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Enhancement Of Mechanical And Microstructural Properties Of Concrete Using Microcrystalline Cellulose And Fly Ash

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ABSTRACT

This study looks into the impact of using Microcrystalline Cellulose (MCC) and Class F fly ash (FA) as partial cement substitutes on the fresh, mechanical, and microstructural properties of concrete. Using the ACI mix design approach, several mix proportions were created, and their performance was assessed using slump tests, compressive strength tests, stress-strain behavior, and Scanning Electron Microscopy. The results showed that the addition of FA increased workability due to its spherical particle morphology, whilst MCC significantly lowered slump values due to its fibrous character. Compressive strength results showed that the optimum mix (M2-cement +0.6% of MCC and 15% of FA) containing both MCC and fly ash achieved higher strength than the conventional mix (M0- concrete with admixtures), suggesting enhanced cementitious reactions and better matrix densification. The stress-strain analysis confirmed increased ductility and energy absorption in the M2 mix. SEM images further revealed a denser, less porous microstructure in the modified mix, with fewer cracks and more compact hydration products. Overall, the study demonstrates that the combined use of MCC and fly ash not only improves concrete performance but also supports sustainable construction practices by reducing cement consumption and utilizing industrial by-products effectively.

Keywords: *Microcrystalline Cellulose (MCC), Fly Ash, Compressive Strength, Stress-Strain Behavior, Scanning Electron Microscopy (SEM)*

1. INTRODUCTION

Cement-based materials are widely utilized in various construction practices due to their availability and performance characteristics. Nevertheless, a notable drawback of these materials is their brittle nature and tendency to develop cracks, which can compromise structural integrity. These materials are inherently heterogeneous and consist of hydration products such as calcium silicate hydrate (C-S-H), calcium hydroxide, ettringite, monosulfate, and residual unhydrated cement particles, all of which form through the hydration process of cement with water (Parveen et al., 2015). Cracking can occur either during the early stages of curing or throughout the service life of the structure, with progressive propagation leading to material degradation or failure. This issue is further exacerbated under adverse environmental conditions, accelerating the deterioration process. To mitigate such problems and enhance mechanical performance, cementitious composites are often strengthened by incorporating various types of fibers—including steel, basalt, carbon, glass, and aramid—which significantly improve their toughness and resistance to crack propagation (Akkaya et al., 2003). Cementitious composites feature a hierarchical or multi-scale structure that includes hydration products at the micron and sub-micron scales as well as millimetre scale elements such as sand. Because the cracks in cementitious composites are multi-scale in nature, the best approach for developing crack-free cementitious composites should include multi-scale reinforcements. Steel fibers, carbon fibers, polyvinyl alcohol fibers, glass fibers, and other materials are commonly used as reinforcements. The reinforcement scale has the greatest influence on the behavior of cementitious composites. Macro-scale fibers, typically defined by diameters exceeding 500 µm, are effective in enhancing the post-cracking behavior of cementitious composites by bridging large cracks and thus improving structural toughness. According to Gdoutos (2009), extremely fine microfibers—less than 50 µm in diameter—are capable of bridging micro-level fissures, delaying their initiation and growth within the cement matrix. More recently, the integration of nanomaterials, such as nanoparticles and nanofibers with diameters in the nanometer range, has gained attention for their ability to bridge nanoscale cracks and densify the microstructure (Nazari et al., 2010;

Siddique & Mehta, 2014). These nanomaterials not only contribute to mechanical enhancement but also impart functional properties, including self-sensing, photocatalytic self-cleaning, and thermal regulation to the cementitious matrix (Liew et al., 2016; Parveen et al., 2013; Le et al., 2014; Sun et al., 2017). Furthermore, the application of natural, bio-derived fibers such as jute, hemp, flax, and sisal in polymeric and cement-based composites has grown significantly, driven by sustainability considerations and the need for eco-friendly alternatives. In line with this trend, bio-based nano and micro reinforcements, including nano-cellulose variants—such as nanofibrillated cellulose (NFC), nanocrystalline cellulose (NCC), and bacterial nanocellulose (BNC)—along with microcrystalline cellulose (MCC), are increasingly utilized across multiple industries due to their renewable nature and high performance. Incorporating fly ash as a partial substitute for Portland cement has been shown to significantly influence the structural performance of concrete. Studies have highlighted that fly ash not only enhances the mechanical properties of concrete but also promotes environmental sustainability by lowering cement consumption, which in turn reduces carbon emissions associated with its production. Research by Kroviakov and Shymchenko (2024) indicates that a replacement level of approximately 20% offers a favorable trade-off between strength and durability. Interestingly, when 10% of cement was replaced with fly ash, the 28-day compressive strength exhibited a 3.8% increase compared to the conventional mix. However, increasing the replacement to 20% led to a reduction in strength by about 14% relative to the control specimen, suggesting the importance of optimizing the substitution ratio to achieve both mechanical performance and ecological benefits. Despite this, another study reported that 20% fly ash yielded the best mechanical properties, including both compressive and flexural strengths, while higher percentages such as 30% led to increased porosity and reduced strength (Vikram, 2024).

Fly ash contributes to enhanced workability and long-term durability of concrete, primarily due to its fine particles which create a lubricating action within the mix—an effect that is particularly noticeable at lower levels of replacement (Vikram, 2024). Experimental evaluations on durability have revealed that incorporating 20% fly ash significantly lowers water absorption rates, thereby increasing the resistance of concrete to moisture ingress and ultimately extending the service life of structural elements (Vikram, 2024). Utilizing fly ash in concrete contributes to environmental sustainability by minimizing the reliance on Portland cement and repurposing industrial by-products that would otherwise contribute to waste (Mahmud et al., 2024; Safitri et al., 2024). However, while FA can improve several concrete properties, excessive replacement beyond 30% may result in diminished strength and increased voids, underscoring the importance of maintaining a balanced proportion for optimal performance.

Cementitious materials, while widely used in construction, suffer from inherent brittleness and susceptibility to cracking, which can compromise structural integrity over time. These limitations are further exacerbated by environmental exposure and microstructural deficiencies. This study examines the pivotal concerns by investigating the application of microcrystalline cellulose (MCC), a sustainable and biodegradable micro-scale reinforcement, in conjunction with FA as a partial substitute for cement. MCC has the capability to bridge microcracks and improve the internal structure of concrete, whilst fly ash promotes sustainability and mechanical performance. By mixing these components, the study intends to produce a more durable and eco-friendly concrete mix.

This research focuses on evaluating both the fresh and hardened mechanical behaviors, along with the microstructural features, of conventional and enhanced concrete mixtures, aiming to develop durable, high-performance cement-based composites.

2. EXPERIMENTAL METHODS

2.1 Materials

To maintain consistency and reliability throughout the experimentation, only high-quality, standard-compliant materials were employed. Ordinary Portland Cement (OPC) of grade CEM I 42.5 R served as the primary binder and was blended with standardized sand as prescribed by NP-EN 196-1 protocols. The study incorporated Class F fly ash, sourced from a thermal power facility in Karnataka, India, conforming to ASTM C618 specifications. This fly ash sample contained a cumulative 90.87% of silica (SiO_2), alumina (Al_2O_3), and ferric oxide (Fe_2O_3), while its sulfate (SO_3) concentration remained well within acceptable limits at 1.36%, significantly below the ASTM ceiling of 5%.

Microcrystalline Cellulose Powder (MCCP102, Grade 101) was supplied by Srihari Balakrishnan, based in Coimbatore, Tamil Nadu, India. The MCC used was of pharmaceutical grade, exhibiting 99% purity. Its particle sizes ranged from 2 µm to 260 µm, with a Sauter mean diameter of 49.1 µm. Moisture content was measured at 3% by weight, and the material had a solid density of 1.54 g/cm³. The morphology consisted primarily of elongated rods and small cuboidal particles.

The fine aggregate was natural river sand that passed through a 4.75 mm sieve, while the coarse aggregate consisted of crushed granite stones, meeting the grading and quality requirements outlined in IS 383:2016. Further specifics regarding the cement and sand properties used in this research are detailed in Thomas et al. (2025).

2.2 Mix Proportion

In this investigation, the concrete mix proportions were tailored based on the ACI mix design methodology, incorporating

FA and MCC as partial replacements for cement to enhance performance and sustainability. Six concrete mix designs (M0 to M4) were created, with M0 serving as the Conventional mix with 400 kg/m³ of cement and no additional components. From M1 to M4, cement content decreased from 360 kg/m³ to 300 kg/m³. Fly ash content increased from 10% (40 kg/m³) in M1 to 25% (100 kg/m³) in M4. Simultaneously, MCC was introduced in increasing amounts, ranging from 0.4% (1.44 kg/m³) in M1 to 1% (3.0 kg/m³) in M4. Water content was kept constant at 180 kg/m³ across all mixes, maintaining a consistent water-to-cementitious ratio. To maintain gradation and workability, fine and coarse aggregates were maintained at 680 and 1200 kg/m³, respectively. Additionally, a superplasticizer dosage of 0.8% by weight of total cementitious materials was uniformly used in all mixes to ensure desired workability. The mix ratios were formulated to evaluate the fresh behavior, mechanical strength, and internal microstructure of concrete incorporating fly ash and microcrystalline cellulose. Table 1 presents the detailed compositions used to investigate these specific performance characteristics.

Table 1. Composition of Concrete Mixes Incorporating OPC, MCC, and FA (Thomas et al., 2025).

Design Mix ID	Cement (kg/m³)	FA (%)	FA (kg/m³)	MCC (%)	MCC (kg/m³)	Water (kg/m³)	Fine Aggregate (kg/m³)	Coarse Aggregate (kg/m³)	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

3. MIXING AND CURING CONDITIONS

A traditional pan mixer was employed to achieve uniform blending of all concrete mixtures used in this study. Initially, the dry constituents—cement, fly ash, microcrystalline cellulose, fine aggregate, and coarse aggregate—were thoroughly mixed for a duration of two minutes to ensure even distribution before the addition of water. Following that, water and superplasticizer were slowly added and combined for another three minutes to ensure a homogenous consistency. After the concrete had been uniformly mixed, it was poured into standard cube and cylinder molds in two layers, each compressed using a table vibrator to remove air gaps. Freshly cast samples were kept in their molds for 24 hours at room temperature, approximately 27 ± 2 °C. Following this initial setting period, the specimens were promptly transferred to a curing tank filled with potable water, maintained consistently at 27 ± 2 °C. They were immersed for curing durations of 7, 14, or 28 days, aligned with the testing timeline. This curing process ensured sufficient hydration and strength gain, enabling accurate assessment of both mechanical performance and microstructural properties.

4. TESTING AND RESULT

The workability of fresh concrete in this study was assessed using the slump cone test, following the procedures outlined in ASTM C143/C143M-20. This test was performed on all six concrete mixtures (M0 through M4) to examine how fly ash and microcrystalline cellulose (MCC) influenced the fresh properties of the mixes. A standard Abrams slump cone was utilized, measuring 300 mm in height, with a base diameter of 200 mm and a top diameter of 100 mm. Accompanying equipment included a tamping rod, 16 mm in diameter and 600 mm long, and a non-absorbent, flat base plate. Prior to testing, the cone's interior surface was cleaned and lightly coated with oil, then set firmly on a rigid, non-absorbent surface.

The fresh concrete was introduced into the cone in three approximately equal layers. Each layer was compacted with 25 evenly distributed strokes of the tamping rod. After placing the final layer, its surface was leveled flush with the top of the cone. The cone was then carefully lifted vertically without any sideways or rotational movement, allowing the concrete to settle naturally. The slump was determined by measuring the vertical difference between the top edge of the mold and the highest point of the concrete after settlement, using a steel ruler.

The recorded slump values showed a progressive increase across the mixes: 85 mm for M0, 92 mm for M1, 100 mm for M2, 105 mm for M3, and 110 mm for M4. This upward trend indicates enhanced workability with the addition of fly ash and MCC. The improvement is largely due to the spherical shape and lubricating properties of fly ash particles, as well as the water retention and fibrous microstructure of MCC, which together improve the cohesiveness and flow characteristics of the fresh concrete. Additionally, the consistent incorporation of a superplasticizer at 0.8% of the total cementitious material helped maintain particle dispersion and overall workability in all mixtures.

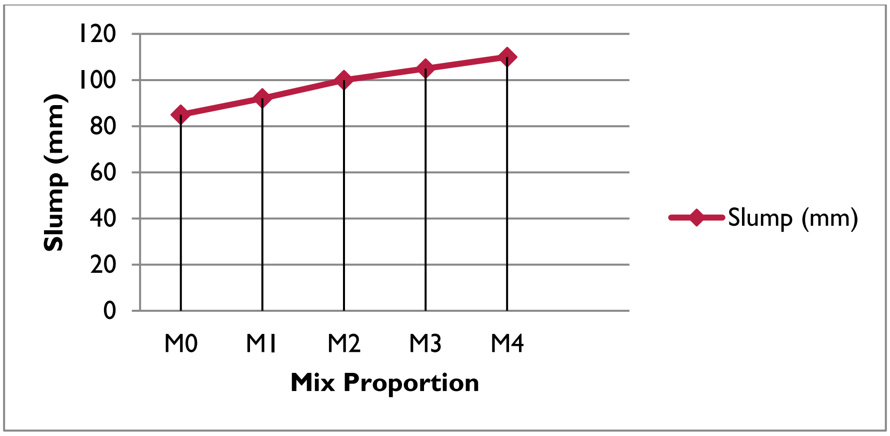


Figure 1. Slump cone test result.

4.1 Mechanical strength

The concrete's mechanical strength was evaluated through compressive strength tests conducted on cube specimens, following the procedures specified in IS 516 (Part 1/Sec 1): 2021, which provides guidelines for assessing the strength of hardened concrete. Cubes measuring 150 mm on each side were prepared for every mix design (M0 through M5) and tested after curing periods of 7, 14, and 28 days. After casting, the specimens were removed from their molds after 24 ± 2 hours and subsequently submerged in potable water maintained at 27 ± 2°C until the designated testing times. The compressive strength tests were carried out using a Compression Testing Machine (CTM) applying a continuous load at a rate of 140 kg/cm² per minute (approximately 0.14 N/mm² per second) until the specimens fractured.

The formula used to compute the compressive strength (f_{ck}) was:

$$f_{Ck} = \left(\frac{P\ (load)}{A\ (area\ of\ cross\ section)}\right) \qquad (1)$$

Table 2: Strength Progression of Concrete Specimens at 7, 14, and 28 Days (N/mm²)

Mix ID	**Curing days**		
	7	**14**	**28**
M0	28.4	35.7	41.5
M1	29.6	36.8	42.8
M2	33.5	41	46.9
M3	32.1	39.5	45.6
M4	31.2	38.7	43.7

The data presented in Table 2 demonstrate a steady increase in compressive strength with the addition of fly ash (FA) and microcrystalline cellulose (MCC) up to an optimal level. The control sample (M0) exhibited a compressive strength of 41.5 N/mm² at 28 days, whereas the mixture containing the optimal blend (M2) reached a peak strength of 46.9 N/mm², representing an improvement of about 13%. This enhancement is primarily attributed to the pozzolanic reaction of Class F fly ash, which facilitates the generation of extra calcium silicate hydrate (C–S–H), resulting in a denser and more refined concrete microstructure. Moreover, the fibrous nature of MCC likely strengthened the internal bonding within the cementitious matrix, further boosting the overall strength. A graphical representation comparing the compressive strengths of the different concrete mixes investigated is shown in Figure 1. The observed trend indicates that a well-balanced incorporation of FA and MCC can significantly improve the mechanical behavior of concrete, supporting sustainable construction practices while ensuring structural performance.

4.2 Stress–Strain Behavior of Concrete Under Compression

The stress-strain behaviour under compression was assessed for all concrete mixes (M0 to M5) in accordance with IS 516 (Part 5/Sec 1): 2018, which specifies a method for assessing concrete stress in compression. 150 mm cube specimens were evaluated utilizing a compression testing machine equipped with a dial gauge and a linear variable differential transformer

(LVDT) to monitor both axial load and axial deformation. The loading was applied gradually and consistently at a rate of 0.2MPa. The shopping and strain (ε) were determined using the following formulas:

$$\sigma = \frac{P}{A} \quad (2)$$

$$\varepsilon = \frac{\Delta L}{L_0} \quad (3)$$

where:

- σ = compressive stress (N/mm²)
- ε = Stain
- ΔL = change in length (mm)
- L_0 = original gauge length (mm)

Each mix's stress-strain data were plotted, and the modulus of elasticity (E_c) in the early linear section of the curve was computed using:

$$E_c = \frac{\sigma_2 - \sigma_1}{\varepsilon_2 - \varepsilon_1} \quad (4)$$

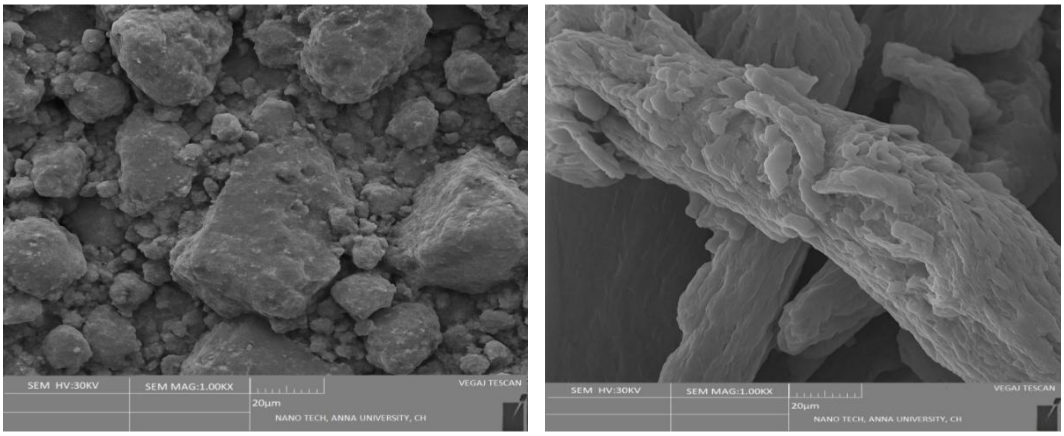
Table 3: Stress–Strain Characteristics of Concrete Mixes at 28 Days

Mix ID	Max. Compressive Strength (MPa)	Peak Stress (MPa)	Peak Strain (×10⁻³)	Modulus of Elasticity (MPa)	Failure Strain (×10⁻³)
M0	41.5	41.5	2.2	32,166	3.3
M1	42.8	42.8	2.35	32,703	3.53
M2	46.9	46.9	2.5	34,179	3.75
M3	45.6	45.6	2.6	33,749	3.9
M4	43.7	43.7	2.7	33,063	4.05

The stress-strain measurements in Table 2 show that integrating FA and MCC improves both the strength and ductility of concrete. The peak compressive stress gradually increased from 41.5 MPa in the control mix (M0) to 46.9 MPa in mix M2. In addition, peak strain values increased somewhat, indicating a transition in the failure mode from brittle to ductile. Furthermore, the modulus of elasticity revealed increased stiffness and energy absorption. These improvements are mostly due to the pozzolanic reaction of FA, which contributes to matrix densification, and the addition of MCC, which acts as micro-fibers to assist reduce micro-crack development, so increasing both strength and ductility.

4.3 Microstructural Characterization

Scanning Electron Microscopy (SEM) analysis is a necessary technique for accepting the microstructural features of concrete at high magnification. It allows for the visualization of the morphology, texture, and distribution of hydration products, unreacted cement particles, and additional cementitious materials. In this study, SEM analysis was performed to compare the microstructural differences between the conventional concrete mix (M0) and the optimized mix (M2) containing Microcrystalline Cellulose (MCC) and FA. The SEM images were captured at a resolution corresponding to a scale of 20 micrometers, which enabled a detailed examination of fine structures such as C-S-H gels, ettringite needles, and the packing of filler materials. This investigation reveals a link between microstructural characteristics and mechanical performance of concrete. It is particularly effective for assessing matrix densification, pore distribution, and crack development. These findings provide unequivocal proof of the increased strength and durability of concrete mixes containing MCC and FA, highlighting their effectiveness in enhancing overall concrete quality.



(a) **(b)**

Figure 2. a) Conventional concrete SEM analysis. b) M2 SEM analysis

Figure 2.a. reveals a heterogeneous and porous microstructure, characterized by loosely packed hydration products and visible micro-cracks. The unreacted cement particles and needle-like ettringite structures suggest incomplete hydration and a relatively less dense matrix. In contrast, figure 2.b. incorporating MCC and FA shows a more compact and homogenous microstructure. The addition of spherical FA particles promotes packing efficiency inside the concrete matrix, whereas MCC helps to refine the pore structure. In mix M2, the noticeable reduction in voids and fracture development is due to improved internal bonding and a more compact microstructure. These microstructural improvements are consistent with the observed advantages in mechanical performance, indicating the complimentary effect of MCC and FA in improving both the durability and strength of concrete.

5. CONCLUSION

Comprehensive experimental analysis reveals that partially replacing cement with fly ash and Microcrystalline Cellulose significantly influences both the fresh and hardened properties of concrete. Slump test results indicate that fly ash enhances workability, attributed to its spherical shape and smooth, lubricating effect. In contrast, the fibrous nature of MCC slightly reduces workability by increasing internal friction within the mix. In terms of mechanical performance, compressive strength findings show that the optimum mix (M2) has higher strength than the conventional mix (M0), suggesting the good synergistic effect of MCC and FA at optimal dose. The stress-strain behavior further supports this finding, with M2 exhibiting higher peak stress and improved ductility, as evidenced by a broader and more gradual descending curve, reflecting its ability to absorb more energy before failure. SEM analysis provides microstructural validation for these observations, showing that the M2 mix has a denser matrix, fewer micro-cracks, and better particle interlocking compared to M0. In conclusion, the addition of MCC and fly ash improves both the mechanical characteristics and workability of concrete, while also contributing to the development of a denser and more durable microstructure. This combination provides a long-term and efficient solution for improving concrete performance.

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