Effect of recycled fine powder as a calcium source on fresh and hardened properties of geopolymer mortar

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ABSTRACT: This study explores the efficacy of Recycled Fine Powder (RFP) derived from construction and demolition waste as a sustainable substitute for Ordinary Portland Cement (OPC) as a Calcium (Ca) source in geopolymer mortar. Fresh properties of Geopolymer Mortar (GPM) were evaluated in terms of slump-flow, initial-final setting time, whereas hardened properties were evaluated in terms of Compressive Strength (CS), Water Absorption (WA), porosity, acid attack, and dry shrinkage at both ambient and heat curing. Results indicate that the RFP performs well in all mixes. However, mix C10R10 shows the overall best results, as 17% higher compressive strength was observed as compared to mix C20R0, which further increases to 88% when compared to mix C0R0. Thus, RFP provides sufficient Ca content as an activator in GPM.

KEY WORDS: Fly ash; Recycled fine powder; Geopolymer mortar; Compressive strength; Water absorption; Acid attack; Dry shrinkage.

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RESUMEN: *Efecto del polvo fino reciclado como fuente de calcio sobre las propiedades frescas y endurecidas del mortero geopolimérico.* Este estudio explora la eficacia del polvo fino reciclado (RFP) derivado de residuos de construcción y demolición como sustituto sostenible del cemento Portland ordinario (OPC) como fuente de calcio (Ca) en morteros de geopolímero. Las propiedades frescas del Mortero Geopolímero (GPM) se evaluaron en términos de asentamiento, tiempo de fraguado inicial-final, mientras que las propiedades endurecidas se evaluaron en términos de resistencia a la compresión (CS), absorción de agua (WA), porosidad, ataque ácido y retracción por secado tanto a temperatura ambiente como con curado a elevada temperatura. Los resultados indican que el RFP funciona bien en todas las mezclas. Sin embargo, la mezcla C10R10 muestra los mejores resultados generales, ya que se observó una resistencia a la compresión un 17% mayor en comparación con la mezcla C20R0, que aumenta hasta un 88% en comparación con la mezcla C0R0. Por tanto, RFP proporciona suficiente contenido de Ca como activador en GPM.

PALABRAS CLAVE: Cenizas volantes; Polvo fino reciclado; Mortero de geopolímero; Resistencia a la compresión; Absorción de agua; Ataque ácido; Retracción por secado.

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1. INTRODUCTION

In today's world, concrete is the next most widely used material after water (1). The increasing demand for concrete is owed to infrastructure development and urbanization around the globe. The high consumption of concrete tends to increase the demand for Ordinary Portland Cement (OPC) as it is utilized as a binder in concrete production. The manufacturing of OPC uses a significant amount of natural resources, as well as emits a huge quantity of carbon dioxide (CO_2) into the atmosphere (2). It is impossible to overstate the increasing effects of the production of OPC on the environment. To address this problem, a geopolymer binder is recommended as an alternate to OPC for use in the concrete industry. In 1978, Geopolymer was first introduced by Prof. Davidovits as an alternative to the conventional binder (3). Geopolymer is a three-dimensional aluminosilicate binder that can reduce CO₂ emissions by 22 % to 72 % and it costs nearly the same as OPC concrete (4). The geopolymer is prepared by a reaction mechanism of a combination of aluminosilicate-rich source materials with a high alkali activator solution to form a 3D structure of tetrahedral bonds of silicate and aluminates (5). Aluminosilicate-rich materials such as Fly Ash (FA), Rice Husk Ash (RHA), Ground Granulated Blast Furnace Slag (GGBS), and Metakaolin (MK) can be used as geopolymer binder (6, 7). The sodium/potassium hydroxide and sodium/potassium silicate are commonly used alkali activator solutions with geopolymer binder (8). The use of FA as an aluminosilicate source material in the geopolymer was suggested by several studies due to the high concentration of silica (Si) and alumina (Al) concentrations, its high abundance, and the ready availability of this material (9-12). Fly ash is a by-product of the burning of coal in thermal power plants (13). Additionally, using FA in the building process lessens the load on landfills, which lowers land contamination. (14). In the geopolymerisation process, the utilization of FA has issues with delayed setting time and low gain in strength at an early age due to their pozzolanic nature. The pozzolanic material (FA) has high silica and alumina content with low cementitious properties which slow down the chemical reaction delays the early setting time and gains in strength. To get around that problem, either heat curing or the use of a high-calcium (Ca) mineral is necessary to shorten the first setting time and speed up the geopolymerization process to obtain early strength. (15, 16). It was observed that the Ca compounds play a vital role in accelerating the chemical reaction and improving the mechanical properties of the Geopolymer Mortar (GPM) (17). The existence of OPC in the geopolymer matrix forms the additional Ca hydrates gel which as a result enhances the Compressive Strength (CS). The presence of dicalcium and tricalcium silicate in OPC

helps to form Ca hydrates which help to improve the strength, and durability performance of geopolymers (18). Shinde, et al. (19) claimed that low calcium FA-based GPM and observed that with the inclusion of a small amount of OPC content, the CS of mortar rises significantly. Lodeiro et al. (20, 21) reported that the addition of Ca compounds in alkali-activated and silica aluminate-rich materials formed the C-A-S-H/N-A-S-H gel which helps to improve the properties. Similarly, Mehta and Siddique (22) also conclude that the substitution of 20% OPC in the place of FA decreases the permeation qualities such as sorptivity, porosity, and Water Absorption (WA). The results were further validated by microstructure analysis, which revealed a considerable enhancement in microstructure with the addition of OPC. However, OPC contains a good amount of Ca content which can overcome the limitations of activation of geopolymer reaction at ambient conditions but on the other hand utilization of OPC can give rise to CO₂ emissions into the environment. Therefore, it is necessary to alter the utilization of OPC with a geopolymer system by considering the other Ca-rich minerals which can reduce the setting time of the FA-based geopolymer system and enhance the geopolymer reactions at early stages of curing.

In addition to this, to maintain sustainability in the construction industry the utilization of Recycled Concrete (RC) is an important factor to concern. The utilization of this RC is also beneficial to reducing CO₂ emissions, which conserves the environment from global warming problems. The RC consists of different particles of coarse and fine recycled aggregates. Fine particles (<4.75 mm) make for about half of the recycled aggregates produced from RC (23, 24). The hydrated cement paste, ettringite, and unhydrated cement are present in the adherent mortar of the fine particles of RC. (25). The un-hydrated cement (25 to 30%) was present in the adhered mortar of fine particles of RC (26). The anhydrous phase of adhered mortar plays an important function in the hydration of cement in concrete and leads to the formation of hydration products by producing more nucleation gel (27). Moreover, according to certain research, if RC fine particles are further milled to increase their fineness and converted into a powder which is termed Recycled Fine Powder (RFP) which can be used as an activator with the primary binder to catalyze the geopolymer reaction content for mortar and concrete production (28, 29). Bordy et al. (27) have conducted an experimental investigation on mortars made with OPC replaced by an RFP and the result shows that the substitution of RFP showed good mechanical properties and resistance against carbonation. The mineral composition of the RFP shows quartz, mullite, calcite ettringite, with un-hydrated cement particles of OPC. Ren et al. (29) demonstrated that the use of RFP in the alkali-activated slag system and it has been found that the substitution of RFP in an alkali-activated slag system enhances the mechanical properties and decreases the WAand porosity value which shows RFP has good reactivity and filling effect.

The literature studies revealed that extensive work has been done on the utilization of OPC as a rich mineral in FA-based GPM for activation at an early stage of curing to improve the strength and durability properties. However, limited information is available on the usage of RFP in FA-based geopolymer mortar. The current study focused on the utilization of RFP in place of OPC as a calcium source in the FA-based geopolymer to achieve sustainability in the construction sector. In this study, OPC was replaced with FA up to 20% in FA-based geopolymer mortar, and further, RFP was substituted with OPC at replacement levels of 5%, 10%, 15%, and 20%. The fresh and hardened properties of GPM were examined for both ambient and heat-curing conditions.

2. EXPERIMENTAL PROGRAM

2.1. Material

2.1.1. Binders

The FA (Class-F) was used as a main binder in the experimental work confirming IS3812:2003 (30). The OPC was firstly partially substituted with FA as a Ca source to enhance the properties of GPM at an early stage of curing. Secondly, RFP was blended with ternary mixtures of FA and OPC at different replacement levels. The RFP was prepared using Recycle Fine Aggregates (RFA) collected from the Concrete Laboratory of the Author's Institute. It was prepared by milling RFA particles up to 75 µ size using a ball mill apparatus. The particle distribution of FA, OPC, and RFP was determined using the Laser diffraction technique as shown in Figure 1. The physical and chemical compositions of FA, OPC, and RFP are listed in Table 1. Natural Fine Aggregate (NFA) was collected locally with a specific gravity of 2.53, a fineness modulus of 2.34, and a WA rate of 1.5%.



FIGURE 1. Particle size distribution of FA, OPC, and RFP.

 TABLE 1. The physical and chemical compositions of FA, OPC, and RFP.

Duon outing	EA	OBC	DED
Properties	ГА	OPC	KFP
Fineness, cm ² /gm	4125	-	3500
Specific Gravity	2.1	3.15	2.5
SiO ₂	56.50%	20.10%	60.0%
Al_2O_3	22.50%	6.80%	11.0%
Fe ₂ O ₃	11.0%	4.30%	4.0%
CaO	3.20%	61.30%	15.0%
MgO	1.25%	3.50%	2.5%
Na ₂ O	0.15%	0.60%	1.85%
K ₂ O	2.2%	1.25%	2.50%
SO ₃	0.25%	1.28%	1.25%
P ₂ O ₅	0.60%	0.08%	0.15%
TiO ₂	2.2%	0.55%	0.65%
Loss of ignition	1.20%	1.20%	1.80%

Characterization of FA, OPC, and RFP

For FA, OPC, and RFP, X-Ray Diffraction (XRD) patterns were observed from Malvern Panalytical's XRD equipment are shown in Figure 2 (a). The figure illustrates that the FA contains two main substances quartz and mullite. The peak of the quartz is coming on 25-30 (2 φ). The RFP contains a higher content of quartz with few peaks of calcite in the form of both hydrated and anhydrous Ca content. According to Table 1, both FA and RFP have a large quantity of silica, about 56.50% and 58% respectively. On the other hand, OPC contains a peak in the range of 25-30 (2 φ) and contains a high proportion of allite, bellite, and calcite which shows a high amount of Ca compound in comparison to FA and RFP.

Furthermore, the Fourier-transform infrared (FTIR) spectroscopy was done using an ATIMATTSON FT-IR-TM series spectrophotometer. In FTIR analysis, infrared radiation is transmitted through a sample, with some of the radiation being absorbed, and transmitted radiation is then recorded at various wavenumbers (cm⁻¹). For FA, OPC, and RFP, the FTIR spectra typically span the range of 400 - 3500 cm⁻¹. Figure 2 (b) shows the existence of Si and Al structure bonds of FA, OPC, and RFP, and the alumino-silicate functional group was presented in the range of 800 and 1100 cm⁻¹ wavenumber. The asymmetric T-O-Si / Si-O-T stretching (T showing either Si or Al) of FA, OPC, and RFP were shown at 1045.57, 882.06, and 1038.88 cm⁻¹ wavenumbers, respectively. Additionally, the peaks observed within the spectral range spanning from 676.80 to 780.11 cm⁻¹ represent the bending vibrations associated with Si-O-T bonds.



FIGURE 2. (a) XRD Analysis of FA, OPC, and RFP used in the experimental work. (b) FTIR Analysis of FA, OPC, and RFP used in the experimental work.

Lime reactivity

Fly Ash is a pozzolanic material derived from industrial waste, rich in aluminosilicates, while RFP is attained from construction and demolition waste. The reactivity test for pozzolanic materials is conducted according to IS 1727:1967 (31) standards. The CS reported for mortar cubes made with FA and RFP was 3.84 MPa and 4.5 MPa, respectively which shows the reactivity of their components.

2.1.2 Alkali activator

The alkali activators used in the experimental work in the form of Sodium Hydroxide (SH) and Sodium Silicate (SS) in combination. The 12M solution of SH was prepared using 98% pure pellets in the experimental work. The pellets were mixed in water to make an SH solution prior to 24 h of casting. Further commercially available solution with a weight ratio of Si/Na of 3.02 was used with chemical compositions of 27.2 % SiO₂,9% Na₂O, and 63.8% H₂O. The properties of SH and SS are shown in Table 2.

TABLE 2. Properties of alkali activators.

Property	NH	NS
Molecular formula	NaOH	Na ₂ SiO ₃
pH value	13-14	13-14
Na ₂ O content (wt %)	-	9
SiO ₂ content (wt %)	-	27.2
H ₂ O content (wt %)	-	63.8

2.2. Mixture design and curing condition

The mixed proportion of GPM is presented in Table 3. The alkali/binder ratio (0.45), SS/SH ratio (2), SH molarity (12M), and water/solid ratio (0.35) were fixed based on a trial-and-error approach. The FA was used as a key binder whereas, OPC and RFP were substituted with FA at different percentages. Firstly, 20% FA was replaced with OPC in the second mixture, after that OPC was replaced with RFP for other mixtures at 5%,10%,15%, and 20% replacement levels while maintaining the FA at 80% in all the GPM mixtures.

For the preparation of geopolymer mortar, NFA and blended binder were mixed dry for 3 minutes in a pan mixer. After that alkali solutions were added to the dry mixture and mixed for more than 3 to 5 minutes. Further, a fixed water content of 57.77 kg/m³ for all mixtures was added to obtain the necessary workability of the geopolymer mixture. After that, the freshly mixed mortar was dispensed into the mould

Mixture No- tation	Mixture description	Mixture Ingredients					
		FA	OPC	RFP	NFA	SS	SH
C0R0	100% FA+ 0% OPC + 0% RFP	500	-	-	1500	150.4	75.22
C20R0	80% FA + 20% OPC + 0% RFP	400	151.42	-	1500	150.4	75.22
C15R5	80% FA +15% OPC + 5% RFP	400	113.59	29.65	1500	150.4	75.22
C10R10	80% FA + 10% OPC +10% RFP	400	69.48	54.54	1500	150.4	75.22
C5R15	80% FA + 5% OPC + 15% RFP	400	37.72	89.06	1500	150.4	75.22
C0R20	80% FA+ 0% OPC + 20% RFP	400	-	118.93	1500	150.4	75.22

TABLE 3. Mix proportion of GPM mortar containing varying replacement of OPC and RFP in kg/m³.

*NFA – Natural Fine Aggregates

and sealed to resist moisture loss. Further, for ambient curing, the GPM samples were placed at room temperature, and heat curing was done at 60°C in the oven for 24 hours.

3. EXPERIMENTAL WORK

3.1. Slump-flow and setting time

The slump-flow and setting time of GPM were observed in their fresh state. The slump-flow was performed by IS 4031- Part 7 (32). The slump should be 110 ± 5 percent as per standards. Further, the initial-final setting time of GPM was examined in accordance with IS: 4031, Part 5 [30].

3.2. Compressive strength test

the 70.6 mm cube specimens were used to examine CS in accordance with IS 4031, Part 7 (32) at, 7, and 28 days of of both ambient and heat curing. The 2000Kn load capacity compression testing machine was used and an average of three specimens were taken to record the CS of GPM for each curing.

3.3. WA and porosity test

the 70.6 mm cube specimens were used to evaluate the WA and porosity of GPM mixtures using ASTM C642 -13 (33) at 28 days of each ambient and heat

curing. The WAand porosity in terms of the percentage of each mixture.

3.4. Dry shrinkage test

The dry shrinkage was observed in accordance with IS 4031: Part 10 (34) using 25 mm x 25 mm x 285 mm prisms. Measurements were obtained at 7, 14, 28, and 56-day intervals throughout two months.

3.5. Acid attack

For conducting the chemical attack test 5% sulphuric acid (H_2SO_4) was used for the current experimental work. The acid resistance was performed as per ASTM C-267 (35). The mortar cubes were first weighed before being placed into an acidic solution in a container. After being placed at 28 days in the acid water, the cubes were removed, cleaned, weighed, and dried for another 24 hours at 100 ± 10 °C. The specimens were then taken out of the oven, weighed, brushed, and re-weighed to determine the mass loss due to acid degradation. In a 28-day soaking period, the acid water was replaced after a 7-day interval.

3.6. Microstructure analysis

The microstructure of GPM samples was examined using SIGMA 500VP Field Emission Scanning Electron Microscopy (SEM). The fine powder obtained by grinding the 28-day geopolymer paste sample to decrease its particle size was subjected to SEM examination with a 1000X scale at 10 and 15 kV.

4. RESULTS AND DISCUSSION

4.1. Fresh properties of GPM

4.1.1. Slump-flow value

The slump-flow results of all GPM mixtures are shown in Figure 3. The attained results show that the addition of OPC and RFP makes the mixture stiffer than the COR0. For mixture C20R0 with 20% OPC, the slump-flow decreases marginally in contrast to the control mixture(C0R0). Further, 5% substitution of RFP with OPC i.e., C15R5 mixture, the slump value declined by 5% relative to the COR0. Similarly for mixture C10R10, the slump value was reduced by 7% comparative to the COR0. Furthermore, for mixtures C5R15 and C0R20, the slump was reduced by less than 10% relative to the COROL Importantly, substituting RFP up to 20% had a limited influence on the flow properties related to the COR0. The reduction in flow or stiffness was attributed to the irregular shape of RFP particles being replaced by the rounded spherical shape of FA particles. Furthermore, for all geopolymer mixes even after a reduction in the slumpflow values all GPM is in satisfactory limits as per IS 4031-Part 7 standard.



FIGURE 3. Slump flow value of geopolymer mortar mixtures incorporating OPC and RFP at different replacement levels.

4.1.2. Setting time

In practical application, the setting times of GPM are significant because they determine the time available for transportation, placement, and compaction. The setting times of all GPMs are presented in Figure 4. The initial-final setting times of the geopolymer paste of the COR0 are 454 and 1400 minutes respectively at room temperature. The geopolymer paste initial-final time was considerably reduced with the substitution of OPC and RFP in the GPM mixtures. For mixture C20R0 i.e., with the substitution

of 20% OPC, the initial setting time decreased significantly to 50 minutes from 454 minutes and the final setting time decreased to 130 minutes from 1400 minutes. Further, for mixture C15R5, i.e., a blend of 15% OPC and 5% RFP, the initial-final setting time also decreases as compared to the COR0. Nath and Sarker (18) also observed that the presence of OPC in FA-based GPM reduces the initial-final setting time significantly. However, in contrast to C20R0, the initial-final setting time increased by 10 and 20 minutes respectively. Similarly, for mixtures C10R10, C5R15, C15R5, and C0R20 the initial-final significantly decreased as related to the COR0 but increased with the rise in the content of RFP. However, an increase in RFP content does not affect both setting times significantly, and still, the initial-final setting time is restricted to 217 and 420 min respectively which is significantly lesser than the COR0 setting time values. The presence of Ca in OPC and RFP plays a key role in accelerating the setting process of geopolymer mortar. The rate at which geopolymerization reactions increase prominently influences the setting time of a geopolymer mixture. Moreover, OPC releases more heat during the hydration process in comparison to RFP due to the higher content of Ca, which as a result accelerates the geopolymerization reactions, leading to faster setting times as compared to RFP. The presence of appropriate Ca content in both OPC and RFP resulted in a considerable drop in the setting times of geopolymer paste. The inclusive results of GPM show that the inclusion of OPC and RFP individually or in combination up to 20% helps to reduce the setting time influentially.



FIGURE 4. Initial and final Setting time of geopolymer mortar mixtures incorporating OPC and RFP at different replacement levels.

4.2. Hardened properties of geopolymer mortar

4.2.1. Compressive Strength

(a) Effect of inclusion of OPC and RFP on the compressive strength of GPM at ambient curing

The CS results of GPM mixtures at different stages of ambient curing are given in Figure 5. The results of GPM mixtures illustrate that the inclusion of both OPC and RFP enhanced the CS. For example, with a 20% substitution of OPC i.e., for the C20R0 mixture, the CS enhanced significantly by 987% and 322% at 3 and 7 days of curing relative to the COR0. Similarly, the CS increased by 57% at 28 days of curing comparative to the COR0. Mehta and Siddique (22) also revealed that the addition of OPC in geopolymers increases the strength significantly due to the development of extra nucleation sites in the form of Ca hydrates along with the gepolymerization of FA and activators. The GPM with 0% OPC i.e. in mixture COR0 only geopolymer matrix is generated, Figure 6 (a), whereas the substitution of OPC in GPM produces supplementary C-A-S-H gel generated with geopolymer matrix as shown in the SEM image in Figure 6 (b). In a previous study, the microstructural analysis also claimed that the presence of OPC in geopolymer forms the C-A-S-H gel in the concrete matrix (36). Further, with the substitution of 5% RFP with OPC i.e. (15% OPC + 5% RFP) C15R5, the strength enhanced by 1190% at 3 days, 382% at 7 days, and 76% at 28 days of ambient curing respectively in contrast to the COR0. In the mixture with 10% OPC and 10% RFP i.e., C10R10, the CS was enhanced by 1383% and 531% at 3 and 7 days of curing respectively, and 88% at 28 days of curing relative to the COR0. Moreover, the CS of C15R5 and C10R10 is higher than the C20R0 mixture, which may be due to the high content of Ca in OPC with a low content of Si and Al in comparison to FA and RFP. The decrease in Si and Al content hinders the geopolymer reaction which tends to reduce the CS. Conversely, crystalline silica present in RFP works as a filler in the concrete matrix which helps to enhance the CS of GPM.



FIGURE 5. Compressive strength result of geopolymer mortar mixtures at 3,7 and 28 days of ambient curing.

In the substitution of RFP with OPC in the GPM mixture, the strength starts decreasing but is still higher than the COR0. For mixtures C5R15 and COR20, the CS increased in comparison to the COR0 but decreased relative to C10R10. However, the decreasing trend of CS with an increase in the content of RFP is due to a lower content of Ca in RFP than in OPC. Therefore, the increasing trend observed in the mixture C20R0 is going to reverse in the mixture C5R15. For mixture C5R15, the CS escalates by 670% at 3 days of curing 197%, and 33% at 7 and 28 days of

curing respectively relative to the C0R0. Similarly, with 20% RFP and 0%, OPC i.e., for the C0R20 mixture, the CS shows an increasing trend concerning the C0R0. The overall observed values of CS show an increasing trend with the inclusion of both OPC and RFP for all geopolymer mixtures and C10R10 shows maximum CS as related to other mixtures. Figure 6 (c), shows the formation of C-A-S-H gel and needle shape particles in the form of N-A-S-H gel along with geopolymer matrix which enhances the CS. Ahmari et al. (28) also show the formation of needle-shaped particles along with a geopolymer matrix with the presence of RFP. Furthermore, the rise in CS up to 7 days is noticeable as compared to 28 days at ambient curing conditions (28).



FIGURE 6. SEM images of geopolymer mortar mixture (a) C0R0 (b) C20R0 and (c) C10R10 at ambient curing.

(b) Effect of inclusion of OPC and RFP on the compressive strength of GPM at heat curing

The CS results of all mixtures for heat curing are shown in Figure 7. A considerable change was found in the strength result of the geopolymer mixtures with the substitution of both OPC and RFP. For example, 20% replacement of OPC with FA in the mixture C20R0 shows a significant increase of 288% at 3 days and 154% at 7 days in CS and 48% at 28 days of curing in comparison to the COR0. This enhancement in the CS was observed due to the provided heat, good reactivity of OPC, and additional Ca silicate gel produced with the geopolymer matrix in comparison to the CORO as shown in Figures 8 (a) and (b). Furthermore, the addition of RFP content with OPC in the mixture C15R5 i.e. (15% OPC + 5%)RFP), C10R10 i.e. (10% OPC + 10% RFP), and C5R15 i.e. (5% OPC + 15% OPC) show a substantial increase in the CS results at all days of curing relative to the C0R0. For example, in mixture C15R5, the increase in CS was observed to be 321%, 170%, and 55%, respectively, on the 3, 7, and 28 days of curing. However, the maximum increase in CS was observed for mixture C10R10 (10% OPC + 10% RFP) i.e., 347%, 170%, and 66% for the same curing ages respectively. Moreover, the increment in the CS in the mixture C10R10 was clearly understood from the SEM image Figure 8 (c) which shows the proper reaction of FA particles with supplementary N-A-S-H and C-A-S-H gel formation. However, the mixture C5R15 and C0R20 having 15% and 20% RFP was substituted with FA and also showed an increase in CS as compared to the C0R0 but showed lower strength than C10R10 as discussed in the above section at ambient curing condition. From the overall result, it was concluded that the inclusion of 20% RFP alone in the FA-based GPM can achieve good CS at an initial stage of heat curing conditions.



FIGURE 7. Compressive strength result of geopolymer mortar mixtures at 3,7 and 28 days of heat curing.

4.2.2. WA and Porosity

(a) Effect of inclusion of OPC and RFP on WA and porosity at ambient curing

The observed values of WA of all mixtures with varying replacement of OPC and RFP at 28 days of

ambient curing are shown in Figure 9. Results obtained illustrate that the substitution of both OPC and RFP content in the mixtures reduces the WA value as related to the C0R0. For example, mixture C20R0 having 20% substitution of OPC in the mixture reduces the WA by 21% relative to the C0R0. The decline in the WA values is due to the densification of the microstructure of the geopolymer mixture with the inclusion of OPC (22). Pangdaeng et al. (36) claimed that the substitution of OPC filled the pores in the concrete matrix which tends to decrease the WA of high Ca FA geopolymers. Moreover, in mixture C15R5 i.e. (15% OPC + 5% RFP) was observed that with the substitution of RFP, a decrease of 27% of WA value was observed in comparison to the C0R0. The maxi-



FIGURE 8. SEM images of geopolymer mortar mixture (a) C0R0 (b) C20R0 and (c) C10R10 at heat curing.

mum reduction of WA was found i.e., 31% in mixture C10R10 having 10% RFP and 10% OPC substituted with the FA in the mixture. However, WA values of mixtures C5R15 and C0R20 also show a 16% and 9% reduction respectively as related to the C0R0. Sharma et al. (37) claimed that the presence of RFP in FA-based GPM reduces their WA due to the formation of additional Ca hydrates along with the geopolymer matrix. The overall results show that substitution of both OPC and RFP up to 20% individually or in combination declines the WA values as compared to the C0R0 and mixture C10R10 shows maximum reduction in WA as compared to other GPM mixtures.

Figure 9 also shows the porosity result of GPM mixtures cured at ambient curing. The result concludes that the substitution of OPC and RFP in the blended mixture reduces the porosity value. For example, the mixture C20R0 having 20% OPC substitution with the FA shows a 13% decrease in the porosity value as compared to the COR0. It results from the pore refinement of the voids in geopolymer mixtures when OPC is added. The previous study also shows the same trend of a reduction in porosity with the addition of OPC in GPM (38). However, in the mixture, C15R5, i.e. (5% RFP + 15% OPC) the reduction in the porosity value was observed as 16% as compared to the COR0. For mixture C10R10 i.e. (OPC 10% + RFP 10%) shows the maximum decrease in porosity value of 20% with respect to the C0R0. Ren et al. (39) claimed that partial substitution of RFP with FA decreases the porosity values due to pore refinement of alkali-activated concrete blocks. A significant decrease in the porosity value was observed due to the enhancement in the microstructure and void filling of the geopolymer mixtures. However, further increasing the content of RFP in mixture C5R15 i.e. (5% OPC + 15% RFP) and C0R20 (0% OPC + 20% RFP) there is a slight increase in the porosity value at 28 days relative to C10R10 but related to the C0R0 it is very low. It shows that the substitution of OPC with

RFP may increase the porosity due to the lower reactivity of RFP as compared to OPC.

(b) Effect of inclusion of OPC and RFP on WA and porosity at heat curing

The WA results of GPM mixtures at heat curing are given in Figure 10. It was observed that the WA value of heat-curing GPM mixtures is less as compared to ambient cured mixtures. The result depicts that with 20% OPC in the mixture C20R0 i.e. (20% OPC + 0%)RFP), the WA value was reduced by 5.63% relative to the COR0. It illustrates that the presence of OPC in the GPM mixture improves their microstructure, Figure 8 (b). However, the accumulation of RFP with OPC in the mixture C15R5 showed a reduction of 22% in WA value as compared to the C0R0. The maximum reduction in the heat curing was observed in the mixture C10R10 i.e. (10% OPC + 10% RFP) which is 29 % as related to the COR0. However, mixture C5R15 and C0R20 having 15% and 20% RFP with FA shows an insignificant increase in the WA value in comparison to C10R10 but as compared to the C0R0 the WA value decreased by 25% and 5% respectively.

Similarly, the porosity value of heat-curing mixtures is shown in Figure 10. The result concludes that the substitution of OPC and RFP helps to decrease the porosity value. For example, the mixture C20R0 shows a 7% decrement in porosity value as related to the C0R0. However, the substitution of OPC and RFP in the GPM mixture shows a significant decrement in the porosity value up to a 10% replacement level. For example, the mixture C15R5 and C10R10 show a 12% and 18 % decline in the porosity value as related to the C0R0. Furthermore, replacing RFP as a place for OPC shows the same trend as ambient curing conditions. For example, in mixtures C5R15 and C0R20, the decrement in the porosity value was 10% and 7 % as compared to the C0R0.



FIGURE 9. Water absorption and porosity values of geopolymer mortar at 28 days of ambient curing.



FIGURE 10. Water absorption and porosity values of geopolymer mortar at 28 days of heat curing.

4.2.3. Correlation between compressive strength, water absorption, and porosity

The relationship between CS with WA and porosity at ambient cured mixtures is shown in Figure 11 (a). For mixture, C10R10, the highest CS of the GPM mixture was found to be 28.4 MPa after ambient curing. The result of CS shows similar trends like WA and porosity for all the GPM mixtures. The GPM mixture having the lowest WA and porosity value shows the highest CS i.e., C10R10. In other words, CS shows an inverse relation with WA and porosity for all mortar mixtures in both curing conditions. Similarly, the relations between water absorption, porosity, and CS at heat-cured GPM mixtures are shown in Figure 11 (b). The heat-cured result shows similar trends as the ambient-cured result in terms of WA and porosity. It was observed that the maximum CS of 32.8 MPa was found for mixture C10R10 with the lowest WA and porosity value.

Figure 12 shows the linear relation between porosity and WA for GPM mixtures at 28 days of both curing. The correlation analysis after correlating the WA with porosity for ambient curing conditions is expressed by the quadratic equation shown in Figure 12 (a) with $R^2 = 0.99$. It shows that WA and porosity change with the presence of OPC and RFP in the same trend. The overall results show that the WA and porosity decrease with the inclusion of 10% OPC and RFP in combination and can further rise with the higher content of RFP. Similarly, the correlation analysis after correlating the WA with porosity for heat curing condition is expressed quadratic equation shown in Figure 12 (b) with $R^2 = 0.94$ with the same trend of ambient curing condition.



FIGURE 11. Compressive strength of mixtures vs water absorption and porosity (a) ambient curing (b) heat curing.



FIGURE 12. Relationship between water absorption and porosity of geopolymer mortars at the age of 28 days of (a) ambient curing (b) heat curing.

4.2.4. Acid attack

Figure 13 shows the change in the mass loss of geopolymer mixtures cured in both curing (ambient and heat) at 28 days of the soaking period. The outcome reveals that the mass loss of GPM mixtures was marginally affected at 28 days of soaking. For example, the COR0 was shown a slight decrement in mass loss i.e., 0.56% at 28 days of sulphuric acid exposure. The calcium sulfates and calcium aluminum sulfates generally cause the deterioration which is generated through a sulphuric acid reaction with Ca present in the binder. However, FA (Class-F) used in the present study has low Ca content which restricts the production of the sulfate products, therefore it shows a negligible effect on FA-based geopolymer mortar. Furthermore, mixture C20R0 shows a maximum decrement in mass loss was 1.61% in ambient curing conditions. Mass loss due to the presence of OPC was mostly driven by the interaction of the Ca hydroxide with the acid. However, the result demonstrates that the inclusion of RFP in the mixture as a replacement for OPC reduces the mass loss percentage. For example, the mass loss of mixture C15R5, C10R10,

C5R15, and C0R20 was observed at 1.61 %, 1.45 %, 1.07 %, and 0.80 % respectively at ambient curing. The depolymerization of the aluminosilicate network of the geopolymer matrix caused by acid assault occurs in the exchange of alkali cations by $H^{3}O^{+}$ or H^{+} ions of acid, trailed by reduction of siliceous phases. This condition, in turn, causes a considerable loss of mass during an acid attack (40).



FIGURE 13. Mass loss due to acid attack on geopolymer mortar mixes at 28 days of soaking period.

Similarly, in the heat curing the mass loss of the geopolymer mixture was higher than the ambient cured mixture. For example, the COR0 shows higher resistance in ambient curing conditions towards sulfuric acid relatively heat-cured mixture. In the heat curing the mass loss of COR0s was 0.79% at 28 days of curing. Moreover, the observed mass loss of heat-cured mixtures was at 1.57%, 1.37%, 1.26%, and 1.57% for C15R5, C10R10, C5R15, and C0R20 respectively. The overall results show that the GPM mixtures show good resistance to acid attack at 28 days of the soaking period.

4.2.5. Dry shrinkage

(a) Effect of inclusion of OPC and RFP on the drying shrinkage values at ambient curing

The observed values of the dry shrinkage test of GPM mixtures cured at ambient temperature are shown in Figure 14. The decrease in dry shrinkage was observed by substituting FA with OPC at 20%, C20R0. The inclusion of OPC fastens the reaction and densifies the microstructure which as a result reduces the evaporation of inner water and aids in lessening the shrinkage. In a previous study, it was stated that the addition of Ca products in GPM reduces dry shrinkage (41). For example, in mixture C20R0 the reduction in drying shrinkage at 7 days was 15.29 %, for 14 days was 6.34 %, and for 28 and 56 days the dry shrinkage reduction was 9.15 and 8.08 % respectively. Similarly, mixture C15R5 having 5% RFP is replaced by OPC the drying shrinkage value is decreased by 12.65% for 7 and 6.06% for 14 days. Further, the decrease in drying shrinkage values was 8.37 % at 28 and 7.36 % at 56 days relative to the COR0. The RFP in the blended GPM mixture shows an insignificant decrement in the shrinkage value. For mixture C10R10 having 10%, RFP content is replaced with OPC the reduction in drying shrinkage for 7 and 14 days was 9.79 % and 4.96 %. At 28 and 56 days of curing, the decrease in the drying shrinkage values was near about 6% each. However, in mixture C5R15 the drying shrinkage reduction was observed at 7.37 %, 4.55 %, 4.57 %, and 5.05 for 7, 14, 28, and 56 days respectively related to the COR0. For mixture C0R20 having 20 % RFP was replaced by fly ash the drying shrinkage value shows a 6.71 % decrement at 7 days. Furthermore, for 14, and 28, 56 days, it shows near about 4 % decrement as related to the COR0. The substitution of RFP helps in pore refinement which in effect reduces the inner water loss as a result reduces the dry shrinkage (42). The overall results show that the substitution of both OPC and RFP shows a decline in drying shrinkage at all ages of ambient curing. Further, for each GPM mixture the shrinkage values increased linearly up to 28 days, and beyond 28 days, no significant variation in dry shrinkage values was observed as shown in Figure 14.



- C0R0 - C20R0 → C15R5 - C10R10 - C5R10 - C0R20

FIGURE 14. Dry Shrinkage value of geopolymer mortar mixes as a function of time at ambient curing.

(b) Effect of inclusion of OPC and RFP replacement on the drying shrinkage value at heat curing

Similarly, the drying shrinkage result of all mixtures at heat curing is shown in Figure 15. The mixture C20R0 having 20% FA was replaced by OPC the decrease in the drying shrinkage value was 11% each at 7 and 14 days each as compared to the C0R0. Further, the drying shrinkage values decreased was 11.37% and 11.61% at 28 and 56 days respectively. Likewise, for the mixture, C15R5 having 5%, RFP was replaced by OPC the reduction in drying shrinkage was observed as near about 11% at all days of curing. For mixture C10R10 having 10% RFP with 10% OPC, the drying shrinkage value was observed as a 9% decrement at 7 and 14 days and 10% at 28 and 10.53 % at 56 days as related to the C0R0. Further mixtures C5R15 and C0R20 show similar trends of dry shrinkage values as of ambient cured mixtures. Further, the dry shrinkage value increased linearly up to 7 days of curing, and after that, there was no significant change in the shrinkage value up to 56 days of curing for each GPM mixture at heat curing conditions.



FIGURE 15. Dry Shrinkage value of geopolymer mortar mixes as a function of time at heat curing.

6. CONCLUSIONS

The overall results show that RFP can be the finest replacement for OPC to enhance the geopolymer reaction in FA-based geopolymer mortars. The substitution of RFP up to 20% alone in GPM can reduce the setting time and enhance their properties.

- 1. The presence of OPC and RFP in the GPM mixtures marginally reduces the slump-flow value relative to the control geopolymer mixture however in acceptable limits with the addition of OPC and RFP for all mixtures as per requirement. Additionally, the initial-final setting time was reduced substantially with the inclusion of OPC and RFP in GPM mixtures.
- 2. Across each of the curing ages, the highest CS was achieved for the GPM mixture having 10% RFP and 10% OPC content in both curing conditions. Further, the utilization of 20% RFP alone also shows promising CS in comparison to the COR0 which can overcome the consumption of OPC in geopolymer mortar. Furthermore, both OPC and RFP are beneficial to improve the CS at an early stage of ambient curing.
- 3. It was concluded that WA as well as porosity values decrease with the substitution of OPC and RFP individually or in combination in all GPMmixtures. The GPM mixture C10R10 shows the lowest WA and porosity value due to its denser microstructure in comparison to other GPM mixtures.
- 4. In comparison to ambient curing conditions, there is lesser dry shrinkage of GPM under heat curing conditions. Further, the dry shrinkage value as related to the COR0 reduced with the inclusion of OPC and RFP in the GPM mixtures.

- 5. The GPM mixtures show good acid resistance in both curing conditions. The maximum percentage decrease in mass of GPM is 1.6% with 20% utilization of OPC in the GPM mixture.
- 6. It was concluded from SEM analysis with the inclusion of the OPC and RFP in the mixture the microstructure of the GPM mixtures is improved in comparison to the COR0. Moreover, the supplementary N-A-S-H or C-A-S-H gel is produced with the inclusion of OPC and RFP along with the geopolymer matrix at 28 days of curing.

Data availability

The authors confirm that the data related to the study is available within the article.

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Authorship contribution statement

Abhishek Sharma: Conceptualization, Data cleansing, Fund raising, Research, Methodology, Software, Writing - original draft.

Paramveer Singh: Conceptualization, Research, Methodology, Visualization, Writing - review & editing.

Kanish Kapoor: Conceptualization, Formal analysis, Project administration, Resources, Supervision, Validation, Visualization.

Declaration of competing interest

The authors of this article declare that they have no financial, professional or personal conflicts of in-terest that could have inappropriately influenced this work.

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