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Investigation of the fresh and hardened properties of fly ash and metakaolin based geopolymer concrete containing of nanoparticles

Ayad Saddam Alwan[1], Mojtaba Fathi[1*]

[1] Water Resources Management Engineering Department, College of Engineering, Al-Qasim Green University, 51013, Babylon, Iraq
* Corresponding author's email: fathim@razi.ac.ir

ABSTRACT

The global construction sector faces significant sustainability challenges due to the high energy consumption and carbon emissions associated with ordinary Portland cement (OPC). Geopolymer concrete (GPC), synthesized through the alkali activation of aluminosilicate-rich by-products such as fly ash (FA) and metakaolin (MK), offers a promising low-carbon alternative. This study examines the effects of combining FA, MK, nano-alumina (NA), and nano-limestone (NL) in binary, ternary, and quaternary GPC blends of GP-40 and GP-60. Hardened properties, including compressive, flexural, and splitting tensile strengths, along with ultrasonic pulse velocity, were evaluated at 7, 28, 56, and 90 days. The results showed that nanomaterials improved pore structure and binder homogeneity, leading to enhanced overall mechanical performance. Optimized mixes achieved compressive strengths exceeding 55 MPa at 90 days, with significant improvements in splitting tensile and flexural strengths. The results indicate that nanomodified GPC blends demonstrated balanced performance, making them a sustainable alternative for future green concrete applications.

Keywords: geopolymer concrete; fly ash; metakaolin; nano-alumina, nano-limestone, fresh and mechanical properties.

INTRODUCTION

In the world, more than any other building material other than water, concrete is used the most. This is a result of its versatility, durability, and superior mechanical characteristics. However, ordinary Portland cement (OPC), the most widely used cement in concrete, has a particularly bad reputation in its environmental impact. The production of this cement alone is responsible for nearly 8% of the global CO_2 emissions, or for every ton of cement produced, 0.8 1.0 tons of CO_2 is released into the atmosphere [1–5]. This also demands a great deal of natural resources and fossil fuel energy. These environmental problems have made people keep working to find greener and better ways to use less carbon without losing strength or safety [6, 7]. In this situation, geopolymer concrete has come up as one of the best low-carbon options [8, 9]. Geopolymers are inorganic binders created through the alkaline activation of aluminosilicate precursors, representing a completely different class of materials from OPC, which sets through the hydration of calcium silicate phases [10, 11]. This different reaction mechanism can decrease CO_2 emissions by as much as 80% compared with OPC [12], and offers better protection against harsh conditions, high temperatures, and chemical damage [13, 14]. FA and MK have been the most used aluminosilicate precursors for geopolymer synthesis. This is due to their abundance and good pozzolanic reactivity, which has attracted a lot of research interest.

Fly ash is a byproduct from the burning of coal in thermal power plants. It contains fine spherical particles that are rich in silica and alumina and have high pozzolanic activity. ASTM

C618 classifies FA into Class F (low-calcium) and Class C (high-calcium) types [15–17]. Class F has better pozzolanic performance than Class C, which has some self-cementing properties. The annual global production of fly ash is around 600–900 million tons, making it an environmental burden as well as a useful material for sustainable concrete technologies [18].

Many studies have reported that replacing 20–40% of ordinary Portland cement with FA improves strength in the long term, reduces permeability to enhance durability, and reduces emissions of CO_2 by about 20–30% [19–21]. In geopolymer systems, FA interacts with alkaline activators—usually sodium hydroxide and sodium silicate—to produce sodium aluminosilicate hydrate (N–A–S–H) gels which fill voids in the microstructure and help develop strength [22]. Its relatively low reactivity at room temperature curing conditions usually results in limited early-age strength [23]. This has led researchers to consider thermal curing or nanomaterial addition as a means of accelerating geopolymerization [24].

MK, is a highly reactive aluminosilicate obtained from calcined kaolin clay between 600 and 800 °C to form an amorphous pozzolanic material with consistent chemical purity [25]. In contrast to FA, MK is deliberately produced which guarantees uniformity and high reactivity. Some studies have reported that the partial replacement of OPC by 5–20% MK enhances compressive as well as tensile strength, increases durability, and lowers the porosity [26–28]. These improvements are mainly due to refining pore structure and forming new binding phases such as C-S-H and hydrated aluminosilicates in alkaline environments [29, 30]. MK-based geopolymers can attain compressive strengths greater than 50 MPa within 28 days under ambient curing conditions with very high resistance to chloride ingress, sulfate attack, and thermal degradation [31, 32].

Although FA is very plentiful and inexpensive, its geopolymerization rates are relatively slow, making it less applicable by itself. On the other hand, MK geopolymerization rates and early strengths are relatively faster but at costs of workability and economy. As a result, there has been great interest in FA/MK mixes and combinations in pursuit of cost, workability, reactivity, and performance optimizations [33, 34]. More recently, there has been great interest in adding nanomaterials like nano-silica, nano-alumina, and nano-lime into FA/MK geopolymer mixes, and it has been shown that nanomaterial incorporation can improve size-scale refinement of geopolymer structure and its overall properties/indices of geopolymerization and polycondensation [35–39].

Several challenges still exist in geopolymer concrete, particularly regarding early-age strength, shrinkage, and long-term durability. Recent studies highlight the importance of investigating binary and ternary systems based on FA and MK, especially when modified with nanomaterials, to achieve a balanced combination of mechanical performance and environmental sustainability.

In response to these challenges, this study focuses on developing and accessing two experimental geopolymer concrete grades (GP-40 and GP-60) based on FA and MK, with nano-alumina and nano-limestone added to improve performance. The mixes were activated using alkaline solutions and cured under ambient conditions to reflect practical applications. Fresh properties, including workability and bulk density, were examined alongside key mechanical properties such as compressive, splitting tensile, and flexural strengths. In addition, ultrasonic pulse velocity (UPV) testing was employed to evaluate internal uniformity and structural soundness.

EXPERIMENTAL PROGRAM

Materials

In this study, a combination of pozzolanic and nano-materials, along with conventional constituents of concrete, was employed to develop geopolymer concrete with enhanced mechanical performance and durability. These materials include FA, MK, NA, NL, in addition to the alkaline activator solution (AAS), fine and coarse aggregates, water, and a superplasticizer admixture. A detailed description of these materials is provided below.

Aluminosilicate materials (FA and MK)

Low-calcium fly ash (Class F, ASTM [13]) and metakaolin were employed as the primary aluminosilicate sources in this study. The fly ash was obtained from local thermal power plants,

whereas metakaolin was produced through the calcination of pure kaolin at 600–800 °C. This endothermic process involves the removal of chemically bonded hydroxyl groups, which disrupts the crystalline lattice of kaolin. This disruption generates a reactive amorphous phase rich in silica and alumina, thereby imparting high pozzolanic activity. Chemical and physical tests for both materials were conducted at the National Center for Construction Laboratories and Research (NCCLR) in Babylon. The results, summarized in Table 1, show that fly ash contains high percentages of SiO_2 and Al_2O_3, providing suitable pozzolanic activity, while metakaolin is one of the purest and most reactive pozzolanic materials with a high specific surface area that enhances geopolymerization reactions.

Nanoparticles (NA (Al_2O_3) and NL ($Ca(OH)_2$))

Nanoparticles were incorporated in this study as highly pure materials to enhance the reactivity of geopolymer concrete. Nano alumina (NA), with 99.99% purity and an average particle size of 60–80 nm, primarily consists of spherical-phase crystals. These crystals provide exceptional reactivity with binder materials, accelerating geopolymerization reactions. The specific surface area ranges from 15 to 40 m²/g. Similarly, nano limestone (NL), composed of calcium hydroxide nanoparticles with an average size of 20–80 nm and a purity of ≥94.5%, exhibits a specific surface area of 30–80 m²/gm and rapid solubility in water, thereby promoting pozzolanic reactions when combined with silica and alumina rich sources such as fly ash and metakaolin. The properties of NA and NL particles, as reported by the manufacturer, are summarized in Table 2.

Alkaline activator solution (AAS)

The alkaline activator solution is a fundamental component in geopolymer production due to its ability to dissolve silica and alumina and to catalyze polymerization reactions [40]. In this study, commercial sodium hydroxide

Table 2. Properties of NL and NA

Property	Test results *	
	NL	NA
Physical form	Powder	Powder
Color	White	White
Particle size, nm	20–80	60–80
Specific gravity	3.2	2.4
purity %	95-99	99.99
Crystal	alpha	spherical
Specific surface area (m²/gm)	30–80	15–40
Fe %	≤0.005	-
Si %	≤0.003	-
Mg %	≤0.001	-
Ca $(OH)_2$ %	-	≥ 94.5

Note: *The test results from the manufacturer

Table 1. Chemical compositions and physical properties of fly ash and metakaolin

Property	Materials	FA	MK
Chemical composition (%)	(SiO_2)	51.0	51.7
	(Al_2O_3)	29.9	35.4
	(Fe_2O_3)	10.6	2.72
	(CaO)	2.24	1.38
	(MgO)	0.77	0.82
	(Na_2O)	0.01	0.73
	(K_2O)	2.02	0.84
	(SO_3)	0.26	0.24
	(P_2O_5)	1.65	0.43
	(TiO_2)	1.71	0.92
	(LOI)	0.46	3.42
Physical properties	Physical form	Powder	Powder
	Colour	Light grey	Off-White
	Median particle size (μm)	12.8	14.3
	Specific surface area (m²/kg)	463	1640
	Specific gravity	2.60	2.62

flakes with 99% purity were dissolved in distilled water to prepare a 12 M solution (362 g/L) following [41, 42], and the solution was left for 24 hours before being mixed with sodium silicate solution. The activator was prepared with a mass ratio of 2.0 between Na_2SiO_3 and NaOH, while maintaining an activator-to-binder ratio of 0.52 for grade GP40 and 0.47 for grade GP60, which enhances the reactivity with the source materials [43]. The properties of the sodium silicate used, as provided by the manufacturer, are presented in Table 3.

Aggregates

In this study, Natural sand from Al-Ukhaider and crushed washed gravel from Al-Nabai'i were used as fine and coarse aggregates, respectively. The physical properties of the fine aggregate included a specific gravity of 2.65, water absorption of 2.63%, fineness modulus of 2.46, sulfate content of 0.38%, and 1.94% passing the 0.075 mm sieve. The coarse aggregate exhibited a specific gravity of 2.6, water absorption of 0.6%, and sulfate content of 0.07%. All tests were conducted according to ASTM standards [44] at the Construction Materials Laboratory, University of Babylon.

Table 3. Properties of sodium silicate utilized in this study

Description	Value
Na_2O % by weight	13.0–13.4
SiO_2% by weight	31–33
Viscosity (CPS) 20 °C	650-1250
Specific Gravity	1.538–1.557
Density - 20 °C	53 ± 0.5
Appearance	Hazy
Ratio of SiO_2 / Na_2O	2.3± 0.05

Super plasticizer (SP)

The high-performance polycarboxylate-based superplasticizer, commercially known as Sika Visco Crete 180G, was used to reduce water demand and improve the workability of geopolymer concrete (GPC). This superplasticizer, produced by Sika (Type F), complies with ASTM standards [45], and its optimal dosage should be determined through site trials to ensure the desired concrete performance.

Mix proportions of geopolymer concrete

The detailed mix proportions of the geopolymer concrete (GPC) used in this study are presented in Tables 4 and 5. The mix design methodology was developed empirically based on the performance of trial mixes and material characteristics, in accordance with general principles of ACI guidelines, but not derived directly from any specific standard procedure. Table 4 summarizes the binary mixes for GP40 and GP60 grades, where fly ash (FA) was partially replaced by metakaolin (MK) at varying percentages (0%, 10%, 20%, 30%, 40%, and 50%). The binder content, alkaline activator solution (NaOH and Na_2SiO_3) at a ratio of 2,

Table 4. The mixing proportions of Binary mixes

Mix. No	Mix grade	L/B	Quantities of ingredients (kg/m³)							
			Binder	FA (%)	MK (%)	NaOH	Na_2SiO_3	F. A	C. A	SP %
M1	GP-40	0.52	415	100	0	72	144	748	1030	1.2
M2	GP-40	0.52	415	90	10	72	144	748	1030	1.2
M3	GP-40	0.52	415	80	20	72	144	748	1030	1.2
M4	GP-40	0.52	415	70	30	72	144	748	1030	1.2
M5	GP-40	0.52	415	60	40	72	144	748	1030	1.2
M6	GP-40	0.52	415	50	50	72	144	748	1030	1.2
M7	GP-60	0.47	520	100	0	81	162	720	947	1.5
M8	GP-60	0.47	520	90	10	81	162	720	947	1.5
M9	GP-60	0.47	520	80	20	81	162	720	947	1.5
M10	GP-60	0.47	520	70	30	81	162	720	947	1.5
M11	GP-60	0.47	520	60	40	81	162	720	947	1.5
M12	GP-60	0.47	520	50	50	81	162	720	947	1.5

and the ratios of fine aggregate, coarse aggregate, and binder are provided for each grade. For GP40 mixes, these ratios are 1:1.8:2.5 with a superplasticizer dosage of 1.2% and a liquid-to-binder (L/B) ratio of 0.52, whereas for GP60 mixes, the ratios are 1:1.4:1.82 with a superplasticizer dosage of 1.5% and an L/B ratio of 0.47. These ratios were chosen to balance workability and mechanical strength, ensuring proper hydration, required flowability for mixing, and adequate strength and durability in the hardened state. The detailed compositions of the ternary and quaternary geopolymer concrete mixes are presented in Table 5. Nano-alumina (NA) and nano-calcite (NL) were added to the optimized blend of 70% fly ash and 30% metakaolin at replacement levels of 2%, 3%, and 4% by mass. The ratios of binder, fly ash, metakaolin, NA, NL, alkaline activator solution, aggregates, and superplasticizer for each mix remain similar to those of the binary mixes, allowing a systematic evaluation of the effects of nano-materials on the performance of GP40 and GP60 concrete.

Mixing, casting, curing, and testing procedures of geopolymer concrete

The primary distinction between geopolymer concrete (GPC) and conventional concrete lies in the type of binder used, where silica and alumina present in FA and MK react with the alkaline solution (Na_2SiO_3 and NaOH) to form a geopolymer paste that binds the coarse and fine aggregates along with other ingredients to produce the concrete. The mixing method plays a critical role in influencing the workability and strength of GPC. All mixing procedures were conducted in a laboratory at 25±2 °C using a pan-type laboratory mixer with a capacity of approximately 0.09 m^3, ensuring that the mixer was kept clean, wet, and free of residual materials before each batch. The alkaline solution was prepared in advance, left for 20 minutes, and then used as the liquid component after adding water and superplasticizer. The mixing steps began with combining the dry materials (coarse and fine aggregates, FA, and MK) for 2.5 minutes, after which the prepared liquid containing the alkaline solution, water, and superplasticizer was gradually added over 3 minutes, with continuous mixing maintained after each addition. According to [46], nanoparticles, such as NA and NL, were initially dispersed in a portion of water and alkaline solution using a hand-electric mixer. The dispersed nanoparticles were then incorporated into the main mixture. To ensure homogeneity, the fresh concrete was hand-mixed for an additional 2–3 minutes before being evaluated for its fresh properties and subsequently cast. The GPC mixture was poured into molds of cubes, cylinders, and prisms until completely filled, and following casting, the specimens were covered with nylon sheets to prevent the evaporation of the alkaline solution [47] and left for 24

Table 5. The mixing proportions of ternary and Quaternary mixes

Mix No.	Mix Grade	L/B	Quantities of ingredients (kg/m^3)									
			Binder	FA (%)	MK (%)	NA (%)	NL (%)	NaOH	Na_2SiO_3	Fine Agg.	Coarse Agg.	SP %
M13	GP-40	0.52	415	0.7	0.3	2	0	72	144	748	1030	1.2
M14	GP-40	0.52	415	0.7	0.3	3	0	72	144	748	1030	1.2
M15	GP-40	0.52	415	0.7	0.3	4	0	72	144	748	1030	1.2
M16	GP-40	0.52	415	0.7	0.3	0	2	72	144	748	1030	1.2
M17	GP-40	0.52	415	0.7	0.3	0	3	72	144	748	1030	1.2
M18	GP-40	0.52	415	0.7	0.3	0	4	72	144	748	1030	1.2
M19	GP-40	0.52	415	0.7	0.3	2	3	72	144	720	947	1.2
M20	GP-60	0.47	520	0.7	0.3	2	0	81	162	720	947	1.5
M21	GP-60	0.47	520	0.7	0.3	3	0	81	162	720	947	1.5
M22	GP-60	0.47	520	0.7	0.3	4	0	81	162	720	947	1.5
M23	GP-60	0.47	520	0.7	0.3	0	2	81	162	720	947	1.5
M24	GP-60	0.47	520	0.7	0.3	0	3	81	162	720	947	1.5
M25	GP-60	0.47	520	0.7	0.3	0	4	81	162	720	947	1.5
M26	GP-60	0.47	520	0.7	0.3	2	3	81	162	720	947	1.5

hours under ambient conditions before demolding, as shown in Figure 1.

The specimens were subjected to curing in a laboratory oven at 60 °C for 24 hours after demolding, followed by curing at room temperature (between 22 and 30 °C) with the specimens covered by plastic sheets until the day of testing. These conditions were chosen to accelerate the geopolymerization process and enhance the binder reaction effectively. The 60 °C temperature promotes faster reaction, while the plastic covers help maintain moisture and prevent moisture loss, ensuring continuous curing in a stable environment [48].

The molds used for specimen preparation included 50×50×50 mm cubes for mortar compressive strength to determine the pozzolanic activity of the materials, 100×100×100 mm cubes for compressive strength and UPV, 100×200 mm cylinders for splitting tensile strength, and 100×100×400 mm prisms for flexural strength.

The comprehensive capturing of the mechanical behavior of the hardened state of GPC has been done using the tests of bulk density, compressive strength, splitting tensile strength, flexural strength, and ultrasonic pulse velocity (UPV) tests which gives a holistic understanding of the structural behavior GPC compared to the conventional OPC concrete. Bulk density was obtained after 7 and 28 days as per ASTM [49] using three 100×100×100 mm cubes for each mix as a result of the fact that higher density is usually indicative of lesser voids and higher porosity, which in turn translates to strength, durability, and permeability of the concrete. Compressive strength is really the key property when it comes to understanding how geopolymerization is going. To measure it, we tested 100 mm cube samples at 3, 7, 28, 56, and 90 days, following BS EN [50]. We used a digital testing machine (1900 kN capacity, see Figure 2A) and kept the loading rate steady at 0.3 MPa per second. For splitting tensile strength-which shows how brittle materials like concrete handle tension we used 100×200 mm cylinders, testing them at 7, 28, and 90 days according to ASTM C496-08 [51], again with the same 1900 kN machine. Plywood strips were placed above and below the specimens to ensure uniform load distribution (Figure 2B). A reduced specimen size was adopted, compared to the standard 150×300 mm dimensions, to minimize the required quantities of geopolymer and nanomaterial components, facilitate casting and handling, and accommodate laboratory equipment limitations, while maintaining the same aspect ratio (L/D = 2.0) to ensure comparable stress distribution. Therefore, this factor was taken into account when interpreting the results and comparing them with previous studies that used standard-sized specimens [52]. Flexural tensile strength was measured on 100×100×400 mm concrete prisms using a digital testing machine with a capacity of 150 kN, following the third-point loading method in accordance with ASTM [53], and tests were conducted specifically at 28 days (Figure 2C), with the average results of three prisms recorded for each mix. In addition to destructive tests, non-destructive evaluation was conducted using the UPV test to assess internal homogeneity and detect potential microcracks without damaging the specimens, following ASTM [54].



Figure 1. Prepared samples of casting samples

The test used the same 100 mm cube specimens prepared for compressive strength at 3, 7, 28, 56, and 90 days (Figure 3) and was carried out with a portable ultrasonic non-destructive digital indicating tester (PUNDIT) equipped with 54 kHz (±5 kHz) piezoelectric transducers, with a thin grease layer applied to ensure effective coupling between the transducers and the concrete surface.

RESULTS AND DISCUSSIONS

Strength activity index

The results of the strength activity index (SAI) for the different mixes at 7 and 28 days, as shown in Table 6, highlight the influence of



Figure 2. Mechanical tests of geopolymer concrete: (a) compressive strength, (b) splitting tensile strength, (c) flexural strength



Figure 3. Ultrasonic pulse velocity test

various additives on the mechanical performance of geopolymer concrete.

Fly ash exhibited SAI values of 87% at 7 days and 98% at 28 days, indicating relatively low pozzolanic activity, likely due to its low calcium content, which slows early-age strength development. Metakaolin showed moderate improvement, with SAI values of 92% and 102%, attributed to its fine, amorphous structure that enhances pozzolanic reactions and matrix strength. Nano-alumina demonstrated a significant increase in SAI to 134% at 7 days and 152% at 28 days, reflecting its role in improving microstructural packing and matrix density. Similarly, nano-lime increased SAI to 129% and 143%, enhancing the alumina-silica reactions and overall mechanical properties. These results indicate that nanomaterials such as nano-alumina and nano-lime substantially improve the early-age strength and reduce porosity in geopolymer concrete, while metakaolin provides moderate enhancement, and fly ash shows the least improvement due to slower pozzolanic activity. Therefore, the inclusion of nanomaterials is recommended to boost mechanical performance, particularly in applications requiring high early strength [35, 55, 56].

Slump test

The results presented in Figures 4 and 5 clearly demonstrate the overall trend in the workability of geopolymer concrete (GPC) mixtures. In the binary blends (FA–MK), increasing the proportion of metakaolin (MK) consistently led to a reduction in slump values. For example, the slump decreased from 165 mm in mix M1 (100% FA) to 121 mm in mix M6 (50% FA–50% MK) at a constant liquid-to-binder ratio (L/B) of 0.52. A similar pattern was observed at L/B =

Table 6. Strength activity index of fly ash, metakaolin and nanoparticles

Mixes	Strength Activity Index (SAI) (%), at 7 days	Strength Activity Index (SAI) (%), at 28 days
Control-PC	100	100
Fly ash	87	98
Metakaolin	92	102
Nano alumina	134	152
Nano lime	129	143

0.47, where the slump value was 155 mm for M7 but dropped to 113 mm for M12.

This reduction in workability can be attributed to the higher surface area and more reactive nature of MK particles compared to FA, which increase water demand and reduce fluidity. These findings are consistent with those reported by [57], who found that higher MK content in geopolymer mixtures decreases workability due to its high fineness and rapid dissolution rate.

In the ternary and quaternary blends (Figure 5), the inclusion of NA and NL further decreased the slump values compared to the binary blends. For instance, mix M13 (with 2% NA) achieved a slump of 130 mm, but when the NA content increased to 4% (M15), the slump decreased to 123 mm. A similar reduction was observed with NL replacement, where mix M16 (2% NL) recorded 116 mm, while M18 (4% NL) dropped to 109 mm.

Quaternary mixes with both NA and NL dropped slump the most. For example, mix M26 reached just 100 mm at L/B = 0.47. Adding NA and NL cut workability because their ultrafine particles and high surface area speed up nucleation and microstructure growth, but they also soak up more water. That extra water demand drags down flowability. These results line up with earlier studies [58,59], adding nanoparticles usually makes the mix less workable, since the particles clump together and boost the viscosity of the fresh mix.

Bulk density

Bulk density results in Table 7 show a steady increase as the material ages, which points to strong geopolymerization and a denser matrix over time. In the binary mixes, density climbs from 2334–2348 kg/m³ at 7 days to 2347–2367 kg/m³ at 28 days. This jump is even more noticeable in mixes with a lower L/B ratio (0.47), thanks to less water and tighter packing of the

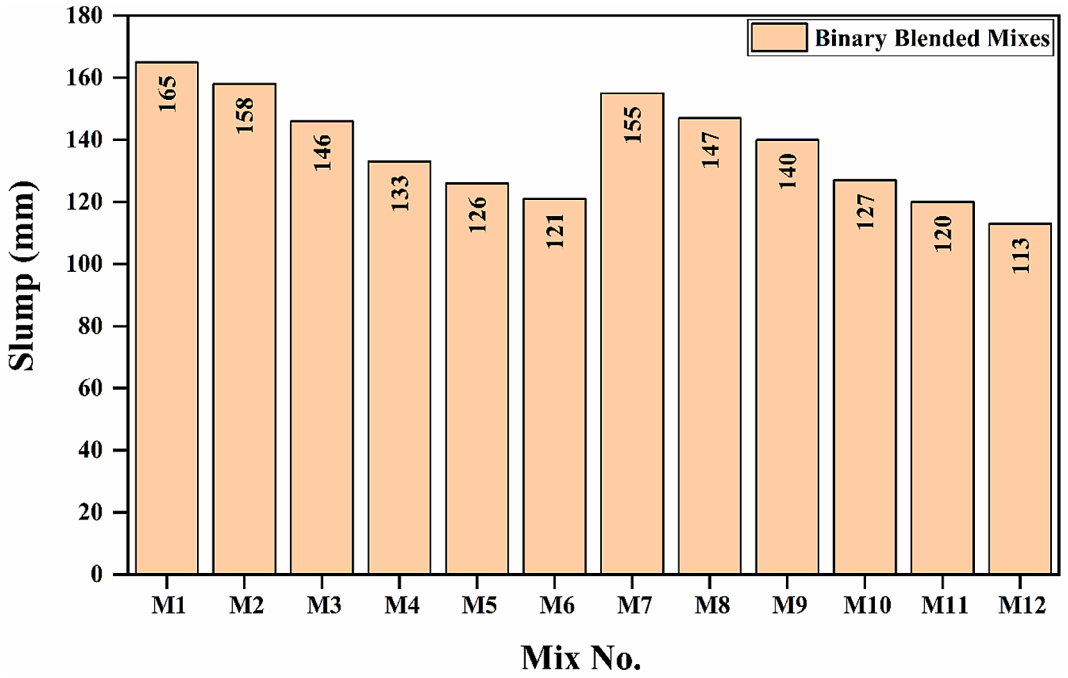


Figure 4. Slump results in binary blended mixes

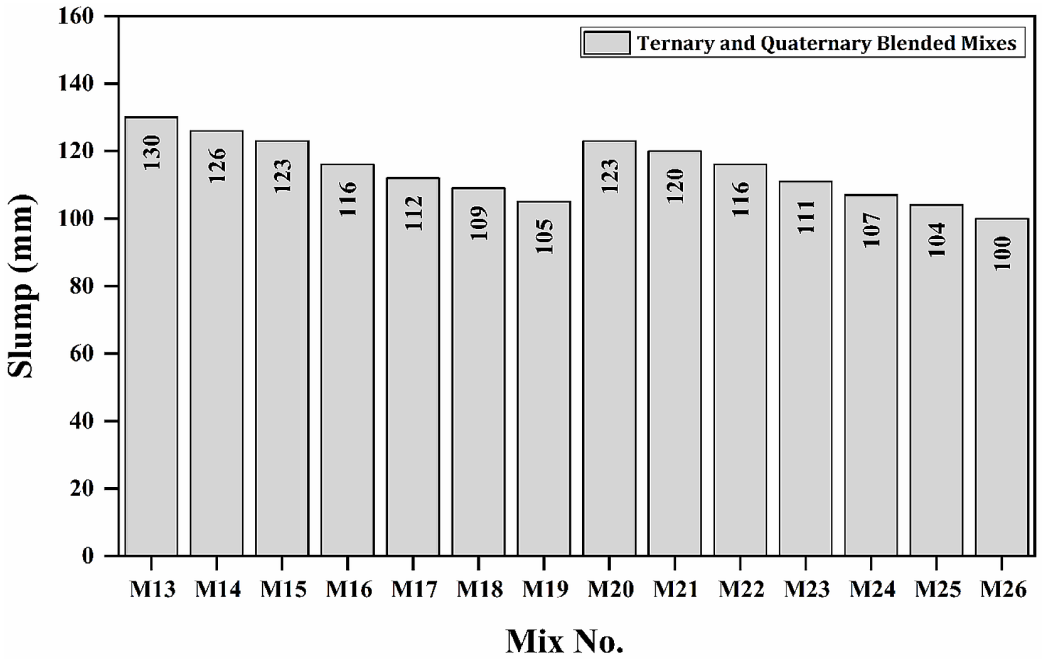


Figure 5. Slump results in ternary and quaternary blended mixes

Table 7. The results of bulk density tested of GPCs

Mix No.	Mix notation	Bulk density (kg/m³)	
		7days	28 days
M1	GP-100FA0MK-40	2334	2347
M2	GP-90FA10MK-40	2338	2349
M3	GP-80FA20MK-40	2341	2353
M4	GP-70FA30MK-40	2348	2367
M5	GP-60FA40MK-40	2347	2365
M6	GP-50FA50MK-40	2344	2361
M7	GP-100FA0MK-60	2343	2444
M8	GP-90FA10MK-60	2347	2447
M9	GP-80FA20MK-60	2349	2451
M10	GP-70FA30MK-60	2358	2458
M11	GP-60FA40MK-60	2354	2456
M12	GP-50FA50MK-60	2352	2453
M13	GP-70FA30MK-2NA0NL-40	2338	2389
M14	GP-70FA30MK-3NA0NL-40	2335	2386
M15	GP-70FA30MK-4NA0NL-40	2331	2379
M16	GP-70FA30MK-0NA2NL-40	2341	2393
M17	GP-70FA30MK-0NA3NL-40	2351	2397
M18	GP-70FA30MK-0NA4NL-40	2344	2391
M19	GP-70FA30MK-2NA3NL-40	2356	2406
M20	GP-70FA30MK-2NA0NL-60	2345	2478
M21	GP-70FA30MK-3NA0NL-60	2340	2470
M22	GP-70FA30MK-4NA0NL-60	2335	2471
M23	GP-70FA30MK-0NA2NL-60	2337	2472
M24	GP-70FA30MK-0NA3NL-60	2347	2481
M25	GP-70FA30MK-0NA4NL-60	2340	2467
M26	GP-70FA30MK-2NA3NL-60	2355	2489

reaction products. When it comes to ternary and quaternary blends, adding NA and NL nanoparticles boosts the bulk density even further. For instance, mix M19 (2% NA + 3% NL) reached 2406 kg/m³ at 28 days, while mix M26 (2% NA + 3% NL at L/B = 0.47) achieved 2489 kg/m³, representing the highest value among all mixes. This improvement matches what [60, 61], found – nanoparticles speed up geopolymerization and tighten up the pore structure, so you get denser, stronger material.

The results make it clear: if you want geopolymer concrete with higher bulk density and better mechanical strength, you need to manage how you add nanoparticles. Doing this shapes a more compact, refined microstructure that really boosts performance.

Compressive strength

The compressive strength results of the binary mixes (FA–MK) presented in Figures 6 and 7 demonstrate that incorporating metakaolin significantly enhanced strength development, particularly up to an optimum level of about 30% replacement. For the GP-40 series, strength increased gradually from 49.3 MPa at 90 days in the control mix M1 (100% FA) to 52.8 MPa in M4 (70% FA–30% MK). This improvement is mainly attributed to the high reactivity of metakaolin, which accelerates geopolymerization and promotes the formation of additional N-A-S-H and C-A-S-H gels, resulting in a denser matrix and reduced porosity. However, when MK content exceeded 30% (M5 and M6), the strength either stabilized or slightly declined (51.1–50.8 MPa at 90 days), due to the disruption in the Si/Al balance and the increased demand for alkalinity that could not be fully compensated, consistent with previous findings on FA–MK systems [62].

In contrast, the GP-60 series, which incorporated higher calcium content, exhibited a remarkable strength improvement starting from 28 days. The compressive strength increased sharply from 63.3 MPa in M7 to 66.3 MPa in M10 (30% MK), with further development reaching 71.4 MPa at 90 days. This behavior is attributed to the synergistic effect of slag, which provides reactive Ca^{2+} ions that facilitate the co-formation of C-(A)-S-H gel along with N-A-S-H gel, leading to a much denser microstructure and superior mechanical performance. To optimize gel formation, it is necessary to balance the calcium-rich precursors with aluminosilicate components due to the enhanced long-term strength of these mixes [63].

Notably, the GP-40 and GP-60 series show different trends: while GP-40 exhibits a reduction in strength beyond 30% MK replacement, GP-60 continues to improve up to 50% MK. This difference can be attributed to variations in binder chemistry, gel formation, and interaction with the alkaline activator, which influence the geopolymerization kinetics and the resulting microstructure. Similar observations have been reported in other studies, where higher-calcium systems or higher activator reactivity enhanced gel formation and strength development even

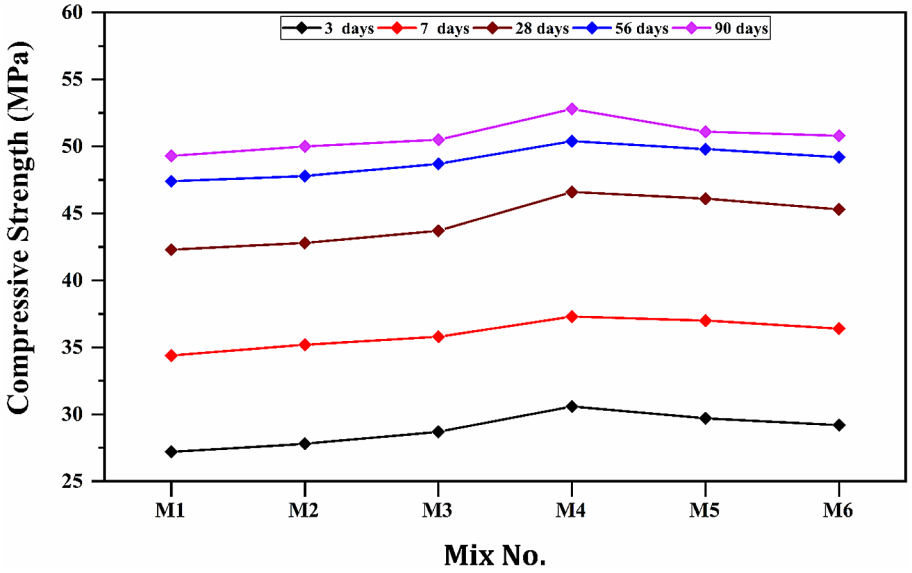


Figure 6. Compressive strength results of binary blended GP-40 mixes at different ages

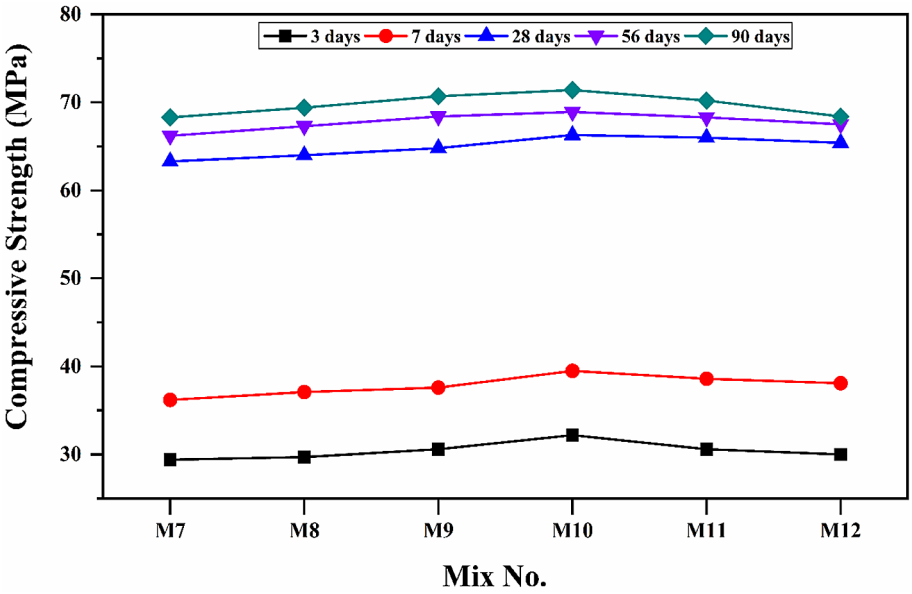


Figure 7. Compressive strength results of binary blended GP-60 mixes at different ages

at higher MK contents [64]. The compressive strength results of the ternary and quaternary mixes presented in Figures (8, 9) demonstrate the positive influence of incorporating nano-additives (NA and NL) into the FA–MK blends. For the GP-40 system, the results show that the addition of either Na_2O NA or NL improved the early- and later-age strengths compared to the binary blends. For example, mixes M13–M18 consistently outperformed the corresponding binary control (M4) at 28–90 days, achieving strengths in the range of 49–57 MPa, while M19 (containing both NA and NL) reached 58.7 MPa at 90 days, highlighting the synergistic effect of combining the two nanomaterials. This enhancement can be attributed to the nucleation effect of nanoparticles, which accelerates geopolymerization, promotes the formation of additional N-A-S-H and C-A-S-H gels, and refines the pore structure, leading to denser matrices.

For the GP-60 system, the strength development was even more remarkable. Ternary and quaternary mixes (M20–M26) showed substantial gains compared to the binary counterparts (M9–M12), with compressive strengths exceeding 70 MPa at 28 days and reaching up to 77.4 MPa in mix M26 at 90 days. The superior performance of these mixes is mainly attributed

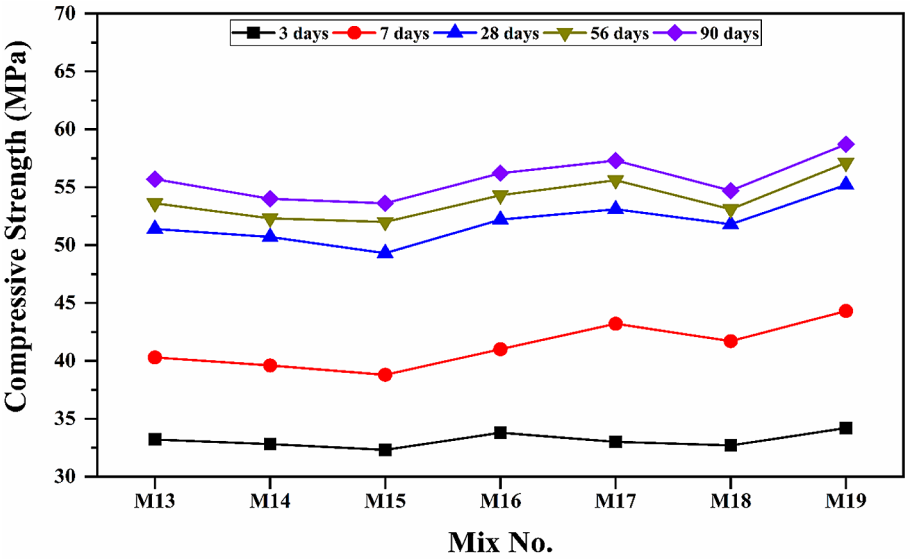


Figure 8. Compressive strength results of ternary and quaternary blended GP-40 mixes at different ages

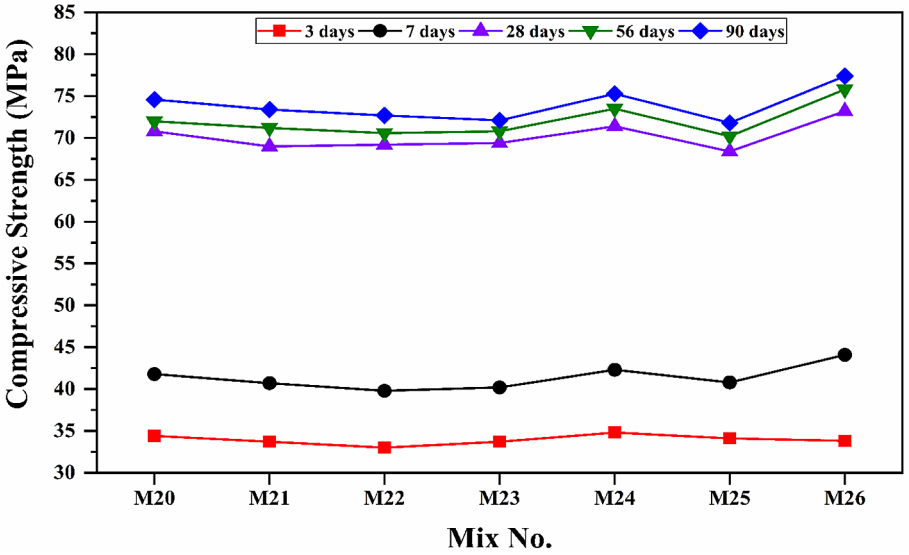


Figure 9. Compressive strength results of ternary and quaternary blended GP-60 mixes at different ages

to the high reactivity of MK in a calcium-rich environment, combined with the nano-induced densification of the matrix. The presence of NA and NL particles not only accelerated the dissolution of aluminosilicate precursors but also contributed to the refinement of the interfacial transition zone (ITZ), thereby improving the cohesion and load transfer capacity of the hardened paste. The observed improvements are consistent with published studies on nanomaterial incorporation in geopolymer systems, which report that nanoparticles act as nucleation centers and pore fillers, resulting in a compact microstructure with superior mechanical properties [65–67]. Furthermore, the continued strength gains up to 90 days aligns with previously reported geopolymer behavior, where these materials progressively develop strength due to the delayed hardening and evolution of the gel structure over time [64].

In general, the results can be summarized as follows: an optimal MK content (30%) enhances strength even in a low-calcium environment, while increasing slag/calcium content leads to rapid and significant improvements at all ages. Meanwhile, nanomaterials (NA and NL) play a crucial role in boosting strength and reducing porosity by enhancing microstructural packing and increasing the density of the geopolymer matrix.

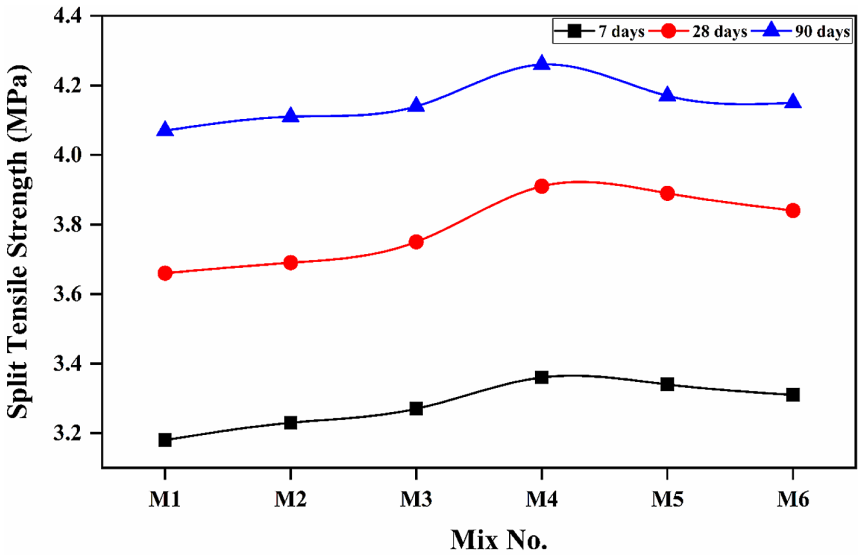


Figure 10. Split tensile strength results of binary blended GP-40 mixes at different ages

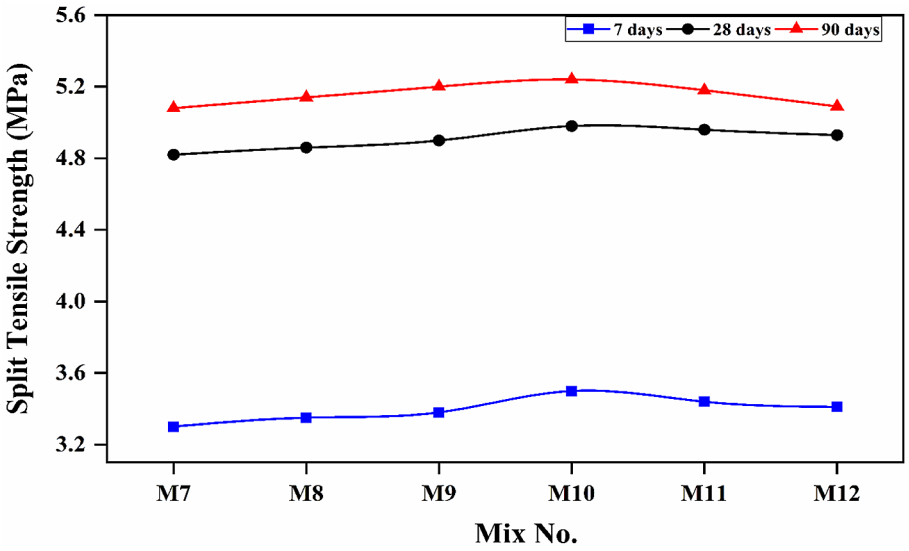


Figure 11. Split tensile strength results of binary blended GP-60 mixes at different ages

Split tensile strength

The split tensile strength results of the binary geopolymer mixes presented in Figures (10, 11) reveal distinct trends governed by the proportion of FA and MK as well as the curing age. At 7 days, all mixes exhibited comparable tensile strength values ranging from 3.18–3.50 MPa, indicating that early-age performance is primarily influenced by the initial dissolution of reactive aluminosilicates rather than by mix composition. However, clear differences appeared at 28 and 90 days.

For the GP-40 system (M1–M6), increasing MK content from 0% to 30% significantly enhanced tensile strength, with mix M4 (GP-70FA30MK-40) reaching 3.91 MPa at 28 days compared to 3.66 MPa in the control mix (M1, GP-100FA0MK-40). At 90 days, the improvement trend continued, with M4 achieving 4.26 MPa, reflecting the beneficial role of MK in accelerating geopolymerization reactions and promoting the formation of additional N-A-S-H and C-A-S-H gels. However, beyond 30% MK replacement, the strength gains plateaued, as seen in mixes M5 and M6, where higher FA replacement limited reactivity due to the lower calcium content of FA. This behavior is in line with like is in agreement in that excessive FA content results in slower geopolymerization kinetics which hinders the development of tensile strength [68].

The GP-60 system (M7–M12) really showed the impact of adding MK. Out of all the mixes, M10 (GP-70FA30MK-60) stood out with the highest tensile strength – 4.98 MPa at 28 days, climbing to 5.24 MPa at 90 days. That's a clear jump compared to M7 (GP-100FA0MK-60), which only hit 4.82 MPa at 28 days and 5.08 MPa at 90 days. This improvement can be ascribed to the greater amount of calcium that is present in the GP-60 system. This allows for quicker geopolymerization, leading to a denser microstructure which has a stronger interfacial bonding between the particles of the binder and the aggregates. Enhanced strength and durability have been reported from a better calcium content in geopolymer systems as described in [69].

Figures 12 and 13 show the split tensile strength results of the ternary and quaternary geopolymer mixes and their enhancements over the binary blends, especially at 28 and 90 days. At younger ages (7 days), the results of all mixes were comparable (3.45–3.78 MPa), however the impact of nanomaterials becomes clearer after continued curing. The addition of nano-alumina (NA) and nano-limestone (NL) improved tensile strength by refining the pore structure, filling microvoids, and enhancing particle packing, which reduced porosity and promoted stronger interfacial bonding within the geopolymer matrix.

The GP-40 series (M13–M19) achieved moderate improvements, with the best performance recorded in the hybrid mix M19 (GP-70FA30MK-2NA3NL-40), which reached 4.58 MPa at 90 days. In contrast, the GP-60 series (M20–M26) showed significantly higher strengths, confirming the positive role of higher calcium availability in accelerating geopolymerization. Among these, M22 (GP-70FA30MK-4NA0NL-60) recorded

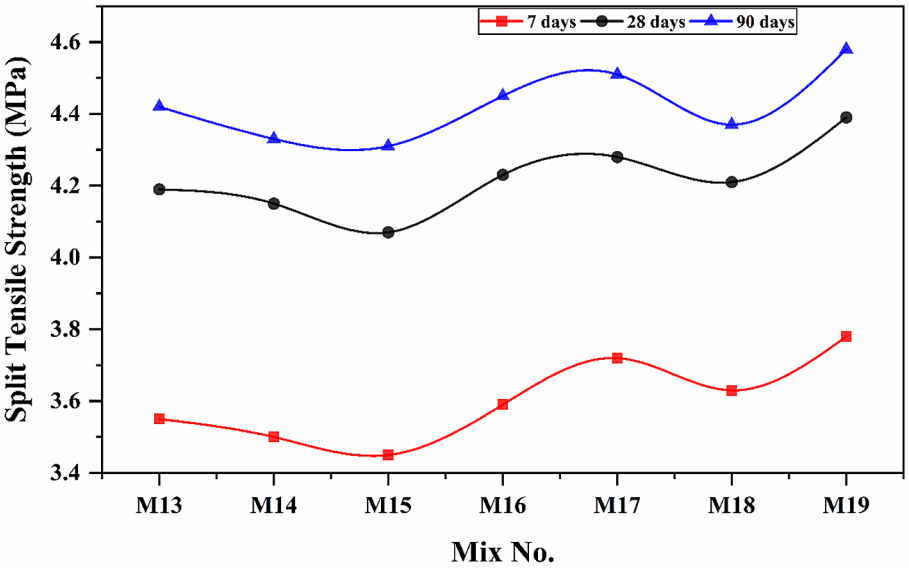


Figure 12. Split tensile strength results of ternary and quaternary blended GP-40 mixes at different ages

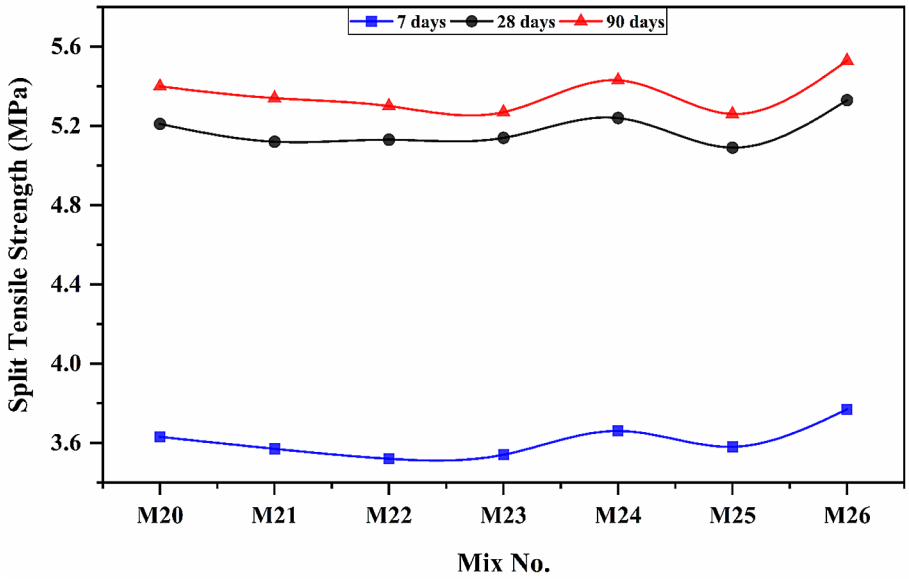


Figure 13. Split tensile strength results of ternary and quaternary blended GP-60 mixes at different ages

the highest strength of 5.30 MPa at 90 days, while M26 (GP-70FA30MK-2NA3NL-60) also performed strongly with 5.53 MPa.

Overall, the results confirm that the combined effect of nanomaterials (NA and NL) and higher MK content (GP-60) provides a synergistic improvement in tensile strength. This enhancement is attributed to the densification of the matrix, improved gel formation, and better stress distribution, in agreement with findings reported by [70].

The highest tensile strength was observed when high MK content was combined with nanomaterials, indicating a synergistic effect that promotes denser microstructure, minimizes microcracks, and maximizes mechanical performance. Overall, increasing MK content gradually improves split tensile strength with age, low-calcium FA requires longer curing to reach full strength, and nanomaterials significantly enhance both tensile performance and microstructural density. The combination of high MK and nanomaterials produces the best results, confirming the synergistic interaction between mix components.

Flexural tensile strength

The flexural tensile strength results in Figures 14, 15 show a clear distinction between the two

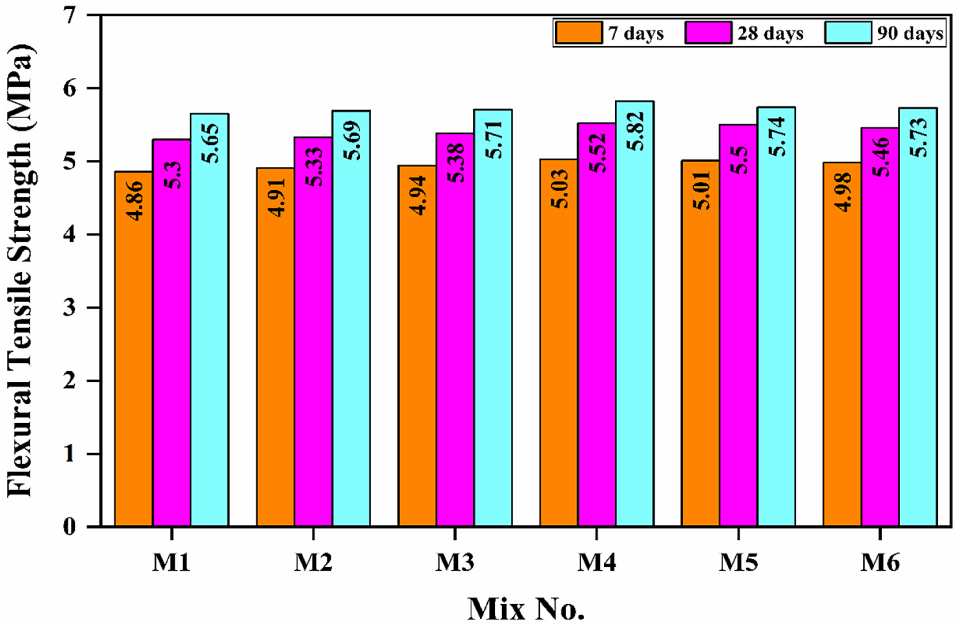


Figure 14. Flexural tensile strength results of binary blended GP-40 mixes at different ages

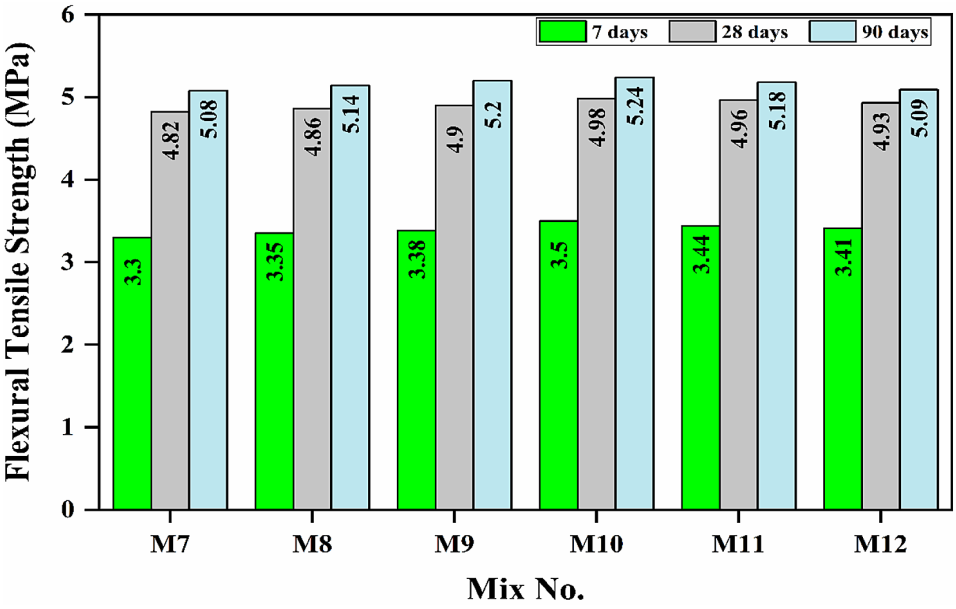


Figure 15. Flexural tensile strength results of binary blended GP-60 mixes at different ages

concrete grades: GP-40 and GP-60, and the effect of varying FA content on performance over time (7, 28, and 90 days).

For the GP-40 mixes, flexural strength values increased gradually with age, ranging from 4.86–5.03 MPa at 7 days, 5.30–5.52 MPa at 28 days, and 5.65–5.82 MPa at 90 days. The highest strength within this grade was observed for M4 (GP-70FA30MK-40), indicating that a balanced FA/MK ratio optimizes the microstructure and gel formation, enhancing matrix cohesion. Slightly lower values at higher FA replacements (40–50%) suggest a minor reduction in strength due to lower reactivity of fly ash and a possible reduction in matrix density. In the GP-60 mixes, the flexural strength was significantly higher at all ages, ranging from 4.97–5.15 MPa at 7 days, 6.28–6.40 MPa at 28 days, and 6.48–6.61 MPa at 90 days, with M10 (GP-70FA30MK-60) achieving the peak value of 6.61 MPa at 90 days. The improvement in GP-60 compared to GP-40 can be attributed to the higher overall binder content and greater compressive strength grade, which enhances the formation of a denser and more

cohesive geopolymer matrix, providing superior flexural performance. The results are consistent with previous findings reported in [71, 72], which confirmed that increasing MK content enhances both the mechanical performance and durability of geopolymer concrete. As shown in Figures 16, 17, the inclusion of nanomaterials such as NA and NL further enhances flexural performance by acting as micro-fillers that reduce porosity and reinforce the interfacial transition zone (ITZ) between aggregates and the matrix, improving bonding and reducing cracking potential [67, 73, 74]. Also, the continuous improvement in strength over the years represents the further geopolymerization that continues to strengthen the aluminosilicate framework and improves structural consolidation [75, 76]. In general, owing to the synergistic effects of the incorporation of MK and nanomaterials that fine tune the microstructural packing and improve the mechanical properties, ternary and quaternary blends have better flexural performance than the binary blends [77].

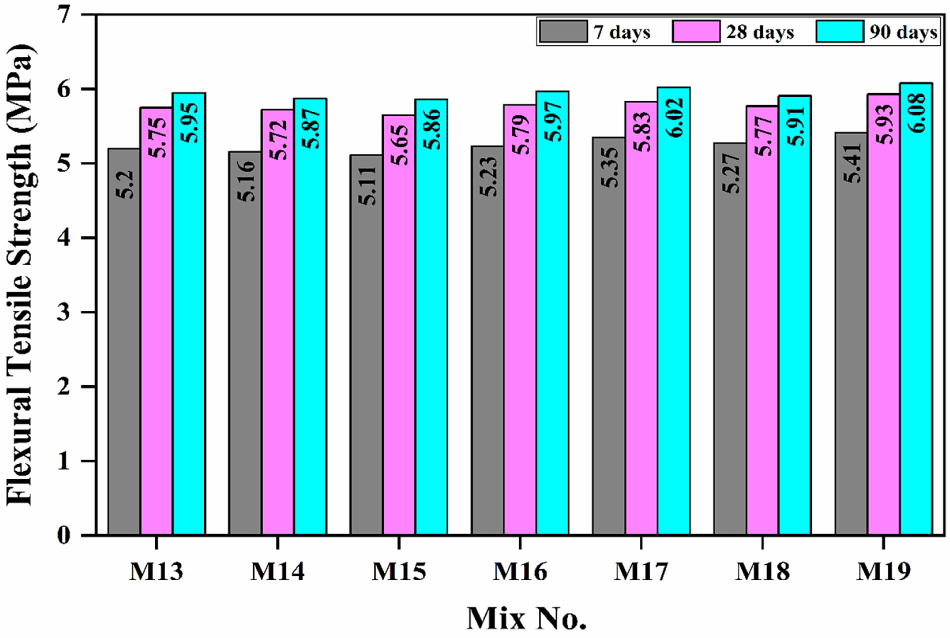


Figure 16. Flexural tensile strength results of ternary and quaternary blended GP-40 mixes at different ages

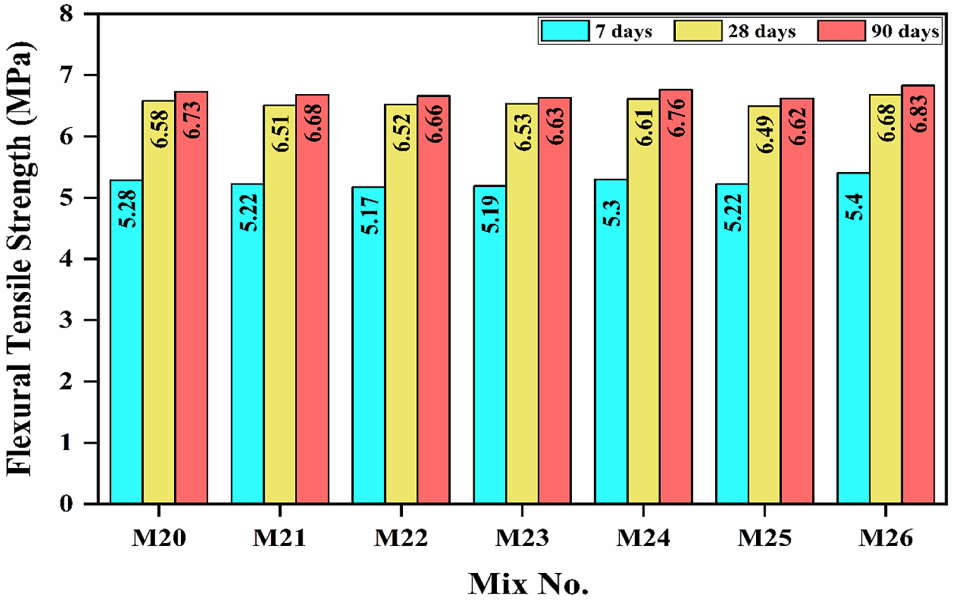


Figure 17. Flexural tensile strength results of ternary and quaternary blended GP-60 mixes at different ages

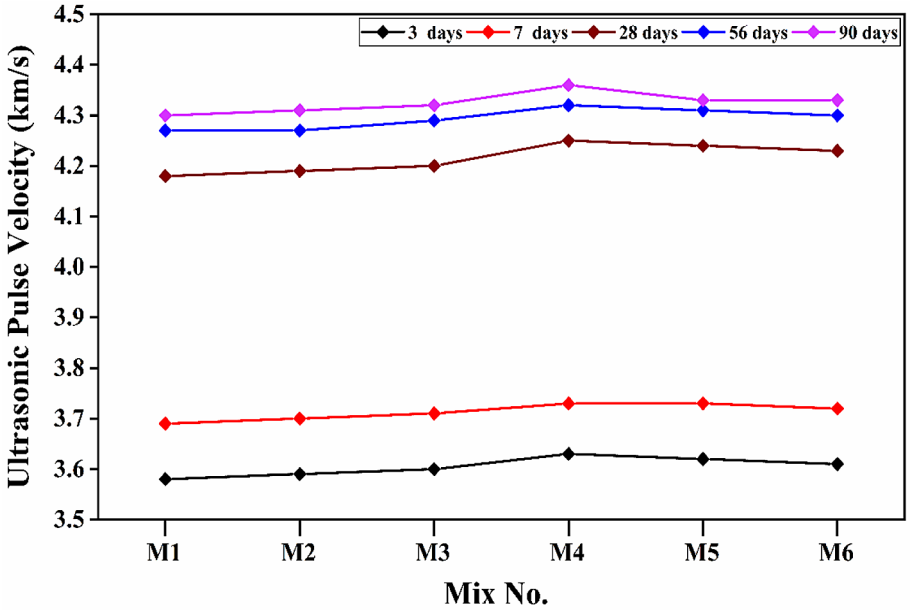


Figure 18. UPV results of binary blended GP-40 mixes at different ages

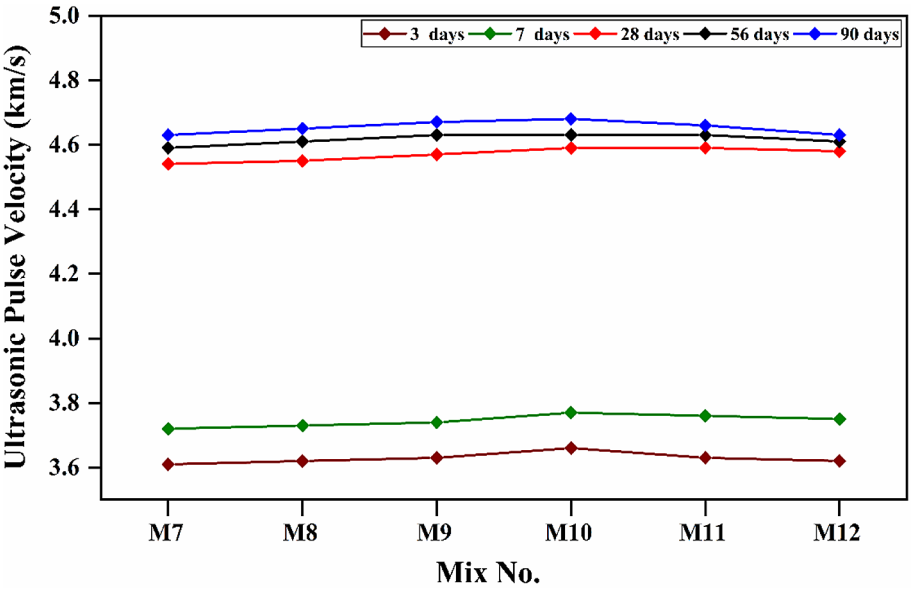


Figure 19. UPV results of binary blended GP-60 mixes at different ages

Ultrasonic pulse velocity

Ultrasonic pulse velocity (UPV) studies of the binary mixes (Figures 18 and 19) show a gradual increase in UPV values with age from 3 to 90 days. This continuous increase is attributed to the gradual geopolymerization of reactions that improve the internal cohesion of a matrix and subsequently result in formation of denser and compact microstructure during the hydration process [78, 79].

In terms of the effect by low grade of concrete, UPV values measured from the GP-60 mixes were consistently larger than those from the GP-40 mixes at all ages. For example, mix M3 (GP-40, FA 20%) achieved 4.32 km/s at 90 days, whereas M9 (GP-60, FA 20%) reached 4.67 km/s. This trend indicates that higher strength grades contribute to a more compact and cohesive matrix, improving particle bonding and minimizing internal voids.

In terms of FA (fly ash) content, although the increase in fly ash added up to 50% in GP-40 mixes contributed slightly to an advancement at most ages. This is related to the poor reactivity of FA compared with GGBS or MK, especially

in early age curing, but it helps create a finer geopolymeric gel. In the case of FA, it was more effective in the GP-60 mix with relatively higher UPV values up to 28–90 days also. For instance, mix M10 (GP-60, FA 30%) attained 4.68 km/s at 90 days. This behavior demonstrates a synergistic effect between FA and higher strength grades, improving matrix compactness and structural homogeneity.

Overall, all mixes showed continuous improvement in UPV values over time, indicating progressive matrix densification. The consistently higher values recorded for GP-60 mixes confirm that higher strength grades enhance internal integrity and durability. These observations align with previous studies [13], which reported that combining FA with highly reactive precursors such as GGBS or MK increases matrix density, strengthens the interfacial transition zone (ITZ), and refines the pore structure – resulting in improved UPV performance.

The results of the ternary and quaternary mixes (Figures 20 and 21) further support these findings. Mixes containing nano-additives (NA or NL) exhibited significantly higher UPV values than the binary ones, particularly at later ages (>28 days). Among these, the quaternary mixes incorporating both NA and NL (e.g.,

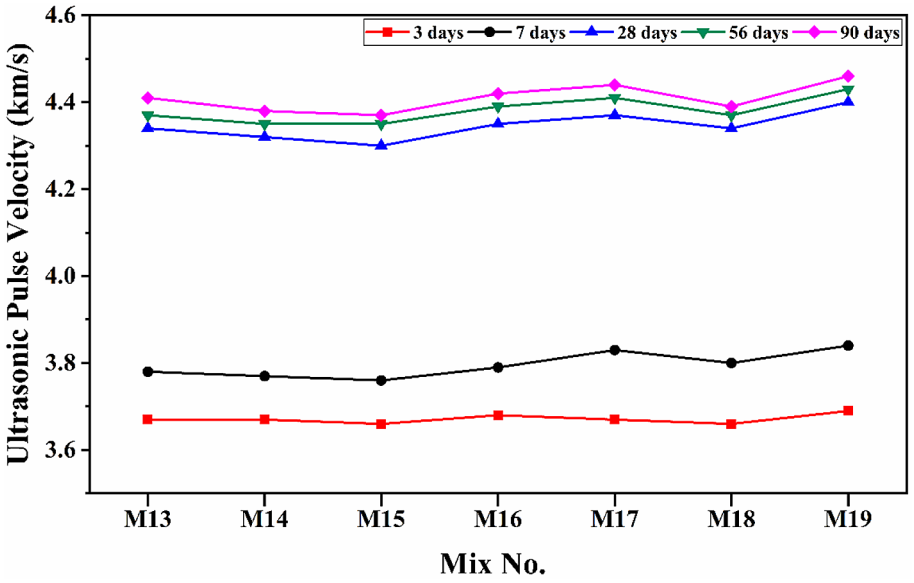


Figure 20. UPV results of ternary and quaternary blended GP-40 mixes at different ages

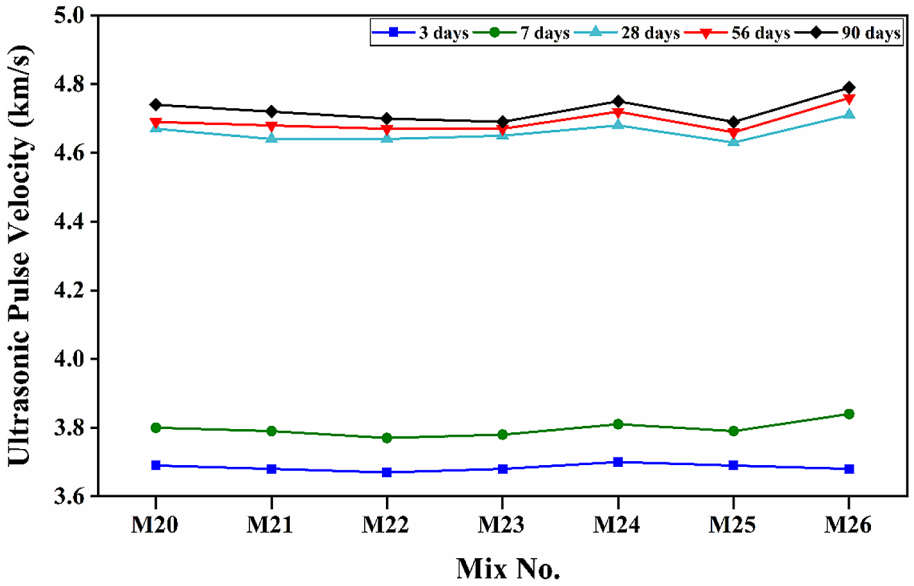


Figure 21. UPV results of ternary and quaternary blended GP-60 mixes at different ages

M19 and M26) achieved the highest UPV values, suggesting that nano-additives play a key role in improving microstructural packing, reducing fine porosity, and thereby enhancing ultrasonic conductivity.

This improvement is consistent with international research [80], which demonstrates that nano-additives act as ultra-fine fillers that increase density and strengthen the ITZ between aggregates and the binder matrix. As the proportion of MK increased or nano-additives were introduced, the internal matrix density improved while overall porosity decreased, resulting in higher UPV values. The elevated UPV readings at 90 days confirm the ongoing enhancement of the geopolymer matrix over time, especially in the quaternary systems combining high MK, FA, and nano-additives (NA/NL).

CONCLUSIONS

The experimental investigation evaluated the mechanical performance of geopolymer concrete incorporating fly ash (FA), metakaolin (MK), and nanomaterials, using compressive, tensile, flexural, and ultrasonic pulse velocity (UPV) tests to provide a comprehensive understanding of the effects of mix composition and curing age on material properties.

Based on the research conducted, the following results were obtained:

1. The incorporation of nano-alumina (NA) and nano-limestone (NL) significantly improved both early- and later-age compressive strength by enhancing microstructural packing, reducing porosity, and promoting densification of the geopolymer matrix.
2. A metakaolin replacement level of 30% was identified as optimal for both low- and high-calcium systems, as it accelerated the geopolymerization process, promoted additional gel formation (N-A-S-H and C-(A)-S-H), and increased overall mechanical performance.
3. Compressive strength of quaternary and ternary blends of MK with nanomaterials was the highest (up to 77.4 MPa at 90 days), with the best split tensile strength indicating a synergetic effect, increasing load bearing capacity and decreasing microcracks.
4. Flexural strength was also significantly improved in premixes with both MK and nanomaterials, due to enhanced interfacial bonding, lower cracking tendency and more compact and uniform matrix.
5. UPV results verified time-dependent densification of the geopolymer matrix with the highest values given for quaternary mixes, thus emphasizing the suitability of MK and nanomaterials as modifiers to improve the internal cohesion and performance of geopolymer concrete.

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