Optimization of Phosphoric Acid Geopolymer Mortar Mixes for Strength and Durability using Industrial Waste Materials

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Abstract. This study investigates the development of sustainable geopolymer mortar using fly ash, metakaolin, and granite dust as primary binders, activated by Phosphoric acid H3Po4. Mortar samples were prepared with varying molarities (6M, 8M, 10M, 12M, 14M, and 16M) and cast into cubes and cylinders using individual and combined material compositions. The specimens were cured at 60°C for durations of 1 and 3 days, and their compressive and tensile strengths were evaluated. Results reveal that mixes incorporating 12M and 14M molarity with 100% fly ash and granite dust achieved compressive strengths of up to 7.5 MPa. Furthermore, blends with 14M and 16M molarity combining granite dust and metakaolin attained compressive strengths exceeding 9.5 MPa. However, 100% metakaolin mixes showed reduced strength due to increased porosity from high aluminum content. Tensile strength testing of cylinders demonstrated superior performance in mixes with 12M and 16M molarity. Overall, mixes with 12M to 16M molarity exhibited optimal performance, suggesting effective geopolymer formulations for sustainable construction applications.

Keywords: Geopolymer Mortar, Metakaolin, Fly ash, Granite dust, Molarity.

1 Introduction

Traditional binders like lime and cement are construction cornerstones, but their production guzzles energy and emits harmful CO_2 [1].In the production of one tonne of cement, about 0.9 tonne of CO_2 is emitted.[2] Industries face significant challenges in safely and effectively disposing of their effluent, sludge, and by-products like fly ash generated from coal combustion for electricity. [3]By 2010, annual fly ash production is estimated to reach 780 million tons, mostly disposed of in landfills. However, landfilling poses financial burdens and future environmental liabilities, highlighting the urgency for alternative disposal methods.[4] As we strive for more sustainable construction practices, finding alternatives to these traditional materials is crucial. However, a pivotal shift occurred in the 1990s with the discovery that fly ash, a by-product of coal combustion, could be activated by alkaline solutions to form high-performance geopolymers. This opened exciting possibilities for the civil engineering field, offering an alternative to traditional Portland cement.[5] The term "geopolymer" was introduced by Joseph Davidovits in the 1970s to describe a class of inorganic polymers made from

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aluminosilicate powders and alkaline solutions. Initially developed for fire-resistant applications, geopolymers found diverse uses in coatings, adhesives, and refractories.[6,7] The utilization of alternative binders like geopolymers, derived from sustainable materials such as industrial by-products (e.g., fly ash), not only enhances environmental sustainability by reducing costs up to 30% and greenhouse emissions by up to 80% but also improves concrete properties, including compressive strength and resistance to various factors like acid and sulphate corrosion. Geopolymers, activated by alkali solutions, have demonstrated compressive strengths exceeding 65 MPa in certain studies.[8]Commercial geopolymer concrete has already been used in Australia for applications like slabs, footpaths, sewer pipes, railway sleepers, and pavers.[9] geopolymer concrete is also used as light weight concrete. [10]Exploring further advancements in 3D printed geopolymers for construction, this paper examines the impact of material selection, reinforcement techniques, curing processes, and printing configurations on both the fresh and hardened properties of the printed material. Additionally, it highlights the crucial connection between key features and printability**.**[11]

1.1 Significance of acid-based binders

Geopolymers result from the interaction of an aluminosilicate precursor with either an alkaline or acidic activator.[7] Extensive research has been conducted on the raw materials, reaction mechanism, modification, and diverse properties of alkali activated geopolymers, as documented in studies.[12–15] Certain research findings have been effectively utilized in engineering applications.[16,17] While evidence suggests that phosphate or acid based geopolymer binders have been utilized since ancient times, unlike alkali activated geopolymer binders, there has been a lack of systematic understanding regarding their properties and applications.[18]

In 2011, Davidovits formally incorporated phosphate geopolymers into his authoritative monograph on geopolymer chemistry and applications.[7] Aluminosilicate phosphate (ASP) geopolymers specifically denote geopolymers resulting from the interaction of aluminosilicate precursors with acidic activators, such as phosphoric acid or phosphate. Previous research has demonstrated the exceptional mechanical properties [19], heat resistance[20] , dielectric properties [21], etc., of ASP geopolymers compared to alkaline based geopolymers.

While most geopolymers use alkali activators, some researchers are exploring the potential of acidic activators like phosphoric acid. This approach yielded promising results, with metakaolin-based geopolymers achieving impressive compressive strengths of up to 93.8 MPa[22,23] . Furthermore, acid-based geopolymers seem to outperform their alkali-based counterparts in terms of temperature resistance (reaching up to 1450°C) and mechanical properties [24]. Recent studies even suggest that a novel phosphoric acid-based geopolymer could serve as a reliable fire or heat insulator, boasting a conductivity as low as 10^-7 S/cm at high temperatures [25]. Therefore, phosphoric acid-based geopolymers represent the optimal substitution for alkaline-based geopolymers.

1.1.1 Geopolymerization process

Geopolymer strength is the result of a chemical reaction, named polymerization, between an amorphous aluminosilicate material and an alkaline solution (fly double dashed ash, metakaolin and slag fines). In phosphate geopolymers, such a formation takes place by reaction of phosphoric acid with the aluminosilicate source leading to a 3D polymeric network. The introduction of phosphoric acid into the network stabilizes it by the introduction of phosphate (PO_4^{3-}) groups into siloxo chains to give Si–O–P–O–Si (phospho-siloxane) linkages [7]. A study of the molar concentration of phosphoric acid is vital to find the optimum for the synthesis and performance of metakaolin-phosphate geopolymers. Davidovits (2011) reported that the geopolymerisation process in low pH includes three fundamental stages: Protonation of siloxane oxygen Cleavage of siloxane bond and rebuilding to form silanol (Si–OH) and Si–O–$PO(OH)_2$, Condensation to form 2D and 3D Si–O–P–O–Si networks. The process leads to a stable macromolecular structure that can be used for a variety of high-performance applications. The process of geopolymerization is presented in Fig 1.

1.1.2 Objectives

- To obtain the Physical properties of Cementitious materials.
- To achieve the Optimal Mix design of Geopolymer Mortar.
- To obtain the Mechanical properties of Geopolymer Mortar.

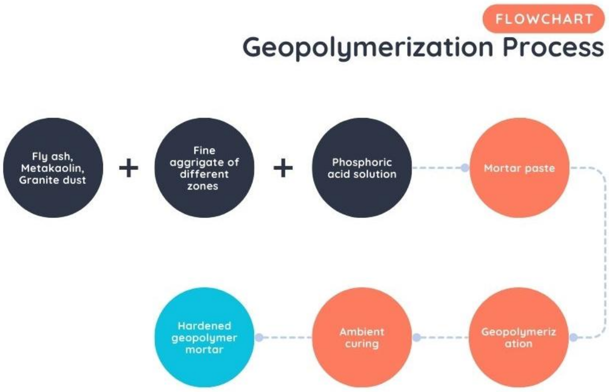

Fig. 1. Flowchart of Geopolymerization Process.

2 Materials Used

Geopolymer mortar is an innovative alternative to traditional cement-based mortar. It's made using geopolymers, which are inorganic polymers synthesized from natural materials. Here's a breakdown of the materials typically used in geopolymer mortar. Table 1 shows the Chemical Composition of Fly Ash, Metakaolin, Granite Dust.

2.1 Fly ash

Fly ash is commonly used in geopolymer mortar because it contains high amounts of alumina and silica, which are essential ingredients for the geo-polymerization process. When activated with a phosphoric acid solution, such as sodium hydroxide or potassium hydroxide, fly ash forms a geopolymer binder that can replace traditional cement. This not only utilizes a waste product but also reduces the environmental impact associated with cement production.

2.2 Metakaolin

Metakaolin is derived from kaolin, which is obtained through mining processes. The kaolin powder is subjected to calcination at 700°C in a programmable electric furnace for 4 hours, with a heating and cooling rate of 5°C/min, to produce metakaolin. The silica and alumina content are: SiO_2- 56.91%, Al_2O_3- 42.35%. Metakaolin is used in geopolymer mortar primarily because of its pozzolanic properties. When mixed with a phosphoric acid solution, metakaolin reacts to form a strong, durable binder known as geopolymer.

2.3 Granite dust

Granite dust, also known as granite fines or granite powder, can often be obtained from granite quarries or stone fabrication shops. These places generate granite dust as a by-product during the cutting and shaping of granite stone. You may be able to purchase granite dust directly from these sources or inquire about its availability. Additionally, some construction material suppliers may also offer granite dust as a product for various applications, including as a binder in certain construction mixes.

Table 1. Chemical Composition of Fly Ash, Metakaolin, Granite Dust.

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	TiO_2	K_2O	MnO	P_2O_5
Fly Ash	55.38	28.14	3.31	3.45	1.85	2.30	-	1.39	-	-
Metakaolin	52.08	46.32	0.26	0.22	-	0.05	-	0.05	-	-
Granite Dust	72.9	14.65	1.7	1.5	0.37	3.85	0.235	3.98	0.026	0.088

2.4 Fine aggregate

Fine aggregates for mortar are materials used to enhance the workability, cohesion and strength of mortar mixes. Fine aggregates are those general granular materials resulting from natural disintegration of rock and used for concrete mixtures, such as natural sand, or crushed stone; or up to 30% of pure natural sand or crushed gravel screenings. In mortar, the well-known fine aggregate is the sand, which is easily available, cost effective, and compatible with the cement paste.

It can be concluded that the fine aggregate plays a role in the expression of volume and filling to the mortar mixture, improving the firmness and the cohesiveness of the same. They fill the gaps between bulk grains to decrease voids and increase density of the mortar. This denser matrix adds to the compressive strength and better crack resistance. Workability of mortar is largely affected by the content and quality of the fine aggregate.

2.5 Phosphoric Acid Solution

Acid-based activators have also been found to cause the activation of geopolymer materials.[7] Acid activators Aspects such as acidity, resistance to chemical attack, strength development and durability all depend on the acid activator system employed. Moreover, the choice of appropriate activator solutions is dictated by the chemical composition and origin of the raw material. Among the acid activating agents, phosphoric acid (H_3PO_4) is the most studied due to its optimal activation potential and its compatibility with specific aluminosilicate materials.

The present experimental work examines the properties of geopolymer concrete activated using phosphoric acid solutions with concentrations of 6M, 8M, 10M, 12M, 14M, and 16M. For 12M, about 480 grams of concentrated phosphoric acid (based on equivalent molarity calculation) are diluted with potable water to prepare one liter of 12M phosphoric acid solution. The ratio of Phosphoric Acid Solution to Sodium Silicate Solution is considered as 2.5, and the mixed solution is stored for 24 hours at room temperature (25±2ºC) and relative humidity of 65% before it is used for casting. Since the dilution of phosphoric acid in water is an exothermic process that releases a significant amount of heat, the solution is allowed to cool to ambient temperature to ensure safe handling and consistent reaction in the concrete mixture.

3. Mix Design

The compressive strength of concrete, particularly achieving a desired 7-day strength of at least 30 MPa, is primarily determined by the water-cement ratio. However, the properties of the aggregate also play a significant role in influencing the strength, alongside the water-cement ratio. Therefore, to ensure optimal strength, it is imperative to minimize the water-cement ratio, although this can impact the workability of the mixture. Through conventional methods of mix compaction and suitable ingredient proportions, concrete meeting these strength requirements can be reliably produced. Table 2 shows the Mix Proportions of Geopolymer Mortar Cube mixtures kg/m3.

Table 2: Mix Proportions of Geopolymer Mortar Cube mixtures kg/m^3.

SNO	Mix ID	FA (Kg)	GD (Kg)	MK (Kg)	Sand (Kg)	Phosphoric acid Sol (Kg)	L/S
1	A6M100F	583.09			1749.27	332.3	0.57
2	A8M100F	583.09			1749.27	314.8	0.54
3	A10M100F	583.09			1749.27	297.3	0.51
4	A12M100F	583.09			1749.27	361.5	0.62
5	A14M100F	583.09			1749.27	394.85	0.6
6	A16M100F	583.09			1749.27	291.54	0.5
7	A6M100M			583.09	1749.27	349.85	0.6
8	A8M100M			583.09	1749.27	384.83	0.66
9	A10M100M			583.09	1749.27	384.83	0.66
10	A12M100M			583.09	1749.27	396.5	0.68
11	A14M100M			583.09	1749.27	384.83	0.66
12	A6M100GD		874.63		1457.72	349.8	0.4
13	A8M100GD		874.63		1457.72	454.81	0.52
14	A10M100GD		874.63		1457.72	437.3	0.5
15	A12M100GD		874.63		1457.72	437.3	0.5

16	A14M100GD	874.63		1457.72	419.8	0.48
17	A16M100GD	874.63		1457.72	437.31	0.5
18	A6M50M50GD	437.31	291.54	1457.72	367.3	0.42
19	A8M50M50GD	437.31	291.54	1457.72	437.3	0.5
20	A10M50M50GD	437.31	291.54	1457.72	419.82	0.48
21	A12M50M50GD	437.31	291.54	1457.72	437.3	0.5
22	A14M50M50GD	437.31	291.54	1457.72	367.3	0.42
23	A16M50M50GD	437.31	291.54	1457.72	454.8	0.52

Procedure for Preparation of geopolymer Mortar 25 g of the dry mix and appropriate weights of the phosphoric acid solution is hand blended by a vigorous scraping-up-and over motion for five to ten minutes and until the mixture is homogeneous. Afterwards, the prepared concrete is cast in 70mm cube moulds to a height of three equal layers. Each of those layers are compacted with 25 rod strokes to ensure a good compaction, and the top layer of each mold is leveled. Once casting is done, the specimens remained undisturbed for at least 24 hours after the demoulding. This conventional procedure provides a good mixing and compact scale of geopolymer concrete and permits the set before testing or use. Fig. 2 shows the Geopolymer Mortar Cubes and Cylinders of Different Molarities.

We prepare in the same way cylinders for the determination of the tensile strength.

Fig. 2. Geopolymer Mortar Cubes and Cylinders of Different Molarities.

4 Methodology

Geopolymer Mortar process commonly has a sequence of physical and chemical operations: the aluminosilicate materials are mixed with phosphoric acid activator form the geopolymer binder, then the geopolymer binder is mixed with aggregates and water to produce the mortar. This combination reacts when mixed, forming a robust, high-strength material that does not required standard cement. The particular method may depend on the properties and uses of the mortar.

4.1 Mixing

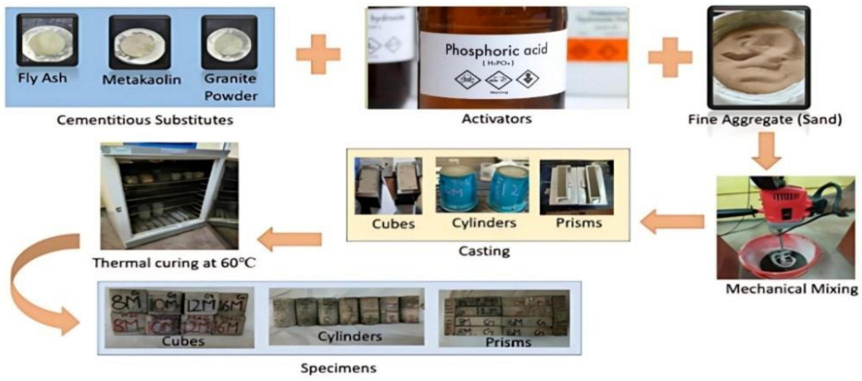

Fig. 3. Flow Chart of Mixing Process of mortar

Fig. 4. Casting of Mortar Cubes and Cylinders

Fig 3 and fig 4 shows the Flow chart of mixing process of mortar and Casting of mortar cubes and cylinders.

4.2 Curing

Thermal Curing of Geopolymer entails curing the geopolymer at an elevated temperature in order to speed up the chemical reaction and improve the strength and durability of the geopolymer. This curing process usually takes place in a controlled environment like in an oven or kiln, at a temperature in a range of, for example, 50°C to 100°C or more, depending on the alumino-silicate composition used and on the sought properties.

Thermal curing is especially powerful to enhance the early strength development and overall properties of geopolymer materials for many construction replacements.

Fly ash, metakaolin, Granite Dust cubes are held in an oven at a temperature of 60°Cfor 1 day and 3 days &7 days respectively.

4.3 Compressive Strength

Compressive strength: Geopolymer mortar specimens are often cast in standard size cube moulds. The mix deign, including the overall and ratios of fly ash, activators, aggregates and water, is closely followed. Following the cast of the material, the samples are conditioned under controlled environment to replicate the desired service environment. Curing may be a curing with ambient air, steam curing or oven curing, according to the need of a particular geopolymer mix at the time. Table 3 shows the Compressive Strength Results of Geopolymer Mortar Cubes.

Table 3. Compressive Strength Results of Geopolymer Mortar Cubes.

SN	Mix ID	L/S	T (°C)	Days	D (Kg/cm^3)	CS (MPa)
1	A6M100F	0.57	60	1	1.86×10^{-3}	2.1
2	A6M100F	0.57	60	3	1.86×10^{-3}	3.6
3	A8M100F	0.54	60	1	1.93×10^{-3}	3.1
4	A8M100F	0.54	60	3	1.93×10^{-3}	4.5
5	A10M100F	0.51	60	1	1.98×10^{-3}	4.4
6	A10M100F	0.51	60	3	1.98×10^{-3}	6.3
7	A12M100F	0.62	60	1	2.05×10^{-3}	6.7
8	A12M100F	0.62	60	3	2.05×10^{-3}	8.3
9	A14M100F	0.6	60	1	2.06×10^{-3}	7.6
10	A14M100F	0.6	60	3	2.06×10^{-3}	9.0
11	A16M100F	0.5	60	1	1.89×10^{-3}	0.9
12	A16M100F	0.5	60	3	1.89×10^{-3}	1.3
13	A6M100M	0.6	60	1	1.82×10^{-3}	0.8
14	A6M100M	0.6	60	3	1.82×10^{-3}	2.3
15	A8M100M	0.66	60	1	1.93×10^{-3}	1.3
16	A8M100M	0.66	60	3	1.93×10^{-3}	2.5
17	A10M100M	0.66	60	1	1.95×10^{-3}	1.1
18	A10M100M	0.66	60	3	1.95×10^{-3}	3.2
19	A12M100M	0.68	60	1	1.96×10^{-3}	2.2
20	A12M100M	0.68	60	3	1.96×10^{-3}	4.2
21	A14M100M	0.66	60	1	1.98×10^{-3}	3.2
22	A14M100M	0.66	60	3	1.98×10^{-3}	4.5

23	A6M100GD	0.4	60	1	1.89×10^{-3}	1.1
24	A6M100GD	0.4	60	3	1.89×10^{-3}	2.3
25	A8M100GD	0.52	60	1	1.98×10^{-3}	2.0
26	A8M100GD	0.52	60	3	1.98×10^{-3}	3.2
27	A10M100GD	0.5	60	1	2.02×10^{-3}	5.5
28	A10M100GD	0.5	60	3	2.02×10^{-3}	7.0
29	A12M100GD	0.5	60	1	2.05×10^{-3}	6.2
30	A12M100GD	0.5	60	3	2.05×10^{-3}	8.3
31	A14M100GD	0.48	60	1	2.09×10^{-3}	6.9
32	A14M100GD	0.48	60	3	2.09×10^{-3}	8.2
33	A16M100GD	0.5	60	1	1.9×10^{-3}	5.4
34	A16M100GD	0.5	60	3	1.9×10^{-3}	6.9
35	A6M50M50GD	0.42	60	1	1.9×10^{-3}	3.5
36	A6M50M50GD	0.42	60	3	1.9×10^{-3}	4.5
37	A8M50M50GD	0.5	60	1	1.96×10^{-3}	4.2
38	A8M50M50GD	0.5	60	3	1.96×10^{-3}	5.5
39	A10M50M50GD	0.48	60	1	2.01×10^{-3}	5.3
40	A10M50M50GD	0.48	60	3	2.01×10^{-3}	7.0
41	A12M50M50GD	0.5	60	1	2.04×10^{-3}	5.6
42	A12M50M50GD	0.5	60	3	2.04×10^{-3}	7.5
43	A14M50M50GD	0.42	60	1	2.06×10^{-3}	7.8
44	A14M50M50GD	0.42	60	3	2.06×10^{-3}	10.7
45	A16M50M50GD	0.52	60	1	2.05×10^{-3}	8.4
46	A16M50M50GD	0.52	60	3	2.05×10^{-3}	12.3

4.4 Tensile Strength

The tensile strength of geopolymer mortar may depend on a number of factors, these include the exact composition of geopolymer mix, curing regime, and how the test was carried out. In general, cylindrical samples are typically employed to assess the tensile strength of geopolymeric mortar.

For tensile strength testing, it is necessary to form specimens of certain size and geometry. For the materials like geopolymer mortar, rectangular or cylindrical specimens are often used. The specimens are well prepared as they're standardized shapes and sizes. After flattened to size samples are rigidly gripped by a tensile testing machine. Great care is taken to ensure that the grips are aligned with the specimens' longitudinal axis to avoid any off-axis loading. The tensile force is increased to the specimen by the test machine with a slowly rising force. The force is usually ramped at a constant rate, so that uniform loading case is maintained during the test. Table 4 shows the Tensile Strength Results of Geopolymer Mortar Cylinders.

Table 4. Tensile Strength Results of Geopolymer Mortar Cylinders.

SN	Mix ID	L/S	T (°C)	Days	D (Kg/cm^3)	TS (MPa)
1	A12M100F	0.54	60	1	2.04×10^{-3}	2.8
2	A12M100F	0.54	60	3	2.04×10^{-3}	4.4
3	A14M100F	0.6	60	1	2.05×10^{-3}	3.3
4	A14M100F	0.6	60	3	2.05×10^{-3}	4.8
5	A8M100GD	0.52	60	1	1.67×10^{-3}	0.6
6	A8M100GD	0.52	60	3	1.67×10^{-3}	1.45
7	A12M100GD	0.5	60	1	1.74×10^{-3}	1.4
8	A12M100GD	0.5	60	3	1.74×10^{-3}	2.2
9	A16M100GD	0.5	60	1	1.86×10^{-3}	2.0
10	A16M100GD	0.5	60	3	1.86×10^{-3}	3.1
11	A8M50M50GD	0.5	60	1	1.93×10^{-3}	1.9
12	A8M50M50GD	0.5	60	3	1.93×10^{-3}	2.8
13	A12M50M50GD	0.5	60	1	2.01×10^{-3}	3.8
14	A12M50M50GD	0.5	60	3	2.01×10^{-3}	5.0
15	A16M50M50GD	0.52	60	1	2.05×10^{-3}	4.6
16	A16M50M50GD	0.52	60	3	2.05×10^{-3}	6.0

4.5 Durability Testing

Durability test is a very important factor for evaluating the long-term behaviour of geopolymer mortars in different environmental conditions. In the present work the durability of the geopolymer mortar has been characterized using the important parameters such as acid resistance, water absorption and porosity. These tests aid in assessing the resistance of the mortar to aggressive chemical environments as well as the ability of the mortar to maintain structural integrity and strength with time. **Acid Resistance Test**: Specimens were immersed in 5% sulfuric acid (H_2SO_4) solution for 28 days. The weight loss and reduction in compressive strength were recorded to assess the degradation levels.

- **Water Absorption Test**: Oven-dried samples were submerged in water for 24 hours, and the percentage of water absorbed was calculated.

- **Porosity Measurement**: Porosity was determined using standard vacuum saturation methods to understand the permeability characteristics of the geopolymer matrix.

The mixtures with highest molarity (12M, 14M and 16M) showed lower water absorption and higher acid resistance which are characteristics of high durability. In particular, a blend of granite dust and metakaolin had a very good chemical resistance as a result of the higher density of the micro structure that was formed during polymerization.

5 Results and Discussions

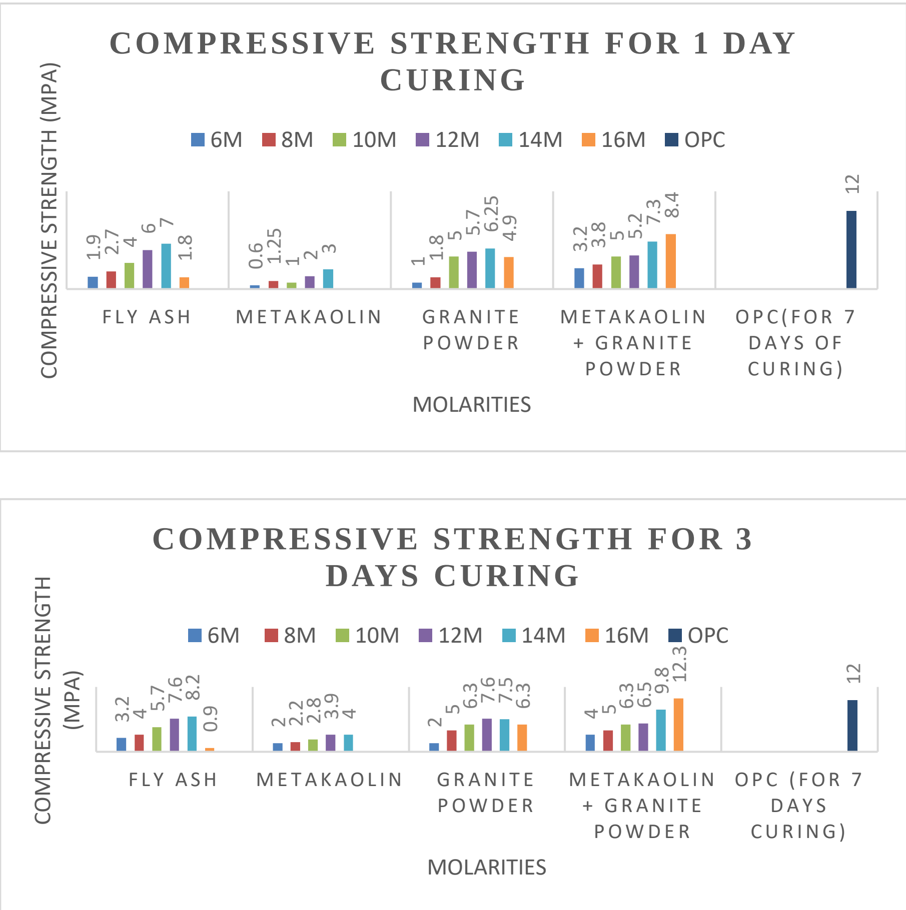

Fig. 5. Comparison of Compressive strength for day 1 strength curing vs day 3 curing.

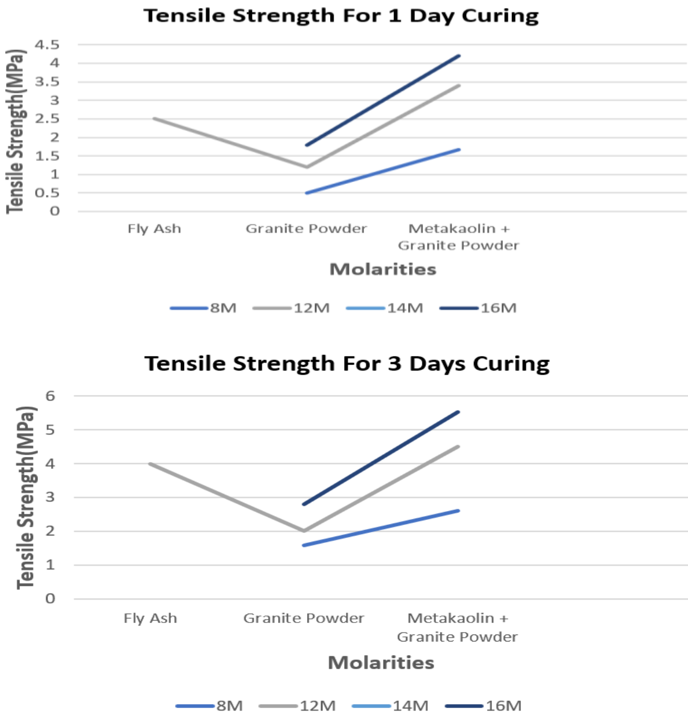

Fig. 6. Comparison of tensile strength for day 1 vs day 3.

Fig. 5. Shows the Comparison of Compressive strength for day 1 strength curing vs day 3 curing. Fig. 6. Shows the Comparison of tensile strength for day 1 vs day 3.

Discussion: The discussion of Geopolymer Mortar tends to focus on its sustainability advantages in contrast to Ordinary Portland Cement (OPC) mortar. Among these, are its low carbon footprint associated with the low production of CO2, possibility to use industrial by products to feed the process and excellent resistance to chemical and environmental degradation. This could touch on its mechanical properties, like compressive strength, flexural strength, and durability as well as uses in construction like infrastructure rehabilitation, precast members, and green building techniques and much more!

6 Conclusion

The Granite Dust, alone is capable of providing a satisfactory compressive strength in geopolymer mortar; however, along with combination of Granite Dust and Metakaolin its compressive strength becomes enhanced in Thermal-curing conditions.

- The mixes containing 12M and 14M with 100% Granite Dust and Fly Ash achieve compressive strengths of 7.61MPa and 8.16MPa.
- Additionally, mixes with 14M and 16M in combination with Granite Dust and Metakaolin achieve compressive strengths of 9.79MPa and 11.22MPa.
- The absence of compressive strength results for 100% Metakaolin is attributed to its

higher aluminum powder content, leading to porosity.

- The mixes with molarities of 12M, 14M, and 16M have achieved satisfactory compressive strength, indicating an optimal mix design.
- Tensile strength is calculated for cylinders, the mixes of 16M with 100% of Granite Dust achieved 2.8MPa. The combinations of Granite Dust and Metakaolin of 16M achieved tensile strength 5.5MPa.
- From above graphs, it concludes that PGPM has achieved greater strength than OPC Mortar.
- Based on cost analysis, it's evident that the cost per m^3 volume of Fly Ash, Metakaolin, and Granite Dust is lower compared to OPC. Specifically, Fly Ash costs Rs 1,166.18 and Granite Dust costs Rs 2,623.89 per m^3 volume.

This makes geopolymer mortar not only technically superior but also economically viable for both rural and large-scale construction projects.

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