Mixes

3.3 Water Absorption

Water absorption tests were conducted on the casted mortar specimens, and results from the test are presented in Fig. 3 and Tables 6.1 and 6.2. The result shows that the RHA 0 mix has lesser water absorption compared to OPC mortar. As RHA content increases, water absorption also increases.This result was described in 2023 [7].When graphene oxide is incorporated, water absorption in the mortar mix is significantly reduced.This result was discussed in [8].

Table 6.1. Water Absorption of Specimens

Specimen	Water Absorption (%)
Cement Mortar	4
RHA 0	3.22
RHA 10	4.35
RHA 20	4.86
RHA 40	5.37
RHA 60	-
RHA 80	-

Table 6.2. Water Absorption of Specimens

Specimen	Water Absorption (%)
RHA 20	4.86
GO 1	2.53
GO 2	1.13

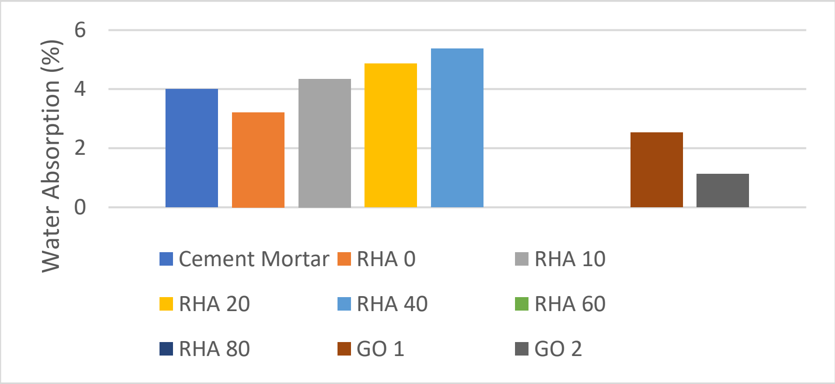


Figure 3. Water Absorption of Design Mixes

3.4 Shrinkage Test

Shrinkage of design mixes was analysed by measuring the percentage change in the linear dimension of specimens. Table 7 shows the results of the shrinkage test.

Table 7. Shrinkage of RHA mixes

Specimen	7-day Reading (mm)	35-day Reading (mm)	Shrinkage (%)
RHA 20	2.611	2.731	0.080
GO 1	2.751	2.844	0.062
GO 2	2.979	3.056	0.051

When Graphene Oxide is incorporated with RHA geopolymer,shrinkage is reduced which was ocncluded from [5].

3.7 Thermal Conductivity Test

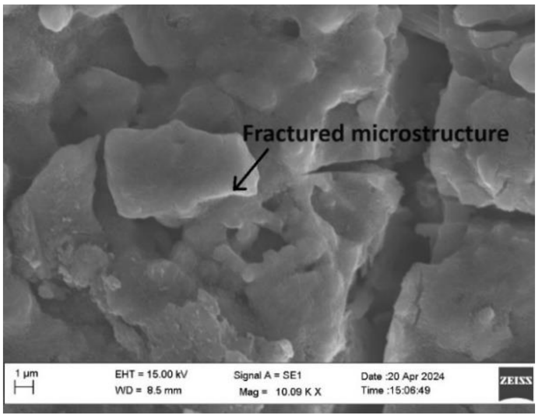
Thermal conductivity and resistivity of different mixes were tested; the result is shown in Table 8. The thermal conductivity of OPC mortar is generally 0.703 to 1.149 W/mK. The thermal conductivity of mortar mixes added with GO was significantly less than that of the RHA 20 mix. As the percentage of GO increased, thermal conductivity also increased.

Table 8. Thermal Conductivity of GO mixes

Specimen	Resistivity (mK/W)	Thermal Conductivity (W/mK)
RHA 20	20.644	0.048
GO 1	19.233	0.052
GO 2	18.649	0.053

3.8 SEM Analysis

SEM images of mortar with RHA 20 and RHA 20 mixed with 0.005%wt GO at seven days are shown in Fig. 4.1, Fig. 4.2, Fig. 4.3, Fig. 4.4, Fig. 4.5 and Fig 4.6.



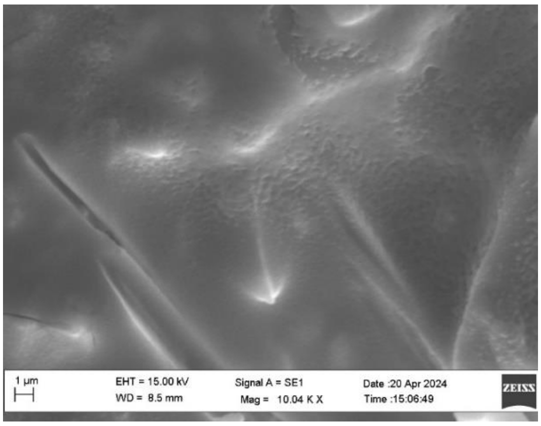
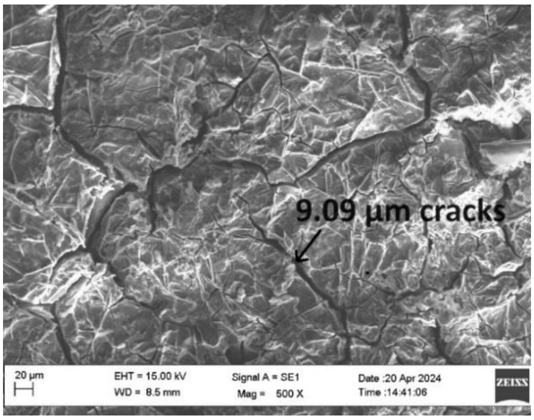


Fig. 4.1

Fig. 4.2



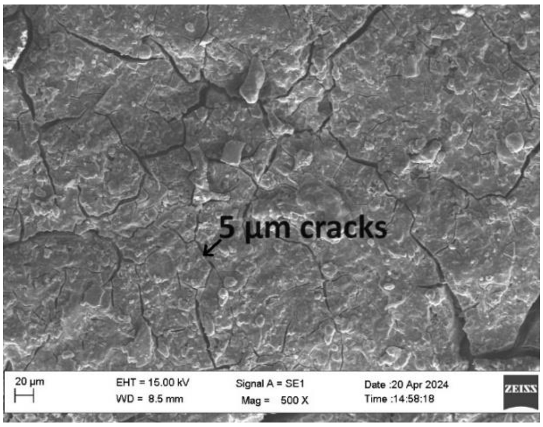
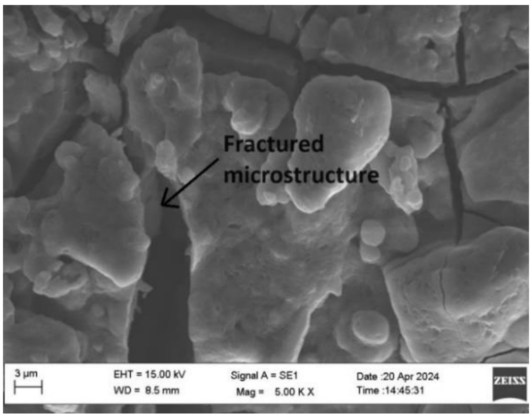


Fig.4.3

Fig.4.4



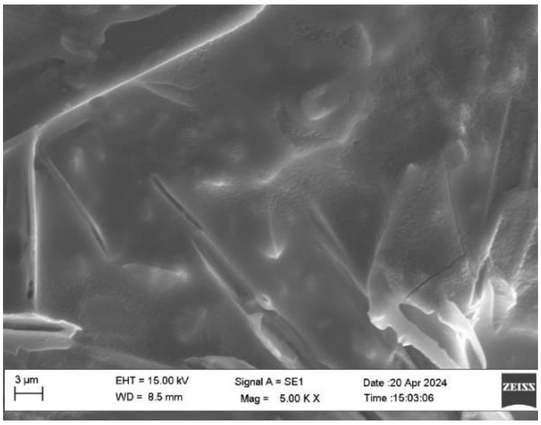


Fig. 4.5

Fig. 4.6

Figure 4. SEM images of RHA 20 mortar with 0% and 0.005% GO content Fig. 4.1, 4.3 and 4.5:0 %GO Fig. 4.2, 4.4 and 4.6:0.005% GO

There are many irregular cracks and wrinkled materials in the view of the sample without graphene oxide. Figures 4.1 and 4.2 show the size of cracks formed in the sample. The RHA 20 sample showed significantly larger crack sizes in its microstructure compared to the sample with GO. The cracks were observed more frequently in the case of RHA 20. From the deep SEM images of 3µm and 1 µm, fissured microstructure was observed in the RHA 20 sample. In comparison, a homogeneous microstructure was observed in the GO sample. It was observed from the SEM images that the GO 1 sample exhibited a denser microstructure compared to the RHA 20 sample. As shown, GO can move through the cracks within the hydration products. This implies that GO can inhibit crack development.

3.9 Cost Analysis

Cost analysis provides insights into the economic performance of selected design mixes. The cost of different materials required for casting 1m^3 of mortar is considered when performing the cost analysis. For materials mentioned in DSR, cost is computed using standard rates. The current market price is considered otherwise. The total cost is calculated by summing the individual cost of the items required for casting the mortar. Table 12 shows cost analysis results. Table 9 shows that the total cost for RHA 20 is less than RHA 0. Replacing 20% GGBS with RHA saved Rs. 1850 per m3. Adding Graphene oxide to the mix increased the cost drastically. The total cost of the GO 2 mix is thrice that of RHA 0. The total cost of casting 1 m3 of OPC cement mortar is Rs.6573/- from Delhi's Schedule of Rates. It costs Rs. 3124 per m3 less than the RHA 20 mix. Upon analysing from a sustainable point of view, the carbon emission is lower for RHA20. Moreover, RHA leaves a lower carbon footprint than cement, which is biodegradable and eco-friendly. Thus, the higher cost of RHA20 mix compared to ordinary Portland cement mix is balanced due to its higher environmental and sustainable benefits.

Table 9. Cost of Selected Mixes

Material	Unit	Rate (Rs.)	Amount Per Specimen (Rs. /m^3)			
			RHA 0	RHA 20	GO 1	GO 2
GGBFS	kg	5	2370	1896	1896	1896
RHA	kg	2.5	-	237	237	237
M-Sand	m^3	900	1117.6	1117.6	1117.6	1117.6
NaOH	kg	50	3950	3160	3160	3160
Na_2SiO_3	kg	52	4108	3286.4	3286.4	3286.4
GO	g	100	-	-	10000	20000
Total Amount (Rs. /m^3)			11545.6	9697	19697	29697

Graphene oxide incorporated mixes, especially GO 1 mix, show better mechanical properties over RHA20. GO 1 is not economically sustainable, but considering its environmental viability and reduced carbon taxes, it could be utilised for small-scale constructions.

4 Conclusions

The purpose of this study was to find optimum ratio of RHA to be incurred to get better mechanical characteristics and reduce shrinkage and water absorption of the selected mix using graphene oxide. RHA 0 mix (made with 100% GGBFS) demonstrates the highest compressive strength. Higher percentages of RHA (60% and 80%) showed fragile structure, and the RHA80 mix failed. Based on the results from the compression test, water absorption test and set time, the optimised mix ratio of RHA-based geopolymer obtained is RHA 20. Despite RHA being porous, increasing RHA content did not significantly affect water absorption. This may result from the polymerisation of the binder by the alkali activator. Therefore, reduction of water absorption through modified fly ash was not necessary. The addition of Graphene Oxide (GO) resulted in better shrinkage and compressive characteristics. The 7-day compressive strength of the GO mortars was comparable to the GGBFS mortar without RHA. The shrinkage value decreased by 22.5% and 36.25% for GO1 and GO2, respectively. However, thermal conductivity showed a slight increase when GO was incorporated. Considering the environmental sustainability and performance of GO-incorporated RHA20 mixes, they can be used for small-scale projects and specific applications where higher water absorption and shrinkage performance are desired.

5 Declaration of Conflict of Interest

The authors declare that there is no known conflict of interest that could have appeared to influence the work reported in this paper.

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