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A Comparative Study on the Microstructure and Mechanical Behavior of Geopolymer and High-Performance Concrete Using SEM and XRD

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ABSTRACT
This paper presents the detailed experimental studies on surface morphology and microstructures of geopolymer concrete compared with high performance concrete using SEM and XRD analysis which reviews in advancement in the field of geopolymer concrete with 16 molarity as a sustainable development in the construction industry. Thus, improving its properties geopolymer concrete is reinforced with glass fibers, GGBS, fly ash is incorporated with percentages of 60:40 along with alkaline fluid ratios of $Na_2SiO_3/NaOH$ is 2.5 and fluid binder ratio 0.26. The modified fiber reinforced geopolymer concrete (MFRGC) enhances its compressive and flexural strength in comparison to high performance fiber reinforced concrete (HPFRC). Though significant research has not been done in the past decades, more challenges need to be addressed. For surface morphology Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) test are performed as per the standard procedures. The test specimens consist of Mix 1 - M-60 (M-60 Grade of concrete, control mix), Mix 2 - M-60+GF (Glass fiber combined with M-60 Grade of concrete), Mix 3 - GPC-16M (Geopolymer concrete with 16 Molarity) and Mix 4 - GPC-16M+GF (Geopolymer concrete with 16 Molarity combined with Glass fiber). The present experimental results have shown that there is improvement in strength properties of modified fiber reinforced geopolymer concrete under consideration. The result proves that compressive and flexural strength has improved for Mix 4 - GPC-16M+GF in comparison with other matrixes used.
Key Words: *Modified fiber reinforced geopolymer concrete, High performance concrete, SEM*

1. INTRODUCTION

Cement production is a major environmental concern, accounting for approximately 7% of global carbon dioxide emissions [24][27]. To reduce its contribution to global warming, supplementary cementitious materials such as fly ash, silica fume, and copper slag are increasingly being used as partial replacements for cement in the construction industry [15][19][28]. These materials not only help decrease CO_2 emissions but also improve the sustainability and performance of concrete [21][26].

In light of the growing concerns over greenhouse gas emissions, geopolymer-based materials have attracted significant research interest [23][30]. Ground Granulated Blast Furnace Slag (GGBS) is a by-product of the iron and steel industries [14]. Geopolymer concrete has been the subject of extensive research over the years to evaluate its viability as a sustainable construction material [17][31]. A significant amount of research has focused on fly ash-based geopolymer concrete [25]. Alkaline liquid activators, including silicates and hydroxides, are widely used to initiate the geopolymerization process [16][29].

The development of compressive strength in geopolymer systems occurs through the polycondensation of silica and alumina [31,32]. The presence of a high alkali content further enhances these reactions, contributing significantly to the material's overall strength and durability [13][20]. To investigate the microstructural characteristics of geopolymer concrete, Energy Dispersive X-ray Spectroscopy (EDX), X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) are key techniques for analyzing the microstructure of cementitious materials [18][22]. Additionally, XRD is used to determine the crystalline phases present, offering complementary insights into the mineralogical composition and enhancing the overall understanding of geopolymer-based materials [26][30]. The findings from these analyses aim to support the development of sustainable fiber-reinforced geopolymer concrete and contribute to the advancement of environmentally friendly, green building, and sustainable development of construction practices in the modern era [15][23][31].

2. EXPERIMENTAL PROGRAM

The present experimental program was designed to investigate the compressive and flexural strength in comparison to high performance fiber reinforced concrete of grade M60.The experimental study includes casting, curing and testing of thirty-six (36) number of cube and flexural specimens having size 150x150x150mm and 500x100x100mm respectively. All the test specimens are cured for 28 days. The four different concrete matrices considered in this present investigations are Mix 1 - M-60 (M-60 Grade of concrete, control mix),Mix 2 - M-60+GF (Glass fiber combined with M-60 Grade of concrete),Mix 3 - GPC-16M (Geopolymer concrete with 16 Molarity) and Mix 4 - GPC-16M+GF (Geopolymer concrete with 16 Molarity combined with Glass fiber) were used.

3. MATERIALS AND MIX PROPORTION

In the present experimental investigation, the binder system for geopolymer concrete consisted of 40% Class-F fly ash and 60% Ground Granulated Blast Furnace Slag (GGBS) [16][24]. Manufactured sand (M-sand) was used as the fine aggregate, while crushed angular coarse aggregates of sizes 20 mm and 12.5 mm were selected for the coarse fraction [21][28]. Aurocast 270M was used as a superplasticizer (chemical admixture), along with glass fibers and potable water for mixing [14][29]. An alkaline activator solution was prepared with a solution-to-binder ratio of 0.25. The ratio of sodium silicate (Na_2SiO_3) to sodium hydroxide (NaOH) within this solution was maintained at 2.5 to ensure effective geopolymerization [18][26]. All physical property tests of the constituent materials were conducted in accordance with relevant Indian Standard specifications [19][30]. The mix design for both modified fiber-reinforced geopolymer concrete and high-performance fiber-reinforced concrete (HPFRC) was carried out as per IS 10262:2019, following a series of preliminary trial mixes [17][31]. The final mix proportions were selected based on the achieved compressive strengths. The four mix combinations considered in this study are:

- **Mix 1 – M60**: Control mix with M60 grade concrete,
- **Mix 2 – M60 + GF**: M60 grade concrete reinforced with glass fibers,
- **Mix 3 – GPC-16M**: Geopolymer concrete using a 16 Molar activator,
- **Mix 4 – GPC-16M + GF**: Geopolymer concrete (16M) reinforced with glass fibers

The compressive strength results for each of these mixes are presented in **Table 1**, providing a comparative overview of their mechanical performance under standard curing conditions.

Table No. 1: Mix Proportions

Material	Mix 1 (kg/m³)	Mix 2 (kg/m³)	Mix 3 (kg/m³)	Mix 4 (kg/m³)
Cement	450	450	-	-
Fly Ash	64	64	184	184
GGBS	128	128	276	276
Fine Agg.	468	468	533.5	533.5
Coarse Agg.	1125	1125	1291	1291

NaOH	-	-	33	33
Na_2SiO_3	-	-	82.5	82.5
Water	169	169	78.5	78.5
Glass Fiber	-	1% vol.	-	1% vol.

TESTS AND RESULTS

Concrete structures are generally designed based on the assumption that concrete can resist compressive forces but has limited tensile strength. Consequently, compressive strength is considered the most important property of concrete in structural design in RCC members, as it provides a reliable indication of the overall quality and performance of the concrete. Compression testing is relatively easy to perform and produces consistent and dependable results, making it the most used method for evaluating concrete's structural suitability. In this study, compressive strength and flexural strength are examined to offer a comprehensive understanding of the mechanical behavior of modified fiber reinforced geopolymer and high-performance fiber reinforced concrete under various loading conditions.

Table No. 2 Summary of 7-, 14 and 28-days Compressive Strength of Test Specimens

Properties	Age in Days	Mix 1	Mix 2	Mix 3	Mix 4
Compressive strength N/mm^2	7	45.18	47.01	48.52	51.01
	14	56.98	58.69	60.74	62.59
	28	68.57	70.95	72.64	75.28

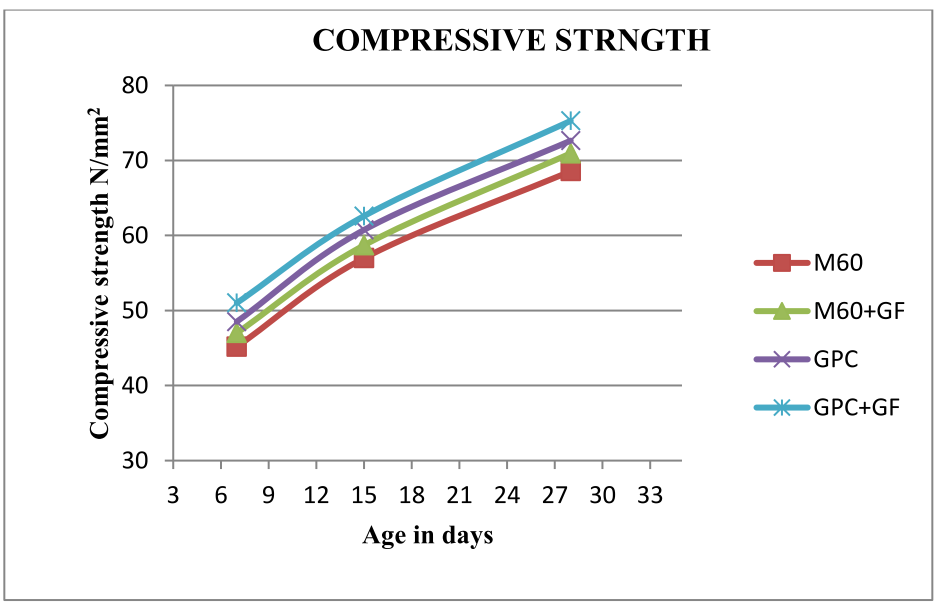


Figure No.1 Compressive Strength with different concrete matrices

Table No. 2 Summary of 7-, 14- and 28-days Flexural Strength of Test Specimens

Properties	Age in Days	Mix 1	Mix 2	Mix 3	Mix 4
Flexural strength N/mm^2	7	5.12	5.54	5.91	6.26
	14	5.64	6.05	6.48	6.97
	28	6.96	7.37	7.88	8.29

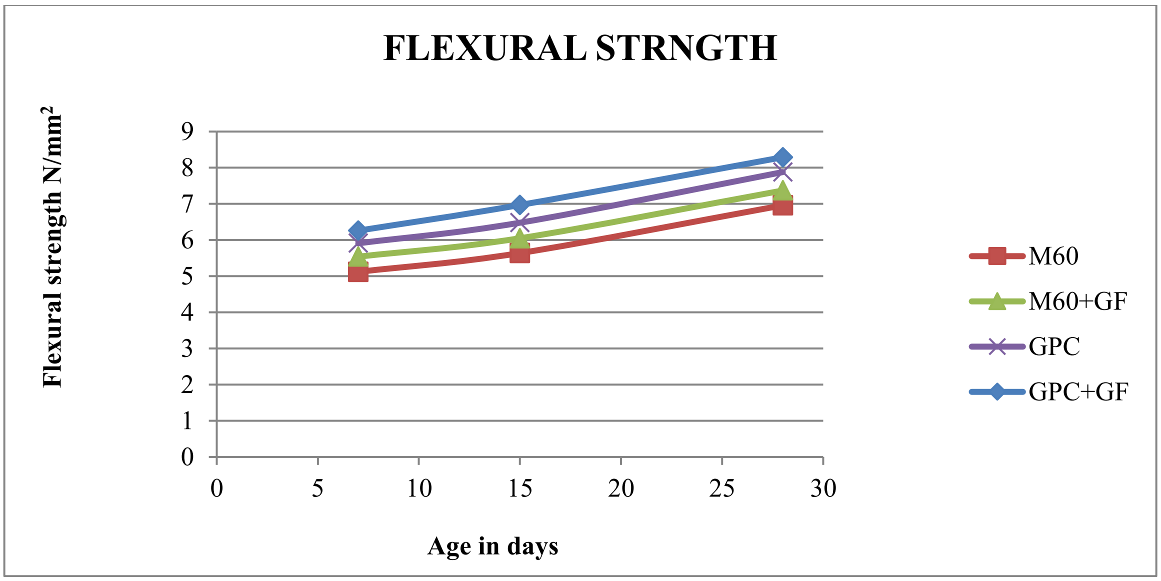
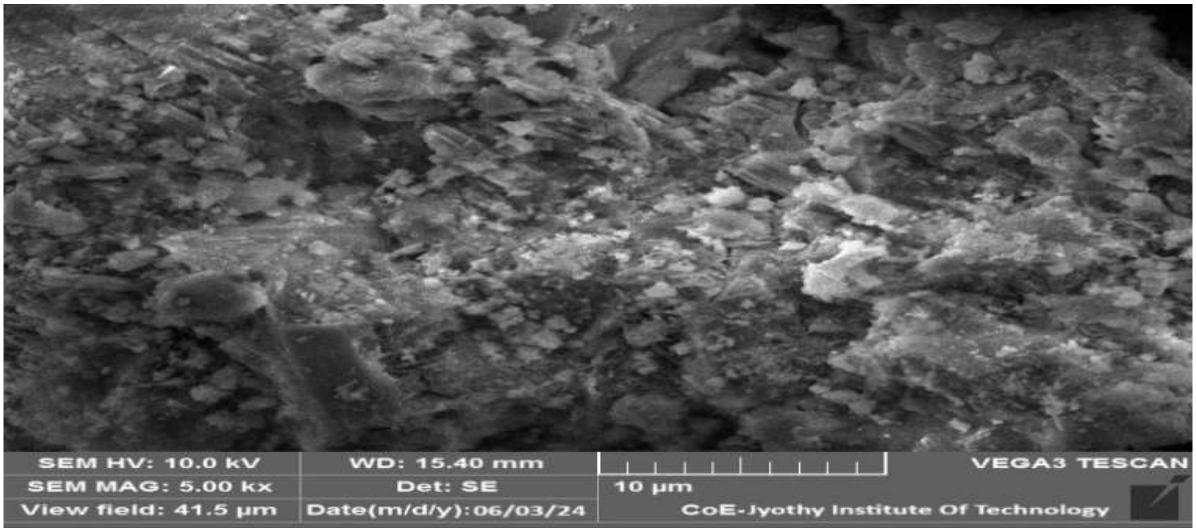


Figure No.2 Flexural Strength with different concrete matrices

SEM (SCANNING ELECTRONIC MICROSCOPE) AND XRD ANALYSIS

For Scanning Electron Microscopy (SEM) analysis, particles of High-Performance Fiber Reinforced Concrete and Modified Fiber Reinforced Geopolymer Concrete are collected in powder form (concrete samples are finely ground into a powder) in accordance with the different concrete matrices. The procedure involves as follows: oven-dried and coated with a conductive material such as gold or carbon to prevent charging during imaging. These samples are then placed in the SEM chamber, where a focused electron beam scans the surface to produce high-resolution images of 5K magnification were considered [33,34]. These images provide detailed information about surface morphology, microcracks, pores, and reaction products such as calcium silicate hydrate (C-S-H) in HPFRC or geopolymer gel in MFRGC. For X-ray Diffraction (XRD) analysis resulting diffraction pattern is analyzed to identify and quantify the crystalline phases present. This data helps assess the degree of hydration in HPFRC and the extent of polymerization in MFRGC, contributing to a better understanding of their microstructural properties.

MIX-1 : M60



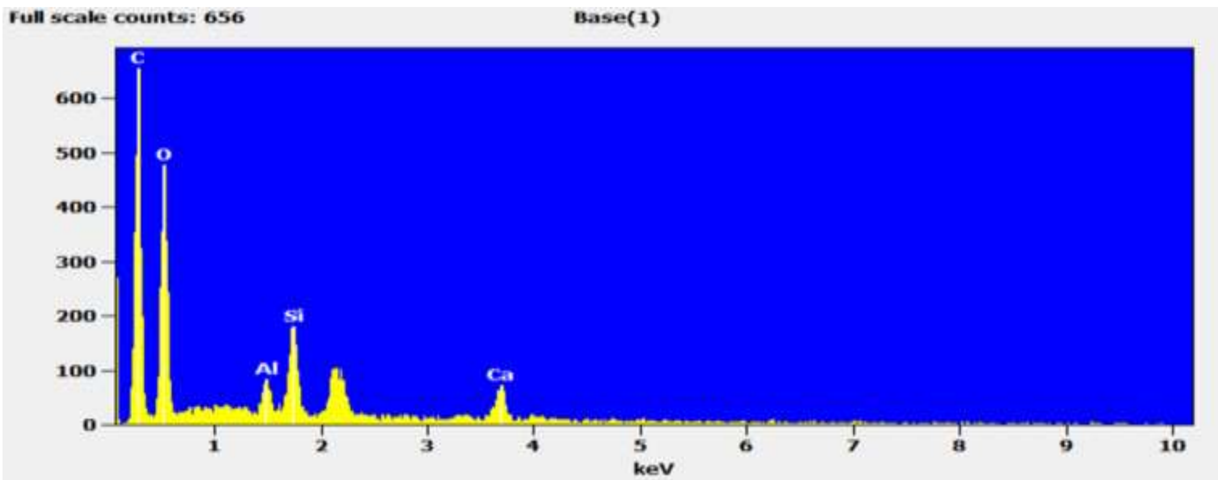
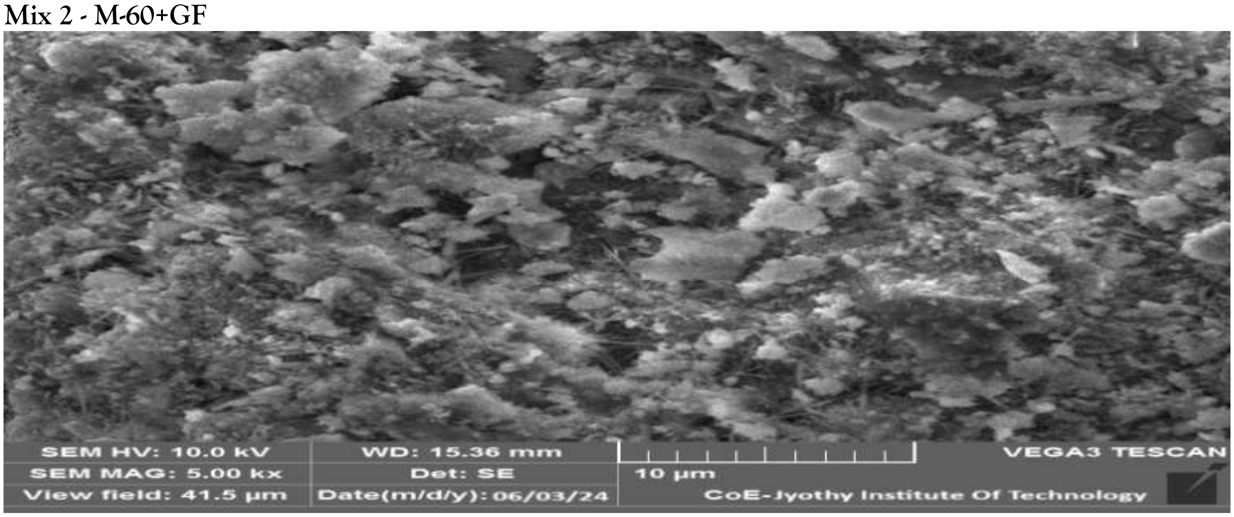


Figure No.3 : Energy Dispersive Micro Analysis on M60 Concrete

Mix 2 - M-60+GF



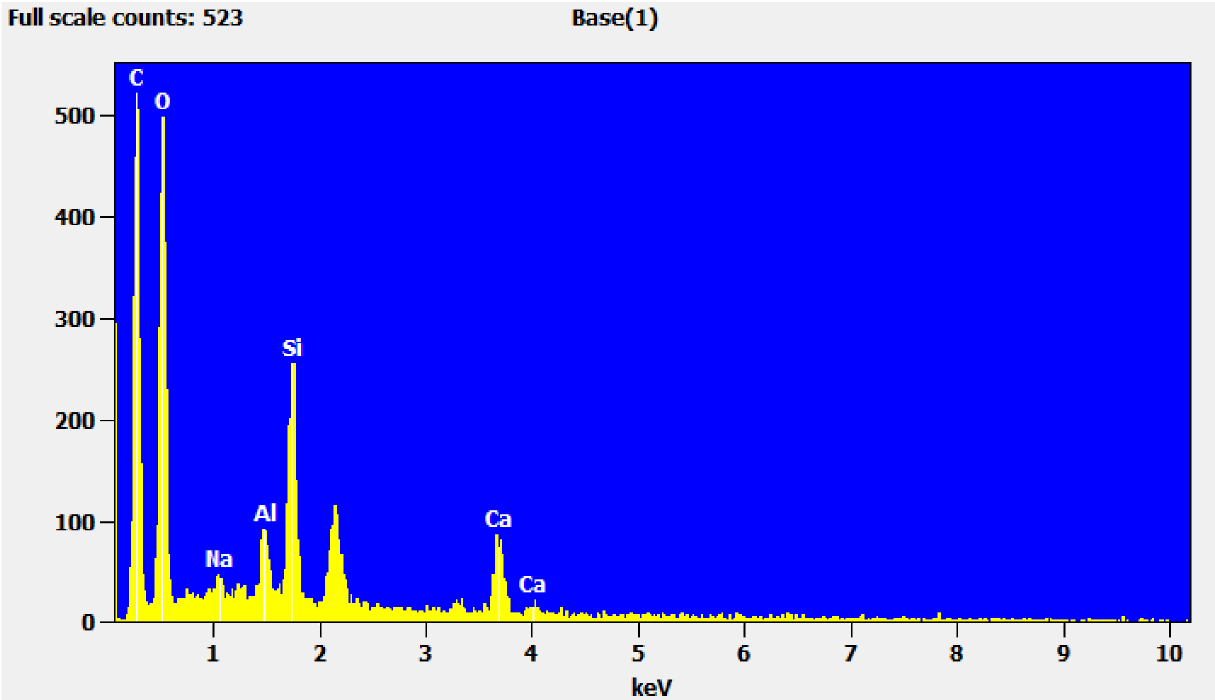
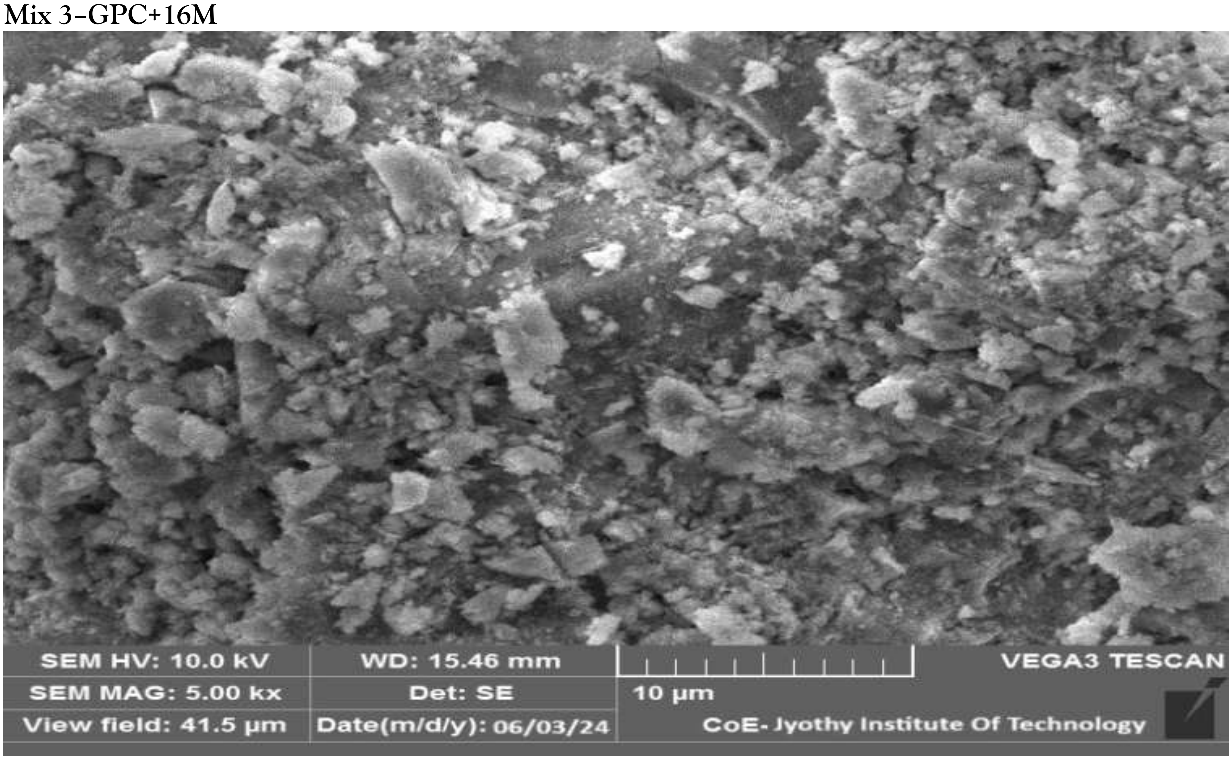


Figure 4: Energy Dispersive Micro Analysis on M60+GF Concrete

Mix 3–GPC+16M



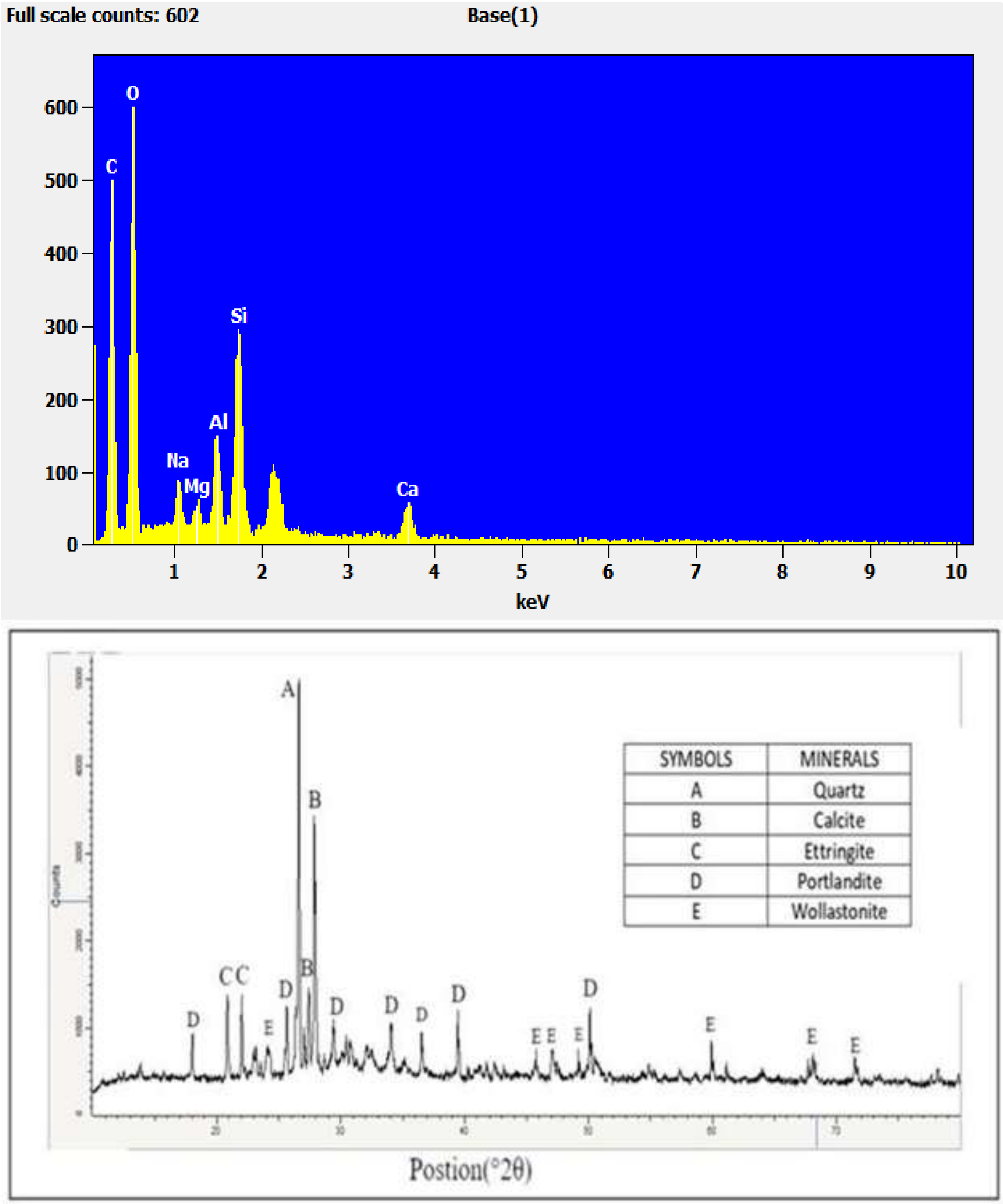
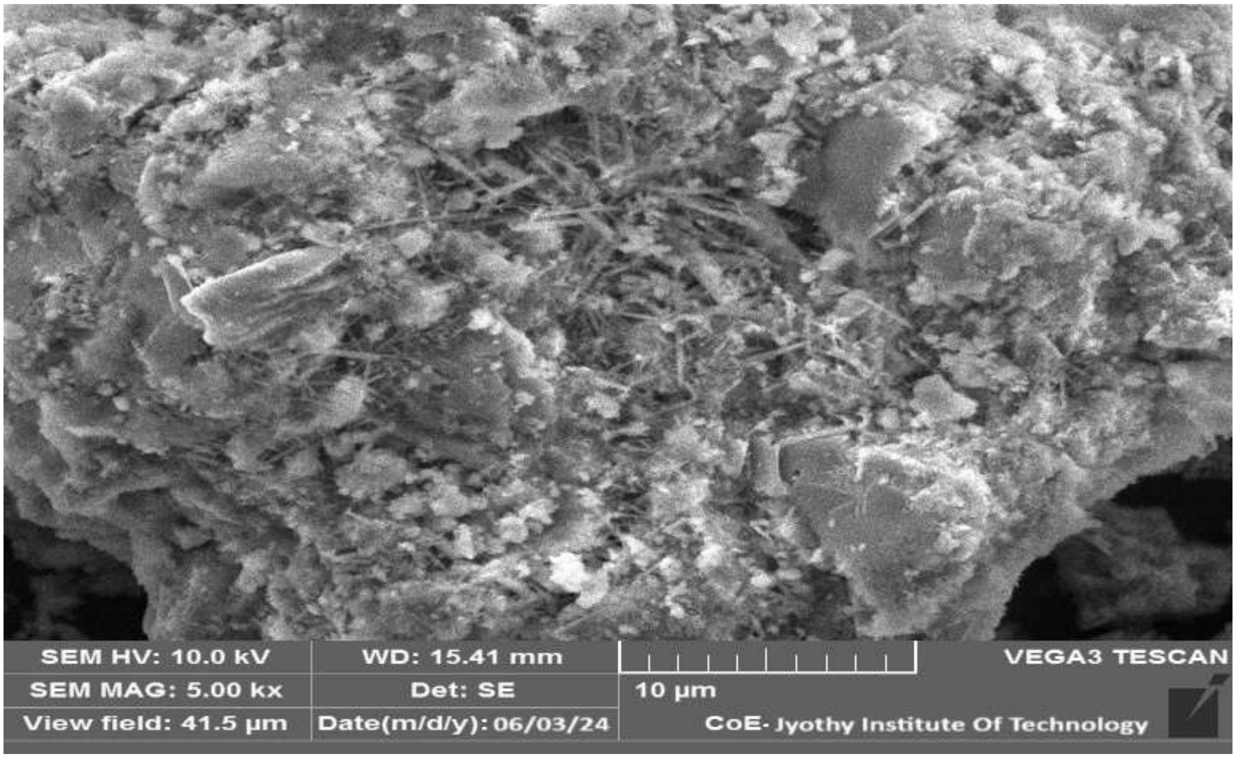
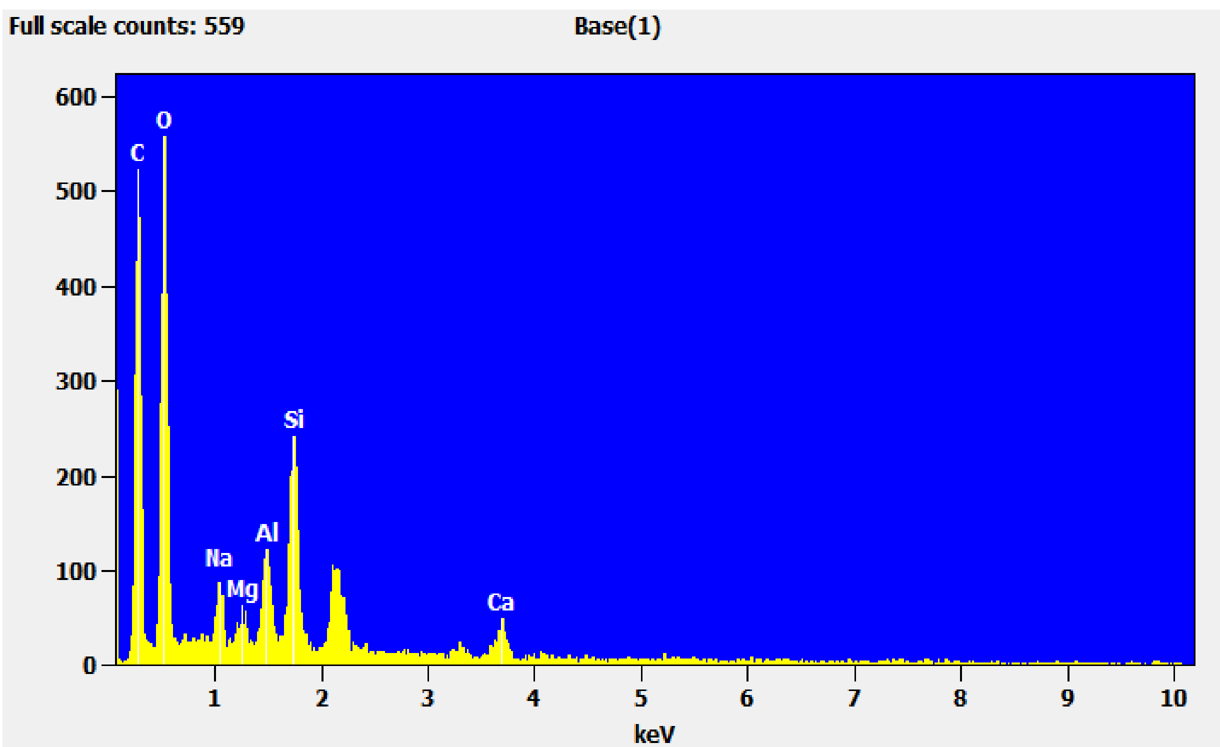


Figure No 5: Energy Dispersive Micro Analysis and XRD Analysis GPC+16M+ Concrete Mix 4–GPC+16M+GF


SEM HV: 10.0 kV
WD: 15.41 mm
VEGA3 TESCAN
SEM MAG: 5.00 kx
Det: SE
10 μm
View field: 41.5 μm
Date(m/d/y): 06/03/24
CoE- Jyothy Institute Of Technology


Full scale counts: 559
Base(1)
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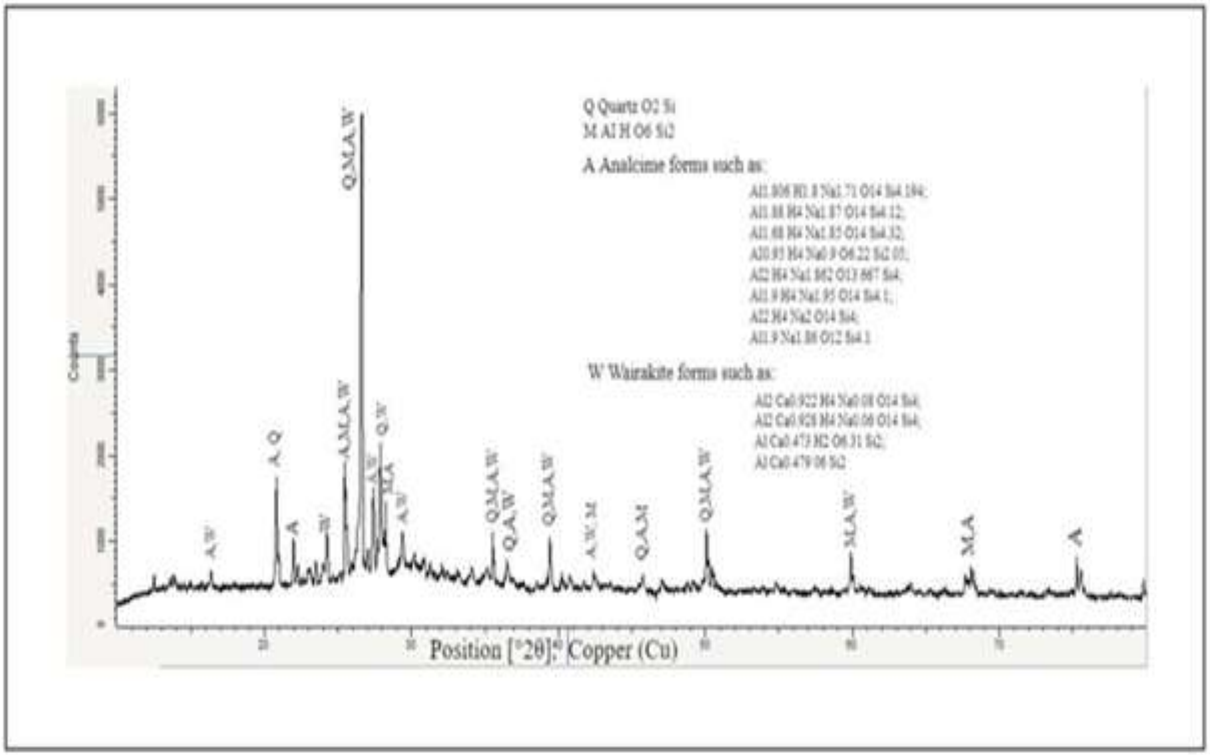

Figure No 6: Energy Dispersive Micro Analysis and XRD Analysis GPC+16M+GF Concrete

4. CONCLUSION

1. The experimental results indicate a substantial improvement in the mechanical performance of the concrete mixes evaluated. At 28 days, the compressive strength of Mix 1, Mix 2, Mix 3 and Mix 4 was obtained as 68.57 N/mm², 70.95 N/mm², 72.64 N/mm², and 75.28 N/mm², respectively. These values represent an increase ranging from 14.28% to 25.47% compared to the Mix 1. Whereas, the flexural strength values are 6.96 N/mm², 7.37 N/mm², 7.88 N/mm², and 8.29 N/mm², reflecting improvements between 39.2%, 47.4%. and 57.6% in comparison of Mix 1. These findings clearly demonstrate that the incorporation of glass fibres and geopolymer constituents significantly enhances both compressive and flexural strength, thereby improving the overall structural performance of the concrete mixes.
2. In SEM analysis. the surface morphology of microstructural examination of the Mix1 to Mix 4 concrete matrices revealed significant variations influencing their mechanical behaviour.

- **Mix 1**, microstructural fissures were present within the hardened matrix, resulting in limited compressive strength. However, a strong bond between the matrix and microcracks was observed at the interface.
- **Mix 2** exhibited similar fissures, but the inclusion of glass fibres improved the post-cracking load-bearing capacity and served as crack arresters, effectively controlling crack propagation.
- **Mix 3** showed fly ash particles embedded within the cracks, with particle sizes closely matching the fissures. This contributed to enhanced performance, as the matrix consisted of geopolymer gel, residual alkaline precipitates, and unreacted or partially reacted fly ash particles in a porous, heterogeneous structure.
- **Mix 4** demonstrated that the addition of fibres led to a denser and more uniformly distributed microstructure, improving particle packing. However, excessive fibre content caused dispersion issues, resulting in clumping and weaker zones at the fibre–matrix interface.

3. The elemental composition of different concrete mixes was analysed using Energy Dispersive X-ray (EDX) analysis.

- Mix 1 consisted of 50.81% calcium oxide (CaO), 10.6% silicon (Si), and 2.98% aluminium (Al), indicating a composition typical of conventional cement-based concrete.
- Mix 2 showed a slightly higher CaO content at 51.30%, with increased amounts of Si (13.88%) and Al (3.37%), suggesting a greater presence of silicate compounds.
- Mix 3, the CaO content decreased to 43.38%, while Si and Al levels rose to 13.81% and 5.53%, reflecting the influence of fly ash and the formation of geopolymer structures.
- **Mix 4** contained 45.46% CaO and 15.65% Si, while aluminium content was not specified but is presumed to be higher due to the geopolymeric nature of the mix. Overall, the results show that the mixes incorporating fly ash (Mixes 3 and Mixes 4) have higher silicon and aluminium content, which are essential for forming strong alumino-silicate gels that enhance concrete strength and durability.

4. The XRD analyses of Mix 3 and Mix 4 reveal distinct yet beneficial mineralogical compositions that contribute to improved performance characteristics.

- Mix 3 exhibits a well-crystallised cementitious matrix rich in Quartz (SiO_2), Calcite ($CaCO_3$), Ettringite, Portlandite, and Wollastonite, with high concentrations of CaO, SiO_2, and Al_2O_3. These components collectively enhance structural strength, durability, and resistance to environmental degradation. In comparison
- Mix 4 demonstrates a chemically stable crystalline matrix dominated by Quartz, Analcime ($NaAlSi_2O_6 \cdot H_2O$), and Wairakite ($CaAl_2Si_4O_{12} \cdot 2H_2O$). The presence of these zeolite minerals, with their alumino-silicate composition, contributes significantly to thermal stability, crack resistance, and ion-exchange capacity, promoting long-term durability. Overall, both mixes are suitable for structural applications, with Mix 3 offering traditional cementitious strength and Mix 4 providing enhanced performance through geopolymeric and zeolitic phases.

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