Behaviour of Fly Ash and Metakaolin Based Composite Fiber (Glass and Polypropylene) Reinforced High Performance Concrete under Acid Attack

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Abstract Improvements in concrete properties have been achieved by researchers with the invention of High-Performance-Concrete (HPC), which can now be improvised by using a combination of mineral admixtures. HPC is usually more brittle which can be made ductile by modifying its composition by adding fibers in the design mix, which led to the development of fiber-reinforced concrete. High-Performance-Concrete made with glass fibers and polypropylene fibers is regarded as Composite Fibre (Glass and polypropylene) Reinforced High Performance Concrete (CFRHPC). The development of cost-effective state-of-the-art procedures for producing, evaluating, and designing with CFRHPC will enhance the performance for each performance characteristic and can be reliably achieved in the field. This investigation evaluates the effect of cement being partially replaced by combined fly ash and metakaolin with glass fibers and polypropylene fibers as an addition to produce high-performance concrete with composite fiber for resistance to hydrochloric acid, magnesium sulphate, and sulphuric acid attack for 30, 60, and 90 days. The water to binder ratios (W/B) of 0.275, 0.300, 0.325, and 0.350 and an aggregate to binder ratio (A/B) of 1.75 were adopted. Fly ash and metakaolin were replaced in the range from 0% to 15% each, glass fibers were added in volume percentages from 0% to 1%, and polypropylene fibers were kept constant at 0.25%. The combined effect of fly ash and metakaolin at 5% each as replacement of cement and the addition of composite fiber dosage of glass fiber=1% and polypropylene fibers=0.25% for W/B of 0.275 was found to be the optimum combination to obtain maximum acid attack resistance, which was also justified by SEM, EDX, and XRD analysis done in this investigation. CFRHPC production minimizes enormous cement production and safeguards the environment from pollution and concrete from environmental pollution throughout its service life.

Keywords Glass Fiber, Polypropylene Fiber, Metakaolin, Fly Ash, Acid Attack

1. Introduction

Concrete is being used extensively all over the world due to its adaptable strength and durability. Durable concrete structures need to be produced as these structures are subjected to severe environments but are anticipated to last with hardly any maintenance for longer periods [1]. Concrete structures deteriorate due to corrosion as they are exposed to detrimental chemicals found in the environment. Chlorides and sulphates are the most
detrimental aggressive chemicals found in seawaters, effluents of industries, and even in groundwater, which affect the long-term durability of concrete structures. The stiffness and strength of concrete structures deteriorate due to their porosity caused by the leaching of portlandite as chloride dissolves in water encountering concrete structures [2]. Hydrated cement paste mixes with toxic calcium, sodium, magnesium, and ammonium sulphates, resulting in cracking and swelling, which eventually results in concrete spalling and loss of strength [2]. Glass fibers (GF) and Polypropylene fibers (PPF) are among the most multipurpose engineering materials acknowledged today all over the world, which are produced from raw materials, which are accessible in almost unlimited supply. These fibers inherit properties such as hardness, transparency, chemical attack resistance, stability, and inertness, which provide strength, flexibility, and stiffness to concrete structures. When both fibers are used in high-performance concrete (HPC), Composite Fibre Reinforced High Performance Concrete (CFRHPC) is developed [3-6].

The supplementary cementing material being used recently to produce high-performance concrete (HPC) is High reactivity Metakaolin (HRM). HRM is produced by driving off the water in interstices of the kaolin by treating kaolin clay of high purity by controlled thermal activation so that its structure collapses, which results in amorphous aluminosilicate, i.e., Metakaolin (MK) [7]. Permeability of concrete decides the durability of concrete. External sulphate attack depends on water binder ratio and porosity at the internal level. Studies at the micro-level reported that with an increase in MK in concrete, the large pores decreased. This enhanced sulphate resistance of MK induced concrete has been reported in various research works, establishing the role of MK as a filler of micropores in the production of HPC [8-12]. Rao et al. [13] studied the deterioration and the relative sulphate resistance of HPC in severe sulphate environments and suggested using Fly ash (FA) by partially replacing natural pozzolana for enhanced performance. Present authors also have investigated the effect of fly ash, metakaolin, and silica fume based composite fiber-reinforced HPC on strength and durability properties and concluded the viability of using fly ash, metakaolin, silica fume, and composite fibers in CFRHPC production for enhanced strength and durability properties [14-16]. The widespread application of FA and MK in the construction industry is the result of extensive investigations on the use of FA and MK in concrete during the past two decades [17–22]. National standards of various countries are established that determine only the degree of attack based on the concentration of hostile substances [23–29].

The chemical resistance of HPC produced using FA and MK as a cement replacement, with the addition of GF and PPF with superplasticizers, are issues that are yet to receive sufficient exploration from the research community as minimal studies are carried out to uphold their effectiveness in the context of durability. Hence, there is a shortfall of research material available. Besides, Indian Standard Codes do not specify the tests to be executed for assessing the durability of HPC. This investigation presents the outcomes of experimental exploration performed to understand FA and MK-based composite fiber-reinforced HPC behavior under acid attack.

2. Materials and Properties

The cement used was OPC of grade 53, having a specific gravity of 3.10. Fine aggregates used were of specific gravity 2.67 collected from a locally available riverbed. Coarse aggregates used were of specific gravity 2.75, which were from a stone quarry available locally with 40% of 12.5 mm and 60% of 20 mm size. The fly ash used had a specific gravity of 2.18, a specific surface area of 0.398 m²/g, and contained 59.16% SiO₂ and 30.64% Al₂O₃. Metakaolin used was of pink colour, having a specific gravity of 2.60 with a specific surface area of 12.7 m²/g and had SiO₂ and Al₂O₃ at 52.4% and 43.18%, respectively. A CemFil AntiCrack HD Glass fiber with 14 μm diameter and 12 mm length was used during concrete production. These fibers are water dispersible, which allows total dispersion of GF into individual filaments upon mixing in an aqueous environment. Polypropylene fibers used were engineered microfibers with a unique triangular cross-section of length 12 mm and 38-μm diameter. Potable fresh water free from organic and acid substances was used for concrete mixing. A chloride-free Superplasticizer (SP) of Fosroc made with a specific gravity of 1.18 was used. The acids used in the investigation were Hydrochloric acid (HCl), Magnesium sulfate (MgSO₄), and Sulfuric acid (H₂SO₄) in the form of 5% concentration solutions.

3. Experimental Procedure

3.1. Mix Proportions

To study the behaviour of CFRHPC, 19 mixes along with one HPC mix without any mineral admixtures and composite fibers were prepared for each water binder ratio. The CFRHPC mixes were designed with W/B of 0.275, 0.300, 0.325, and 0.350 with a constant A/B of 1.75. FA and MK of 5%, 10% and 15% each were adapted as cement replacement with addition of 0%, 0.25%, 0.5%, 0.75% and 1% GF content along with constant PPF of 0.25% of concrete volume. SP was used at 0.8% by weight of the binder. These relative proportions were obtained by the absolute volume method. Recently
manufactured single batch OPC of 53 grades has been used. The first letter in the mix designation indicates composite matrix containing GF and PPF, second letter indicates percentage of GF and PPF used, i.e. P=0%GF & 0%PPF, Q=0.25%GF & 0.25%PPF, R=0.5%GF & 0.25%PPF, S=0.75%GF & 0.25%PPF and T=1%GF & 0.25%PPF. F indicates FA, and M indicates MK. The following number indicates the total percentage of cement replaced by FA and MK. Last alphabet indicates water binder ratios, i.e. A=0.275, B=0.300, C=0.325 and D=0.350. CPMF0A indicates a plain high-performance concrete mix without any cement replacement by mineral admixtures and without the addition of any fibers for W/B of 0.275 with the cement of 805.43 kg/m³. For the CTFM10A mix, the cement of 719.15 kg/m³ was used, and the quantity of FA and MK used was 39.95 kg/m³ each. The proportions of ingredients used for W/B of 0.275 are tabulated in Table 1. Similar patterns of ingredients were used for W/B of 0.300, 0.325, and 0.350.

| Mix Designation | W/B | A/B | SP | FA (%) | MK (%) | GF (%) | PPF (%) |
|-----------------|-----|-----|----|--------|--------|--------|--------|
| CPFMOA          | 0.275 | 1.75 | 0.8 | 0      | 0      | 0      | 0      |
| CPFM10A         | 0.275 | 1.75 | 0.8 | 5      | 5      | 0      | 0      |
| CPFM20A         | 0.275 | 1.75 | 0.8 | 10     | 10     | 0      | 0      |
| CPFM30A         | 0.275 | 1.75 | 0.8 | 15     | 15     | 0      | 0      |
| CQFM0A          | 0.275 | 1.75 | 0.8 | 0      | 0      | 0.25   | 0.25   |
| CQFM10A         | 0.275 | 1.75 | 0.8 | 5      | 5      | 0.25   | 0.25   |
| CQFM20A         | 0.275 | 1.75 | 0.8 | 10     | 10     | 0.25   | 0.25   |
| CQFM30A         | 0.275 | 1.75 | 0.8 | 15     | 15     | 0.25   | 0.25   |
| CRFM0A          | 0.275 | 1.75 | 0.8 | 0      | 0      | 0.5    | 0.25   |
| CRFM10A         | 0.275 | 1.75 | 0.8 | 5      | 5      | 0.5    | 0.25   |
| CRFM20A         | 0.275 | 1.75 | 0.8 | 10     | 10     | 0.5    | 0.25   |
| CRFM30A         | 0.275 | 1.75 | 0.8 | 15     | 15     | 0.5    | 0.25   |
| CSFM0A          | 0.275 | 1.75 | 0.8 | 0      | 0      | 0.75   | 0.25   |
| CSFM10A         | 0.275 | 1.75 | 0.8 | 5      | 5      | 0.75   | 0.25   |
| CSFM20A         | 0.275 | 1.75 | 0.8 | 10     | 10     | 0.75   | 0.25   |
| CSFM30A         | 0.275 | 1.75 | 0.8 | 15     | 15     | 0.75   | 0.25   |
| CTFMOA          | 0.275 | 1.75 | 0.8 | 0      | 0      | 1      | 0.25   |
| CTFM10A         | 0.275 | 1.75 | 0.8 | 5      | 5      | 1      | 0.25   |
| CTFM20A         | 0.275 | 1.75 | 0.8 | 10     | 10     | 1      | 0.25   |
| CTFM30A         | 0.275 | 1.75 | 0.8 | 15     | 15     | 1      | 0.25   |

W/B - Water Binder ratio
A/B - Aggregate Binder ratio
SP - Superplasticizer
FA – Fly ash
MK - Metakaolin
GF - Glass fiber
PPF - Polypropylene fiber
Table 2. Water cured compressive strength at 28 days, and HCl immersed residual compressive strength results

| Mix Designation | Compressive strength | Residual Compressive strength | Mix Designation | Compressive strength | Residual Compressive strength |
|-----------------|----------------------|-------------------------------|-----------------|----------------------|-------------------------------|
|                 | MPa                  | Days                          |                 | MPa                  | Days                          |
| CQFM0A          | 76.20                | 69.74                         | CQFM0B          | 72.40                | 66.03                         |
| CQFM10A         | 88.31                | 82.03                         | CQFM10C         | 84.78                | 78.57                         |
| CQFM20A         | 86.06                | 79.45                         | CQFM20C         | 82.61                | 76.08                         |
| CQFM30A         | 77.13                | 70.72                         | CQFM20D         | 86.92                | 72.72                         |
| CQFM30B         | 78.04                | 71.76                         | CQFM30C         | 74.14                | 67.92                         |
| CQFM10A         | 91.17                | 84.96                         | CQFM10D         | 87.05                | 80.91                         |
| CQFM20A         | 89.19                | 82.66                         | CQFM20C         | 85.16                | 78.70                         |
| CQFM30A         | 79.79                | 73.47                         | CQFM30C         | 87.75                | 79.67                         |
| CRFM0A          | 79.88                | 73.90                         | CRFM0C          | 75.89                | 69.92                         |
| CRFM10A         | 94.02                | 88.01                         | CRFM10C         | 89.32                | 83.36                         |
| CRFM20A         | 92.32                | 86.00                         | CRFM20C         | 87.70                | 81.44                         |
| CRFM30A         | 82.46                | 76.25                         | CRFM30C         | 78.34                | 72.18                         |
| CSFM0A          | 84.00                | 77.98                         | CSFM0C          | 80.63                | 74.47                         |
| CSFM10A         | 98.37                | 92.20                         | CSFM10C         | 93.45                | 87.25                         |
| CSFM20A         | 95.55                | 89.23                         | CSFM20C         | 90.78                | 84.43                         |
| CSFM30A         | 86.40                | 80.17                         | CSFM30C         | 82.08                | 75.82                         |
| CTFM0A          | 86.14                | 80.34                         | CTFM0C          | 81.84                | 76.11                         |
| CTFM10A         | 100.35               | 94.40                         | CTFM10C         | 96.34                | 90.43                         |
| CTFM20A         | 98.92                | 92.58                         | CTFM20C         | 94.96                | 88.67                         |
| CTFM30A         | 88.50                | 82.44                         | CTFM30C         | 84.96                | 78.94                         |
| CTFM0B          | 74.30                | 67.88                         | CTFM0D          | 71.63                | 65.10                         |
| CTFM10B         | 86.55                | 80.30                         | CTFM10D         | 83.01                | 76.76                         |
| CTFM20B         | 84.34                | 77.76                         | CTFM20D         | 80.89                | 74.31                         |
| CTFM30B         | 75.19                | 68.85                         | CTFM30D         | 72.03                | 65.69                         |
| CQFM0B          | 76.09                | 69.84                         | CQFM0D          | 73.34                | 66.97                         |
| CQFM10B         | 89.11                | 82.93                         | CQFM10D         | 85.23                | 79.03                         |
| CQFM20B         | 87.17                | 80.68                         | CQFM20D         | 83.37                | 76.86                         |
| CQFM30B         | 77.79                | 71.52                         | CQFM30D         | 74.36                | 68.07                         |
| CRFM0B          | 77.88                | 71.90                         | CRFM0D          | 75.06                | 68.95                         |
| CRFM10B         | 91.67                | 85.68                         | CRFM10D         | 87.44                | 81.43                         |
| CRFM20B         | 90.01                | 83.72                         | CRFM20D         | 85.86                | 79.53                         |
| CRFM30B         | 80.40                | 74.21                         | CRFM30D         | 76.69                | 70.47                         |
| CSFM0B          | 82.32                | 76.22                         | CSFM10D         | 78.96                | 72.71                         |
| CSFM10B         | 95.91                | 89.72                         | CSFM10D         | 92.47                | 86.13                         |
| CSFM20B         | 93.16                | 86.83                         | CSFM20D         | 89.82                | 83.34                         |
| CSFM30B         | 84.24                | 77.99                         | CSFM30D         | 81.21                | 74.81                         |
| CTFM0B          | 83.99                | 78.22                         | CTFM0D          | 79.29                | 73.53                         |
| CTFM10B         | 98.34                | 92.41                         | CTFM10D         | 93.33                | 87.41                         |
| CTFM20B         | 96.94                | 90.62                         | CTFM20D         | 92.00                | 85.70                         |
| CTFM30B         | 86.73                | 80.69                         | CTFM30D         | 82.30                | 76.27                         |

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Table 3. Water cured compressive strength at 28 days, and MgSO4 immersed residual compressive strength results

| Mix Designation | Compressive strength | Residual Compressive Strength |
|-----------------|----------------------|-------------------------------|
|                 | MPa                  | MPa                           |
|                 | 28                   | 30  | 60 | 90 | 28 | 30 | 60 | 90 |
| Days            |                      |     |    |    |    |    |    |    |
| CQFM0A          | 76.20                | 69.56 | 66.35 | 61.09 | 72.40 | 65.90 | 62.76 | 57.38 |
| CQFM10A         | 88.31                | 81.61 | 78.05 | 72.89 | 84.78 | 78.15 | 74.64 | 69.27 |
| CQFM20A         | 86.06                | 78.65 | 74.87 | 69.36 | 82.61 | 75.31 | 71.57 | 65.84 |
| CQFM30A         | 77.13                | 70.54 | 67.29 | 61.65 | 73.25 | 66.81 | 63.63 | 57.88 |
| CQFM0B          | 78.04                | 71.28 | 67.99 | 63.16 | 74.14 | 67.50 | 64.31 | 59.46 |
| CQFM10B         | 91.17                | 84.37 | 80.88 | 75.71 | 87.05 | 80.34 | 76.94 | 71.71 |
| CQFM20B         | 89.19                | 81.82 | 77.92 | 72.74 | 85.16 | 77.88 | 74.08 | 68.85 |
| CQFM30B         | 79.79                | 72.97 | 69.61 | 64.34 | 75.79 | 69.08 | 65.82 | 60.56 |
| CRFM0A          | 79.88                | 73.39 | 70.22 | 65.36 | 75.89 | 69.40 | 66.38 | 61.67 |
| CRFM10A         | 94.02                | 87.25 | 83.85 | 78.67 | 89.32 | 82.63 | 79.37 | 74.29 |
| CRFM20A         | 92.32                | 85.11 | 81.10 | 76.30 | 87.70 | 80.58 | 76.72 | 72.02 |
| CRFM30A         | 82.46                | 75.70 | 71.94 | 67.08 | 78.34 | 71.62 | 68.04 | 63.30 |
| CTFM0A          | 84.00                | 77.52 | 74.01 | 69.11 | 80.63 | 73.99 | 70.57 | 65.90 |
| CTFM10A         | 98.37                | 91.26 | 87.88 | 82.78 | 93.45 | 86.35 | 83.08 | 78.18 |
| CTFM20A         | 95.55                | 88.30 | 84.52 | 79.79 | 90.78 | 83.53 | 79.87 | 75.34 |
| CTFM30A         | 86.40                | 79.68 | 76.09 | 70.67 | 82.08 | 75.31 | 71.84 | 66.71 |
| CTFM0B          | 86.14                | 79.94 | 76.71 | 71.50 | 81.84 | 75.71 | 72.33 | 67.30 |
| CTFM10B         | 100.35               | 93.28 | 89.78 | 85.43 | 96.34 | 89.35 | 85.68 | 81.39 |
| CTFM20B         | 98.92                | 91.58 | 87.76 | 83.59 | 94.96 | 87.70 | 83.71 | 79.62 |
| CTFM30B         | 88.50                | 82.00 | 78.47 | 72.69 | 84.96 | 78.49 | 74.78 | 69.20 |
| CTFM0B          | 74.30                | 67.73 | 64.55 | 59.23 | 71.63 | 65.01 | 61.81 | 56.37 |
| CTFM10B         | 86.55                | 79.88 | 76.34 | 71.07 | 83.01 | 76.34 | 72.80 | 67.40 |
| CTFM20B         | 84.34                | 76.98 | 73.22 | 67.59 | 80.89 | 73.55 | 69.78 | 64.04 |
| CTFM30B         | 75.19                | 68.67 | 65.46 | 59.76 | 72.03 | 65.51 | 62.29 | 56.52 |
| CTFM0B          | 76.09                | 69.39 | 66.14 | 61.30 | 73.34 | 66.58 | 63.33 | 58.42 |
| CTFM10B         | 89.11                | 82.35 | 78.91 | 73.70 | 85.23 | 78.47 | 75.04 | 69.78 |
| CTFM20B         | 87.17                | 79.85 | 76.00 | 70.79 | 83.37 | 76.06 | 72.23 | 66.96 |
| CTFM30B         | 77.79                | 71.03 | 67.71 | 62.44 | 74.36 | 67.58 | 64.29 | 59.01 |
| CRFM0B          | 77.88                | 71.39 | 68.29 | 63.51 | 75.06 | 68.41 | 65.33 | 60.59 |
| CRFM10B         | 91.67                | 84.94 | 81.60 | 76.48 | 87.44 | 80.70 | 77.41 | 72.29 |
| CRFM20B         | 90.01                | 82.84 | 78.90 | 74.15 | 85.86 | 78.68 | 74.97 | 70.04 |
| CRFM30B         | 80.40                | 73.66 | 69.98 | 65.18 | 76.69 | 69.90 | 66.31 | 61.54 |
| CSFM0B          | 82.32                | 75.75 | 72.29 | 67.50 | 78.96 | 72.22 | 68.77 | 64.11 |
| CSFM10B         | 95.91                | 88.80 | 85.48 | 80.47 | 92.47 | 85.23 | 81.91 | 76.89 |
| CSFM20B         | 93.16                | 85.91 | 82.19 | 77.56 | 89.82 | 82.44 | 78.71 | 74.08 |
| CSFM30B         | 84.24                | 77.49 | 73.96 | 68.68 | 81.21 | 74.29 | 70.76 | 65.58 |
| CTFM0B          | 83.99                | 77.82 | 74.52 | 69.39 | 79.29 | 73.11 | 69.75 | 64.75 |
| CTFM10B         | 98.34                | 91.31 | 87.72 | 83.40 | 93.33 | 86.36 | 82.71 | 78.38 |
| CTFM20B         | 96.94                | 89.64 | 85.73 | 81.60 | 92.00 | 84.75 | 80.77 | 76.68 |
| CTFM30B         | 86.73                | 80.25 | 76.62 | 70.94 | 82.30 | 75.82 | 72.12 | 66.61 |
| Mix Designation | Compressive strength | Residual Compressive Strength | Mix Designation | Compressive strength | Residual Compressive Strength |
|----------------|----------------------|------------------------------|----------------|----------------------|------------------------------|
|                | MPa                  | Days                         |                | MPa                  | Days                         |
| CQFM10A        | 76.20                | 69.11                        | CQFM0C         | 72.40                | 65.41                        |
| CQFM10B        | 88.31                | 80.80                        | CQFM10C        | 84.78                | 77.37                        |
| CQFM20A        | 86.06                | 78.24                        | CQFM20C        | 82.61                | 74.90                        |
| CQFM30A        | 77.13                | 70.08                        | CQFM30C        | 73.25                | 66.35                        |
| CQFM10A        | 78.04                | 71.19                        | CQFM10C        | 74.14                | 67.35                        |
| CQFM10B        | 91.17                | 83.65                        | CQFM10C        | 87.05                | 79.64                        |
| CQFM20A        | 89.19                | 81.40                        | CQFM20C        | 85.16                | 77.47                        |
| CQFM30A        | 79.79                | 72.87                        | CQFM30C        | 75.79                | 68.97                        |
| CQFM10A        | 79.88                | 73.10                        | CQFM10C        | 75.89                | 69.18                        |
| CQFM10B        | 94.02                | 86.63                        | CQFM10C        | 89.32                | 82.02                        |
| CQFM20A        | 92.32                | 84.70                        | CQFM10C        | 87.20                | 80.17                        |
| CQFM30A        | 82.46                | 75.42                        | CQFM30C        | 78.34                | 71.38                        |
| CQFM10A        | 84.00                | 76.81                        | CQFM30C        | 80.63                | 73.38                        |
| CQFM10B        | 98.37                | 90.72                        | CQFM10C        | 93.45                | 85.81                        |
| CQFM20A        | 95.55                | 87.89                        | CQFM10C        | 90.78                | 83.11                        |
| CQFM10B        | 86.40                | 78.97                        | CQFM10C        | 82.08                | 74.67                        |
| CQFM20A        | 86.14                | 78.81                        | CQFM10C        | 81.84                | 74.67                        |
| CQFM10B        | 100.35               | 92.86                        | CQFM10C        | 96.34                | 88.93                        |
| CQFM20A        | 98.92                | 91.16                        | CQFM10C        | 94.96                | 87.28                        |
| CQFM20B        | 88.50                | 80.87                        | CQFM10C        | 84.96                | 77.42                        |
| CQFM20B        | 74.30                | 67.26                        | CQFM10C        | 71.63                | 64.46                        |
| CFM10B         | 86.55                | 79.09                        | CQFM10D        | 83.01                | 75.56                        |
| CQFM10B        | 84.34                | 76.57                        | CQFM10D        | 80.89                | 73.13                        |
| CQFM20B        | 75.19                | 66.22                        | CQFM10D        | 72.03                | 65.05                        |
| CQFM20B        | 76.09                | 66.27                        | CQFM10D        | 73.34                | 66.38                        |
| CQFM10B        | 89.11                | 81.64                        | CQFM10D        | 85.23                | 77.77                        |
| CQFM20B        | 87.17                | 79.44                        | CQFM10D        | 83.37                | 75.64                        |
| CQFM20B        | 77.79                | 70.92                        | CQFM10D        | 74.36                | 67.46                        |
| CFM10B         | 77.88                | 71.14                        | CQFM10D        | 75.06                | 68.23                        |
| CFM10B         | 91.67                | 84.32                        | CQFM10D        | 87.44                | 80.09                        |
| CFM20B         | 90.01                | 82.43                        | CQFM10D        | 85.86                | 78.27                        |
| CFM20B         | 80.40                | 73.39                        | CQFM10D        | 76.69                | 69.67                        |
| CFM20B         | 82.32                | 75.09                        | CQFM10D        | 78.96                | 71.66                        |
| CFM10B         | 95.91                | 88.26                        | CQFM10D        | 92.47                | 84.68                        |
| CFM20B         | 93.16                | 85.50                        | CQFM10D        | 89.82                | 82.02                        |
| CFM20B         | 84.24                | 76.81                        | CQFM10D        | 81.21                | 73.67                        |
| CFM10B         | 83.99                | 76.74                        | CQFM10D        | 79.29                | 72.13                        |
| CFM10B         | 98.34                | 90.89                        | CQFM10D        | 93.33                | 85.94                        |
| CFM20B         | 96.94                | 89.22                        | CQFM10D        | 92.00                | 84.33                        |
| CFM30B         | 86.73                | 79.14                        | CQFM10D        | 82.30                | 74.79                        |

Table 4. Water cured compressive strength at 28 days and H₂SO₄ immersed residual compressive strength results.
3.2. Sample Preparation, Curing, and Testing

Samples were prepared by mixing cement, fine aggregate, FA, and MK thoroughly by manual means first to achieve a uniform mix, and then composite fibers were added to the mixture, followed by coarse aggregates and water mixed with a superplasticizer.

80 mixes were prepared, and for each mix, 30 cubes, specimens of 100 mm were cast for each water binder ratio.

As initial curing, a wet cloth was used for covering the exposed portion of all 30 specimens before demoulding. After the concrete was set, specimens were demoulded, and out of 30 cube specimens cast for each mix, 3 cubes were cured in a transparent water tank at 27° ± 2°C until 28 days testing age. And out of the remaining 27 cubes, 9 cubes each was immersed in 5% concentration solutions of HCl, MgSO₄, and H₂SO₄. Out of 9 cubes immersed in acids, 3 cubes each was immersed for 30, 60, and 90 days.

After the curing period for the specified testing age, all 30 samples were removed from the water and acids and dried under the shade. Among these mentioned 28 days, compressive strength was tested for 3 water cured cubes on the 3000 kN AIMIL make digital compression testing machine by applying a constant rate of loading up to the failure of the specimens. AIMIL compression testing machines are compliant with IS: 14858(2000) and are calibrated to a ± 1% accuracy, as required by 1828 (Class1). And out of the remaining 27 acids immersed cubes, 3 cubes each was tested at 30, 60, and 90 days for residual compressive strength for each type of acid immersion on the same machine.

4. Results and Discussion

Residual compressive strengths of various fly ash and metakaolin-based CFRHPC are presented in Tables 2, 3, and 4.

4.1. Effects of Water Binder Ratios on Residual Compressive Strength of CFRHPC

The 30, 60, and 90 days residual compressive strength after acid immersions with varying water binder ratios are presented in Figs. 1, 2, 3, 4, 5, 6, 7, 8 and 9 respectively.

Values presented in Tables 2, 3, and 4 represent the 30, 60, and 90 days residual compressive strength results immersed in HCl, MgSO₄, and H₂SO₄ respectively. It is evident that 30, 60, and 90 days residual compressive strength of mixes tried decreased when water binder ratio was increased, and all other mixes followed the same trend. Maximum residual compressive strength was obtained for a mix with a 0.275 water binder ratio and was valid for all other mixes with different percentages of cement replacements and the addition of composite fibers.

The maximum compressive strength retained after 30, 60, and 90 days for HCl immersion were 94.40 MPa, 91.03 MPa, and 85.90 MPa, respectively, for the CTFM10A mix. Further, for the same mix, when the water binder ratio was increased to 0.3, its 30 days residual compressive strength was reduced by 2.11% with respect to the CTFM10A mix, and it was further reduced to 4.21% and 7.40% for W/B ratios of 0.325 and 0.35 respectively with respect to CTFM10A mix. Similarly, 60 days residual compressive strength was reduced by 2.27%, 4.53%, and 7.83%, and 90 days residual compressive strength was reduced by 2.36%, 4.71%, and 8.20%, respectively, with reference to the residual compressive strength of the respective 0.275 water binder ratio.

Similar behaviour was witnessed for mixes immersed in MgSO₄ and H₂SO₄ (Refer Figs 4 to 9), wherein deterioration of compressive strength increased with increasing water binder ratios. The maximum residual strength was obtained in mixes with 0.275 water to binder ratio, as less water was available for hydration in the concrete leading to early strength gain.
Figure 2. Residual compressive strength versus water binder ratios for various CFRHPC mixes immersed in HCl for 60 days

Figure 3. Residual compressive strength versus water binder ratios for various CFRHPC mixes immersed in HCl for 90 days
Figure 4. Residual compressive strength versus water binder ratios for various CFRHPC mixes immersed in MgSO₄ for 30 days.

Figure 5. Residual compressive strength versus water binder ratios for various CFRHPC mixes immersed in MgSO₄ for 60 days.
Figure 6. Residual compressive strength versus water binder ratios for various CFRHPC mixes immersed in MgSO$_4$ for 90 days

Figure 7. Residual compressive strength versus water binder ratios for various CFRHPC mixes immersed in H$_2$SO$_4$ for 30 days
Figure 8. Residual compressive strength versus water binder ratios for various CFRHPC mixes immersed in H$_2$SO$_4$ for 60 days

Figure 9. Residual compressive strength versus water binder ratios for various CFRHPC mixes immersed in H$_2$SO$_4$ for 90 days
4.2. Effects of Cement Replacement by Fly Ash and Metakaolin and Age of Curing on Residual Compressive Strength of CFRHPC

The deterioration in compressive strength after 30, 60, and 90 days of acid immersion for W/B of 0.275 with varying percentages of FA and MK is presented in Figs. 10, 11, and 12, respectively.

Figure 10. Residual compressive strength versus percentages of FA and MK for various CFRHPC mixes immersed in HCl immersion.

It can be witnessed from these figures that the depreciation of compressive strength decreased with cement being replaced by combined FA and MK percentages from 0 to 10. The addition of FA and MK enhanced the load-carrying capacity of the mix as both FA and MK improved the pore size distribution and the pore shape of concrete. Further increase in the percentage of mineral admixtures, i.e., 20 and 30, decreased the residual compressive strength as these admixtures acted only as fillers, but not less than the plain CPFM0A mix and was true for all mixes of CFRHPC designed in this analysis. Maximum residual compressive strength was obtained for 10% cement replacement for all ages of curing and type of acid immersions. From these figures, it was also evident that as the age of acid immersion was increased, residual compressive strength decreased as the deteriorating effect of the acid increased and was true for all CFRHPC mixes. The maximum residual compressive strengths obtained for the CTFM10A mix were 94.40 MPa, 91.03 MPa, and 85.90 MPa for 30, 60, and 90 days HCl immersion. Similarly, 93.28 MPa, 89.78 MPa, and 85.43 MPa for 30, 60, and 90 days MgSO4 immersion and 92.86 MPa, 89.10 MPa, and 83.59 MPa for 30, 60, and 90 days H2SO4 immersion. Compressive strength of CFRHPC mix CTFM10A attacked by HCl reduced by 5.93%, 9.29%, and 14.40% for 30, 60, and 90 days compared to the same water cured mix at 28 days. Similarly, MgSO4 reduced compressive strength by 7.04%, 10.54%, and 14.87%, and H2SO4 by 7.47%, 11.21%, and 16.71% for 30, 60, and 90 days compared to the same water cured mix at 28 days. Residual compressive strength decreased with the age of acid immersion, with a maximum depreciation in compressive strength witnessed at 90 days of all three acid curing due to an increase in the formation of ettringites with the age of acid immersion.

Figure 11. Residual compressive strength versus percentages of FA and MK for various CFRHPC mixes immersed in MgSO4 immersion.
4.3. Effects of Volumes of Composite Fibers on Residual Compressive Strength of CFRHPC

The deterioration in compressive strength after acid immersions for W/B of 0.275 with varying percentages of composite fibers is presented in Figs. 13, 14, and 15, respectively.

From Figs. 13, 14, and 15, it can be perceived that with an escalation in the composite fiber volume, the residual compressive strength increased. This trend was true for all the types of CFRHPC mixes tried in this study. For every increase in 0.25% GF and constant PPF of 0.25% dosages, there was an average increase in residual compressive strength by 3.67%, 7.48%, 12.84%, and 16.59% for mixes with the same composite fiber dosages when compared to mixes without composite fibers for HCl cured mixes. Similarly, the residual compressive strength increased by 3.62%, 7.51%, 12.91%, and 16.59 % for MgSO₄ cured mixes and by 4%, 8.03%, 13.60%, and 17.39% for H₂SO₄ cured mixes. The residual compressive strength of mixes prepared without composite fibers was considerably lesser than those prepared with composite fibers, as these fibers were effective in arresting both macro and micro level cracks. The development of microcracks was stopped by fibers at a micro level. The quantity of fibers played a significant role in controlling the growth of microcracking. At the macro level, crack openings were controlled by fibers, increasing the composite's energy absorption capacity.

Figure 12. Residual compressive strength versus percentages of FA and MK for various CFRHPC mixes immersed in H₂SO₄.

Figure 13. Residual compressive strength versus percentages of composite fibers for various CFRHPC mixes immersed in HCl.
Figure 14. Residual compressive strength versus percentages of composite fibers for various CFRHPC mixes immersed in MgSO$_4$.

Figure 15. Residual compressive strength versus percentages of composite fibers for various CFRHPC mixes immersed in H$_2$SO$_4$. 
4.4. Effects of Type of Acid on Residual Compressive Strength of CFRHPC

CFRHP mixes were subjected to 5 percent concentrated solutions of HCl, MgSO₄, and H₂SO₄. The effect of these acids on the deterioration of compressive strength in different CFRHPC mixes with W/B of 0.275 is presented in Figs. 16, 17, 18, and 19, respectively.

From Figs 16, 17, 18, and 19, it is evident that maximum depreciation of compressive strength was reported for cubes with H₂SO₄ acid immersion than that of HCl and MgSO₄. The same trend was followed by all the three dosages of mineral admixtures tried in the present investigation. Out of the three acids HCl, MgSO₄, and H₂SO₄, the HCl acid immersion reported the least loss of compressive strength for all mineral admixtures replacement levels of cement and the addition of composite fibers. For the CTFM10A mix, the deteriorated compressive strength after 30 days of HCl immersion was 94.40 MPa, and it was reduced to 93.28 MPa and 92.86 MPa for MgSO₄ and H₂SO₄ with a reduction of 1.18% and 1.63% in comparison with 30 days HCl cured CTFM10A mix. Similarly, there was a reduction of 1.38% and 2.12% and 0.55% and 2.70% for 60 and 90 days residual compressive strengths, respectively indicating the severity of H₂SO₄ as it led to the formation of CaSO₄ which reacted with C3A, resulting in the formation of expansive ettringite which leads to deterioration of concrete’s compressive strength. Similar trends were observed for other mixes as well. It can be concluded that the attack of H₂SO₄ was major on CFRHPC, and that of HCl was the least out of three acids tried in this study.

Figure 16. Residual compressive strength versus days of acid curing for various CFRHPC mixes with 0% mineral admixtures (0% Fly ash and 0% Metakaolin)
**Figure 17.** Residual compressive strength versus days of acid curing for various CFRHPC mixes with 10% mineral admixtures (5% Fly ash and 5% Metakaolin)

**Figure 18.** Residual compressive strength versus days of acid curing for various CFRHPC mixes with 20% mineral admixtures (10% Fly ash and 10% Metakaolin)
4.5. Microstructure of CFRHPC

The SEM, XRD, and EDX analysis were used to assess the microstructural properties of CFRHPC blends. The experiments were also conducted to confirm the improvements that occurred as a result of the substitution of fly ash and metakaolin for cement and additions of glass and polypropylene fiber.

The micromorphology characteristic images of fly ash and metakaolin are shown in Figs. 20 and 21, respectively. Figs. 22 and 23 show SEM images of plain cement concrete at W/B of 0.275.
Figure 23. SEM image of plain CPFM0A mix at W/B of 0.275 showing poor interface.

Figure 24. SEM image of FA and MK based CTFM10A mix.

Figure 25. SEM image of FA and MK based CTFM30A mix.

Figure 26. SEM image of ettringites in CTFM30A mix due to acid immersion.

Figure 27. SEM image of Glass fiber.

Figure 28. SEM image of Polypropylene fiber.
Figs. 22 and 23 illustrate the SEM representation of a plain CPFM0A mixture. It exhibited a lack of strong interfacial transition zones between the mortar phase and the aggregates, as well as the occurrence of microcracks due to the lack of fibers. This reduces the compressive strength of concrete.

From Fig. 24, a dense mortar mix with a low number of voids was observed in the SEM micrograph of the CTFM10A mix. Improved mechanical efficiency was achieved by proper proportioning and distribution of FA, MK, and cement. In Fig. 25, the CTFM30A mix showed a partially dense mortar matrix and a greater number of voids than the CTFM10A mix, indicating that FA and MK serve solely as fillers at that proportion of admixtures. As shown in Fig. 26, the \( \text{H}_2\text{SO}_4 \) resulted in the formation of expansive ettringite as a result of the formation of \( \text{CaSO}_4 \), which reacted with C3A, resulting in the weakening of the compressive strength of the concrete.

SEM images of glass fiber in a CTFM10A mix specimen with a w/b of 0.275 are shown in Fig. 27. As seen in the figure, the GF and cement matrix were extremely tightly bonded, which could be clarified by the fact that GF is a mineral fiber material with a high degree of hydrophilicity. The high quality of the fiber-matrix bonding created favorable conditions for GF to increase the concrete's strength characteristics and decrease its water absorption. The GF, which had its surface coated with a densely hardened cement matrix, acted as a filler in the pores, thus reducing porosity.

SEM images of polypropylene fiber in a CTFM10A mix specimen with a w/b of 0.275 are shown in Fig. 28. The micromorphology of PPF is seen in the figure; the loose bonding between the PPF and matrix is visible, degrading the concrete's strength properties. In comparison to Fig. 27, the bond quality between the PPF and the cement matrix was significantly lower than that between the GF and the cement matrix due to the inherent hydrophobicity of PPF as a synthetic fiber. However, it was discovered that the micro bumps (a cement hydration product) on the surface of the PPF created an irregular fiber surface and created a physical interlocking effect between the interfaces, thus increasing the interface strength between the PPF and matrix. From this vantage point, adding PPF improves the strength properties of concrete [30-31].

Along with the SEM test, the EDX test (Energy Dispersive X-Ray Analysis) was performed to determine the level of CH in plain concrete and the level of CSH in CTFM10A and CTFM20A concrete containing fly ash and metakaolin, as illustrated in Fig. 29, 30, and 31.

The EDX analysis revealed the existence of C–S–H and CH as the primary hydrated products in ordinary Portland cement, while FA and MK paste contained C–A–S–H and C–A–H.
Additionally, the Na/Si, Al/Si, and Ca/Si ratios depicted in the figures decrease as the FA and MK replacement levels are increased. These findings are consistent with the pore solution’s alkalinity data and the high Al and low Ca contents of the FA and MK. The low Ca/Si ratios observed in these mixtures can account for the amorphous nature of the alkali-silica reaction products produced.

The present investigation authenticates the dominance of CFRHPC manufactured with FA and MK as partial replacement of cement to resist aggressive acidic environments. The conclusions of the present analysis are as below.

- The loss in compressive strength of CFRHPC mixes increased along with increased water to binder ratio. Maximum residual compressive strength was obtained for a mix with a 0.275 water binder ratio and is valid for all other mixes with different percentages of cement replacements, the addition of composite fibers, and the type of acid immersed.
- FA and MK based CFRHPC mix counterattacked the acid attack effectively in comparison to plain CPM0A/B/C/D mixes at all ages of HCl, MgSO₄, and H₂SO₄ exposure.
- It was perceived that the deterioration in compressive strengths of all CFRHPC mixes are substantially lower than that of plain CPM0A/B/C/D mix at all ages of HCl, MgSO₄, and H₂SO₄ exposures.
- The degradation of compressive strength in CFRHPC mixes due to acid attack was least at 10% (5%FA+5%MK) combined cement replacement. Hence, 10% replacement, i.e., 5% each of FA and MK, is considered the optimum dosage.

5. Conclusions

The XRD patterns of plain CPM0A mix and CTFM10A mix are shown in Figs.32 and 33. The XRD pattern in Fig. 33 demonstrates the existence of the gismondine phase, which can form in cementitious systems containing a high proportion of FA and MK and a high Al/Ca ratio. The formation of calcium aluminate silicate is favored by the high initial pH and fast dissolution of Si and Al amorphous phases.
The degradation of compressive strength in CTFM10A mix is 16.71% for FA and MK based CFRHPC mixes after 90 days of dipping in H₂SO₄ acid, while a loss of 22.09% was reported in similar exposure for plain CFPM0A mix. This proves the superiority of FA and MK based CFRHPC in fighting acid attacks.

The loss in compressive strength of CFRHPC increased with an increase in the duration of acid curing. Maximum degradation of compressive strength has been witnessed at 90 days of acid immersion. The behaviour of all acids tried in the present analysis followed the same trend.

Degradation of compressive strength decreases with the addition of composite fibers. The minimum degradation in compressive strength was obtained for mixes with 1% GF and 0.25% PFF dosage and is applicable for all mixes tried in this analysis.

The maximum harm to compressive strength happened in the circumstance of H₂SO₄ acid curing compared to HCl and MgSO₄ acids. Out of the three acids, the least degradation of compressive strength was documented for HCl acid immersion.

Thus, it can be concluded that the combined effect of FA and MK at 5% each as replacement of cement and the addition of composite fiber dosage of GF=1% and PPF=0.25% for W/B of 0.275 was found to be the optimum combination to obtain maximum acid attack resistance for CFRHPC.

The SEM, XRD, and EDX analysis confirm the improvements that occurred as a result of the substitution of fly ash and metakaolin for cement and additions of glass and polypropylene fiber in composite fiber-reinforced high-performance concrete.

The conclusions above demonstrate the viability of using FA and MK and composite fibers (GF and PFF) in CFRHPC production, which minimizes enormous cement production and safeguards the environment from pollution throughout its service life.

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