

AKADÉMIAI KIADÓ



UNIVERSITY of
DEBRECEN

International Review of
Applied Sciences and
Engineering

15 (2024) 1, 44–54

DOI:

10.1556/1848.2023.00638

© 2023 The Author(s)

ORIGINAL RESEARCH
PAPER



Assessment of mechanical and durability performance of silica fume and metakaolin as cementitious materials in high-performance concrete

B. Sankar* and P. Ramadoss

Department of Civil Engineering, PTU, Puducherry, 605014, India

Received: March 2, 2023 • Accepted: July 3, 2023

Published online: September 28, 2023

ABSTRACT

The present study aims to determine the effects of blending cementitious materials on the mechanical and durability properties of high-performance concrete (HPC). Densified silica fume and fine-grounded metakaolin are used as supplementary cementitious materials (SCMs). A total of 16 mixes containing both binary and ternary blending of SCMs were chosen for w/b ratios of 0.4 and 0.3 respectively. The hardened properties tested for the HPC mixes were compressive strength at 7, 28, and 90 days, flexural strength at 28 days, and modulus of elasticity at 28 days. Maximum strength gains up to 15%, 38%, and 23% for compression, flexure, and elastic modulus were observed in ternary mixes compared to binary mixes. Stress-strain behaviour of ternary mixes indicates increased tolerance of stress for the least amount of strain in the specimens. Based on the experimental results, empirical relations were developed and checked with the existing codes and by earlier researchers. The durability properties tested for HPC were water absorption at 28 days, acid attack, and sulphate attack at 28, 56, and 90 days. Ternary mixes improved the pore structure of HPC, resulting in a 56% reduction in water absorption and a 34% reduction in compressive strength loss due to immersion in 5% H₂SO₄ at 90 days. The findings of the study endorse that ternary blending of SF and MK can improve the engineering properties of HPC, and a mix containing SF 10% and MK 10% is recommended for the best results.

KEYWORDS

high-performance concrete, metakaolin, silica fume, ternary blended concrete, acid attack, empirical relations

1. INTRODUCTION

Cement consumption in construction sectors is rapidly increasing around the world, consequently escalating the CO₂ emissions during cement manufacturing. Excessive utilization of raw materials raises global concerns over the search for alternate sustainable and environmentally friendly materials [1]. Supplementary cementitious materials (SCMs) such as silica fume, metakaolin, blast-furnace slag, fly ash, rice husk ash, etc. are by-products of production industries that are being used as cement replacements due to their ecological benefits. High-performance concrete (HPC) necessarily contains such SCMs as part of its exclusive design for the durability and sustainability of structures by tailing off the necessity for repair and rehabilitation in the long run. The addition of ultrafine SCMs such as SF and MK in HPC shows improved strength and durability properties [2–4]. The addition of SF helps in the early development of strength in concrete due to its higher rate of pozzolanic activity. The maximum strength improvement attained for SF concrete was for a 15% replacement of cement [5, 6]. In addition, the finer particle size of SF helps in reducing the pore volume by acting as filler and by forming a secondary C-S-H gel, ultimately leading to a reduction in harmful agents penetrating the concrete [7]. SF addition in concrete tends to reduce mass loss when introduced to an acidic environment [8]. Jagan and Neelakantan [9]

*Corresponding author.

E-mail: samkarboomibalan180@pec.edu



studied the effects of immersing SF concrete in an acidic medium containing 5% HCl and concluded that the mix containing 15% SF provided better acid resistance with minimal compressive strength loss. MK, on the other hand, is highly reactive and provides excellent strength and durability properties when added to concrete [10]. MK addition was limited to 10%, beyond which the matrix showed poor workability and reduced strength [11]. Dubey et al. [12] observed an increase in compressive strength, flexural strength, and electrical resistance for MK-added HPC. Even though the benefits of SCMs in concrete are widely discussed, some may possess shortcomings such as inadequate early strength, challenges in case of higher replacement levels, and availability of key constituents in forming C-S-H gel for pore refinement. One possible way of mitigating this limitation is through the blending of SCMs. In a study involving HPC, SF and MK were added in the fly ash concrete to improve early-age strength development [13, 14]. Ahmed et al. [15] observed a reduction in chloride ion penetration in concrete, containing 25% fly ash and 10% SF. Jung et al. [16] reported the benefits of ternary blending of cementitious materials in reducing the heat of hydration in mass concretes. Acid resistance is an important durability index when the concrete is being used in harsh environments such as sewer linings and industrial effluent pathways where the pH levels of the wastewater are low. The acid resistance of FA and SF ternary blended concrete was superior compared to the binary mixes [17]. Low-density high porous foam concrete made with blending of SF and MK showed enhancement in mechanical properties [18]. The dense particle packing also helps in bringing out the unreacted water molecules present in voids for the lubrication of binder materials [19]. Chu et al. [20] studied the synergistic effects of using SF and MK in particle packing and observed improved mechanical performance of mortars. With the acquired knowledge in the area of ternary blending of cementitious materials, the present study aims to investigate the effects of ultrafine SCMs such as SF and MK on the mechanical performance, stress-strain relationship, and durability performance of HPC which seems to be absent in most of the studies. Two different w/b ratios of 0.3 and 0.4 with similar mixes were chosen to investigate the effect of strength grades on the mechanical and durability properties of HPC. The experimental studies included in the present work are compressive strength, modulus of elasticity, flexural strength, water absorption, acid attack, and sulphate attack. Following the experimental program, empirical relations between mechanical properties were also investigated.

2. MATERIALS AND METHODS

2.1. Materials

Ordinary Portland cement of 53-grade with a compressive strength of 54.5 MPa at 28-day and a specific gravity of 3.14 conforming to IS 12269 [21] is used. Silica fume and

metakaolin obtained from Astrra Chemicals Pvt Ltd. conforming to IS 15388 [22] and IS 16354 [23] were taken as binder materials. Particle size distribution and chemical compositions of OPC, SF, and MK are shown in Fig. 1 and Table 1 respectively. Manufactured sand conforming to zone II of sieve passing percentage from IS: 383-1970 [24] is used as fine aggregate. Coarse aggregate with nominal size not less than 12.5 mm is used. Polycarboxylate ether based high-range water reducer conforming to IS: 9103-1999 [25] with a specific gravity of 1.2 ± 0.05 is used as a superplasticizer. The physical properties of the materials from the current study are well within the limits of Bureau of Indian Standards (BIS) specifications and are presented in Table 2.

2.2. Mix details

Mixture proportioning was carried out using the guidelines recommended by ACI 211.4R-93 [26]. Two sets of 8 mixes for w/b ratios of 0.3 and 0.4 were chosen for the study. The mixes contained both binary and ternary mixes of SF and MK with a maximum cement replacement of 20%. The dosage of superplasticizer was varied to attain slump values of more than 75 mm. Fine aggregate adjustments were carried out for every percentage replacement of SCM. The mixture details of concrete are presented in Table 3. A total of 3 samples per mix were cast and the average of the sample results were taken as the final result for compressive strength, flexural strength, modulus of elasticity, and all the

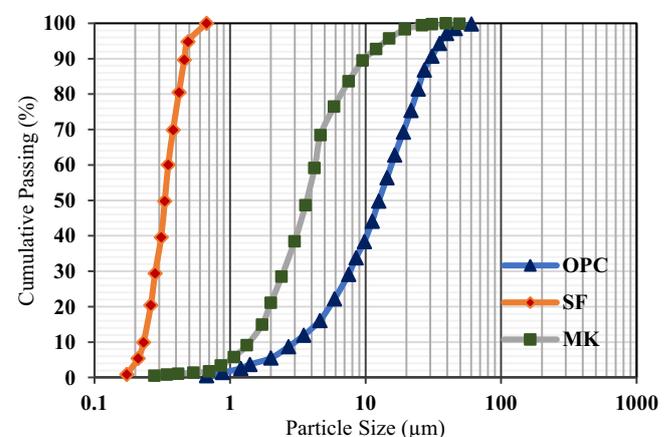


Fig. 1. Particle size distribution of cementitious materials

Table 1. Chemical composition of OPC, SF, and MK

Chemical composition	OPC	SF	MK
SiO ₂	21.07	92.06	57.4
Al ₂ O ₃	5.54	0.48	35.26
CaO	64.26	0.4	0.02
Fe ₂ O ₃	5.16	2.11	0.94
MgO	0.86	0.63	0.18
K ₂ O	0.37	1.24	3.17
P ₂ O ₅	<0.9	0.02	0.09
Others	1.2	0.52	0.42
Loss on ignition	1.54	2.54	2.52



Table 2. Physical properties of constituent materials

S No.	Raw material	Property	Tested value	BIS limits
1	Cement	Specific gravity	3.14	3.15
		Initial setting time (mins)	68	≥30
		Final setting time (mins)	220	≤600
2	SF	Specific gravity	2.21	2.20 to 2.30
		Surface area (m ² kg ⁻¹)	22,000	>15,000
		Bulk density (kg m ⁻³)	650	>500
3	MK	Specific gravity	2.5	–
		Surface area (m ² kg ⁻¹)	14,000	>9,000
		Bulk density (kg m ⁻³)	900	–
4	Water	pH	8	>6.00
5	Manufactured sand (FA)	Specific gravity	2.59	2.50–3.00
		Fineness modulus	2.9	2.00–4.00
		Water absorption (%)	1.48	0.30–2.50
6	Gravel (CA)	Specific gravity	2.66	2.50–3.00
		Fineness modulus	7.25	6.75–8.00
		Water absorption (%)	1.12	<2.00

durability properties tested in the present study. The specimens were cured in water at $27 \pm 2^\circ\text{C}$ and 95% relative humidity. Details of specimen preparation and testing mechanisms are shown in Fig. 2.

2.3. Testing methods

2.3.1. Slump test. The fresh state property was determined using the slump test to measure the ease of placement of concrete. The test was carried out as per the specifications provided by ASTM C143 [27].

2.3.2. Compressive strength. The compressive strength test was carried out on cube specimens after a curing period of 7, 28 and 90 days. The test was carried out on cube specimens of size $100 \times 100 \times 100$ mm in accordance with the specifications provided by ASTM C469 [29].

2.3.3. Flexural strength. The flexural strength test was carried out after a curing period of 28 days. The test was carried out on prism specimens of size $500 \times 100 \times 100$ mm in a four-point bending setup following the specifications provided by ASTM C78 [28].

2.3.4. Modulus of elasticity. Modulus of elasticity of the concrete specimens was carried out after a curing period of 28 days. The test was carried out on cylindrical specimens of size 150×300 mm in accordance with the specifications provided by ASTM C469/C469M-14 [29]. From the results,

Table 3. Mixture details of HPC

Mix No.	Mixes	w/b ratio	Binder (kg m ⁻³)	Cement (kg m ⁻³)	SF (kg m ⁻³)	MK (kg m ⁻³)	Fine aggregate (kg m ⁻³)	Coarse aggregate (kg m ⁻³)	Water (kg m ⁻³)	SP (kg m ⁻³)
M1	Control	0.4	450	450	0	–	635.00	1,099	179	0.00
M2	SF 15		450	382.5	67.5	–	582.11	1,099	179	1.15
M3	MK 10		450	405	–	45	582.96	1,099	179	1.73
M4	SF-5 MK-10		450	382.5	22.5	45	607.65	1,099	179	1.90
M5	SF-10 MK-5		450	382.5	45	22.5	596	1,099	179	1.70
M6	SF-15 MK-5		450	360	67.5	22.5	567	1,099	179	1.80
M7	SF-5 MK-15		450	360	22.5	67.5	591	1,099	179	2.30
M8	SF-10 MK-10		450	360	45	45	579.15	1,099	179	2.00
M9	Control	0.3	555	555	0	–	699.00	1,088	167	1.15
M10	SF 15		555	471.75	83.25	–	632.09	1,088	167	1.73
M11	MK 10		555	499.5	–	55.5	634.82	1,088	167	2.54
M12	SF-5 MK-10		555	471.75	27.75	55.5	582	1,088	167	2.00
M13	SF-10 MK-5		555	471.75	55.5	27.75	574	1,088	167	2.00
M14	SF-15 MK-5		555	444	83.25	27.75	555	1,088	167	2.40
M15	SF-5 MK-15		555	444	27.75	83.25	570	1,088	167	3.20
M16	SF-10 MK-10		555	444	55.5	55.5	562	1,088	167	3.00





Fig. 2. Casting and testing of concrete specimens

the stress-strain relationship of the mixes in the present study was developed.

2.3.5. Water absorption. Water absorption values for the binary and ternary mixes were determined according to ASTM C1585-13 [30] using the $100 \times 100 \times 100$ mm cube specimens. The specimens were preconditioned in a hot oven and the dry weight was noted before immersing it in water. The percentage weight of water absorbed by the sample is measured by calculating the difference in weight of the water-immersed sample and oven-dried sample to that of the oven-dried sample.

2.3.6. Acid attack. For the acid attack test, H_2SO_4 solution was used to provide the acidic medium for the concrete specimens. The test was carried out in accordance with ASTM C1898-20 [31]. Water with 3 times the volume of the cube specimens was taken in a tub and in that 5% H_2SO_4 by volume of the total water was poured. The cube specimens were taken after 28-day curing period and the surface moisture was removed using a dry towel. The specimens were weighed before immersing in the acidic solution for 28, 56, and 90 days, after which a loss in mass and compressive strength of the specimens was found.

2.3.7. Sulphate attack. For sulphate attack test, Na_2SO_4 solution was used as sulphate medium for the concrete specimens. The test was carried out in accordance with ASTM C1012 [32]. Water with 3 times the volume of the

cube specimens was taken in a tub and in that 5% Na_2SO_4 by volume of the total water was mixed. The cube specimens were taken after 28-day curing period and immersed in the solution for 28, 56, and 90 days, after which a loss in mass and compressive strength of the specimens was found.

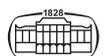
3. RESULT AND ANALYSIS

3.1. Fresh concrete property

The workability of concrete is an important factor that relates directly to the mechanical property and durability of the structure. It is the measure of ease in placement with which there will be the least amount of segregation and bleeding without loss in homogeneity. The dosage of superplasticizer is adjusted so that the slump values are more than 75 mm. The slump details of the mixes are mentioned in Fig. 3. In the case of both SF and MK, with an increase in percentage replacement, the water demand also increased due to the finer particle size and increased surface area. The water demand for MK was more compared to SF due to the cohesive nature of MK particles. The ternary blending of SF and MK improved the workability of the mixes.

3.2. Hardened properties of HPC

3.2.1. Compressive strength. The compressive strength test of the specimens was carried out after a curing period of 7,



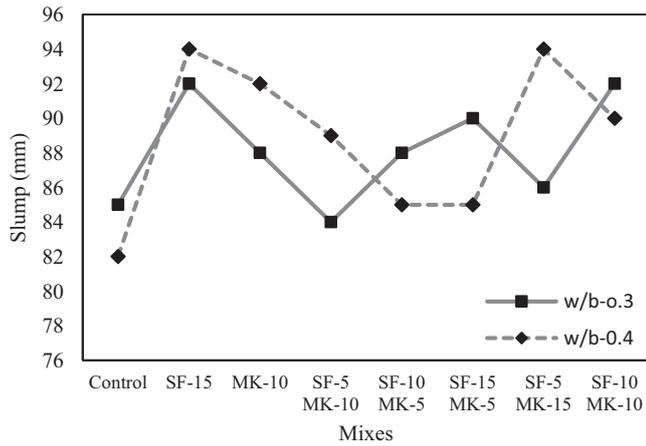


Fig. 3. Slump values of the concrete mixes

28, and 90 days. The maximum replacement levels that provide ultimate strength for binary SF and MK mix were chosen from the literature. The compressive strength results of the mixes tested under a universal testing machine are presented in Table 4. The development in the strength of the mixes with curing age is also shown in Fig. 4. From the figure, it is observed that for w/b ratios of 0.3 and 0.4, the SF binary mix with 15% replacement of cement resulted in a strength increment of 13.1% and 20.8% with respect to the reference mix. In the case of MK binary mix, the strength increment for a 10% replacement was observed to be 17.3% and 23.1% for w/b ratios of 0.3 and 0.4. Ternary mixes containing a total of 20% replacement of SF and MK showed superior strength properties compared to a net 15% replacement. A ternary mix containing SF10% and MK 10% showed increments in strength up to 29.7% and 30.2% with respect to the reference mix, 14.7% and 7.8% increment

with respect to binary SF 15% mix, and 10.5% and 5.8% increment for binary MK 10% mix for w/b ratio of 0.3 and 0.4. The development of strength for ternary mixes was more pronounced in mixes with a lower w/b ratio. The compressive strength increment for the 28-day curing period of the mixes M8 and M16 from the present study was significantly large compared to a 10% increment for ternary mixes containing 10% RHA and 10% FA [33], 26.6% increment for a mix containing 10% SF and 10% ultrafine GGBS [34] and 17.6% increment for a mix containing 10% AL and 20% FA [35] with respect to the reference mix.

3.2.2. Flexural strength. The flexural strength test was carried out on prism specimens using a four-point bending loading. The tested results for all the mixes after 28-day curing are presented in Fig. 5. From the figure, a maximum of 60% and 48% increment in flexural strength of ternary mixes is noticed for w/b ratio of 0.3 and 0.4 compared to the reference mixes. Ternary mixes with a net 20% replacement level showed better results compared to a net 15% replacement of cement. A ternary mix containing SF 10% and MK 10% showed 37.8% and 22.3% increased bending strength compared to binary SF 15% mix for w/b ratios of 0.3 and 0.4. Similarly, the ternary mix of SF 10% and MK 10% showed 35.3% and 19.5% increased bending strength for w/b ratios of 0.3 and 0.4 compared to the binary MK 10% mix. The experimental results showed improved performance when compared with similar studies containing 10% AL + 20% FA [35] and mortar mix with 10% SF + 10% MK [20], where an increment in flexural strength of 10.6% and 30% was observed with respect to reference mix. The flexural strength of the mixes are in relation with the compressive strength and show similar trends. The enhancement in flexural strength of the ternary mixes is attributed to more

Table 4. Experimental results of mechanical and durability tests of concrete mixes

Mix No.	Compressive strength (MPa)		Flexural strength 28-day (MPa)	Young's modulus 28-day (GPa)	Water absorption 28-day (%)	Compressive strength loss in 5% H ₂ SO ₄ at 90-day immersion (%)	90-day compressive strength after immersion in 5% Na ₂ SO ₄ solution (MPa)
	28-day	90-day					
M1	50.12	52.81	4.92	28.67	4.02	48.84	44.80
M2	60.56	64.78	5.96	30.27	2.95	31.20	60.12
M3	61.70	66.00	6.10	32.43	3.40	33.94	60.00
M4	58.50	61.71	6.05	32.95	2.98	33.60	58.40
M5	56.50	59.48	5.94	32.33	2.64	32.45	57.70
M6	59.89	62.92	6.94	33.72	2.37	29.78	60.78
M7	62.90	65.95	6.67	34.81	2.65	33.13	63.14
M8	65.30	68.33	7.29	35.46	2.13	28.65	68.40
M9	73.90	78.25	6.59	32.67	3.72	44.94	67.98
M10	83.60	89.60	7.95	35.27	2.57	29.60	84.46
M11	86.70	93.02	8.10	36.43	2.80	32.23	86.10
M12	84.36	89.22	8.76	35.95	2.41	31.83	86.30
M13	83.32	87.93	8.00	35.33	2.11	30.90	85.12
M14	87.50	92.16	8.87	38.72	1.95	25.56	92.00
M15	93.10	97.86	9.60	41.81	1.99	30.70	93.20
M16	95.92	100.62	9.96	43.16	1.64	24.93	101.06



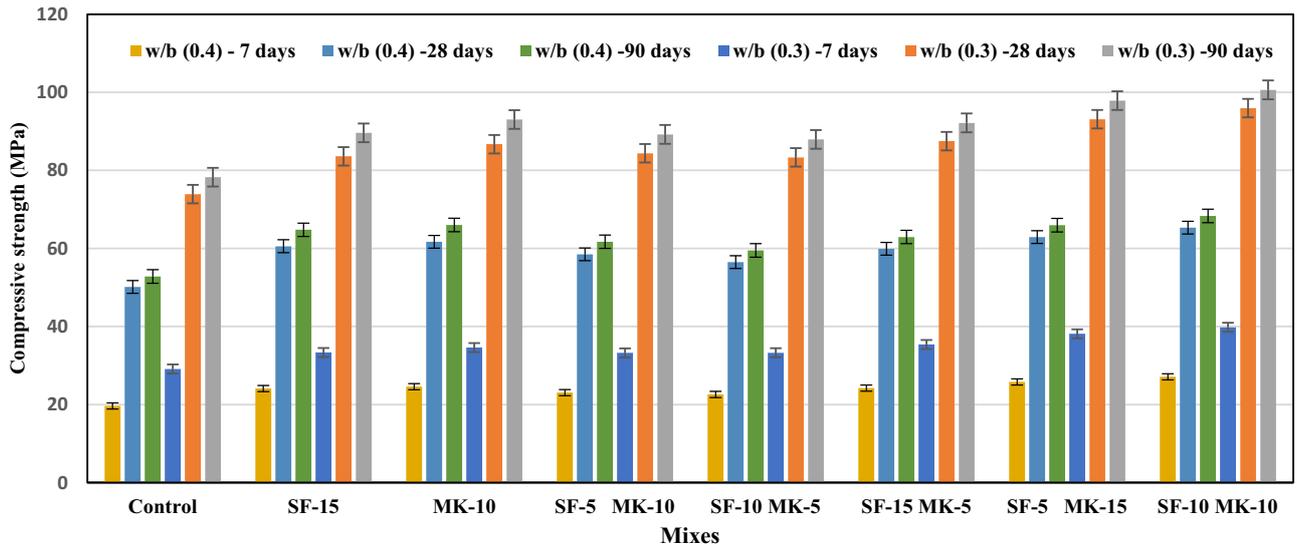


Fig. 4. Compressive strength variation of concrete mixes with curing ages

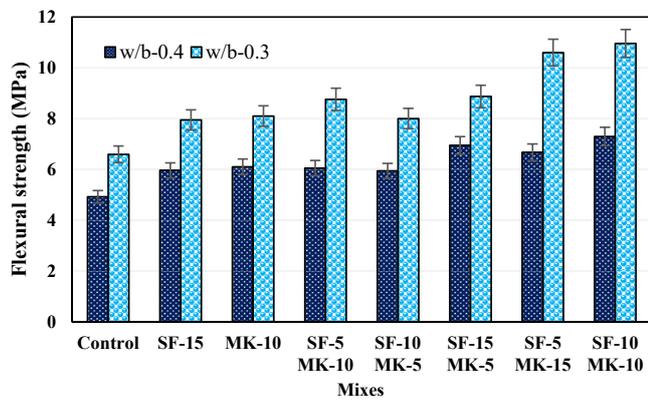


Fig. 5. Flexural strength results of concrete mixes

volume of ettringite formation leading to an increase in the bond between aggregate and matrix, which is considered the weakest link in concrete.

3.2.3. Modulus of elasticity. The modulus of elasticity of concrete represents the extent to which the specimen can take elastic deformation. Cylindrical specimens were used for testing under the universal testing machine. For every gradual loading, the strain values were recorded using a dial gauge mounted on the specimen. The stress-strain relationship of the mixes from the present study is shown in Fig. 6. The values of the modulus of elasticity of the mixes at 28 days using the secant modulus are mentioned in Table 2. From the figure, it is observed that both binary and ternary mixes showed higher tolerance towards stress. The binary mix containing MK 10% showed a 13.1% increment in elastic modulus compared to a 5.5% increment for SF 15% with respect to the reference mix. However, the binary MK mix shows more brittleness compared to the SF mix. Ternary mixes M6, M7 and M8 of w/b ratio 0.4 showed elastic modulus increments of 17.6%, 21.4%, and 23.6% compared to the reference mix. Similarly, ternary mixes M14, M15, and M16 of w/b ratio 0.4 showed elastic modulus increments of

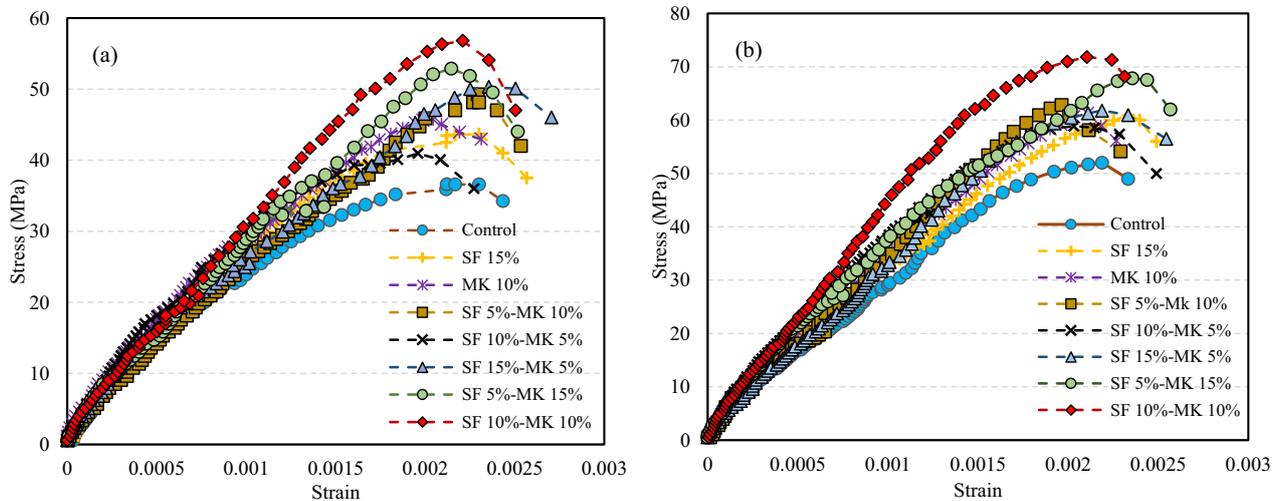


Fig. 6. Stress-strain relationship of concrete mixes a) w/b-0.4 b) w/b-0.3



18.5%, 27.9%, and 32.1% compared to the reference mix. A ternary mix containing SF 10% and MK 10% showed increased stress tolerance of 22.3% and 17.1% compared to binary SF 15%, whereas 18.4% and 9.3% increment in the case of binary MK 10% mix was observed for w/b ratio of 0.3 and 0.4 respectively. MK particles contribute more to the rapid hardening and increase in stiffness of the ternary mixes. The increase in stiffness of the concrete mixes also increases the brittleness nature, which is clearly visible from the sudden drop down of the stress-strain curve.

3.3. Relationship between mechanical properties of HPC

The standard relationship between mechanical properties of normal and high-strength concrete are provided in different codes such as CEB-FIP MC 90 [36], ACI 318 [37], ACI 363 [38], IS 456 [23], NS 3273 [39], NZS-3101 [40] & BS-8110 [41]. Since modern concretes essentially contain additional pozzolanic materials to improve their performance, the codal values have to be checked. Some of the earlier researchers such as Mindess et al. [42], Rashid et al. [43], Ahmad and Shah [44] have derived empirical relations between compressive strength flexural strength and modulus of elasticity. In the present study, the relation between compressive strength (f_c) and flexural strength (f_t) from the experimental results of HPC containing SF and MK is derived using regression analysis. The proposed equation from the present study for the prediction of f_t as a function of f_c is given in Eq. (1).

$$f_t = 0.145 (f_c)^{0.92} \quad 50 \text{ MPa} \leq f_c \leq 110 \text{ MPa} \dots \quad (1)$$

Also, the proposed equation for the relation between compressive strength (f_c) and Elastic modulus (E_c) derived from the regression analysis is given in Eq. (2).

$$E_c = 3.72 (f_c)^{0.53} \quad 50 \text{ MPa} \leq f_c \leq 110 \text{ MPa} \dots \quad (2)$$

The fitted regression from the experimental results along with the values obtained from the equations provided by various researchers and code of practices are shown in Figs 7

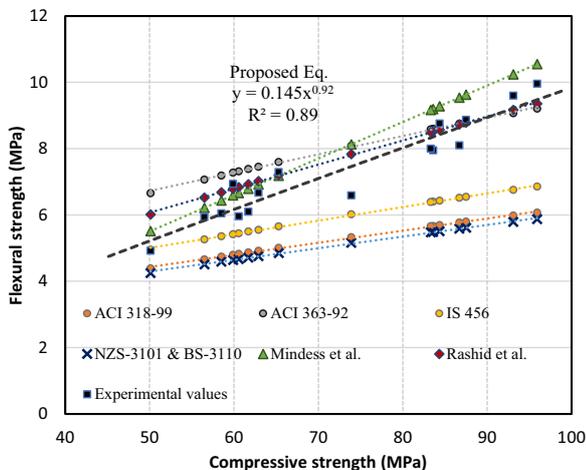


Fig. 7. Relationship between compressive strength and flexural strength

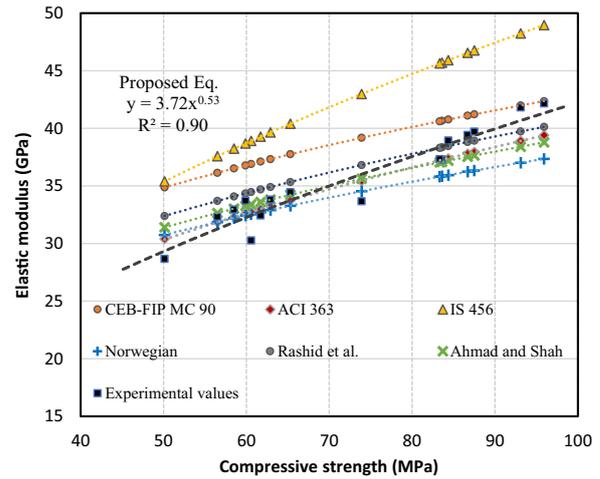


Fig. 8. Relationship between compressive strength and modulus of elasticity

and 8. To determine the effectiveness of the proposed equation, performance indices such as root mean square error (RMSE) and integral absolute error (IAE) were used. The percentage IAE and RMSE values for equations from various codes, by earlier researchers, and from the present study are given in Table 5. The results of error metrics indicate that the proposed equation shows the least error in predicting f_t with RMSE and IAE as 0.58 MPa and 6.06% and for E_c , the RMSE and IAE values were 1.15 MPa and 2.84%. The predicted values from Eq. (1) and Eq. (2) are in agreement with the values derived from equations provided by Mindess et al. [42], Rashid et al. [43], Ahmad and Shah [44]. The comparative analysis demonstrates that the proposed equations can be effectively used to predict f_t and E_c values for HPC.

3.4. Durability properties

3.4.1. Water absorption. Water absorption in concrete is the basic durability property that determines the quality of concrete in resistance to water ingress. Water ingress through the pores present in concrete can damage the steel reinforcement and can cause severe structural damage. Water absorption is directly proportional to the porosity of concrete. To carry out the test, cube samples were taken after curing for 28 days and kept in the oven for 24 h, then the over-dried samples were immersed in water for 48 h. The mass percentage of water absorbed by the oven-dried sample is represented as the percentage of water absorption of the concrete samples. The values are presented in Table 2 and a graphical representation is also given in Fig. 9. Binary SF mix shows more resistance towards water ingress compared to binary MK mix, due to the smaller particle size of SF which helps in pore refinement. The difference in strength grades also shows a significant difference due to denser particle packing. A maximum of 55.9% reduction in water absorption for the ternary mix was observed compared to the reference mix. A ternary mix containing SF 10% and MK 10% showed 36.2% and



Table 5. Comparison of empirical relations from various codes and by earlier researchers

Compressive strength vs Flexural strength				Compressive strength vs Elastic modulus			
Code of Practice/ Researchers	Empirical Relation	IAE (%)	RMSE (MPa)	Code of Practice/ Researchers	Empirical Relation	IAE (%)	RMSE (MPa)
ACI 318 (99)	$f_t = 0.62(f_c')^{0.5}$	19.80	2.29	CEB-FIP MC 90	$E_c = 21.5 (f_c'/10)^{0.3}$	8.42	3.75
ACI 363 (92)	$f_t = 0.94(f_c')^{0.5}$	10.12	0.94	ACI 363-92	$E_c = 3.32 (f_c')^{0.5} + 6.9$	3.70	1.57
IS 456 (2000)	$f_t = 0.7(f_c')^{0.5}$	13.90	1.66	IS 456-2000	$E_c = 5 (f_c')^{0.5}$	16.20	6.98
NZS 3101 & BS - 8110	$f_t = 0.6(f_c')^{0.5}$	24.46	2.45	Norwegian	$E_c = 9.5 (f_c')^{0.3}$	5.88	2.45
Mindess et al. [42]	$f_t = 0.11(f_c')$	8.78	0.82	Rashid et al. [43]	$E_c = 8.8 (f_c')^{0.325}$	4.08	1.89
Rashid et al. [43]	$f_t = 0.42(f_c')^{0.68}$	7.18	0.65	Ahmad and Shah	$E_c = 8.9 (f_c')^{0.33}$	4.54	2.00
Proposed Eq. (1)	$f_t = 0.145(f_c')^{0.92}$	6.09	0.59	Proposed Eq. (2)	$E_c = 3.72 (f_c')^{0.53}$	2.88	1.18

f_c' - cube compressive strength (MPa), f_c' - cylinder compressive strength (MPa), f_t - flexural strength (MPa), E_c - Modulus of elasticity (GPa).

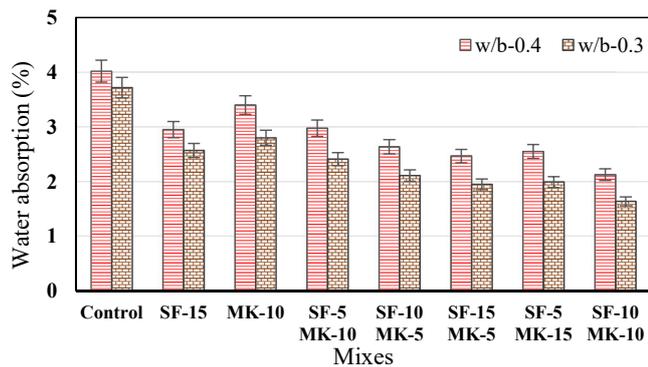


Fig. 9. Water absorption percentages of concrete mixes

27.9% reduction in water absorption compared to binary SF 15% mix for w/b ratio of 0.3 and 0.4. Similarly, the ternary mix of SF 10% and MK 10% showed 41.5% and 37.5% reduction in water absorption for w/b ratio of 0.3 and 0.4 compared to the binary MK 10% mix.

3.4.2. Acid attack. Concretes are exposed to acidic environments such as waste sewage, industrial effluent lining, and the atmosphere near industries. Hence acid resistance of concrete is considered an important durability index. In the present study, sulfuric acid with a 5% concentration is chosen resembling the pH levels of the harsh environment. The cubic specimens were taken after a 28-day curing period

and immersed in the acidic solution and tested for mass loss in specimen and compressive strength loss after 28, 56, and 90 days. The mass loss percentages of the mixes are shown in Fig. 10. From the figure, it is seen that initially there is an increase in the mass majorly due to the accumulation of calcium sulphate and softening of the interfacial transition zone (ITZ) of specimens. The maximum increase in weight of binary SF and MK mix was 2.17% and 2.43%. The maximum increase in weight for the ternary mix was 2.28%. A ternary mix containing SF 10% and MK 10% showed a 43.4% and 36.3% reduction in mass loss compared to the reference mix. After 4 weeks, the concrete started deteriorating and weight loss was observed. Ternary mixes showed reduced weight loss compared to binary mixes. The compressive strength loss percentages of the mixes with standard deviation are shown in Fig. 11. The mass loss in specimens and the loss in their compressive strength are in correlation with each other. Ternary mix containing SF 10% and MK 10% showed an increase in compressive strength for 28, 56, and 90 days up to 52.3%, 42.8%, and 44.5% compared to the reference mix for a w/b ratio of 0.3. Similarly, a 52%, 43.5%, and 41.4% increase for a w/b ratio of 0.4 was observed in comparison to the reference mix. Mixes containing SF showed better acid resistance compared to MK.

3.4.3. Sulphate attack. Deterioration of concrete can occur in the presence of sulphate as salts in a physical or

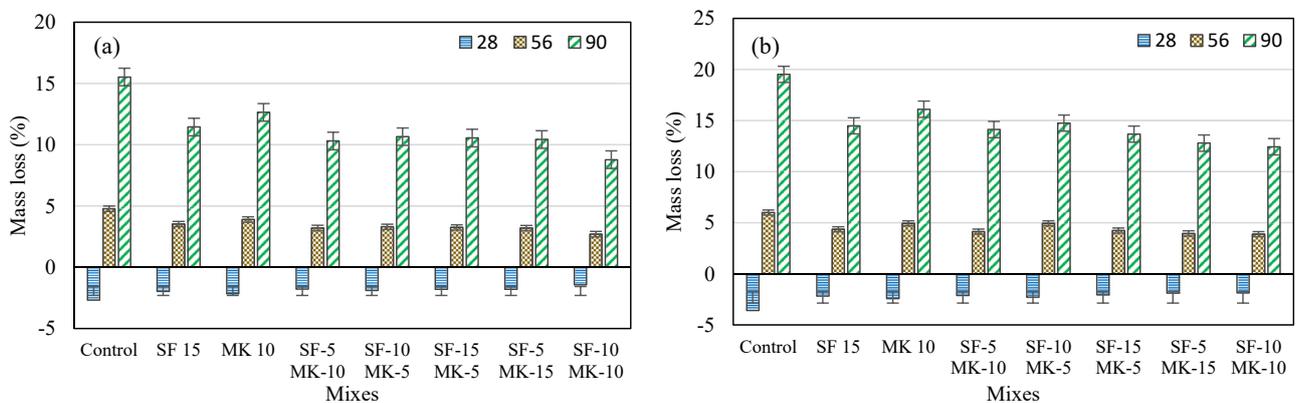
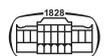


Fig. 10. Mass loss of concrete mixes with immersion ages in 5% H₂SO₄ solution a) w/b-0.3 b) w/b-0.4



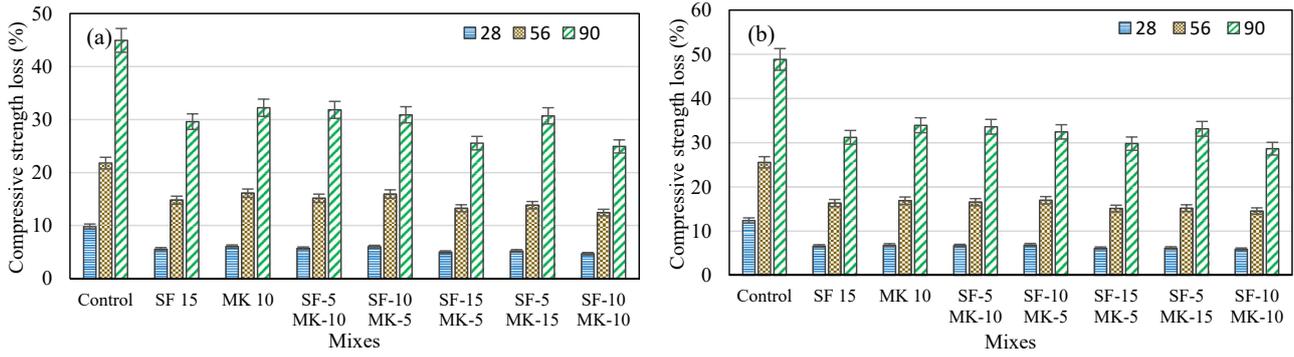


Fig. 11. Compressive strength loss of concrete mixes with immersion ages in 5% H₂SO₄ solution a) w/b-0.3 b) w/b-0.4

chemical state such as in marine environments and underground soil. In the present study, sodium sulphate with a 5% concentration is chosen resembling the severe environmental condition. The cubic specimens were taken after a 28-day curing period and immersed in the acidic solution and tested for mass loss and compressive strength loss after 28, 56, and 90 days. The mass loss percentages of the mixes are shown in Fig. 12. From the figure, a marginal loss in weight is observed for all the mixes except the reference mixes. The compressive strength results of the specimens immersed in sulphate

solution are shown in Fig. 12. Ternary mixes exhibited better acid resistance compared to binary mixes. The compressive strength of almost all the mixes increased till 56 days of immersion, after which deterioration of the concrete started to begin. A ternary mix containing SF 10% and MK 10% showed increased compressive strength even after 90 days of immersion. The presence of calcium alumina silicate hydrate (C-A-S-H) and secondary calcium silicate hydrate (C-S-H) refines the pore structure and prevents harmful agents from penetrating the concrete surface (Fig. 13).

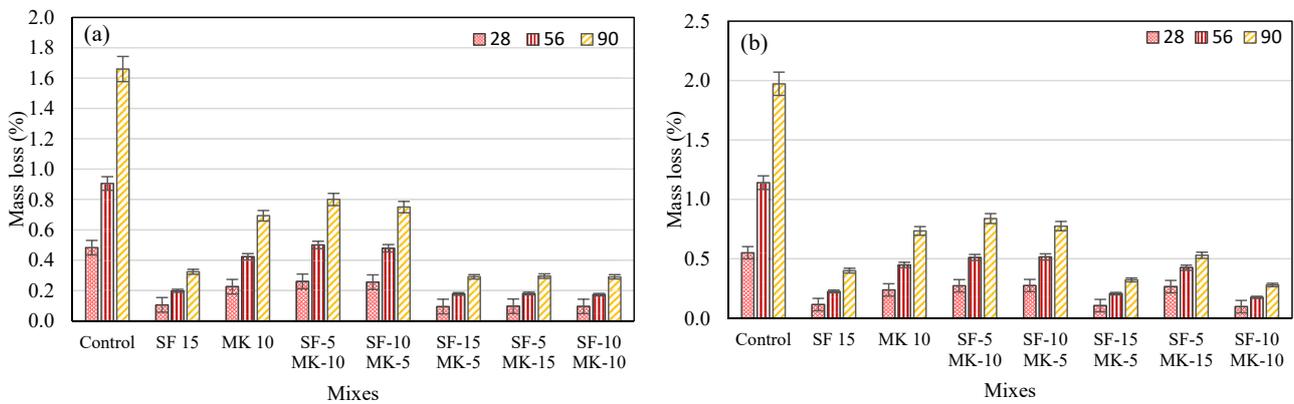


Fig. 12. Mass loss of concrete mixes with immersion ages in 5% Na₂SO₄ solution a) w/b-0.3 b) w/b-0.4

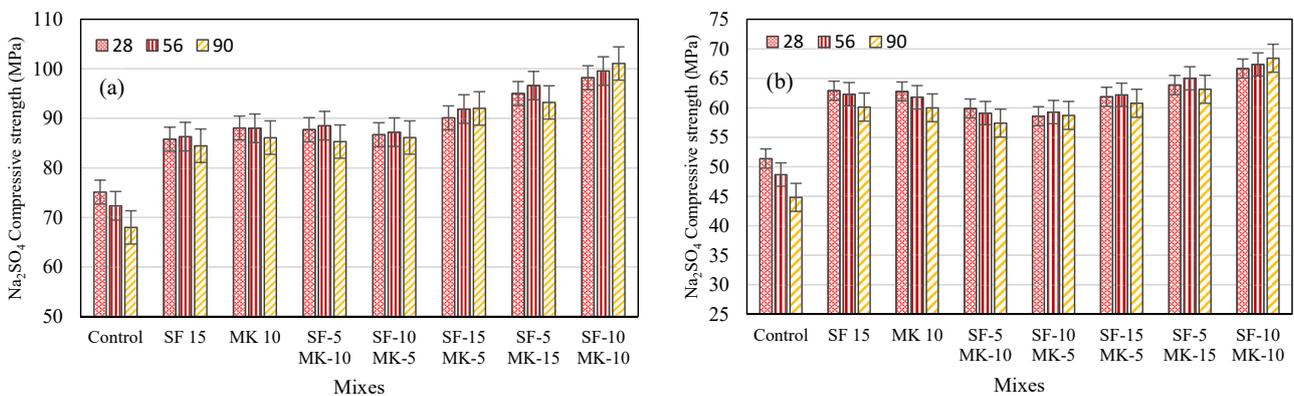


Fig. 13. Compressive strength variation in concrete mixes with immersion ages in 5% Na₂SO₄ solution a) w/b-0.3 b) w/b-0.4



3.5. Recommendations for further research

The present study can be extended by determining other durability properties such as chloride diffusion, carbonation, water and gas permeability, etc. Predictive models can be developed by correlating the durability properties with the mechanical properties of the HPC containing SF and MK and validating the proposed models with the experimental values provided by earlier researchers. The present study aims at understanding the effects of ultrafine SCMs in HPC, a comparison of the results with similar HPC mixes containing other SCMs can help us understand and properly choose the SCM for a targeted environment.

4. CONCLUSIONS

Based on the experimental investigation carried out in this study the following conclusions were drawn:

The mechanical and durability characteristics of HPC containing both binary and ternary mixes of SF and MK were studied.

The blending of SF and MK in concrete made the matrix denser through increased volume of ettringite formation resulting in enhanced strength and durability properties.

The role of SF is more attributed to the particle packing and secondary C-S-H gel formation, whereas MK with higher pozzolanic activity helps in the formation of C-A-S-H resulting in rapid hardening and stiffening of the matrix.

The ternary mixes showed a maximum improvement in compressive strength of 30.2% and 14.7% with respect to the reference mix and binary mix.

Similarly, the flexural strength and modulus of elasticity test results showed a maximum strength improvement of about 37.8% and 32.1%.

The denser particle packing using the ultrafine SF and MK improved the pore structure, preventing the ingress and capillary action of water. A 55.9% reduction in water absorption was noticed for a ternary mix containing SF 10% and MK 10%.

Ternary mixes showed reduced mass loss and compressive strength loss compared to binary SF and binary MK mix when immersed in an acidic medium.

No significant mass loss was observed for concretes immersed in sulphate solution, although strength loss was noticeable after 56 days of immersion.

A ternary mix containing SF 10% and MK 10% provided better results in terms of strength and durability when compared with other mixes.

Alterations in the empirical relation between compressive strength, flexural strength, and elastic modulus were suggested for concretes containing SCMs.

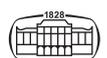
The empirical relationships proposed in the present study to predict flexural strength and elastic modulus as a function of compressive strength shows the least RMSE and IAE of 0.59 MPa, 1.18 MPa and 6.09%, 2.88% compared to earlier researchers and code of practice.

ACKNOWLEDGEMENT

The authors would like to express their thanks to the All India Council for Technical Education (AICTE) for providing financial assistance towards the research work.

REFERENCES

- [1] P. K. Mehta, *Pozzolanic and Cementitious By-Products in Concrete-Another Look*, 1989. <https://doi.org/10.14359/1835>.
- [2] K. H. Khayat, M. Vachon, and M. C. Lanctôt, "Use of blended silica fume cement in commercial concrete mixtures," *ACI Mater. J.*, 1997. <https://doi.org/10.14359/9873>.
- [3] S. Bhanja, and B. Sengupta, "Investigations on the compressive strength of silica fume concrete using statistical methods," *Cem. Concr. Res.*, vol. 32, no. 9, 2002. [https://doi.org/10.1016/S0008-8846\(02\)00787-1](https://doi.org/10.1016/S0008-8846(02)00787-1).
- [4] B. Sankar, and P. Ramadoss, "Modelling the compressive strength of high-performance concrete containing metakaolin using distinctive statistical techniques," *Results Control Optim.*, vol. 12, p. 2023, Paper no. 100241. <https://doi.org/10.1016/j.rico.2023.100241>.
- [5] B. Sankar, and P. Ramadoss, *Mechanical and Durability Properties of High Strength Concrete Incorporating Different Combinations of Supplementary Cementitious Materials: A Review*, 2022. http://dx.doi.org/10.1007/978-981-16-4321-7_45.
- [6] B. Sankar, and P. Ramadoss, *Review on Fiber Hybridization in Ternary Blended High-Performance Concrete*, 2021. <https://doi.org/10.1016/j.matpr.2021.01.366>.
- [7] H. W. Song, S. W. Pack, S. H. Nam, J. C. Jang, and V. Saraswathy, "Estimation of the permeability of silica fume cement concrete," *Constr. Build. Mater.*, 2010. <https://doi.org/10.1016/j.conbuildmat.2009.08.033>.
- [8] E. K. Attiogbe, and S. H. Rizkalla, "Response of concrete to sulfuric acid attack," *ACI Mater. J.*, 1988. <https://doi.org/10.14359/2210>.
- [9] S. Jagan, *Effect of Silica Fume on the Hardened and Durability Properties of Concrete*, 2020, pp. 6–11. <https://doi.org/10.1556/1848.2020.00129>.
- [10] G. Lizia Thankam, and N. T. Renganathan, "Ideal supplementary cementing material - metakaolin: A review," *Int. Rev. Appl. Sci. Eng.*, 2020. <https://doi.org/10.1556/1848.2020.00008>.
- [11] R. Siddique, and J. Klaus, "Influence of metakaolin on the properties of mortar and concrete: a review," *Appl. Clay Sci.*, vol. 43, nos 3–4, pp. 392–400, 2009. <https://doi.org/10.1016/j.clay.2008.11.007>.
- [12] M. Dubey, S. V. Deo, and G. Ramtekkar, "Evaluating properties of high performance concrete containing metakaolin as cementitious material," *Int. Rev. Appl. Sci. Eng.*, vol. 13, no. 3, pp. 257–62, 2022. <https://doi.org/10.1556/1848.2021.00359>.
- [13] E. A. B. Khalil, and M. Anwar, *Carbonation of Ternary Cementitious Concrete Systems Containing Fly Ash and Silica Fume*, Water Sci., 2015. <https://doi.org/10.1016/j.wsj.2014.12.001>.
- [14] M. H. Shehata, and M. D. A. Thomas, "Use of ternary blends containing silica fume and fly ash to suppress expansion due to alkali-silica reaction in concrete," *Cem. Concr. Res.*, 2002. [https://doi.org/10.1016/S0008-8846\(01\)00680-9](https://doi.org/10.1016/S0008-8846(01)00680-9).



- [15] M. S. Ahmed, O. Kayali, and W. Anderson, "Evaluation of binary and ternary blends of pozzolanic materials using the rapid chloride permeability test," *J. Mater. Civ. Eng.*, 2009. [https://doi.org/10.1061/\(asce\)0899-1561\(2009\)21:9\(446\)](https://doi.org/10.1061/(asce)0899-1561(2009)21:9(446)).
- [16] S. H. Jung, Y. C. Choi, and S. Choi, "Use of ternary blended concrete to mitigate thermal cracking in massive concrete structures—a field feasibility and monitoring case study," *Constr. Build. Mater.*, 2017. <https://doi.org/10.1016/j.conbuildmat.2017.01.108>.
- [17] P. Murthi, and V. Sivakumar, *Studies on Acid Resistance of Ternary Blended Concrete*, Asian J. Civ. Eng. (Build. Hous.), 2008.
- [18] J. Jiang, Z. Lu, Y. Niu, J. Li, and Y. Zhang, "Investigation of the properties of high-porosity cement foams based on ternary Portland cement-metakaolin-silica fume blends," *Constr. Build. Mater.*, 2016. <https://doi.org/10.1016/j.conbuildmat.2015.12.190>.
- [19] J. J. Chen, P. L. Ng, S. H. Chu, G. X. Guan, and A. K. H. Kwan, "Ternary blending with metakaolin and silica fume to improve packing density and performance of binder paste," *Constr. Build. Mater.*, 2020. <https://doi.org/10.1016/j.conbuildmat.2020.119031>.
- [20] S. H. Chu, J. J. Chen, L. G. Li, P. L. Ng, and A. K. H. Kwan, "Roles of packing density and slurry film thickness in synergistic effects of metakaolin and silica fume," *Powder Technol.*, vol. 387, p. 2021. <https://doi.org/10.1016/j.powtec.2021.04.029>.
- [21] *Bureau of Indian Standards (BIS)*, IS 12269: 2013 Ordinary Portland Cement, 53 Grade Specification, Indian Stand., 2013.
- [22] *IS:15388*, Silica Fume-Specification, New Delhi, India, Bur. Indian Stand., 2003, pp. 1–13.
- [23] *IS 16354*, Metakaolin for Use in Cement, Cement Mortar and Concrete - Specification, New Delhi, India, Bur. Indian Stand., 2015.
- [24] *IS:383*, Specification for Coarse and Fine Aggregates from Natural Sources for Concrete, Indian Stand., 1970, pp. 1–24.
- [25] *B. of Indian Standards*, IS 9103: 1999, Specification for Concrete Admixtures BIS, Delhi, Bur. Indian Stand., 1999.
- [26] *ACI Committee 211*, ACI 211.4R-93 Guide for Selecting Proportions for High-Strength Concrete with Portland Cement and Fly Ash, Man. Concr. Pract., 1998.
- [27] *ASTM C143/C143M*, Standard Test Method for Slump of Hydraulic-Cement Concrete, Astm C143, no. 1, 2015, pp. 1–4. <https://doi.org/10.1520/C0143>.
- [28] *ASTM*, Astm C78/C78M -18," Stand. Test Method Flexural Strength Concr, vol. 04.02, USA, (Using Simple Beam with Third-Point Loading)ASTM Int, 2002, pp. 1–3.
- [29] *ASTM International*, ASTM Standard C469/C469M – 14, Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression, ASTM Int., 2014.
- [30] *ASTM C1585-13*, Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic Cement Concretes, ASTM Int., 2013.
- [31] *ASTMC1898-20*, Standard Test Methods for Determining the Chemical Resistance of Concrete Products to Acid Attack, ASTM Internation. <https://doi.org/10.1016/j.asej.2013.05.002>.
- [32] *ASTM C1012*, Standard Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution, ASTM C157 West Conshohockem, PA, ASTM, 2003.
- [33] R. A. Khan, and M. Haq, "Long-term mechanical and statistical characteristics of binary- and ternary-blended concrete containing rice husk ash, metakaolin and silica fume," *Innov. Infrastruct. Solut.*, 2020. <https://doi.org/10.1007/s41062-020-00303-0>.
- [34] B. Sankar, and P. Ramadoss, "Experimental and statistical investigations on alccofine based ternary blended high-performance concrete," *Int. J. Eng. Trans. B Appl.*, vol. 35, no. 8, pp. 1629–40, 2022. <https://doi.org/10.5829/IJE.2022.35.08B.19>.
- [35] B. Sagar, and S. M.V.N, "Mechanical and microstructure characterization of alccofine based high strength concrete," *Silicon*, vol. 14, no. 3, 2022. <https://doi.org/10.1007/s12633-020-00863-x>.
- [36] *C.-F. I. F. for Structural*, Structural Concrete: Textbook on Behaviour, Design and Performance: Updated Knowledge of the CEB/FIP Model Code 1990, Bulletin, 2009.
- [37] C. V. Baker, et al., *Building Code Requirements for Structural Concrete (ACI 318-95) and Commentary (ACI 318R-95)*, american concrete institute".
- [38] *ACI*, Report on High-Strength Concrete Reported by ACI Committee 363, 1992, ACI 363R-92.
- [39] *NS-3473 Concrete Structures-Design Rules*. Norwegian standards, Oslo, Norway, 1992".
- [40] *New Zealand Standards*, NZS-3101-Concrete Structures Standard.
- [41] *British Standards Institution*, *Bs 8110-1:1997*, London, Br. Stand. Inst., 1997, p. 168, no. 1.
- [42] D. D. Sidney Mindess, and J. Francis Young, *Concrete Second Edition*, Saddle River, NJ 07458, Pearson Educ. Inc. Up., 2003.
- [43] M. A. Rashid, M. A. Mansur, and P. Paramasivam, "Correlations between mechanical properties of high-strength concrete," *J. Mater. Civ. Eng.*, 2002. [https://doi.org/10.1061/\(asce\)0899-1561\(2002\)14:3\(230\)](https://doi.org/10.1061/(asce)0899-1561(2002)14:3(230)).
- [44] S. P. Shah, and S. H. Ahmad, "Structural properties of high strength concrete and its implications for precast prestressed concrete," *J. Prestress. Concr. Inst.*, 1985. <https://doi.org/10.15554/pcij.11011985.92.119>.

Open Access statement. This is an open-access article distributed under the terms of the Creative Commons Attribution-NonCommercial 4.0 International License (<https://creativecommons.org/licenses/by-nc/4.0/>), which permits unrestricted use, distribution, and reproduction in any medium for non-commercial purposes, provided the original author and source are credited, a link to the CC License is provided, and changes - if any - are indicated.

