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EVALUATION OF PERFORMANCE, MECHANICAL AND DURABILITY CHARACTERISTICS OF ENVIRONMENTAL FRIENDLY LIMESTONE CALCINED CLAY CEMENT AND FLY ASH - A SUSTAINABLE ALTERNATIVE

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Abstract- The chemical and thermal processes that occur during cement production are significant contributors to CO_2 emissions, with 60 percent attributed to direct emissions from heating limestone to create clinker, while 40 percent results from the burning of fuels in cement kilns. Overall, cement manufacturing is responsible for approximately 8 percent of worldwide CO_2 emissions. Limestone Calcined Clay Cement (LC^3) offers an innovative low-carbon blended cement that facilitates a decrease in CO_2 emissions during production. The utilization of supplementary cementitious material such as fly ash particularly in partial replacement with LC^3, has the potential to significantly reduce carbon emissions and resource consumption in cement production. This research focuses on blending Fly ash with LC^3 at various combinations to determine the optimum percentage replacement of LC^3 with fly ash. From the research It has been discovered that the blend of LC^3 with fly ash at 30% replacement exhibits superior performance in terms of various mechanical and durability properties of concrete.

Keywords: Limestone Calcined Clay Cemen; Supplementary cementitious material;, compressive strength of concrete;, CO_2 emission

1. INTRODUCTION

Concrete is the most commonly used construction material globally, with cement being its primary component [1]. India's cement consumption was around 445 Million Tonnes in FY24, which is expected to grow to 670 MTPA by 2030 which is a 50% increase[2]. Cement is an extremely valuable, essential, and ubiquitous material, while also being a significant source of emissions. If cement were considered a nation, it would rank as the third largest emitter of greenhouse gases globally, surpassed only by China and the United States. Cement is the most extensively produced product on the planet by weight, and it plays an essential part in promoting and aligning various sustainability objectives[1]. Portland cement is acknowledged as one of the most reliable materials which is extensively used in nearly all construction activities, with a wide range of applications across various environmental conditions. Despite advancements in the manufacturing process of Ordinary Portland Cement (OPC), the fundamental rheology and chemistry of cement have remained unchanged. Portland cement is being increasingly substituted with traditional Supplementary Cementitious Materials (SCMs), which have been investigated by numerous researchers; however, achieving a complete replacement of Portland cement with alternative binding systems continues to pose a challenge in civil engineering research. Clinker substitution has been recognized as a crucial solution for achieving sustainability in the cement industry [3]. Globally, cement production is responsible for 7% of CO_2 emissions. Approximately half of these emissions result from combustion processes, while the other half is due to calcination. On average, The application of blended cements can reduce CO_2 emissions from 0.82 to 0.65 kg/kg. Furthermore, the Energy Technology Perspective 2020 report by the International Energy Agency estimates that about 0.654 tons of CO_2 are emitted for every ton of cement produced. Since the availability of cementitious industrial by-products are limited, their utilization as a binder is also restricted [4]. Assessing the appropriateness of alternative cementitious materials is currently essential. In this context, limestone calcined clay cement has surfaced as an outstanding substitute for ordinary Portland cement. The abundant availability of calcined clay can meet the growing need for materials that can replace cement. As a highly reactive pozzolan, calcined clay promotes the development of early pore structure and microstructure. The characteristics of binders are influenced differently based on the quantity of limestone present. LC^3 represents a novel type of blended cement, comprising 50% clinker, 30% calcined clay, 15% limestone, and 5% gypsum. From an environmental perspective, LC^3 serves as a low-carbon alternative when compared to Portland cement. It has the potential to decrease CO_2 emissions during the process of cement manufacturing by reducing the amount of clinker used and substituting it with LC^3. From an environmental standpoint, LC^3 can diminish CO_2 emissions in cement production by reducing the clinker content[5]. Limestone calcined clay cement, represents a promising technical solution aimed at making cement more environmentally friendly while achieving a balance among economic, environmental and social goals. The manufacturing of cement with lower CO_2 emissions poses significant technical challenges, as 60% of these emissions arise from the decomposition of limestone at a temperature of 1450 degrees Celsius. LC^3 cement has the potential to lower the clinker factor by combining portland cement with calcined clay and un burnt limestone, making it one of the most promising and cost-effective alternatives; it can lead to a reduction of up to 40% in CO_2 emissions when compared to Portland Cement. Furthermore, LC^3 can be implemented on a global scale, as suitable clays are nearly universally available, and the establishment of a production facility necessitates considerably lower investments and operational costs than those linked to a portland cement facility. Additionally, it serves as a more economical option compared to carbon capture and storage technologies. The incorporation of supplementary cementitious materials (SCMs), such as fly ash, along with limestone, has the capacity to greatly diminish carbon emissions and resource usage in cement production. The analysis of environmental impact indicated that LC^2 has decreased the relative energy consumption and carbon emissions associated with cement, achieving a reduction of approximately fifty percent at a substitution rate of 45 to 60% [6].

2. MATERIALS AND METHODOLOGY

2.1 Limestone Calcined Clay Cement (LC^3):

LC3 represents an innovative form of cement derived from a mixture of limestone and calcined clay. This type of cement has the potential to decrease CO2 emissions by as much as 40% and is produced using limestone along with low-grade clays that are readily available in large quantities, is cost effective and does not require capital intensive modifications to existing cement plants. LC^3 contains 50% clinker, 30% calcined clay, 15% limestone and 5% gypsum[1]. Clay with over 40% kaolin content is suitable for LC3. Besides conventional rotary kilns, the clay can also be calcined using flash calcination units, shuttle kilns, muffle furnaces and roller hearth kilns. The physical properties of LC^3 is represented in Table 1. Figure 1 represents the XRD pattern for analysis conducted on LC^3 sample. The analysis confirms the presence of quartz, hematite, calcite, illite and other minerals as seen in ordinary portland cement. The permeability tests conducted on LC^3, OPC and FA has suggested that the LC^3 binder system attains much lower permeability with respect to the OPC and FA [7]. The coarsening of the pore-structure and a reduction in the compressive strength occurs for low clinker cements, when cured at the higher temperature [8]. The proportion of the reactive phase, the ratio of reactive SiO_2/Al_2O_3, and the fineness are considered to be the essential attributes of calcined clays [9]. The initial rate of capillary water absorption has also exhibited a suitable correlation with the outcomes of water penetration and electrical resistivity testing methods [10].

Table 1: Physical properties of LC^3

Description	Specific Gravity	Fineness(%)	Normal Consistency (%)	Initial Setting time (minutes)	Final Setting time (minutes)
Results	3.79	12	35	35	240

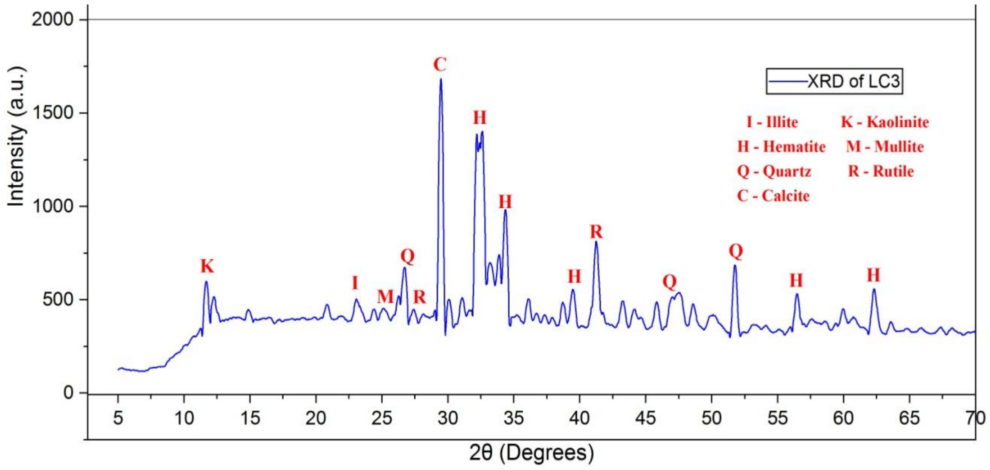


Figure1: XRD pattern of LC^3

2.2 Flyash:

Fly ash is a fine, powdery byproduct generated from the combustion of pulverized coal in coal-fired power plants. It is collected from the flue gases through filtration systems, such as electrostatic precipitators or bag houses. Fly ash is commonly utilized as a supplementary cementitious material in manufacturing concrete. Fly ash offers multiple benefits in concrete production, including improved strength, durability, workability, and environmental sustainability. Its utilization helps in reducing the carbon footprint of the construction industry while making efficient use of a waste material. Figure 2 represents the XRD pattern for analysis conducted on fly ash sample.

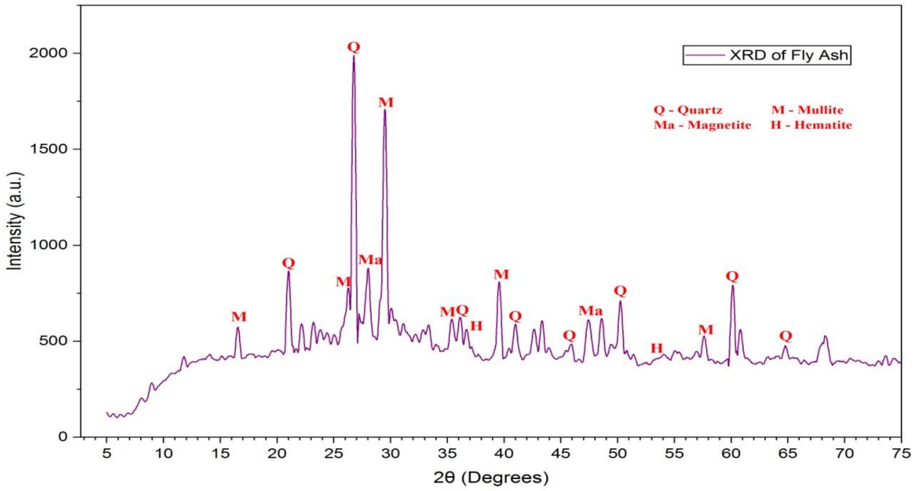


Figure 2: XRD pattern of Fly ash

2.3 Fine aggregate:A well graded sample of fine aggregate typically consisting of particles ranging in size from 0.075 mm to 4.75 mm has been used in the current study. Fine aggregate particles play a tremendous role in binding the cement paste together. They provide a surface for the hydration of cement particles, forming a cohesive paste that binds the aggregates together and develops strength in the concrete.

2.4 Coarse aggregate: Coarse aggregate are larger in size compared to fine aggregate and typically composed of particles ranging from 4.75 mm to 20 mm in diameter are used for the present research work. The shape and texture significantly influence the workability, bonding and interlocking properties of the concrete mix.

2.5 Mix proportioning: The mix proportioning for M25 grade concrete was formulated in accordance with IS: 10262-2019 guidelines to achieve the desired strength of the concrete. Trial mixes incorporating fly ash in varying proportions into the LC^3 concrete mix were conducted. Using these optimized mix ratios, the concrete mix was evaluated for workability, mechanical, and durability properties. Initially, coarse aggregate and fine aggregate were introduced into the mixer and blended for 2 minutes. Subsequently, the necessary amount of LC^3 and fly ash was added to the mixture and mixed for an additional 2 minutes in a dry state. Finally, water and super plasticizer were incorporated into the mixer and mixed thoroughly. The mix proportions of concrete with LC^3 and fly ash is represented in table 2.

Table 2: Mix proportion of concrete with LC3 and fly ash

Material (kg/m^3)	100LC^3	20FA 80LC3	25FA 75LC3	30FA 70LC3	35FA 65LC3	40FA 60LC3
LC^3	394	315.2	295.5	275.8	256.1	236.4
Fly ash	-	78.8	98.5	118.2	137.9	157.6
Fine aggregate	693.41					
Coarse aggregate	1249.9					

3. EXPERIMENTAL INVESTIGATION

The current research evaluates about incorporating fly ash into the LC^3 concrete mixture. The mechanical, durability and workability properties of the LC^3 concrete with different percentages of fly ash ranging from 20% to 40%, were investigated.

3.1 Workability

The slump test is adopted to assess the consistency or workability of fresh concrete. This process entails filling a standard slump cone with concrete, compacting the material, and subsequently removing the cone to observe the slump or subsidence of the concrete. The resulting slump value reflects the flow and cohesiveness of the fresh concrete. Slump test was performed on concrete sample with fly ash and LC^3 in accordance with IS 1199 (Part 2): 2018 norms.

3.2 Compressive strength test on concrete

The compressive test on hardened concrete is a standard test method used to determine the compressive strength of concrete. It involves subjecting a cylindrical or cubical concrete specimen to a gradually applied compressive load until failure occurs. The test results provide valuable information about the strength of concrete and its ability to withstand compressive forces. The primary standard referred is IS 516 (Part 1/Sec 1) : 2021, detailing methods for concrete strength, with IS 456:2000 providing acceptance criteria.

3.3 Split tensile strength test on concrete

This test refers to the measure of the tensile strength of concrete or other materials in a cylindrical specimen. It is determined by subjecting the specimen to a diametric compressive load, causing the concrete to split along the diameter, and calculating the tensile strength based on the applied load and specimen dimensions as per IS 516 (Part 1/Sec 1) : 2021.

3.4 Flexural strength test on concrete

The flexural strength of concrete is one measure of the tensile strength of unreinforced concrete. It refers to the ability of the concrete beam that is being tested to resist bending. The codes referred for flexural strength of concrete are IS 456:2000 and IS 516 (Part 1/Sec 1): 2021.

3.5 Carbonation test on concrete

The carbonation test of concrete is a procedure to determine the depth of carbonation penetration into the concrete surface. It involves exposing the concrete specimen to a carbon dioxide (CO_2) environment and then conducting chemical tests using phenolphthalein solution to identify the presence of carbonation. This test helps assess the susceptibility of concrete to carbonation-induced corrosion as per IS 516 (Part 5/Sec 3):2021.

4. RESULTS AND DISCUSSIONS

4.1 Workability test on concrete`

The workability of concrete mixes containing LC3 and Fly ash was assessed using the slump cone test as prescribed by IS 1199 (Part 2): 2018, with the results presented in figure 3 demonstrating a clear trend in slump values under a constant water-to-cement (W/C) ratio. The incorporation of LC^3 with FA has continuously increased slump values till 35% replacement and a slight decrease at 40% replacement. However, even at 40% replacement of LC^3 with fly ash, a 14.3% of increase in slump can be witnessed. The smaller fly ash particles, in contrast to the LC3 particles, act as a filler within the mixture and are often the cause for this increase in workability. Due to its spherical particle configuration, fly ash can create a "ball-bearing" effect in the concrete mixture. This effect results in increased fluidity and improved workability, reducing friction among particles and facilitating their movement past each other. However, at higher replacement levels, the increased surface area of the particles leads to a rise in water absorption, necessitating a increased amount of super plasticizer to ensure workability.

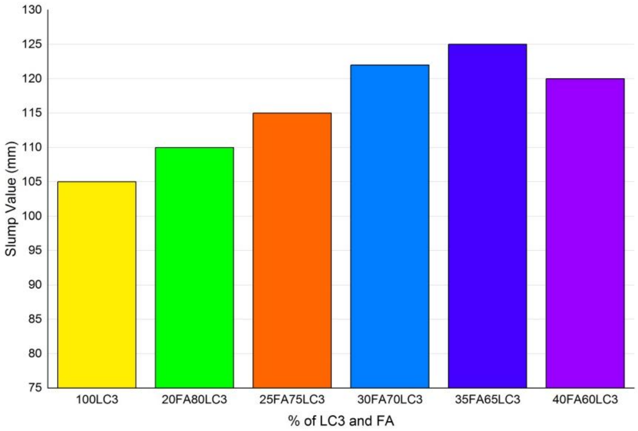


Figure 3: Workability of LC3 with Fly ash by slump test

4.2 Compressive strength test on concrete

Figure 4 illustrates that the concrete mixture containing 30% FA and 70% LC^3 achieves the highest compressive strength of 30.97 N/mm^2 when compared with other combinations. This specific ratio surpasses 100% LC^3 mix by 8.8% increase in strength. Although the it tends to decline with a higher FA content, the blend of 30% FA and 70% LC^3 exhibits exceptional performance. The silica and alumina present in fly ash interact with calcium hydroxide, which is a by-product of cement hydration, resulting in the formation of additional cementitious compounds. This process contributes to a denser microstructure. Furthermore, the fine particles of fly ash occupy voids, enhancing particle packing and decreasing permeability, which ultimately leads to a substantial increase in long-term strength. Furthermore, the transition zone between the aggregates and cement paste, which is typically the weakest point in conventional concrete, is enhanced by the finer particles of FA and LC^3. This improvement results in a more effective load distribution, thereby minimizing micro cracking.

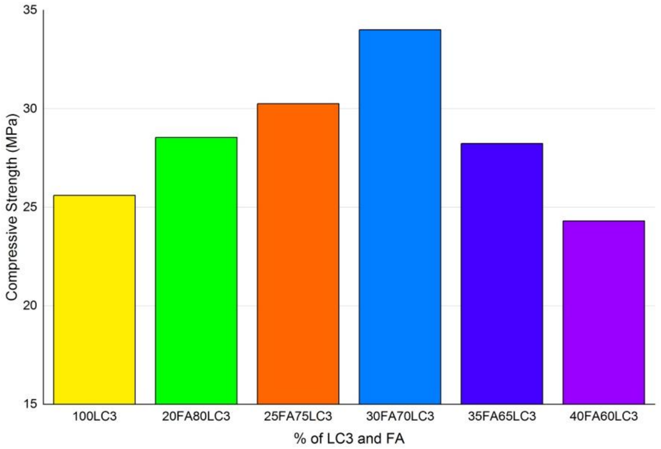


Figure 4: Compressive strength of concrete with LC3 and Fly ash

4.3 Split tensile strength test on concrete

Figure 5 represents the variation in split tensile strength of concrete specimens of 100%LC3, 20%FA80%LC3, 25%FA75%LC3, 30%FA70%LC3, 35%FA65%LC3, 40%FA60%LC3. The tensile strength of specimens with flay ash is relatively at par with the base mix 100% LC^3 till 25% replacement. Whereas there is slight increase in strength of around 5.6% for mix with 30% FA and 70% LC^3. But there is a drastic decrease in split tensile strength beyond 30% replacement of LC^3 with FA.

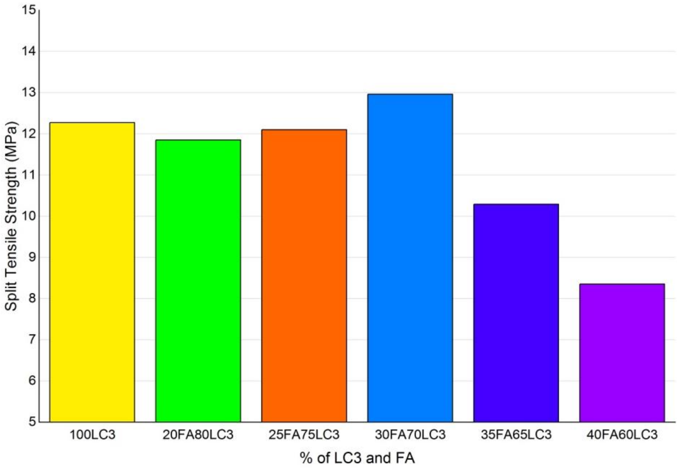


Figure 5: Split tensile strength of concrete with LC^3 and fly ash

The incorporation of fly ash can alter the hydration products generated in LC^3 concrete. The inclusion of fly ash may result in the development of additional C-S-H gel, thereby enhancing the strength of the concrete. Nevertheless, an overabundance of C-S-H can also result in a denser microstructure, which may reduce tensile strength [11]. Furthermore, the addition of other supplementary cementitious materials (SCMs) such as GGBS, silica fume, and metakaolin, whether used in binary, ternary, or quaternary mixtures alongside a partial replacement of ordinary Portland cement (OPC), has been observed to significantly decrease split tensile strength. When LC^3 and fly ash are combined, they enhance each other's strengths. The pozzolanic reaction provided by fly ash guarantees a long-term increase in strength, whereas the early-age strength of LC3 cement helps to alleviate any potential decrease in strength during the initial curing phases. In comparison to concrete mixtures that include fly ash with OPC or PPC, the collaboration between fly ash and LC^3 cement in the concrete mixture demonstrates a more balanced and possibly less significant reduction in split tensile strength.

4.4 Flexural Strength test on concrete

Figure 6 illustrates the flexural strength of specimens with various LC^3 and FA replacements. Incorporation of LC^3 & FA has shown slight improvement in flexural strength up to 30% replacement of LC^3 with fly ash. However higher FA content over 30% exhibit decrease in flexural strength of around 13.6% for up to 40% replacement. Maximum increase in flexural strength is obtained for 30% replacement that is around 4.5% more than the reference mix.

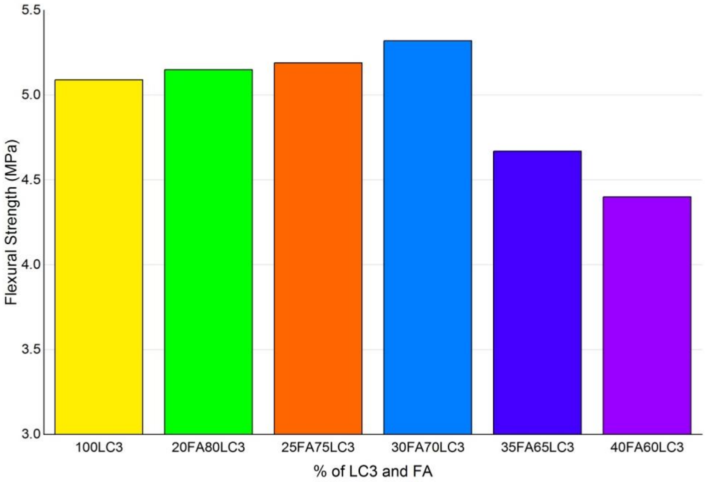


Figure 6: Flexural strength of concrete with LC^3 and fly ash

The incorporation of fly ash into LC^3 concrete results in finer pore structures, increased porosity, and modifications in the distribution of hydration products. These changes in the microstructure can influence the bonding between aggregates and the matrix, thereby affecting the flexural strength [12]. Additionally, variations in micro structural properties can also impact the overall elasticity and deformation behaviour of the concrete, which are essential elements in assessing flexural strength.

4.5 Carbonation test on concrete

It is essential to strike a balance between achieving high compression strength and managing carbonation depth when selecting a mix ratio for specific project requirements. So we must consider both factors to ensure the desired strength and durability of concrete structures in carbonation-prone environments. Figure 7 illustrates the performance of concrete specimens on exposure to carbon di oxide rich environment over a long period whereas figure 8 represents the carbonation depth of concrete cubes after using Phenolphthalein indicator. Carbonation depth has been found to be increasing as the percentage of fly ash is increased up to 40% but the depth of carbonation is well within the reinforcement zone of RCC members typically located at more than 20mm.

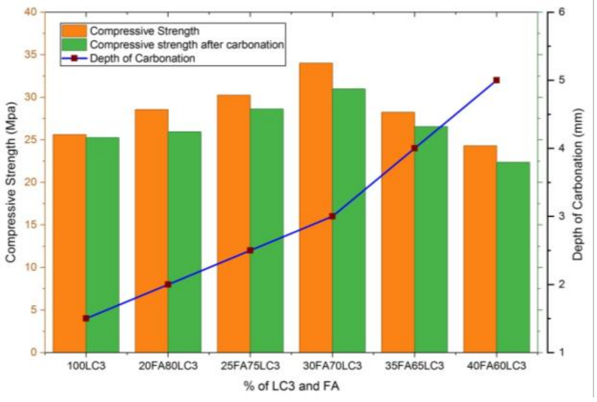


Figure7: Performance of concrete cubes after carbonation

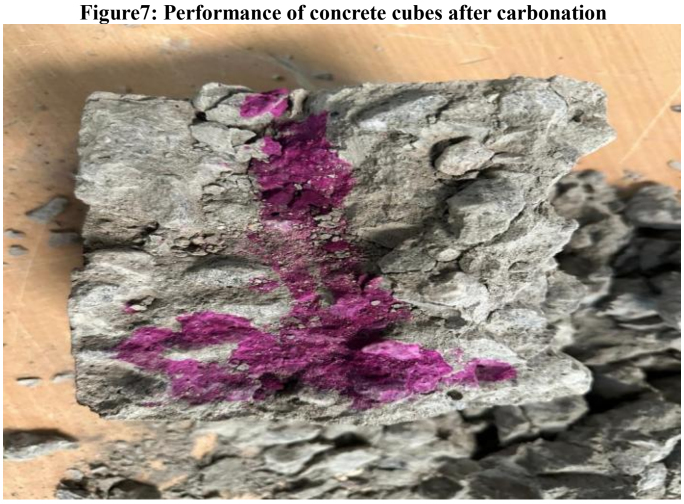
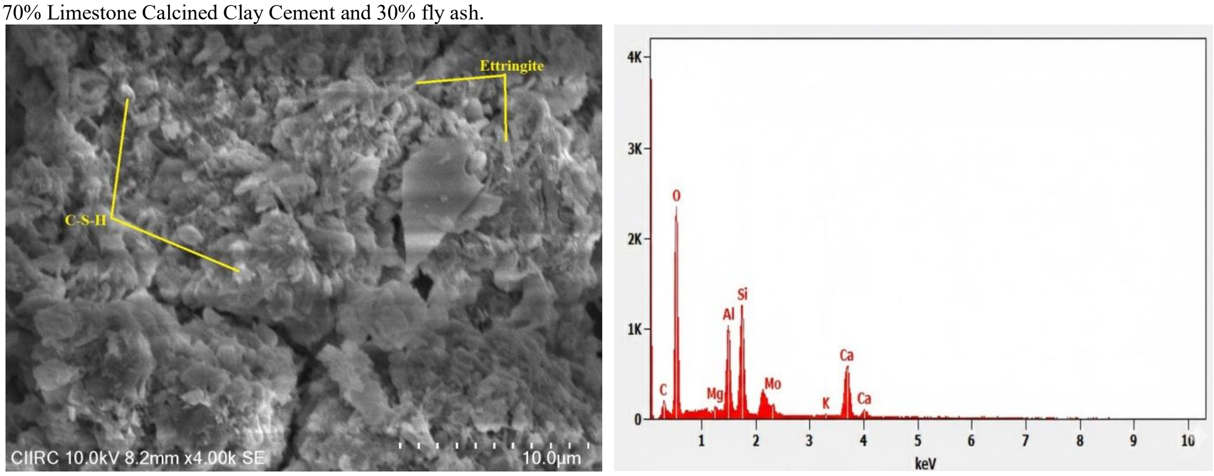


Figure 8: Concrete specimen subjected to carbonation

Carbon dioxide present in the atmosphere permeates the porous structure of concrete. CO₂ interacts with moisture to produce carbonic acid. This carbonic acid subsequently reacts with calcium hydroxide found in the concrete, resulting in the formation of calcium carbonate and water, which leads to a decrease in pH levels. The reduction in pH compromises the passive layer that safeguards the steel reinforcement, rendering it susceptible to corrosion. Whereas in the research, it was determined that the depth of CO_2 penetration falls within the acceptable limits.

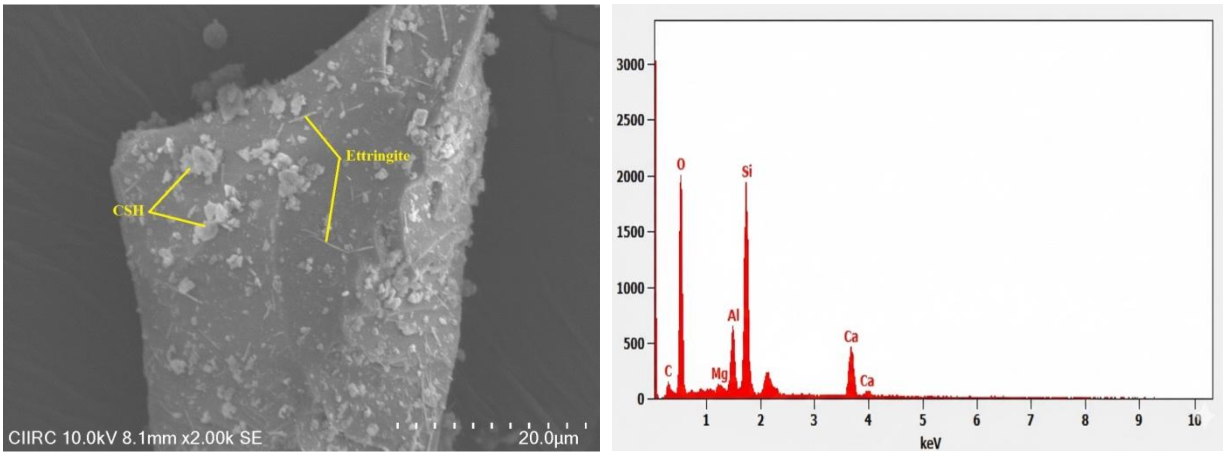
4.6 Microstructural analysis

Samples of LC^3 concrete SEM micrographs at a magnification of 10μm were utilized to investigate the different arrangements of hydration products, such as calcium hydroxide, CSH gel, and un hydrated cement within the concrete samples. Figure 9 (a) and (b) represents the microscopic and EDX image of concrete with only Limestone Calcined Clay cement after 28 days of curing. Whereas, Figure 10 (a) and (b) represents the microscopic and EDX image of concrete with 70% Limestone Calcined Clay Cement and 30% fly ash.



(a) (b)

Figure 9: (a) SEM & (b) EDX image of LC3 concrete specimens



(a) (b)

Figure 10: (a) SEM & (b) EDX image of 30FA70LC3 hardened concrete

The SEM analysis enables the characterization of the microstructure of both the concrete and the cement. The sample for SEM analysis was acquired from the compressive strength assessments conducted to perform a micro-structural analysis on the samples. When observed at higher magnifications, the hydration products and particles of un hydrated cement become apparent in the samples. From the SEM analysis of sample obtained after subjecting the specimen to compression test, it can be observed that several hexagonal pillars were observed in both the specimens, primarily composed of calcium, sulfur, aluminum, and oxygen. The calcium-aluminum-hydrate of these elements reacts with a significant amount of sulfur to produce calcium-aluminum-sulfur-hydrate, commonly known as ettringite [13]. The EDX image of the LC^3 and LC3+FA sample indicates that the elemental composition, which includes elements such as Ca, Si, Al, Mg, K, and O, is represented as peaks in the spectrum. The most prevalent elements in the concrete are Ca, Si, Al, and O. The high percentage of these elements implies the presence of calcium silicate hydrate (C-S-H) phases. The LC^3 pastes exhibits a densification process as hydration progress and additionally, it is found evident that the LC^3 pastes displays a more refined pore structure with a decrease in pore size at different curing ages [14]. Furthermore, the incorporation of fly ash reacts with calcium hydroxide, which is generated during the hydration of cement, to create a calcium silicate hydrate gel. This gel serves to bind the particles together and enhance the strength of the concrete. The EDX spectrum indicates that the concrete primarily consists of Calcium (Ca), Silica (Si), Aluminum (Al), Magnesium (Mg) and Potassium (K). The most pronounced peaks in the spectrum are attributed to calcium, which serves as the principal component of cement. The peaks observed at lower energies correspond to the other elements present in the concrete. A notable reduction in the quantity of calcium hydroxide was observed. Furthermore, it is clear that the incorporation of fly ash contributes to filling the voids within the microstructure. Certain particles of fly ash remain unhydrated due to their slower reactivity. These particles manifest as angular, glassy inclusions in the SEM image. Their existence may influence the long-term strength development and shrinkage characteristics of the concrete. SEM analysis can also uncover the presence of microcracks and pores within the concrete. The incorporation of fly ash can improve the pore structure, resulting in a more compact and less permeable concrete. C_2S and C_3S reached their peak values, while CH attained its lowest level. The predominant elements include Si, C, Fe, O, and Ca. It is believed that C-S-H, tetra-calcium ferrite (C4F), and carbo-aluminates are formed.

5. CONCLUSIONS

Utilizing LC^3 combined with fly ash significantly reduces the carbon footprint of construction, as LC^3 alone can lower CO2 emissions by up to 40% compared to traditional Portland cement by reducing clinker content. Based on the research findings regarding the use of FA in LC^3 concrete, the following conclusions can be drawn:

- The research identifies a 30% replacement of LC^3 with fly ash as the optimum blend, as it exhibits superior performance across various properties for M30 grade concrete.
- The combination of 30% FA and 70% LC^3 achieved 8.8% increase in compressive strength than the mix containing 100% LC^3.
- The inclusion of FA generally increases the workability of fresh concrete due to the "ball-bearing" effect of its spherical particles, which reduces friction and increases fluidity.

- While mechanical strengths peak at 30% replacement, exceeding this threshold leads to a drastic decrease in split tensile strength and a notable decline in flexural strength (approximately 13.6% reduction at 40% replacement).
- Although carbonation depth increases as the addition of FA is increased, the penetration depth remains within permissible limits and is well within the typical reinforcement protection zone.
- SEM and EDX analysis reveal that FA reacts with calcium hydroxide to form additional C-S-H gel, which binds particles and creates a denser, less permeable microstructure.
- The addition FA with LC^3 is a cost-effective solution because it utilizes abundant low-grade clays and industrial waste without requiring capital-intensive modifications to existing cement production facilities.

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