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# Investigating the Effects Of Sustainable Additives On The Performance And Microstructure Of Concrete

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**ABSTRACT**

This research aims to explore the possibility of a dual binder system which is based on Fly Ash (FA) and Microcrystalline Cellulose Powder (MCC) as an alternative to Ordinary Portland Cement (OPC) concrete. The aim was to enhance both the mechanical and microstructural properties of concrete, and to increase sustainable construction practices. Concrete mixtures were designed to achieve a desired compressive strength of 30 MPa with w/c = 0.45. 10–25% of the cement was replaced by FA and 0.4–1.0% of MCC was added by the weight of the binder. The performance was evaluated in terms of the flexural strength, X-ray diffraction analysis (XRD), and bulk density. The blend with 15 wt% FA and 0.6 wt% MCC (M2) had best performance, with 5.5 MPa while 22.2% increase in flexural strength when compared to control mixture. Moreover, the density of M2 rose, implying denser microstructure. (2013) observed a decrease in the portlandite ($Ca(OH)_2$) peak intensity in mix M2, which suggested that a greater pozzolanic reaction was taking place. This reaction mechanism led to formation of additional C-S-H and enhanced both microstructure and strength. These findings demonstrate that FA with MCC-incorporated concrete can manufacture durable and sustainable concrete material containing high mechanical strengths. However the strengths and densities of specimens obtained by exceeding the optimal levels of FA and MCC, decreased, indicating the importance of dosage optimization. This study may provide an available route to develop green construction materials for sustainable infrastructure.

***Keywords:*** *MCC, Fly Ash, Flexural Strength, X-ray diffraction.*

## 1. INTRODUCTION

The growing emphasis on sustainable concrete solutions is driven by the urgent need to address the environmental challenges posed by conventional concrete production. One of the major concerns is the high level of greenhouse gas emissions and the extensive consumption of natural resources, primarily associated with Portland cement manufacturing. The cement industry alone is estimated to contribute nearly 5% of global carbon dioxide emissions, as the production of one ton of cement typically releases an equivalent amount of $CO_2$ into the atmosphere (Naik, 2008; Singh, 2024). In light of these environmental impacts, researchers and industry professionals are increasingly turning to eco-friendly alternatives such as green concrete. This innovative approach incorporates industrial waste materials and recycled aggregates, thereby reducing the demand for non-renewable resources and minimizing the volume of waste directed to landfills (Findik, 2022; Singh, 2024).

In the global pursuit of decarbonization, countries like India are placing increasing emphasis on identifying sustainable alternatives to Ordinary Portland Cement (OPC). One such promising alternative is the use of alkali-activated binders, which are synthesized by activating aluminosilicate-rich industrial by-products such as fly ash and ground granulated blast furnace slag (GGBS). These binders are gaining traction due to their substantially lower carbon dioxide emissions and reduced contribution to global warming (Bajpai et al., 2020; Habert et al., 2011). In India alone, approximately 226 million tons of fly ash (Haque et al., 2013) and 7.2 million tons of GGBS (IMY, 2020) are produced each year, providing a significant resource base for large-scale development of alkali-activated construction materials.

Despite their environmental benefits, alkali-activated materials (AAMs) often suffer from high shrinkage rates—typically four to five times greater than those seen in OPC-based systems—which can lead to micro-cracking and long-term durability concerns (Ye and Radlinska, 2016; Collins and Sanjayan, 2000). To mitigate these issues, researchers have explored the use of internal curing agents such as superabsorbent polymers (SAPs), cenospheres, and recycled aggregates (Li et al., 2020; Chen et al., 2019; Lee et al., 2017). For example, Li et al. (2020) demonstrated that incorporating just 0.25% SAP into the mix could reduce autogenous shrinkage by 55–60%, primarily by countering the effects of internal drying or self-desiccation.Among the various sustainable materials, cellulose stands out as one of the most abundant, renewable, and biodegradable biopolymers on Earth. Thanks to its environmentally friendly nature, cellulose finds extensive application in developing composite materials. Microcrystalline cellulose (MCC), a form of cellulose with a diameter and length exceeding 1 mm and an aspect ratio of around 1, is obtained through acid hydrolysis followed by homogenization and is readily available commercially (Maria et al., 2014; Rebouillat and Pla, 2013). MCC demonstrates excellent mechanical properties, including an axial elastic modulus ranging from 120 to 200 GPa and a tensile strength of 7.5 GPa (Rebouillat and Pla, 2013), and is already widely used in food, cosmetics, pharmaceutical, and hygiene products.

Research on the use of nanocellulose and MCC as reinforcement in polymeric matrices is well established (Stephen et al., 2010; Kiziltas et al., 2014), yet their application in cementitious composites for construction purposes remains relatively limited. Recent studies show that the incorporation of well-dispersed nanocellulose can enhance the flexural strength of cement composites by up to 30% (Cao et al., 2015).

The incorporation of Microcrystalline Cellulose (MCC) and fly ash in concrete mixtures has shown considerable promise in enhancing mechanical performance, particularly in flexural strength. Studies have indicated that cellulose fibers can improve the flexural fatigue strength of concrete by as much as 68% compared to conventional mixes, with even more pronounced improvements when used alongside steel fibers (Zong-cai, 2008). Furthermore, the integration of cellulose microfibers and MCC within cementitious systems has been found to facilitate better hydration and strength development. However, an excessive amount of fibers may hinder workability and uniform dispersion, ultimately reducing overall strength (Bilcati et al., 2024; Zubair, 2017).

Mineralogical investigations using X-ray diffraction (XRD) confirm that the inclusion of MCC does not significantly alter the primary crystalline phases of cement-based composites, suggesting that the core characteristics of Portland cement are preserved (Bilcati et al., 2024). In summary, the combined application of MCC and fly ash not only improves the flexural behavior of concrete but also contributes to greater durability and crack resistance (Xiaopan et al., n.d.; Nasir et al., 2022).

## 2. EXPERIMENTAL STUDIES

### 2.1. Raw materials used

The study utilized Microcrystalline Cellulose Powder (MCCP 102, Grade 101) of 99% pharmaceutical grade purity. Srihari Balakrishnan from Coimbatore, Tamilnadu, India sent a 500 g sample. The particle size distribution of MCC ranges from 2μm to 260μm, and the Sauter mean diameter is about 49.1μm, as declared by the manufacturer. The powder contains about 3% moisture by weight and has a solid density of 1.54 g/cm³. Structurally, particles are composed of elongated rod-shaped fibers and fine cuboidal forms. The fly ash for the experimentation was obtained from an adjacent thermal power plant in Karnataka, India. The chemical composition was categorized as Class F, based on criteria of ASTM C618, with a combined 90.87% of alumina ($Al_2O_3$), silica ($SiO_2$) and iron oxide ($Fe_2O_3$). The sulfate level ($SO_3$) was found at 1.36%, which is significantly below the ASTM C618 criterion of 5%.

Fly ash used in the experiment was collected from an adjacent thermal power plant in Karnataka, India. It was identified the chemical composition as Class F in accordance with ASTM C618 standard, and the sum of the alumina ($Al_2O_3$), silica ($SiO_2$) and iron oxide ($Fe_2O_3$) was of 90.87%. The level of sulfate ($SO_3$) was determined as 1.36%, which is substantially lower than the ASTM C618 standard limit of 5 %.

The principal binder was ordinary Portland cement (CEM I 42.5 R type), which was mixed with standardized sand in accordance with NP-EN 196-1 specifications. The coarse aggregate used was crushed granite that met IS 383:2016 specifications and had a nominal maximum size of 20mm. Fine aggregate was created by putting river sand through a 4.75 mm filter. The principal binder was ordinary Portland cement (CEM I 42.5 R type), which was mixed with standardized sand in accordance with NP-EN 196-1 specifications. The coarse aggregate used was crushed granite that met IS 383:2016 specifications and had a nominal maximum size of 20mm. Fine aggregate was created by putting river sand through a

4.75 mm filter.

**Table 1. Major Chemical Constituents of Cement and Pozzolanic FA**

| Constituents | OPC | Fly Ash |
|---|---|---|
| **CaO** | 65.8 | 3.23 |
| **$Al_2O_3$** | 3.9 | 25.4 |
| **$SiO_2$** | 16.5 | 60.3 |
| **$Fe_2O_3$** | 5.64 | 5.17 |
| **$SO_3$** | 3.17 | 1.36 |
| **$Na_2O$** | 2.2 | — |
| **$K_2O$** | 0.09 | 1.42 |
| **MgO** | — | 0.89 |
| **$TiO_2$** | 0.62 | 1.4 |
| **$P_2O_5$** | 0.73 | 0.46 |

### 2.2 Mix Design, Mixing, and Curing Conditions

The concrete mix designs in this study were created in line with ACI 211.1 criteria for conventional concrete, with a goal compressive strength of 30 MPa (M30 grade) at 28 days. To achieve the best mix of mechanical performance and workability, the water-to-cementitious materials ratio (w/cm) was set at 0.45. Ordinary Portland Cement (CEM I 42.5 R) was the primary binding medium.

To improve the mix's sustainability, fly ash was used as a supplemental cementitious ingredient, replacing cement at 10% to 25% by weight. Microcrystalline Cellulose Powder (MCC) was also used as a separate additive, with dosages ranging from 0.4% to 1.0% based on the total weight of cementitious component (cement + fly ash). Each level of FA replacement—10%, 15%, 20%, and 25%—was balanced by reducing an equivalent amount of cement, whereas MCC was added separately for each mix variation. Table 2 shows the detailed mix compositions.

To ensure uniform distribution, the dry ingredients (cement, fly ash, MCC, fine aggregates, and coarse aggregates) were completely blended in a pan mixer for about 2 minutes. Following that, the premeasured amount of water and the required dose of superplasticizer were gradually injected. The mixing procedure was then extended for an additional 3 minutes to produce a consistent and workable concrete mix.

After the concrete was cast into the molds, the specimens were immediately covered with plastic sheets to prevent moisture evaporation during the early setting phase. After 24 hours, the hardened specimens were carefully demolded and immersed in a curing tank filled with potable water maintained at a controlled temperature of 27 ± 2 °C. The samples were submerged in the curing liquid until they reached their testing ages. Compressive strength tests were performed at 7, 14, and 28 days in accordance with established testing criteria.

**Table 2. Composition of Concrete Mixes Incorporating OPC, MCC, and FA**

| Design Mix ID | Cement ($kg/m^3$) | FA (%) | FA ($kg/m^3$) | MCC (%) | MCC ($kg/m^3$) | Water ($kg/m^3$) | Fine Aggregate ($kg/m^3$) | Coarse Aggregate ($kg/m^3$) | Superplasticizer (% of Cementitious) |
|---|---|---|---|---|---|---|---|---|---|
| M0 | 400 | 0 | 0 | 0 | 0 | 180 | 680 | 1200 | 0.8 |
| M1 | 360 | 10 | 40 | 0.4 | 1.44 | 180 | 680 | 1200 | 0.8 |
| M2 | 340 | 15 | 60 | 0.6 | 2.04 | 180 | 680 | 1200 | 0.8 |
| M3 | 320 | 20 | 80 | 0.8 | 2.56 | 180 | 680 | 1200 | 0.8 |
| M4 | 300 | 25 | 100 | 1 | 3 | 180 | 680 | 1200 | 0.8 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| M5 | 300 | 25 | 100 | 1.2 | 3.6 | 180 | 680 | 1200 | 0.8 |

### 2.3 Test methods

The flexural strength of the concrete specimens was measured using a three-point bending test setup, as shown in figure 1. Flexural stress is a critical mechanical parameter of brittle materials, indicating their capacity to endure deformation under applied pressures. Concrete treated with Microcrystalline Cellulose (MCC) demonstrated higher flexural performance, which was attributable to the increased flexibility provided by the presence of nanofibers within the matrix.

Concrete specimens (38 mm × 13 mm × 120 mm) were carefully put on supports of a Universal Testing Machine (UTM). During testing, a force was applied at a constant rate of 100 mm/min to the specimens' midpoint. The maximum force measured right before the specimen failed was used to calculate the flexural stress using the following equation:

$$f_{Cf} = \frac{3\,FL}{2d_1 d_2^2} \qquad (1)$$

Where:

F is the maximum applied load at failure (N),

L is the span length between the supports (mm),

$d_1$ is the width of the specimen (mm),

$d_2$ is the thickness of the specimen (mm).

Specimens with varying MCC dosages (0.4%, 0.6%, 0.8%, and 1.0%), as well as plain (control) concrete samples, were tested. To verify the reproducibility of the results, each mix was tested three times, with the average of the three maximum load values used to determine the associated flexural stress.

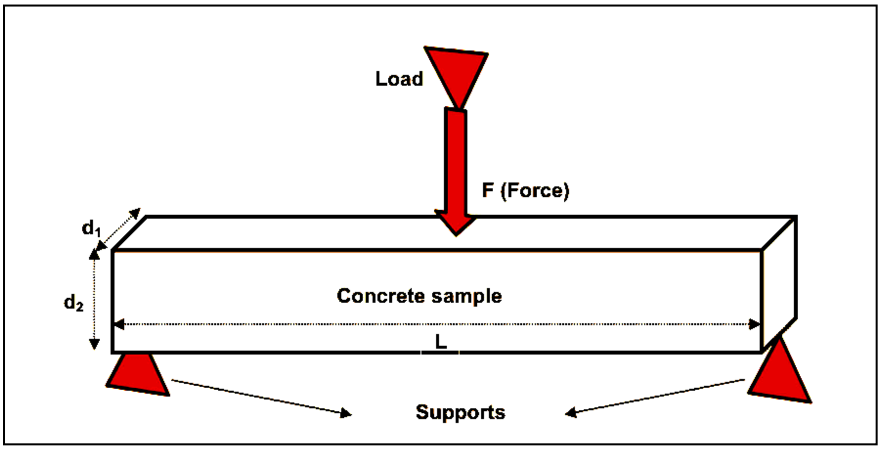


**Fig. 1. Three point loading test.**

## 3. RESULTS AND DISCUSSIONS

### 3.1 Concrete Density

The observed increase in toughened density of mixes M1 and M2 over the control mix (M0) can be attributed to the synergistic actions of FA and MCC. FA, which is recognized for its pozzolanic capabilities, reacts with calcium hydroxide produced during cement hydration, promoting the development of extra calcium silicate hydrate (C-S-H) gel, which adds to increased density. This secondary C-S-H product serves to fill capillary pores, which densifies the cementitious matrix. In addition, MCC functions as a nano-scale filler, improving particle packing and efficiently minimizing microvoids inside the concrete structure. This synergy produces a more compact, densely linked microstructure, especially when the FA and MCC contents are kept at ideal values (10-15% FA and 0.4-0.6% MCC).

However, when the FA and MCC dosages were increased above these ideal proportions, such as in mixes M3 and M4, density decreased. This drop is primarily caused by the addition of excessive FA, which may leave behind unreacted particles, as well as an excess of MCC, which can have a detrimental impact on the mix's homogeneity, resulting in greater porosity and poor compaction.

As a result, the highest density values found in M1 and M2 indicate that a key balance of pore refinement and workability was attained at these compositions.

Table 3 shows the dry bulk density values for both the control and modified concrete mixtures (MCC and FA). These results demonstrate the impact of the additional components on the density and overall compactness of the concrete matrix.

**Table 3. Density of Concrete Incorporating Varying Proportions of Fly Ash and MCC.**

| Mix ID | Density at Fresh State ($kg/m^3$) | Density at Hardened State ($kg/m^3$) |
|---|---|---|
| M0 (Control) | 2430 | 2510 |
| M1 (10% FA + 0.4% MCC) | 2415 | 2515 |
| M2 (15% FA + 0.6% MCC) | 2405 | 2535 |
| M3 (20% FA + 0.8% MCC) | 2390 | 2480 |
| M4 (25% FA + 1.0% MCC) | 2375 | 2465 |

### 3.2 Three-Point Bending Test

The flexural performance of concrete modified with varied concentrations of Fly Ash (FA) and Microcrystalline Cellulose Powder (MCC) provides important insights into how these extra ingredients affect the mechanical properties of the mix. In the control mix (M0), which contained no FA or MCC, the flexural strength reached 4.5 MPa after 28 days, reflecting the typical behavior of M30 grade concrete. When 10–20% FA and 0.4–0.8% MCC were incorporated into Mixes M1 to M3, a notable enhancement in flexural strength was observed. Mix M2, which included 15% FA and 0.6% MCC, achieved the highest flexural strength of 5.5 MPa at 28 days. This improvement is primarily due to the synergistic effects of fly ash's pozzolanic reaction, which generates additional calcium silicate hydrate (C-S-H) to densify the cement matrix, and MCC's micro-filling action, which refines the pore structure and bridges microcracks, improving load distribution and crack resistance.

However, when the levels of FA and MCC were further increased in Mixes M4 and M5 (with 25% FA and MCC content exceeding 1.0%), a decline in flexural strength was observed. For instance, Mix M5 exhibited a reduced flexural strength of 4.3 MPa at 28 days. This reduction is mostly due to MCC's high water absorption capacity at increasing doses, which has a negative impact on workability and compaction, resulting in an uneven internal structure. Additionally, the higher levels of fly ash substitution tend to slow the early hydration process, hindering the development of mechanical strength during the initial stages.

The analysis of strength development over the curing periods (7, 14, and 28 days) demonstrated a consistent trend of gradual hydration in all mixes. However, mixes with higher fly ash (FA) and microcrystalline cellulose powder (MCC) content showed slower strength gain after 14 days. This delay is mostly caused by the reduced reactivity of fly ash compared to typical cement during the early phases of hydration.

Finally, the results emphasize the importance of carefully regulating the amounts of fly ash and MCC to produce appropriate flexural strength. The mix comprising 15% fly ash and 0.6% MCC (Mix M2) was the most successful, providing significant gains in flexural strength while maintaining good workability and improving the microstructure of the concrete. Table 3 shows the flexural strength evolution of various mixes comprising FA and MCC after 7, 14, and 28 days of curing, demonstrating the effect of these extra elements on concrete performance over time.

**Table 3. Development of Flexural Strength in Concrete Mixes with Varying Fly Ash and MCC Contents at Different Curing Ages.**

| Design Mix ID | 7 Days (MPa) | 14 Days (MPa) | 28 Days (MPa) |
|---|---|---|---|
| M0 (Control) | 2.8 | 3.5 | 4.5 |
| M1 (10% FA + 0.4% MCC) | 3.2 | 3.9 | 5.2 |
| M2 (15% FA + 0.6% MCC) | 3.4 | 4.1 | 5.5 |
| M3 (20% FA + 0.8% MCC) | 3.1 | 3.8 | 5 |
| M4 (25% FA + 1.0% MCC) | 2.9 | 3.6 | 4.7 |


**Fig 2: Effect of Various Fly Ash and MCC Proportions on Concrete Flexural Stress.**

### 3.3 X-ray diffraction analysis

To investigate the effect of combining Fly Ash (FA) and Microcrystalline Cellulose (MCC) on concrete hydration and phase development, X-ray Diffraction (XRD) and Thermogravimetric Analysis (TGA) were performed on both the control mix (M0) and the optimized blend. Concrete samples were crushed and submerged in acetone for 24 hours to prevent additional hydration, then dried in an oven at 60°C for 8 to 10 hours to eliminate any leftover moisture. To ensure reliable phase identification in the XRD analysis, the powdered samples were scanned in a nitrogen environment (flow rate: 40 mL/min).

The calcium hydroxide (CH) concentration was calculated from the mass loss between 420°C and 540°C during the TGA using the formula presented by Gupta et al. (2021). The mass of anhydrous cement (MCp) was used to calculate the weight of the paste at 600°C, as described by Vance et al. (2013).

$$C_H(\%) = \left(\frac{4.12\ L_{dx}}{M_{C_P}}\right) X100 \qquad (2)$$

A detailed comparison of the XRD patterns for the control mix (M0) and the M2 mixture (Figure 3) indicated the effect of the binary binder system on the crystalline structure. The control mixture showed strong diffraction peaks associated with quartz, portlandite, and calcium silicate hydrate (C-S-H) phases. Quartz was found at 2θ values of 26.53°, 50.29°, 60.11°, and 68.34°, while portlandite was found at 17.95°, 34.04°, and 63.96°, indicating the presence of calcium hydroxide, a critical product of cement hydration (Costafreda et al., 2013; Kontoleontos et al., 2013; Sumra et al., 2020). C-S-H, a key component in concrete's mechanical strength and longevity (Scrivener and Nonat, 2011), was found at a 2θ value of 29.35°.

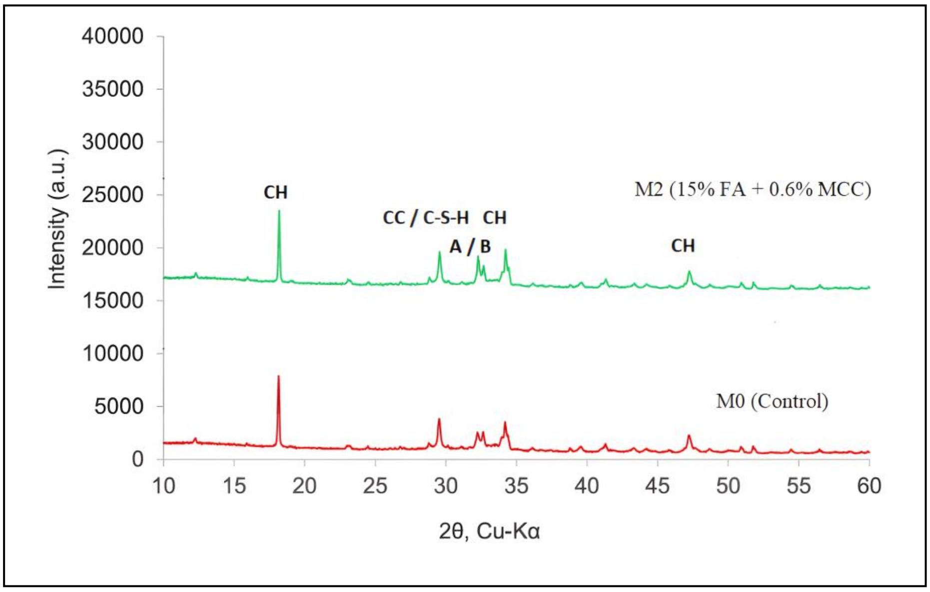


**Fig. 3. X-ray Diffraction Pattern of Concrete after 28 Days of Curing.**

Minor phases, such as hydrotalcite, were also found in the M2 mix, which is consistent with earlier research on alkali-activated slag and FA systems in which magnesium content enhances hydrotalcite formation (Oh et al., 2009; Wang & Scrivener, 1995). Because M2 contains Class F FA, the production of hydrotalcite promotes the presence of further hydration processes inside the concrete matrix.

The M2 mix showed significant differences in the hydration process when compared to the control mix (M0). Shifts in the portlandite peaks at 2θ values of 18.02°, 34.03°, and 47.12° were detected, indicating that the addition of MCC may alter the crystallization of portlandite, resulting in smaller, less crystalline structures. This change not only decreased the calcium hydroxide level, but also increased the formation of calcium silicate hydrate (C-S-H), resulting in a denser and stronger microstructure.

Furthermore, the C-S-H peaks in the M2 samples had increased intensity and small changes, indicating a more ordered and crystalline structure. This matrix densification is directly related to observed improvements in mechanical properties, specifically an increase in flexural strength, which is consistent with the microstructural study.

## 4. CONCLUSION

An experimental study was done to assess the impact of binary binder systems containing Fly Ash (FA) and Microcrystalline Cellulose Powder (MCC) on the mechanical and microstructural qualities of concrete. The study focused on many essential metrics, including flexural strength, bulk density, and phase composition, which were examined using X-ray diffraction.

The findings from the experiments revealed the following insights:

The concrete mix known as M2 performed the best in terms of split tensile strength, achieving 5.5 MPa after 28 days of curing. In addition, M2's flexural strength increased by 22.2% as compared to the control mix (M0). This improvement demonstrates the benefits of include both FA and MCC in the binder system.

The density of the concrete was tested in both its fresh and hardened form. The results showed a rise in density up to Mix M2 compared to the control, implying higher packing density and greater matrix integrity. However, density decreased gradually beyond the M2 composition. This decrease is mostly owing to higher levels of MCC and FA replacement, which, due to their lower specific gravities and proclivity to absorb more water, resulted in a less dense microstructure with more entrapped air.

The XRD analysis revealed information about the microstructural changes caused by the changing binder composition. The M2 mix resulted in lower intensity of portlandite ($Ca(OH)_2$) peaks compared to the control, indicating an accelerated pozzolanic reaction. This change fostered the creation of new calcium silicate hydrate (C-S-H) phases, which contributed to increased microstructural compactness and strength.

The experimental study clearly indicated that adding fly ash and microcrystalline cellulose at the appropriate doses considerably improved both the mechanical and microstructural qualities of concrete. The M2 mix, in particular, delivered the highest overall performance, with higher tensile and flexural strengths and a denser microstructure. However, exceeding the recommended amounts of FA and MCC resulted in a decrease in density and could jeopardize mechanical integrity. As a result, striking an optimal balance in replacement levels is crucial for producing high-performance concrete with sustainable supplemental cementitious materials.

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