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THESIS
on
**Mechanical and Microstructural Characterization of Lime Sludge-
Metakaolin Blended Concrete Composite**

Submitted in partial fulfilment of the requirements for the award of the degree

of
DOCTOR OF PHILOSOPHY
in Civil Engineering

Submitted By

Arti Chouksey

(2K18/PHD/CE/27)

To



CIVIL ENGINEERING DEPARTMENT
DELHI TECHNOLOGICAL UNIVERSITY

2026

CANDIDATE'S DECLARATION

I hereby declare that the work which is being presented in the dissertation, entitled "Mechanical and Microstructural Characterization of Lime Sludge- Metakaolin Blended Concrete Composite", in Partial fulfillment of the requirements for the award of the degree of **Doctor of Philosophy in Civil Engineering** and submitted to **Delhi Technological University, Delhi** is an original piece of research work carried out by me during the period from August 2018 to July 2026 under the supervision of Dr. **NIRENDRA DEV**, Professor, Department of Civil Engineering, Delhi Technological University, Delhi and Co- Supervision of **Dr. V. V. L. KantaRao** The matter embodied in this thesis has not been submitted by me for the award of any other degree of any other University/Institute.

Arti Chouksey

Candidate Name & Signature

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

Signature of Supervisor (s)

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1. Prof. Nirendra Dev

2. Dr. V.V.L. Kanta Rao

CERTIFICATE

Certified that Name of student **Arti Chouksey** Roll No. **2k18/phdce/27** has carried out their search work presented in this thesis entitled "Mechanical and Microstructural Characterization of Lime Sludge-Metakaolin Blended Concrete Composite" for the award of Doctor of Philosophy from Department of Civil Engineering, Delhi Technological University, Delhi, under our supervision. The thesis embodies results of original work, and studies are carried out by the student herself and the contents of the thesis do not form the basis for the award of any other degree to the candidate or to anybody else from this or any other University/Institution.

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PROF. NIRENDRA DEV	Dr. V V L K Rao
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Arti Chouksey

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LIST OF ABBREVIATIONS

Abbreviation	Full Form
LS	Lime Sludge
CLS	Calcined Lime Sludge
MK	Metakaolin
SCM	Supplementary Cementitious Material
SP	Superplasticizer
FA	Fine Aggregate
CA	Coarse Aggregate
W/B	Water-to-Binder Ratio
FS	Flexural Strength
WA	Water Absorption
RCPT	Rapid Chloride Penetration Test
UPV	Ultrasonic Pulse Velocity
SEM	Scanning Electron Microscopy
EDS	Energy Dispersive X-ray Spectroscopy
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
FTIR	Fourier Transform Infrared Spectroscopy
TGA	Thermogravimetric Analysis
DTG	Derivative Thermogravimetry
DSC	Differential Scanning Calorimetry
C-S-H	Calcium Silicate Hydrate
C-A-S-H	Calcium Aluminosilicate Hydrate
C-H	Calcium Hydroxide (Portlandite)
CaCO ₃	Calcium Carbonate

40	SiO ₂	Silicon Dioxide
	Al ₂ O ₃	Aluminum Oxide
	Fe ₂ O ₃	Ferric Oxide
	CaO	Calcium Oxide
	MgO	Magnesium Oxide
	SO ₃	Sulfur Trioxide
	LOI	Loss on Ignition
	LCA	Life Cycle Assessment
	LCI	Life Cycle Inventory
	LCIA	Life Cycle Impact Assessment
	EPD	Environmental Product Declaration
19	GWP	Global Warming Potential
	ODP	Ozone Depletion Potential
	AP	Acidification Potential
	EP	Eutrophication Potential
	POCP	Photochemical Ozone Creation Potential
	ADP	Abiotic Depletion Potential

ABSTRACT

In this study, two by-products, lime sludge (LS) and metakaolin (MK), were used as partial replacements for cement to enhance the mechanical, durability and microstructural properties of concrete. MK is a reactive aluminosilicate pozzolan, whereas LS is a calcium-rich by-product of the paper industry. The investigation was carried out in two linked phases. In the first phase, M25 grade concrete was prepared with LS and MK at replacement levels of 0–15% each (a 4×4 factorial set of 16 mixes), and the effect of these materials was studied in terms of workability, density, compressive strength, split tensile strength, flexural strength, water absorption, acid resistance and chloride ion penetration. Regression analysis was performed to examine the relationships among strength, density and water absorption, and scanning electron microscopy and energy-dispersive spectroscopy were employed to examine the hydration products and the microstructure. The mix with 10% LS and 10% MK (C80M10L10) performed best among all the LS–MK mixes. Compared with the control mix with 0% LS and 0% MK, it attained about 23% and 26% higher compressive strength at 28 and 90 days, 13–15% higher split tensile strength and 13–15% higher flexural strength. Its water absorption was 20% lower (6.4% versus 8.0%), its mass loss after 12 weeks in 5% HCl was 8% against 10% for the control, and its 56-day RCPT charge was about 64% lower (800 C versus 2,200 C). Regression analysis showed positive but moderate correlations between density and the strength properties ($R^2 = 0.24\text{--}0.36$) and a stronger inverse correlation between water absorption and density ($R^2 = 0.54$). The complete 4×4 factorial matrix of MK–LS blends corresponds to a Taguchi L16 (4^2) orthogonal array, with MK and LS as the two control factors at four levels (0, 5, 10 and 15%). ANOVA of the signal-to-noise ratios showed that MK was the dominant factor, contributing about 41–89% of the total variation in compressive strength, water absorption, RCPT charge and acid-induced mass loss, while LS contributed about 8–31%; because each mix was tested as a single run, the MK $\times$ LS interaction could not be separated from the error term. The cost analysis showed that replacing 20% of the cement with 10% MK and 10% LS increased the material cost of the superplasticised M25 concrete by about 10.2%, mainly because of the higher price of MK, as lime sludge adds very little cost; this

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marginal increase is justified by the improvements in strength, durability, microstructure and sustainability. Overall, the mix with 10% MK and 10% LS is technically and economically feasible and suitable for durable and eco-friendly concrete applications. Microstructural analysis indicated a denser matrix and a refined interfacial transition zone (ITZ) for C80M10L10, and the EDS-derived Ca/Si ratios were consistent with the formation of additional C–S–H/C–A–S–H gel, although these phases were inferred rather than directly identified. In the second phase, the performance of M40 grade concrete incorporating MK and calcined lime sludge (CLS) at 0–20% each was investigated. Workability decreased with increasing replacement owing to the higher specific surface areas of MK and CLS, whereas the mechanical properties improved at moderate replacement levels. The mix containing 15% MK and 10% CLS (M15CL10) exhibited the highest compressive strength (55.2 MPa at 90 days against 49.0 MPa for the control), the highest split tensile and flexural strengths and the lowest water absorption, indicating a synergistic effect between the pozzolanic activity of MK and the calcium-rich composition of CLS; beyond this replacement level, strength decreased and water absorption increased. Quadratic response surface models fitted to the CLS–MK data confirmed 15% MK and 10% CLS as the optimum (desirability 0.95). A cradle-to-gate life cycle assessment showed that the optimum mixes reduce the global warming potential by about 18% (C80M10L10) and 21% (M15CL10) relative to their respective controls. Therefore, this study establishes LS–MK and CLS–MK blended concretes as technically feasible and more sustainable alternatives to conventional concrete, contributing to waste valorisation and a reduced carbon footprint.

Keywords: Lime sludge, metakaolin, concrete, cementitious materials, mechanical properties, durability, carbon footprint, calcined clay, calcined lime sludge.

CHAPTER 1: INTRODUCTION

The most commonly used construction material in the world is concrete, and the rising demand for concrete with the expansion of infrastructure and urbanisation leads to its large-scale production. The clinker manufacturing for the production of cement is associated with the emission of carbon dioxide, depletion of natural resources and high energy consumption (Albidah et al., 2021; Topçu & Sarıdemir, 2008). For sustainable development, the construction practices should include low-carbon, energy-efficient and resource-efficient materials in place of cement in concrete (Berodier & Scrivener, 2015; Zunino & Scrivener, 2019).

For enhancing the performance of concrete, supplementary cementitious materials have emerged as energy-efficient, resource-efficient, and low-carbon materials. There are various materials which are highly pozzolanic, for example, fly ash, rice husk ash, Metakaolin, etc, which are recognised as supplementary cement materials as they can refine the microstructure of concrete by reducing pores and permeability of concrete (Brooks & Megat Johari, 2001; Diaz-Loya et al., 2019).

The strong pozzolanic reaction of MK with the calcium hydroxide released during cement hydration results in the formation of additional C–S–H and C–A–S–H gels, which enhance the durability and strength of concrete. Lime sludge, on the other hand, is a calcium-rich by-product of paper mills, sugar industries and water treatment plants, which has gained attention as a supplementary cementitious material (Vashistha, Singh, et al., 2019). The use of LS in concrete not only decreases environmental pollution but also reduces landfilling. The calcium present in LS helps in enhancing hydration and reducing pores by densifying the concrete matrix (Abo-El-Enin et al., 2016; Buruberry et al., 2015). LS exhibits lower pozzolanic activity than MK, but its filler and nucleation effects provide a favourable environment for the hydration process.

14 The benefits of LS and MK have been documented individually, but their combined use as a partial replacement of cement in concrete has received comparatively little attention; the most closely related studies have combined limestone or lime sludge with calcined clay or silica fume rather than paper-mill LS with high-purity MK (Antoni et al., 2012; Murali et al., 2023). Therefore, this study explores the combined effect of LS and MK on the mechanical, durability and microstructural properties of M25 grade concrete and, after calcination of the sludge, of calcined lime sludge (CLS) and MK in M40 grade concrete. The interaction between the pozzolanic action of MK and the filler effect of LS needs to be understood thoroughly: the calcium for the hydration process is provided by LS, and the pozzolanic action is performed by MK, which is expected to result in the formation of C–S–H and C–A–S–H gels and lead to high-performance and sustainable concrete.

10 The present study focuses on evaluating the synergistic effects of LS and MK as partial replacements of cement in concrete. It includes experimentation, durability assessment and microstructural analysis by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The study aims to identify the optimum LS–MK mix that attains high performance in terms of mechanical, durability and microstructural properties, contributing to sustainable development through waste valorisation and reduced cement consumption.

The present experimental work therefore addresses a research gap and aims to provide a cost-effective, environmentally friendly and high-performance concrete for sustainable development.

1.1 Traditional Concrete

Traditional concrete refers to the mixture of cement, fine aggregates, coarse aggregates and water. In the past years, the above ingredients have been used to make traditional concrete. Concrete is the most extensively used construction material, which depletes resources and degrades the environment due to its high aggregate and cement content. So, advancement in the concrete industry is required for New Age Concrete, which offers better properties of concrete than traditional concrete. New age concrete means

the addition of any new additives in the concrete other than traditional ingredients of concrete, which results in the enhancement of the properties of concrete. Sustainable construction is the demand of society, so special concrete can be prepared by considering the concept of sustainability. Sustainable concrete reduces the environmental effects of manufacturing and disposal while retaining structural integrity and performance (Embong et al., 2016; Memon et al., 2019; Srividya & Kannan Rajkumar, 2022). Recycled rice husk, sugarcane bagasse, ground granulated blast furnace slag, micro silica, fly ash, and bamboo leaf ash may be used to make sustainable concrete (Abbass et al., 2021; Abbass & Akhai, 2025b; Abbass & Singh, 2022b, 2023b, 2024; Li et al., 2010; Verma & Dev, 2022).

1.2 Economic Impact of the Construction Industry

The construction industry plays a pivotal role in driving national and global economic development, contributing significantly to GDP growth, employment generation and infrastructure expansion (Chiang et al., 2015; Pheng & Hou, 2019). The demand for materials, technology and labour in buildings, housing, utilities and transportation networks creates a multiplier effect on investment and supports long-term socio-economic growth (Berk & Biçen, 2017; Chiang et al., 2015). However, the industry's economic importance is connected with resource consumption and environmental impact, highlighting the need for sustainable construction practices that balance ecological responsibility with economic growth (Cronan et al., 2022). Because cement is the most expensive and carbon-intensive ingredient of concrete, its partial replacement with low-cost industrial by-products such as LS, together with reactive pozzolans such as MK, offers a direct economic lever for the industry.

1.3 Environmental Impact of the Construction Industry

Due to the high consumption of natural resources, the construction industry exerts a substantial environmental footprint, with significant waste generation and a high energy demand in the production of cement. Nearly 8% of global carbon dioxide emissions are attributed to cement production (Y. H. M. Amran et al., 2020). The large-

scale extraction of gravel, sand, clay and limestone depletes non-renewable resources, and improper disposal of demolition waste, chemical leaching and particulate emissions cause water and air pollution (Kurniati et al., 2023; N. B. Singh & Middendorf, 2020). Reducing reliance on clinker-intensive binders and recycling industrial by-products have therefore become important strategies for a circular economy (Murali et al., 2024; Qaidi et al., 2022; A. Wang et al., 2020). The partial replacement of clinker by MK and by industrial residues such as LS directly targets this footprint, while diverting LS from landfill.

1.4 Social Impact of the Construction Industry

The construction industry plays a vital social role by providing employment for unskilled, semi-skilled and skilled labour and by delivering housing, schools, hospitals and transport systems (Soundarya Priya & Anandh, 2024). It also faces social challenges, including workplace hazards, inadequate social security and the effects of construction activities on nearby communities (Andersen et al., 2018; Bajracharya et al., 2023). The use of locally available industrial by-products such as LS also has a social dimension, as it reduces the landfill burden on communities living near paper mills.

1.5 Technological Impact of the Construction Industry

Technological advances such as automation, building information modelling (BIM), digital project management tools and robotics have improved productivity, accuracy and safety in construction (S. Zhang et al., 2013; W. Wu et al., 2013), while advanced materials such as geopolymer concrete, fibre-reinforced composites, 3-D printed and high-performance concretes have widened the possibilities for efficient and durable construction (Ngo et al., 2018; Vanderroost et al., 2017). Advances in materials characterisation (SEM/EDS, XRD, TGA and calorimetry) and in the statistical design of experiments, which are used in this thesis, now allow the performance of new binder combinations such as LS–MK to be quantified reliably.

1.6 Need for Sustainable Materials in the Construction Industry

The cement production is associated with the extensive environmental footprint, high carbon emissions and resource depletion, which generates an urgent need for SCMs in concrete (M. Amran, Debbarma, et al., 2021; Cancio Díaz et al., 2017; Krishnan et al., 2018). The rising production cost, ecological degradation and climate change are caused by the traditional construction practices, which heavily rely on the energy-intensive practices, including manufacturing processes and the use of virgin raw materials. An effective approach is offered to reduce the carbon footprint, use of raw materials and reduction of landfills by the provision of sustainable materials, such as pozzolanic materials, recycled aggregates, industrial waste, and low carbon binders for maintaining the structural performance of the concrete (Y. H. M. Amran et al., 2020; de Brito & Kurda, 2021; Lima et al., 2012). By diverting waste from landfills, lowering greenhouse gas emissions, and conserving national resources, these materials support circular economy principles. The increase in demand for infrastructure worldwide, integrating SCMs in construction, is essential for attaining economic viability, long-term environmental stewardship and compliance with evolving green building materials and standards (Ahmed, Mohammed, Rafiq, et al., 2021; Danish et al., 2024; Xia et al., 2024). This demand for greener materials underscores the importance of research into eco-friendly and innovative materials such as metakaolin and lime sludge.

1.7 Supplementary Cementitious Materials

The finely divided mineral additives, which, when utilised in combination with cement in concrete, contribute to the fresh and mechanical properties of concrete through pozzolanic activity or hydration, are termed supplementary cementitious materials (SCMs) (M. Amran, Al-Fakih, et al., 2021; Suraneni et al., 2019; L. Zhang et al., 2023). SCMs like silica fume, fly ash, ground granulated blast furnace slag, Metakaolin, Calcined clay, etc, have become integral to the modern design of concrete due to their properties of enhancing strength, durability, and microstructural properties while decreasing consumption of cement. The formation of binding gels, which refine pore structure, lowers the permeability and improve the resistance to chemicals, is

governed by their pozzolanic reaction with calcium hydroxide. SCMs support sustainable construction by utilising industrial waste, reducing landfills, reducing the use of natural resources, and decreasing the carbon footprint associated with cement (Aprianti S, 2017; S. Gupta & Chaudhary, 2022; Scrivener et al., 2018). SCMs play an important role in developing high-performance and low-carbon concrete, supporting the circular economy by providing greener and durable infrastructure.

1.8 Metakaolin

Metakaolin is a highly reactive aluminosilicate material obtained through the controlled calcination of purified kaolinite clay, typically between 650°C and 800°C. Its amorphous structure and fine particle size contribute to exceptional pozzolanic activity, allowing it to react readily with calcium hydroxide released during cement hydration (Antoni et al., 2012; M. Singh & Garg, 2006). This reaction leads to the formation of additional calcium silicate hydrate (CSH) and calcium aluminosilicate hydrate (CASH) gels, which significantly enhance the strength, durability, and microstructural refinement of concrete. Metakaolin also reduces permeability, mitigates deleterious reactions, and improves long-term performance, making it a superior supplementary cementitious material for sustainable and high-performance concrete (Sarath Chandra Kumar & Ramesh, 2018; Wild et al., 1996; Yue, 2021). Due to its ability to lower clinker content and enhance mechanical properties simultaneously, metakaolin has gained considerable attention in modern low-carbon construction research.

1.9 Lime Sludge

Lime sludge is an industrial waste generated from paper mills, water treatment facilities, and sugar processing industries, which is rich in calcium (Murali et al., 2024). The calcium-rich lime sludge exhibits latent cementitious and filler properties, which make it a potential supplementary cementitious material for the partial replacement of cement in concrete. The fine particles of lime sludge contribute to early hydration, enhance packing density and support the formation of additional CSH and

CASH gel by improving microstructural and strength properties (Abo-El-Enein et al., 2016; Brás et al., 2016; Oliveira et al., 2017). The use of lime sludge in concrete lowers clinker consumption, reduces landfill burden and promotes sustainable management of waste lime sludge offers a promising pathway for developing economically viable and greener cement material for low-carbon and resource-efficient construction practices (Buruberri et al., 2015; IEA, 2018; Simão et al., 2017).

According to the Technology Roadmap of the International Energy Agency (IEA, 2018), calcined clay and limestone are expected to be major drivers of clinker substitution by 2050, as shown in Figure 1.1.

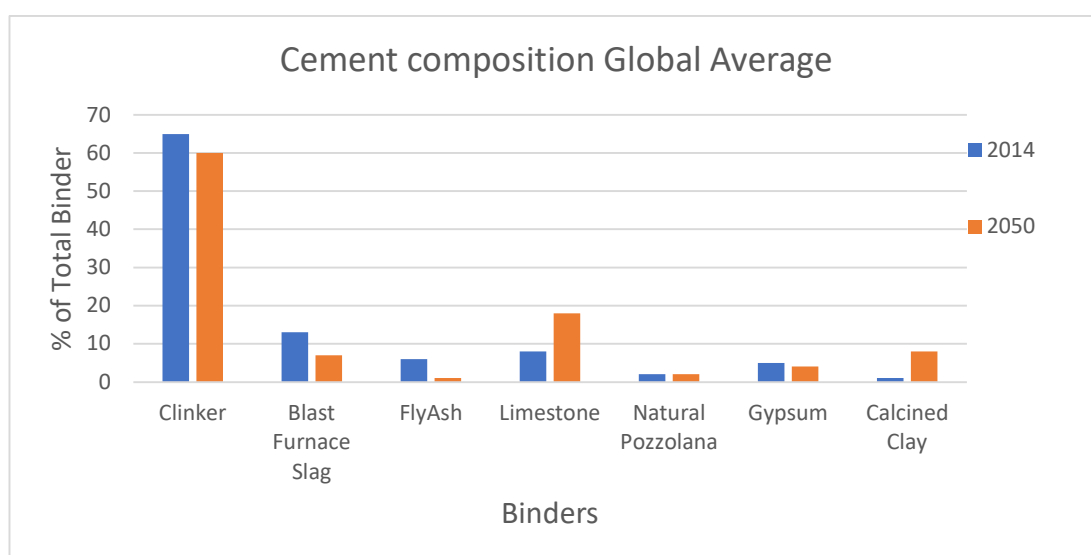


Figure 1.1: Cement composition pathway towards a low-carbon cement sector (IEA, 2018)

India produces about 20–25 million tonnes of paper per year, against a global production of about 400–420 million tonnes. The total (dry) sludge generated by the paper industry is about 4.5–5 million tonnes per year in India and 16–20 million tonnes per year globally, of which lime-sludge-rich streams constitute a significant fraction (Bajpai, 2015; Mandeep et al., 2020). Table 1.1 summarises the paper industry and sludge generation scenario in India and globally.

Table 1.1: Paper industry and sludge generation scenario in India and globally

Parameter	India	Global
Paper production	20–25 million tonnes/year	~400–420 million tonnes/year
Lime sludge generation factor	0.5–1.6 tonnes/tonne paper (lime sludge rich streams)	4.3–50 kg/tonne (dry sludge overall)
Total sludge generation (dry)	4.5–5 million tonnes/year	~16–20 million tonnes/year
No. of paper mills	700–900 mills	70+ countries, hundreds of large mills
Growth rate	5–7% (developing sector)	~2–3% (mature global avg.)
Major contributors	Agro-based & recycled fiber mills	Agro Based
Waste management practice	Landfill + partial reuse	Increasing recycling & circular use
Utilization level	Moderate (emerging applications)	High (cement, soil, energy recovery)

1.10 Calcined Clay

Calcined clay is a highly reactive pozzolanic material produced through the thermal activation of kaolinitic clay. Due to its high silica and alumina content and fine particle size, it can significantly enhance the mechanical strength and durability of concrete through pozzolanic reactions that form additional calcium silicate hydrate (C–S–H) gel (Bhandari et al., 2023; M. Singh & Garg, 2006; N. B. Singh & Middendorf, 2020). In this thesis, the term calcined clay (CC) refers to the broader class of thermally activated clays of variable kaolinite content, whereas metakaolin (MK) denotes the high-purity, highly reactive calcined kaolin. All the experimental mixes of this study contain MK; the abbreviation CC is used only when discussing the literature.

Kaolin, also known as china clay, is widely distributed across India and forms the primary raw material for metakaolin production. Major states with kaolin deposits are Gujarat, Rajasthan, West Bengal, Madhya Pradesh, Jharkhand, and Odisha, with total Reserves of around 2.8 -3 billion tonnes and an annual production of about 3-5 million tonnes.

1.11 Calcined Lime Sludge

Lime sludge is an industrial waste generated from paper and pulp industries, which is rich in calcium compounds. When properly calcined, lime sludge can act as a potential cementitious material and contribute to the formation of additional hydration products in concrete (Murali et al., 2023; Murali et al., 2024; Venu et al., 2024). The utilization of such industrial by-products not only improves the engineering properties of concrete but also helps in reducing waste disposal problems and environmental pollution. In this thesis, calcined lime sludge is used in combination with MK in M40 grade concrete (Phase II of the experimental programme).

1.12 Scope of the Study

The scope of this study encompasses a comprehensive experimental investigation into the combined use of LS and MK as partial replacements for cement in M25 grade concrete. The research evaluates their individual and synergistic effects on fresh properties, mechanical strength parameters, durability performance, and microstructural characteristics. The study includes systematic variations in LS and MK replacement levels to identify an optimal blend that enhances strength and long-term performance while reducing cement consumption. Advanced analytical techniques such as SEM and EDS are employed to validate the hydration behaviour and microstructural refinement resulting from the incorporation of LS and MK. In addition to performance assessment, the study also addresses the sustainability potential of utilising industrial by-products in concrete, focusing on waste valorisation, resource efficiency, and reduction of environmental impact. Overall, the work aims to generate reliable scientific evidence, establish mix design recommendations, and contribute to the advancement of sustainable and high-performance concrete materials.

The investigation was structured in two linked phases. Phase I examined raw LS in combination with MK in M25 concrete, in which LS acts mainly as a calcium carbonate-rich filler and nucleation agent and MK supplies a reactive aluminosilicate. Because the reactivity of raw LS is limited, Phase II examined LS after calcination (CLS), which converts part of its CaCO_3 into reactive CaO , in combination with MK in a higher-strength M40 concrete. The two phases are therefore connected by a common hypothesis: that a calcium source (LS or CLS) and a reactive aluminosilicate (MK) act synergistically, and that increasing the reactivity of the calcium source through calcination extends this synergy to higher-grade structural concrete. Phase II also widened the replacement range to 20% of each material and added calorimetric, XRD and TGA analyses, and both phases were evaluated through life cycle and cost assessments.

1.13 Research Objectives

The study investigates the utilisation of metakaolin and lime sludge as a sustainable building material in an effort to resolve resource depletion, waste management, and sustainable practices in the construction industry. It further seeks to accomplish specific objectives.

1. To characterise lime sludge, calcined lime sludge and metakaolin in terms of their physical properties, chemical composition and mineralogy (XRF, XRD, TGA and SEM).
2. To evaluate the pozzolanic reactivity of lime sludge–metakaolin blends through XRD, TGA and mechanical testing against a reference mortar, and to identify the calcination condition and blend proportion giving the highest reactivity.
3. To optimise the proportion of the lime sludge–metakaolin blend in concrete on the basis of strength properties and to verify the optimum through microstructural characterisation (SEM/EDS).
4. To determine the rheological (slump and slump retention) and mechanical (compressive, split tensile and flexural strength at 7, 28 and 90 days) properties of the blended concrete.

5. To assess the performance of the blended concrete in terms of water absorption, acid attack resistance and chloride ion penetration, together with its environmental (life cycle) and economic performance.

1.14 Organisation of the Thesis

The thesis is organised into five chapters, as outlined below:

Chapter 1: Introduction

The chapter represents an overview of the construction industry. It includes using traditional concrete, economic and environmental aspects and its impact. It further discusses the new developments in sustainable concrete and identifies the scope of binary blend concrete.

Chapter 2: Literature review

This chapter reviews the recent literature on sustainable concrete, metakaolin, calcined clay, lime sludge and calcined lime sludge, critically synthesises the most closely related studies, identifies the research gaps and states the research problem.

Chapter 3: Experimental Programme

This chapter covers the properties of materials and mixes proportions. Design of the binary blended mix proportions as per the guidelines of IS: 10262-2019. Design of experiments matrix with Factor 1 (Metakaolin) and Factor 2 (Lime sludge).

Chapter 4: Results & Discussions

This chapter presents and discusses the experimental results for the approved objectives, including the statistical (Taguchi and RSM), environmental and cost analyses.

Chapter 5: Conclusion and Future Scope

The conclusions to be summarised from the influence of binary blend concrete (Metakaolin and lime sludge) on the mechanical characteristics, microstructural analysis and optimisation technique. This chapter will conclude the research of this experimental and analytical study, with a discussion of the study's future scope.

CHAPTER 2: LITERATURE REVIEW

The literature review presents a comprehensive analysis of previous research related to the use of supplementary cementitious materials in sustainable concrete development, with a specific focus on lime sludge and metakaolin. This chapter examines their individual characteristics, chemical composition, pozzolanic behaviour, and influence on mechanical and durability properties as reported in existing studies. It also highlights the advancements in microstructural analysis, waste valorisation, and environmental benefits associated with these materials. Despite the substantial research available on lime sludge and metakaolin separately, the literature indicates that their combined utilisation in concrete has received comparatively little attention. Therefore, this chapter systematically synthesises the current state of knowledge while identifying the limitations in existing work, thereby establishing the foundation and rationale for the present investigation on the synergistic performance of lime sludge–metakaolin blended concrete.

2.1 Literature Search Methodology

The literature reviewed in this chapter was identified through a structured search of the Scopus, Web of Science, ScienceDirect and Google Scholar databases. Combinations of the keywords “lime sludge”, “lime mud”, “paper mill sludge”, “calcined lime sludge”, “metakaolin”, “calcined clay”, “limestone calcined clay cement”, “supplementary cementitious material”, “pozzolanic reactivity”, “mechanical properties”, “durability” and “microstructure” were used. The search focused mainly on studies published between 2000 and 2026, supplemented by earlier seminal works on metakaolin and pozzolanic reactions. Peer-reviewed journal articles, review papers, conference papers and relevant standards and technical reports in English were included when they reported experimental data or mechanistic insight on the use of metakaolin, calcined clay, lime sludge or calcined lime sludge in cementitious systems. Studies dealing exclusively with unrelated binders, records without accessible full text and duplicate records were excluded, and geopolymers or alkali-activated studies were retained only where they provide transferable insight into the reactivity of metakaolin or calcium-rich residues. The selected studies are

synthesised thematically in Sections 2.2–2.6, and the research gaps derived from them are presented in Section 2.7.

2.2 Metakaolin in Concrete

Metakaolin (MK) is a highly reactive aluminosilicate material obtained through the calcination of high-purity kaolinite clay, typically between 650°C and 800°C. Due to its fine particle size, high pozzolanic activity, and rich silica–alumina composition, MK has emerged as one of the most effective supplementary cementitious materials for enhancing concrete performance (Bhandari et al., 2023; Diaz-Loya et al., 2019). Its incorporation in cementitious systems promotes secondary hydration through the consumption of calcium hydroxide, leading to the formation of additional CSH and CASH gels that significantly improve strength, refine pore structure, and enhance durability (Biswal & Dinakar, 2021; Garcia-Lodeiro et al., 2011). Numerous studies have reported improvements in compressive strength, flexural strength, chemical resistance, and overall microstructural densification when MK is used in appropriate proportions. Given its ability to reduce permeability, mitigate deleterious reactions, and support low-carbon construction practices, metakaolin continues to receive considerable attention in sustainable concrete research (Antoni et al., 2012; H. Wu et al., 2022). Tables 2.1–2.3 summarise the main findings of the existing literature on the role of metakaolin in concrete, its effect on mechanical properties and its effect on durability and microstructure, respectively.

Table 2.1: Role of metakaolin in concrete

Author / Source	Focus Area	Main Findings on Metakaolin	Relevance to the Present Study
(Davidovits, 2018)	Pozzolanic behaviour & MK chemistry	MK is a highly reactive aluminosilicate obtained by calcination of kaolinite (650–800°C). Shows high pozzolanic reactivity and forms	Supports the study's emphasis on MK's contribution to strength and microstructural refinement.

		strong CSH and CASH gels.	
(Belmokhtar et al., 2017)	Microstructure comparison	MK-based geopolymers show denser matrices and improved bonding due to higher SiO ₂ –Al ₂ O ₃ content.	Justifies microstructural improvement observed in LS+MK concrete (dense ITZ, reduced porosity).
(Alanazi et al., 2019)	Durability of MK blends	MK improves sulphate/chloride resistance, reduces permeability, and enhances durability.	Aligns with reduced water absorption and acid attack recorded for L10M10.
(Ahmed, Mohammed, Mohammed, et al., 2021)	Mechanical performance	MK improves compressive, flexural and tensile strength due to accelerated pozzolanic reactions.	Correlates with 23–26% compressive strength increase in L10M10.
(N. B. Singh & Middendorf, 2020)	MK in geopolymer systems	Metakaolin improves early strength and reduces voids; enhances overall binder reactivity.	Reflects similar behaviour in LS–MK concrete (high 7-day strength).
(Habert et al., 2011)	Environmental benefits	MK reduces clinker consumption and CO ₂ footprint.	Supports sustainability objective of LS+MK blend.
(Nana et al., 2024)	Thermal behaviour & reactivity	MK shows high reactivity when activated with suitable alkali or calcium-rich phases.	Lime sludge provides CaO, enhancing MK's

			pozzolanic reaction.
(Oliveira et al., 2017)	SCMs from industrial wastes	MK improves hydration and reduces porosity when blended with Ca-rich residues.	Confirms synergy between MK and LS reported in the manuscript.

Table 2.2: Effect of metakaolin on the mechanical properties of concrete

Author / source	Mechanical property	Main findings on metakaolin	Relevance to the present study
(Wild et al., 1996)	Compressive strength	In superplasticised concrete, strength increased with MK content up to an optimum replacement of about 20%, the largest enhancement occurring at early ages (7–14 days).	Supports the strength gain of MK-containing mixes and the use of a superplasticiser.
(Brooks & Megat Johari, 2001)	Compressive strength	Replacing 5–15% of cement by MK increased compressive strength and reduced creep and shrinkage.	Consistent with the higher strength of MK mixes (Tables 4.7 and 4.8).
(Sabir et al., 2001)	Compressive, tensile and flexural strength	Review: MK accelerates early strength development and improves long-term strength; optimum replacement is commonly about 10–15%.	Supports the 10–15% MK optimum identified in both series.
(Nandanam et al., 2021)	Compressive, split tensile and flexural strength	MK improved the compressive, split tensile and flexural strength of self-compacting concrete with recycled aggregate.	Matches the parallel improvement of all three strengths in LS–MK and CLS–MK mixes.
(Antoni et al., 2012)	Compressive strength (mortar)	Combining MK with limestone allowed high cement substitution with strengths comparable to plain cement.	Supports the synergy between calcium carbonate-rich LS and MK.

(Nadeem et al., 2014)	Compressive strength	MK concrete attained higher compressive strength than the control at ambient temperature.	Corroborates the strength contribution of MK.
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Table 2.3: Effect of metakaolin on the durability and microstructure of concrete

Author / Source	Durability Aspect	Findings About MK	Relation to Present Study
(El-Gamal, Amin, et al., 2017)	Fire resistance & microstructure	MK improves thermal stability and reduces pore connectivity.	Supports creation of dense, stable matrix seen in SEM images.
(Gopalakrishnan et al., 2020)	Water permeability	MK significantly reduces water penetration due to pore refinement.	Matches reduction in water absorption and acid attack.
(Abo-El-Enein et al., 2016)	Chemical resistance	MK decreases $\text{Ca}(\text{OH})_2$ content, increases durability.	Present study: Ca/Si ratio improves and acid resistance increases.
(Ghrici et al., 2007)	Long-term performance	MK reduces porosity, enhances densification.	SEM confirms dense C–S–H & C–A–S–H structures in L10M10.

2.3 Calcined Clay in Concrete

Calcined clay is a thermally activated aluminosilicate material produced by heating kaolinitic or clay-rich soils at temperatures typically ranging from 600°C to 850°C (Bhandari et al., 2023; Murali et al., 2024). The calcination process removes chemically bound water and transforms the crystalline clay structure into a highly

reactive, amorphous pozzolanic phase (Murali et al., 2024; S. K. Singh et al., 2020). Due to its high silica and alumina content, calcined clay readily reacts with calcium hydroxide released during cement hydration, forming additional binding gels, such as CSH and CASH (Patil et al., 2023; Suraneni et al., 2019). This contributes to enhanced early and long-term strength, improved density, and reduced permeability of cementitious composites. Along with performance benefits, calcined clay offers significant sustainability advantages by reducing clinker demand, lowering CO₂ emissions, and enabling the utilisation of abundant, low-cost clays (Murali et al., 2024). Its effectiveness is further pronounced in blended systems where it improves durability parameters such as resistance to sulphate attack, chloride penetration, and alkali–silica reactions (Scrivener et al., 2018). With growing interest in low-carbon construction, calcined clay has emerged as a promising supplementary cementitious material for producing high-performance and environmentally responsible concrete. In this thesis, metakaolin (MK) denotes the high-purity, highly reactive calcined kaolin used in the experimental programme, whereas calcined clay (CC) refers to the broader class of thermally activated clays with variable kaolinite content; the two terms are therefore not used interchangeably. Table 2.4 compares calcined clay and metakaolin.

Table 2.4: Comparison between calcined clay and metakaolin

Author/Source	Aspect	Metakaolin (MK)	Calcined Clay (CC)
(Sabir et al., 2001)	Source	Produced by calcining high-purity kaolinite at 650–800°C	Produced by calcining general clay deposits (kaolinite, illite, montmorillonite)
(Acchar et al., 2009)	Reactivity	Very high due to high amorphous aluminosilicate content	Moderate to high, depends on clay mineralogy
(Nybo Jakobsen, 1994)	Calcination Temperature	650–800°C	600–850°C

(Maraghechi et al., 2018)	Chemical Composition	High SiO ₂ & Al ₂ O ₃ ; low impurities	Variable composition; may contain Fe, K, Mg
(Antoni et al., 2012)	Strength Development	Excellent early & long-term strength	Good strength gain; improved further in LC ³ systems
(Monticelli et al., 2016)	Durability	Strong resistance to chloride penetration, sulphate attack, ASR	Improves durability; especially effective when combined with limestone (LC ³)
(Sabir et al., 2001)	Microstructure	Highly refined pore structure; dense gel formation	Moderately refined structure; improved with balanced calcination
(Acchar et al., 2009)	Workability	Slight reduction due to high fineness	Slight reduction or neutral depending on clay type
(Alvarez et al., 2021)	Sustainability	Moderate CO ₂ reduction; high performance	Very high CO ₂ reduction; large-scale availability
(Maraghechi et al., 2018)	Applications	HPC, UHPC, repair mortars, durability-critical structures	LC ³ cement, mass concrete, low-carbon infrastructure

2.4 Lime Sludge in Concrete

Lime sludge is an industrial waste generated from paper mills, water treatment facilities, and sugar processing industries, which is rich in calcium (Murali et al., 2024). The calcium-rich lime sludge exhibits latent cementitious and filler properties, which make it a potential supplementary cementitious material for the partial replacement of cement in concrete. The fine particles of lime sludge contribute to early hydration, enhance packing density and support the formation of additional CSH and

CASH gel by improving microstructural and strength properties (Abo-El-Enein et al., 2016; Brás et al., 2016; Oliveira et al., 2017). The use of lime sludge in concrete lowers clinker consumption, reduces landfill burden and promotes sustainable management of waste lime sludge offers a promising pathway for developing economically viable and greener cement material for low-carbon and resource-efficient construction practices (Buruberri et al., 2015; Simão et al., 2017). Tables 2.5 to 2.7 present a consolidated review of the existing literature on lime sludge, covering its composition and environmental role, its effect on mechanical properties and its effect on durability and microstructure, respectively.

Table 2.5: Composition, sources and environmental role of lime sludge

Author / Source	Focus Area	Main findings on Lime Sludge	Relevance to the Present Study
(Xu et al., 2014)	Utilisation of industrial lime sludge	Lime sludge (LS) is a calcium-rich waste (mostly CaCO_3), suitable for reuse in cement-based products.	Supports LS as a sustainable calcium source and binder replacement.
(Vashistha, Kumar, et al., 2019)	Valorisation of paper mill LS	LS can partially replace cement and contribute to the circular economy by reducing waste and clinker demand.	Justifies using LS to reduce cement consumption and landfill load.
(Buruberri et al., 2015)	Chemical composition of LS	LS contains Ca(OH)_2 , CaCO_3 and minor impurities; usable in clinker production and binder formulation.	Aligns with chemical contribution of LS in strengthening reactions.
(Samaras et al., 2008)	Environmental benefit of LS	LS reduces landfill, stabilises waste	Important for sustainability goal

		materials, and improves eco-efficiency.	of LS–MK concrete.
(Murali et al., 2024)	LS as low-clinker binder	Recycled LS contributes to hydration and enhances strength when combined with supplementary materials.	Supports strength gain seen in L10M10 mix.

Table 2.6: Effect of lime sludge on the mechanical properties of concrete

Study	Mechanical Property	Main findings on Lime Sludge	Relevance to the Present Study
(Kumar & Prasad, 2019)	Strength & durability	LS improves compressive and flexural strength when blended with pozzolanic materials.	Matches synergy seen in LS+MK mixes, especially L10M10.
(Murali et al., 2023)	Fibre-reinforced LS mixtures	LS improves matrix packing and contributes calcium for pozzolanic reactions with SCMs.	Explains LS role in densifying the matrix in combination with MK.
(Maheswaran et al., 2015)	Belite formation using LS	LS reacts with silica sources and produces strength-enhancing belite phases at low temperatures.	Parallels LS–MK reaction leading to secondary C–S–H formation.
(Nandanam et al., 2021)	Hybrid pozzolan–Ca blends	Calcium-rich additives boost early strength but require balance with reactive SCMs.	Confirms optimum proportion (10% LS + 10% MK) found in study.

Table 2.7: Effect of lime sludge on the durability and microstructure of concrete

Author / Source	Microstructural Aspect	Main findings about LS	Connection to Present Study
(Bhagath Singh & Subramaniam, 2019)	Hydration & microstructure	LS particles act as nucleation sites, improving hydration product distribution and densification.	SEM images show improved ITZ and dense C–S–H gel in LS–MK mixes.
(De Soares et al., 2016)	Pozzolanic behaviour	LS provides reactive Ca for enhanced C–S–H formation when mixed with aluminosilicates.	MK uses LS-derived calcium to form strong binding phases.
(Abo-El-Enein et al., 2016)	Solid waste cementitious behaviour	Ca-rich wastes reduce free lime content and enhance stability and microstructural uniformity.	Explains reduced porosity and improved microstructure in L10M10.
(Khater & Ezzat, 2018)	Engineered composites	LS helps densify matrix and reduce large pores when blended with reactive SCMs.	Matches SEM findings (dense matrix, minimal pore networks).

2.5 Calcined Lime Sludge

Calcined lime sludge is an industrial by-product generated primarily from the causticizing stage of the paper and pulp industry, where lime mud composed mainly of calcium carbonate is recovered, dried, and thermally treated at high temperatures (Maheswaran et al., 2011). The calcination process converts the carbonate-rich sludge into reactive calcium oxide, significantly improving its chemical reactivity and suitability for use as a supplementary material in construction applications (S. K.

Singh et al., 2020). After calcination, the material exhibits a finer particle structure, enhanced surface area, and increased pozzolanic potential, enabling it to participate in secondary hydration reactions when incorporated into cementitious systems. Its high calcium content supports the formation of additional binding phases, contributing to densification of the matrix, reduction in pore connectivity, and improvement in mechanical strength (Gu et al., 2025). Utilising calcined lime sludge supports waste valorisation, reduces dependence on energy-intensive clinker, and contributes to a circular economy approach by transforming an environmental burden into a valuable resource for sustainable construction (Abo-El-Enein et al., 2016; Oliveira et al., 2017). Table 2.8 shows the comparison between lime sludge and calcined lime sludge.

Table 2.8: Comparison between lime sludge and calcined lime sludge

Author/Source	Aspect	Lime Sludge (LS)	Calcined Lime Sludge (CLS)
(S. K. Singh et al., 2020)	Source	By-product of water treatment and paper mills; primarily CaCO_3 with moisture and impurities	Produced by calcining lime sludge at 600–900°C, converting $\text{CaCO}_3 \rightarrow \text{CaO} +$ reactive phases
(Vashistha, Kumar, et al., 2019)	Reactivity	Low reactivity due to the dominance of crystalline CaCO_3	High pozzolanic/latent hydraulic reactivity due to the formation of CaO and amorphous phases
(S. K. Singh et al., 2020)	Calcination Temperature	No calcination; material remains largely inert	700–900°C optimal; excessive temperature may cause sintering
(Gu et al., 2025; Murali et al., 2024)	Chemical Composition	High CaCO_3 (~70–90%), minor SiO_2 , Al_2O_3 , MgO	Higher CaO content after calcination; improved availability of reactive Ca^{2+}

(Kumar & Prasad, 2019) & (Buruberri et al., 2015)	Strength Development	Limited contribution to strength; acts mainly as filler	Significant enhancement in early strength due to CaO hydration forming C–S–H and C–A–H
(Gu et al., 2025; Murali et al., 2024)	Durability	Slight improvement due to filler effect	Higher resistance to sulphate attack, chloride ingress, and carbonation when blended optimally
(S. K. Singh et al., 2020)	Microstructure	Coarse particles; limited pore refinement	Denser microstructure with refined pores due to enhanced hydration
(Gu et al., 2025; Murali et al., 2024)	Workability	Generally improves workability (acts like fine filler)	May reduce workability due to higher fineness and water demand (post-calcination)
(Vashistha, Kumar, et al., 2019)	Sustainability	Good sustainability benefit; reduces landfill dumping	Very high sustainability; reduces CO ₂ emissions by replacing clinker + reuses industrial waste
(S. K. Singh et al., 2020)	Applications	Filler in mortar, bricks, and sub-base stabilisation	SCM in blended cement, geopolymer activator ingredient, durable & low-carbon concretes

2.6 Critical Synthesis of Closely Related Studies

Although the combined use of paper-mill lime sludge and metakaolin has not been reported systematically, several closely related systems in which a calcium carbonate-rich or calcium-rich material is combined with a reactive aluminosilicate have been investigated, and these provide the most relevant benchmarks for the present work. Antoni et al. (2012) showed that metakaolin and limestone act synergistically, the formation of carboaluminate phases allowing high clinker substitution, a concept developed into limestone calcined clay cement (LC³) (Scrivener et al., 2018). Murali and co-workers used recycled lime sludge with calcined clay and silica fume in low-

clinker binders and fibrous concretes (Murali et al., 2023, 2024; Venu et al., 2024), Singh et al. (2020) activated lime sludge thermo-chemically to produce low-clinker binders, Maheswaran et al. (2016) synthesised belite cements from calcined lime sludge and silica fume, and calcined paper sludge has been shown to react pozzolanically (Fernández et al., 2010).

A critical reading of these studies reveals three sources of variability that limit the direct transfer of their findings to the present system. First, the composition of the sludge depends strongly on its source (recausticising lime mud, de-inking sludge or water-treatment sludge) and contains variable proportions of CaCO_3 , free lime, organic matter and moisture (Vashistha, Kumar, et al., 2019). Second, the reported calcination conditions vary widely, from about 550 °C to 900 °C, and the optimum depends on whether the aim is decarbonation to reactive CaO or the preservation of CaCO_3 for carboaluminate formation (Chouksey et al., 2024; S. K. Singh et al., 2020). Third, the reported optimum replacement levels range from about 5% to more than 30%, depending on the binder system, the grade of concrete and the use of a superplasticiser. Claims regarding the novelty of the LS–MK combination in this thesis are therefore made with reference to these closely related studies, which are compared with the present work in Table 2.9.

Table 2.9: Comparison of closely related studies with the present work

Study	Materials / system	Main findings	Difference from the present study
Antoni et al. (2012)	MK + limestone in OPC mortar	Synergy through carboaluminate formation; high substitution with comparable strength	Natural limestone, not paper-mill LS; mortar rather than concrete
Scrivener et al. (2018)	Limestone calcined clay cement (LC ³)	Calcined clay–limestone blends enable large reductions in clinker content	Blended cement; does not use industrial LS
Murali et al. (2023)	Recycled LS + calcined clay + silica fume in fibrous concrete	Improved impact strength and fracture toughness	Ternary binder with fibres; calcined clay instead of high-purity MK
Murali et al. (2024)	Recycled LS in a low-clinker cementitious binder	Hydration and strength development of low-clinker concrete	Different binder formulation; no LS–MK factorial optimisation
S. K. Singh et al. (2020)	Thermo-chemically activated LS	Activated LS used to produce low-clinker binders	Binder production; concrete performance with MK not studied
Maheswaran et al. (2016)	Calcined LS + silica fume	β -belite cement synthesised from calcined LS	Clinker-synthesis route rather than cement replacement

Study	Materials / system	Main findings	Difference from the present study
Fernández et al. (2010)	Calcined paper sludge	Pozzolanic reaction and evolution of hydrated phases	Paste-level study of a different sludge type
Present study	Raw LS + MK (M25) and CLS + MK (M40)	Factorial evaluation of mechanical, durability, microstructural, environmental and cost performance	—

2.7 Identified Research Gaps

The following research gaps were identified from the literature review and the comparison with closely related studies (Section 2.6).

1. To the best of the author's knowledge, the combined use of paper-mill lime sludge (raw or calcined) and high-purity metakaolin as a partial replacement of cement in structural concrete has received little systematic attention; related studies (Table 2.9) mainly combine limestone or lime sludge with calcined clay or silica fume. Consequently, the optimum LS–MK proportions have not been established, and a comprehensive evaluation of their fresh, mechanical and durability properties (workability, density, strength, water absorption, acid and chloride resistance) within a single factorial programme is lacking.
2. The microstructural interaction between calcium-rich lime sludge and aluminosilicate-rich metakaolin, i.e., the hydration behaviour, gel formation and refinement of the interfacial transition zone, has not been characterised using complementary SEM/EDS, XRD, TGA and calorimetric techniques.
3. Information on the chemical compatibility of LS–MK systems, including the Ca/Si and Si/Al ratios of the hydration products and their relationship with strength development, is limited.
4. The effect of calcining lime sludge on its synergy with metakaolin, and the extension of LS–MK blends from normal-strength (M25) to higher-strength (M40) concrete, have not been examined.
5. Statistical design and optimisation tools, such as the Taguchi method and response surface methodology, have rarely been applied to LS–MK or CLS–MK concretes.

6. The sustainability benefits of the combined use of LS (or CLS) and MK, namely the reduction in clinker, waste valorisation, carbon footprint and cost, have not been quantified through life cycle and cost assessments.

2.8 Research Problem Statement

Concrete production continues to impose significant environmental burdens due to high cement consumption, increasing CO₂ emissions, and the growing challenge of managing industrial by-products. Although supplementary cementitious materials such as lime sludge (LS) and metakaolin (MK) have individually demonstrated potential as sustainable alternatives, their combined use has received comparatively little attention in the existing literature. Limited research provides clarity on how LS and MK interact chemically or microstructurally when incorporated together, nor on how calcination of the sludge modifies this interaction, nor on the optimum replacement levels required to enhance the strength, durability, and overall performance of structural-grade concrete. Additionally, the absence of detailed investigations into hydration mechanisms, pore refinement, microstructural synergy, and long-term durability behaviour (such as water absorption and acid resistance) further limits the understanding of their joint contribution. This lack of comprehensive studies on the combined application of LS and MK creates a critical knowledge gap, hindering the development of a sustainable, high-performance, and waste-utilising binder system capable of meeting modern construction requirements. Therefore, a systematic evaluation of the mechanical, durability, and microstructural characteristics of LS–MK blended concrete is essential to address this research deficiency.

CHAPTER 3: EXPERIMENTAL PROGRAMME

This chapter describes the materials used, their characterisation, the mix designs and the testing procedures adopted. The experimental programme comprised two linked phases: Phase I, in which raw lime sludge (LS) and metakaolin (MK) were used in M25 grade concrete, and Phase II, in which calcined lime sludge (CLS) and MK were used in M40 grade concrete. The mixes were designed in accordance with IS 10262:2019 and IS 456:2000, and the mechanical and durability tests were performed in accordance with the relevant IS and ASTM standards.

3.1 Materials Used

Cement

Coarse aggregates

Fine aggregates

Water

Lime sludge

Calcined lime sludge

Metakaolin

Superplasticiser

The source, grade and principal specifications of these materials are summarised in Table 3.1.

Table 3.1: Source, grade and specifications of the materials used

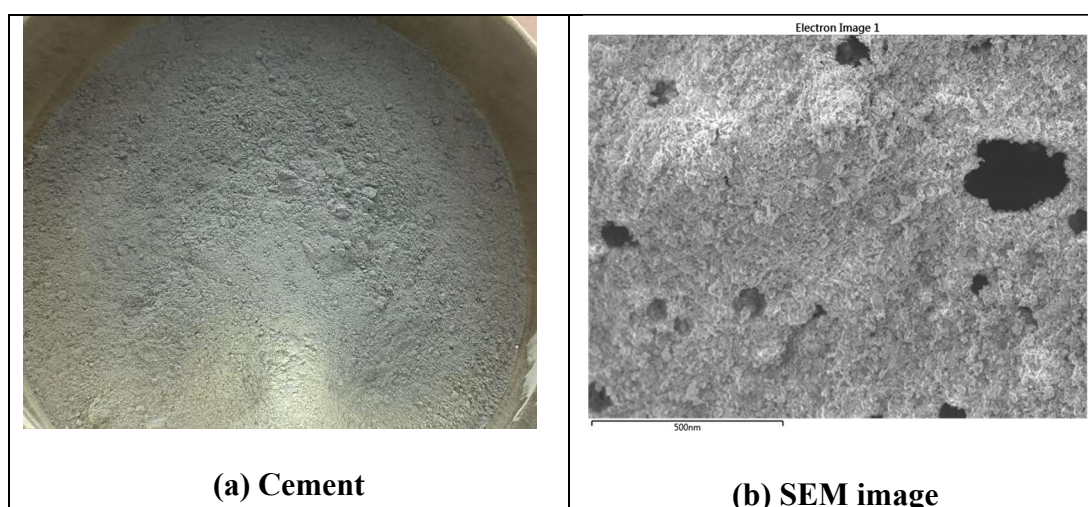
Material	Source / supplier	Grade / type	Specification / reference
Cement	Local supplier (commercially available)	OPC 43 grade	IS 8112:2013; Tables 3.2–3.3
Coarse aggregate	Local supplier	Crushed stone, 20 mm nominal maximum size	IS 383:2016; Tables 3.4–3.5
Fine aggregate	Local supplier	Locally available sand, Zone II	IS 383:2016; Tables 3.6–3.8
Water	Laboratory tap water	—	IS 456:2000 (Clause 5.4); Table 3.9
Metakaolin (MK)	TARA (Technology and Action for Rural Advancement), Qutub Institutional Area, New Delhi	High-reactivity metakaolin ($\text{SiO}_2 + \text{Al}_2\text{O}_3 \approx 93\%$)	Tables 3.10–3.11
Lime sludge (LS)	Bilt Paper Industry, Yamunanagar, Haryana (≈ 200 km from the laboratory)	Paper-mill lime sludge, dried and ground (85% passing 90 μm)	Tables 3.12–3.13

Material	Source / supplier	Grade / type	Specification / reference
Calcined lime sludge (CLS)	Produced in the laboratory from the above LS	Calcined at 750 °C for 2 h, slow-cooled and ball-milled (95% passing 90 µm)	Section 3.2.7; Tables 3.14–3.15
Superplasticiser (SP)	CemWet SP-3000 (commercial product)	Polycarboxylate ether (PCE) based	Section 3.2.8

3.2 Testing of Materials

3.2.1 Cement

Ordinary Portland cement (OPC) of 43 grade conforming to IS 8112:2013 was used for the preparation of the concrete. To ensure conformity with the IS specifications, the following tests were conducted to evaluate the physical properties of the cement. The physical properties and chemical composition of the cement are given in Tables 3.2 and 3.3, respectively. The sample, SEM, XRD and TGA images of the cement are shown in Figure 3.1. The tests performed on the cement are described below:



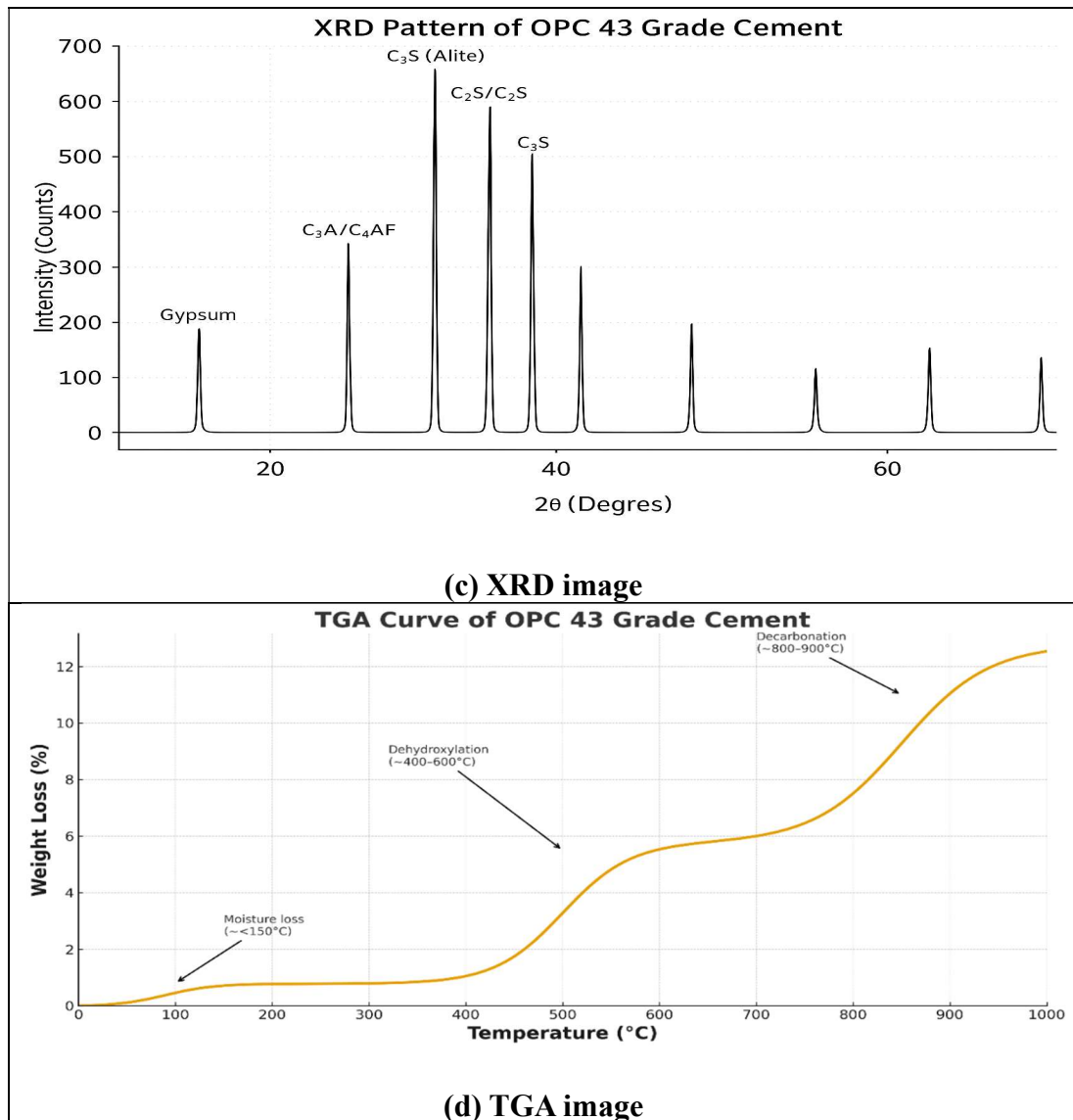


Figure 3.1: Cement: (a) sample, (b) SEM image, (c) XRD pattern and (d) TGA curve

3.2.1.1 Fineness of Cement (IS 4031 Part 2: 1988)

The fineness test determines the particle size of cement, which affects the rate of hydration and strength development. It was measured using the dry sieve method with a 90 μm IS sieve. About 100 g of cement was sieved for 15 minutes, and the residue retained was weighed. The detailed procedure is given below

1. Weigh 100 g of cement accurately.
2. Then place it on a 90 μm IS sieve.
3. Sieve the sample (15 minutes) continuously using a circular and vertical motion.

4. Weigh the residue left on the sieve after sieving.
5. Calculate the percentage of residue retained using equation 3.1.

$$\text{Fineness} = \frac{\text{Residue}}{\text{Original sample}} \times 100 \dots\dots\dots (3.1)$$

3.2.1.2 Standard Consistency Test (IS 4031 Part 4: 1988)

The standard consistency was determined using the Vicat apparatus. Cement paste of varying water contents was tested to identify the percentage of water required to penetrate the plunger 5–7 mm from the bottom of the Vicat mould. Figure 3.2 shows the image of Vicat apparatus. The detailed procedure for the test is given below:

1. Prepare a paste of 300 g cement with a known percentage of water.
2. Fill the Vicat mould with the paste and level the surface.
3. Lower the standard plunger (10 mm diameter, 50 mm long) gently into the paste.
4. Note the depth of penetration.
5. Repeat with varying water percentages until the plunger penetrates to 5–7 mm from the bottom.
6. Record the water content as standard consistency.



Figure 3.2: Vicat apparatus

3.2.1.3 Initial and Final Setting Time (IS 4031 Part 5: 1988)

This test measures the time taken for the cement to begin and complete its setting process. The Vicat apparatus with initial and final setting needles was used. **Initial Setting Time (IST) should not be less than 30 minutes, and Final Setting Time (FST) should not exceed 600 minutes.** The detailed procedure for the test is given below:

1. Mix cement paste using 300 g of cement and water equal to standard consistency.
2. Fill the Vicat mould with the paste.
3. For IST, use the initial needle and apply it every 10 minutes until the needle does not penetrate beyond 5 mm from the bottom.
4. For FST, replace the needle with a circular annular needle and apply until it leaves only an impression on the surface (not on the bottom).
5. Record both times from water addition.

3.2.1.4 Specific Gravity of Cement (IS 2720 Part 3: 1980 – Pycnometer Method)

Specific gravity was determined using a Le-Chatelier Flask or Pycnometer. It indicates the density of cement compared to water and is essential for mix design. The detailed procedure for the test is given below:

1. Dry and weigh an empty pycnometer (C1).
2. Add about 64 g of cement and weigh (C2).
3. Fill the pycnometer with kerosene (no water reaction) and weigh (C3).
4. Clean, fill with kerosene only, and weigh (C4).
5. Equation 3.2 gives the value of specific gravity.

$$\text{Specific Gravity} = \frac{C2-C1}{(C2-C1)-(C3-C)} \dots\dots\dots (3.2)$$

3.2.1.5 Compressive Strength of Cement Mortar (IS 4031 Part 6: 1988)

Cement was mixed with standard sand in a 1:3 ratio by weight and cast into 70.6 mm cubes. **The cubes were tested for compressive strength after 3, 7, and 28 days of curing** using a compression testing machine (CTM). Minimum Required 28-Day Strength for OPC 43 Grade: ≥ 43 MPa. Figure 3.3 shows the image of compressive strength moulds. The detailed procedure for the test is given below:

1. Mix 1 part cement to 3 parts standard sand (by weight).

2. Add water equal to 25% of total mass of cement + sand.
3. Mix thoroughly and fill in 70.6 mm cube moulds.
4. Compact in two layers using a vibrating table or hand tamping.
5. Cure cubes in clean water at $27 \pm 2^\circ\text{C}$ for 3, 7, and 28 days.
6. Test the cubes using a compression testing machine at a loading rate of 35 N/mm²/min.
7. Record and report the compressive strength.



Figure 3.3: Moulds for the compressive strength test of cement mortar

3.2.1.6 Soundness Test (IS 4031 Part 3: 1988)

This test was done using the Le Chatelier apparatus to ensure that the cement does not show excessive expansion after setting, which can lead to cracking. The permissible Expansion is < 10 mm. Figure 3.4 shows the sample made for the soundness test of cement, and the detailed procedure for the test is given below:

1. Lightly oil the Le Chatelier mould and place it on a glass plate.
2. Fill the mould with cement paste of standard consistency.
3. Cover with another glass plate and place a small weight to ensure contact.
4. Submerge in water at room temperature for 24 hours.
5. Measure the initial distance between indicator points.

6. Boil the water for 3 hours while the mould is submerged.
7. Allow cooling and measure final distance between indicators.
8. The difference gives the expansion.

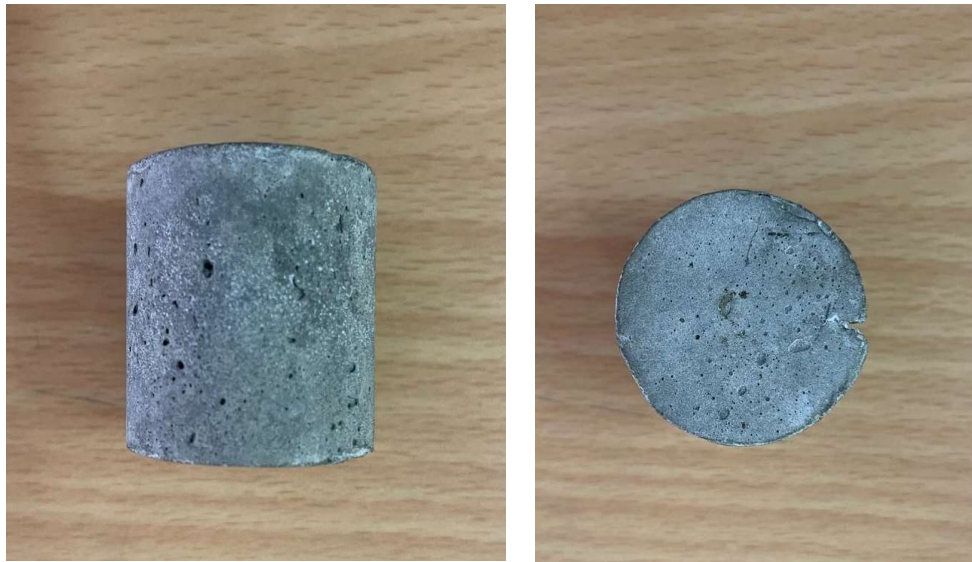


Figure 3.4: Soundness test of cement (Le Chatelier method)

Table 3.2: Physical properties of cement

Property	Result	IS Code and limit
Standard Consistency (%)	28	IS 4031 Part 4 (26–33%)
Fineness (% retained on 90 μ m)	6.4	IS 4031 Part 2 ($\leq$ 10%)
Specific Gravity	3.13	IS 2720 Part 3 (3.10–3.15)
Initial Setting Time (min)	41	IS 4031 Part 5 ($\geq$ 30 min)
Final Setting Time (min)	318	IS 4031 Part 5 ($\leq$ 600 min)
Compressive strength (28 days, MPa)	43	IS 4031 Part 6 ($\geq$ 43)
Soundness (mm expansion)	3	IS 4031 Part 3 ($\leq$ 10 mm)

Table 3.3: Chemical composition of cement

Oxide	Content (%)	Role
-------	-------------	------

CaO	64	Forms C ₃ S and C ₂ S, responsible for strength development
SiO ₂	22	Combines with CaO to form C ₃ S and C ₂ S (strength phases)
Al ₂ O ₃	6	Contributes to C ₃ A phase; affects setting time
Fe ₂ O ₃	2	Forms C ₄ AF; minor role in strength
MgO	3	Should be < 6%; excess causes expansion
SO ₃	2	Controls setting; reacts with C ₃ A
Na ₂ O	0.4	Part of alkalis; excessive content causes efflorescence

3.2.2 Coarse Aggregates

To ensure the suitability of the coarse aggregates, procured from a local supplier, the following tests were performed as per IS 2386 (Parts I–IV) before their use in this study. Figure 3.5 shows the coarse aggregates, and Tables 3.4 and 3.5 show the sieve analysis and the other test results, respectively. The detailed procedures for the tests are given below:



Figure 3.5: Coarse aggregates

3.2.2.1 Sieve Analysis (IS 2386 Part I: 1963)

For determining the particle size distribution and ensure proper grading of coarse aggregates following procedure was followed:

1. Weigh a sample of coarse aggregates (typically 5–10 kg).
2. Sieve the sample through a standard set of IS sieves (80 mm to 4.75 mm).

3. Record the weight retained on each sieve.
4. Calculate the cumulative percentage retained and percentage passing.
5. Plot a grading curve if required.

Table 3.4: Sieve analysis of coarse aggregates

IS Sieve Size (mm)	Observed % Passing	IS 383:2016 Limits (% Passing)	Range	Compliance
40	100	100	Fixed	✓ Within limit
20	95	85 – 100	85–100	✓ Within limit
16	72	Not Specified	–	For reference only
12.5	30	Not Specified	–	For reference only
10	5	0 – 20	0–20	✓ Within limit
4.75	0	0 – 5	0–5	✓ Within limit

3.2.2.2 Specific Gravity and Water Absorption (IS 2386 Part III: 1963)

For the determination of the density and water-absorbing capacity of aggregates, the following testing procedure was followed

1. Take a 2 kg sample and soak it in water for 24 hours.
2. Remove surface water and weigh the saturated surface dry sample (A1).
3. Weigh the sample while submerged in water (A2).
4. Oven dry the sample at 100–110°C for 24 hours and weigh it (A3).
5. Compute specific gravity and water absorption with the help of equations 3.3 and 3.4, respectively.

$$\text{Specific Gravity} = \frac{A3}{(A1-A2)} \dots \dots \dots (3.3)$$

$$\text{Water absorption} = \frac{A1-A3}{A3} \times 100 \dots \dots \dots (3.4)$$

3.2.2.3 Aggregate Crushing Value (IS 2386 Part IV: 1963)

To assess the compressive strength of aggregates under a gradually applied load, the following procedure has been adopted:

1. Fill the cylindrical mould with aggregate and compact in three layers.
2. Apply a compressive load of 40 tonnes in 10 minutes.
3. Sieve the crushed material through a 2.36 mm sieve.

4. Calculate the percentage passing:

$$\text{Aggregate Crushing value} = \frac{\text{weight passing 2.36 mm sieve}}{\text{Total weight}} \times 100 \dots \dots \dots (3.5)$$

3.2.2.4 Aggregate Impact Value (IS 2386 Part IV: 1963)

For determining the value of impact of aggregates due to blows following procedure has been adopted

1. Fill the impact mould with aggregates in three layers and compact.
2. Subject the sample to 15 blows from the hammer.
3. Sieve through 2.36 mm and weigh fines.
4. Compute impact value:

$$\text{Aggregate impact value} = \frac{\text{weight of fines}}{\text{original weight}} \times 100 \dots \dots \dots (3.6)$$

Table 3.5: Test results of coarse aggregates

S. No.	Test Name	Test Method / IS Code	Measured value	Permissible Limits / Remarks
1	Sieve Analysis	IS 2386 (Part I): 1963	Well graded (20 mm nominal)	Should conform to IS 383:2016 grading zone
2	Specific Gravity	IS 2386 (Part III): 1963	2.7	Typical range: 2.6 – 2.9
3	Water Absorption (%)	IS 2386 (Part III): 1963	0.8%	≤ 2% (IS 383:2016 recommendation)
4	Aggregate Crushing Value (%)	IS 2386 (Part IV): 1963	21.4%	≤ 30% for concrete work
5	Aggregate Impact Value (%)	IS 2386 (Part IV): 1963	18.7%	≤ 30% for non-wearing surfaces

3.2.3 Fine Aggregates

Natural river sand conforming to IS 383:2016 was used as the fine aggregate, and the testing procedures are given in the following subsections. Tables 3.6, 3.7 and 3.8 show the grading zone classification, the sieve analysis and fineness modulus, and the other test results of the fine aggregates, respectively. The observed percentages passing (Table 3.6), computed from the sieve analysis in Table 3.7, fall within the limits of Zone II, and the fineness modulus is 2.60. The particle size distribution curves of the coarse and fine aggregates are shown in Figure 3.6.

3.2.3.1 Sieve Analysis (IS 2386 Part I: 1963)

To determine particle size distribution, IS 2386 (Part 1) – 1963 has been followed, and the procedure is given below

1. Take 1 kg of oven-dried sand.
2. Stack IS sieves (4.75 mm, 2.36 mm, 1.18 mm, 600 μ m, 300 μ m, 150 μ m).
3. Place sand in top sieve and shake mechanically for 10 minutes.
4. Record the weight retained on each sieve.

Table 3.6: Grading zone classification of fine aggregates (IS 383:2016)

IS Sieve Size (mm)	% Passing for Zone I	% Passing for Zone II	% Passing for Zone III	% Passing for Zone IV	Observed % Passing
4.75	90–100	90–100	90–100	95–100	98
2.36	60–95	75–100	85–100	95–100	91
1.18	30–70	55–90	75–100	90–100	76
0.6	15–34	35–59	60–79	80–100	51
0.3	5–20	8–30	12–40	15–50	21
0.15	0–10	0–10	0–10	0–15	3

Table 3.7: Sieve analysis and fineness modulus of fine aggregates

Sieve Size (mm)	Weight Retained (g)	Cumulative Weight Retained (g)	% Weight Retained	Cumulative % Retained
4.75	20	20	2.00	2.00
2.36	70	90	7.00	9.00
1.18	150	240	15.00	24.00
0.600	250	490	25.00	49.00
0.300	300	790	30.00	79.00
0.150	180	970	18.00	97.00
Pan	30	1000	3.00	100.00

Fineness modulus =

$$\frac{\sum \text{cumulative percentage retained on IS sieves (4.75 mm to 150 } \mu\text{m)}}{100} \dots\dots\dots (3.7)$$

Fineness modulus 2.6

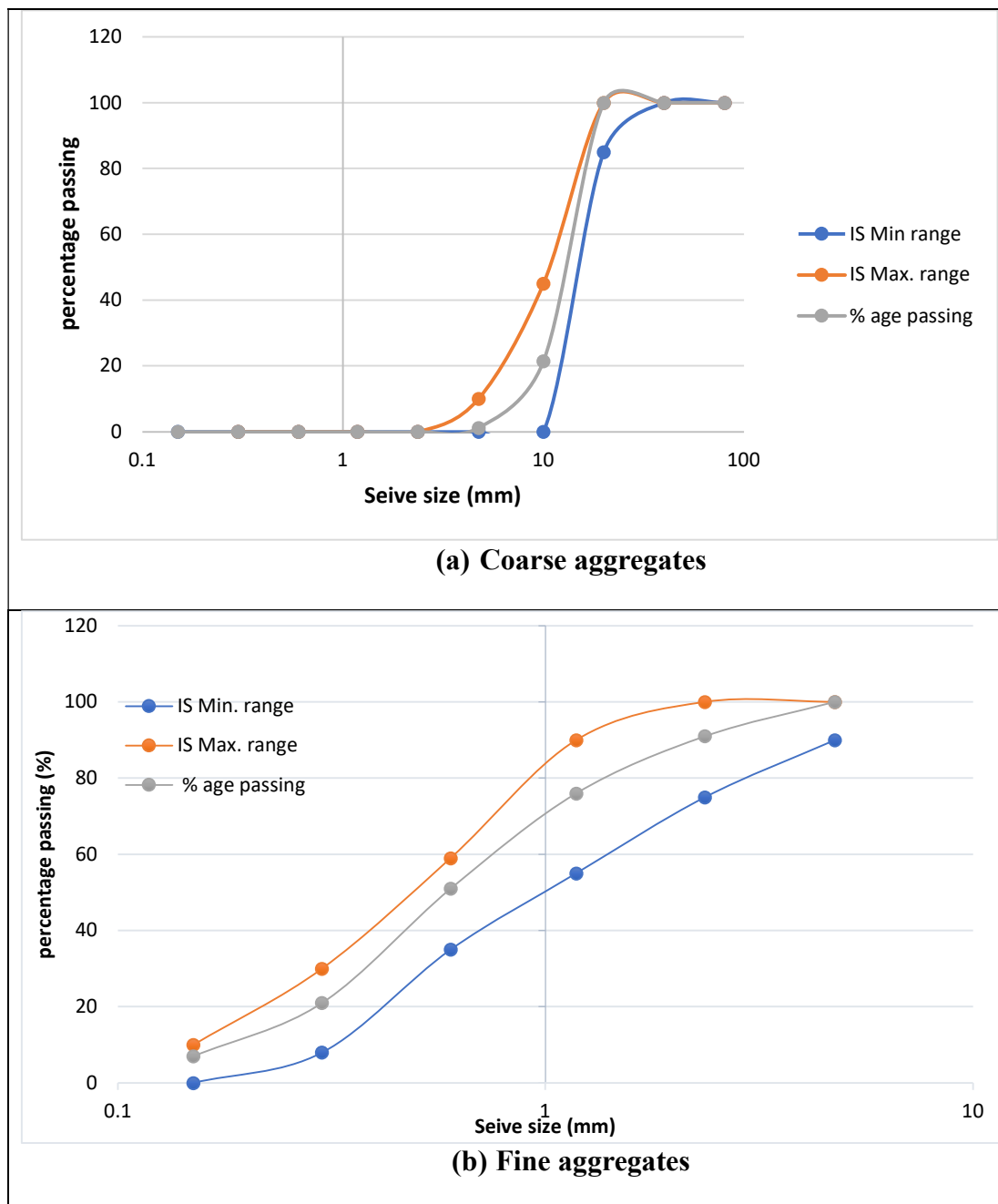


Figure 3.6: Grading curves of (a) coarse aggregates and (b) fine aggregates

3.2.3.2 Specific Gravity (IS 2386 Part III: 1963)

For the determination of the specific gravity of fine aggregates, the following procedure has been followed, and equation 3.8 has been used to calculate final results.

1. F1 = Weight of empty pycnometer
2. F2 = Weight of pycnometer + dry sample

3. F3 = Weight of pycnometer + sample + water
4. F4 = Weight of pycnometer filled with water only

$$\text{Specific Gravity} = \frac{F2-F}{(F2-F1)-(F3-F4)} \dots\dots\dots (3.8)$$

3.2.3.3 Bulking of Sand (IS 2386 Part III: 1963)

For the determination of bulking of fine aggregates following procedure has been followed, and equation 3.9 has been used to calculate final results.

1. Fill the measuring cylinder with 200 ml of moist sand (Vm).
2. Add water to completely saturate it.
3. Measure reduced volume (Vs).
4. Vm = Volume of moist sand
5. Vs = Volume of fully saturated sand (i.e., after flooding with water)

$$\text{Bulking (\%)} = \frac{V_m - V}{V_s} \times 100 \dots\dots\dots (3.9)$$

3.2.3.4 Silt Content Test (IS 2386 Part II: 1963)

For the determination of silt content of fine aggregates following procedure has been followed, and equation 3.10 has been used to calculate final results.

1. Add sand to 100 ml mark in measuring jar.
2. Add water and 1% salt solution, shake thoroughly.
3. Allow to settle for 3 hours.
4. Measure the height of silt and sand separately.
5. Hs is height of silt layer after 3 hours of settling (in mm or cm)
6. Ht is total height of sand + silt layer (in same unit)

$$\text{Silt Content (\%)} = \frac{H_s}{H_t} \times 100 \dots\dots\dots (3.10)$$

3.2.3.5 Moisture Content (IS 2386 Part III: 1963)

For the determination of moisture content of fine aggregates following procedure has been followed, and equation 3.11 has been used to calculate final results.

1. Weigh the moist sample.
2. Oven-dry at 100–110°C for 24 hrs.
3. Weigh the dry sample.
4. Mw is the Weight of the **wet sample** (before drying)
5. Md is the Weight of the **dry sample** (after oven drying at 100–110°C)

$$\text{Moisture content} = \frac{M_w - M_d}{M_d} \times 100 \dots\dots\dots (3.11)$$

Table 3.8: Test results of fine aggregates

Test Name	IS Code	Purpose	Standard Limit	Experimental Value
Sieve Analysis	IS 2386 (Part 1), IS 383	Grading zone classification	As per Zone I–IV (IS 383)	Zone II
Fineness modulus	IS 2386 (Part 1), IS 383	Average particle size	As per (IS 383)	2.6
Specific Gravity & Absorption	IS 2386 (Part 3)	Assess density and porosity	Absorption ≤ 2%	2.63 & 1.2
Bulking	IS 2386 (Part 3)	Volume increases due to moisture	Should be corrected in batching	18
Silt Content	IS 2386 (Part 2)	Check silt impurity	≤ 8%	5
Moisture Content	IS 2386 (Part 3)	Water present in the sand	Depends on site/season	4.76

3.2.4 Water

Water is an essential ingredient of concrete, as hydration is not possible without it. The water used was free from mud, debris and harmful chemical compounds and was tested according to IS 456:2000 (Clause 5.4). Table 3.9 shows the test results of the water. The tests performed on the water, along with their procedures, are given below:

3.2.4.1 pH Value Test

For the determination of pH value of water following procedure has been followed

1. Calibrate the pH meter using standard buffer solutions.

2. Pour the water sample into a clean beaker.
3. Dip the electrode into the water.
4. Record the reading once stabilized.

3.2.4.2 Chloride Content Test

For the determination of chloride content of water following procedure has been followed.

1. Take a known volume of water sample (e.g., 100 ml).
2. Add potassium chromate indicator.
3. Titrate with silver nitrate solution until brick-red colour appears.
4. Calculate chloride concentration using titration volume.

3.2.4.3 Sulphate Content Test

For the determination of sulphate content of water following procedure has been followed

1. Add barium chloride to water sample.
2. Heat the solution and allow barium sulphate to precipitate.
3. Filter, dry, and weigh the precipitate.
4. Calculate sulphate content (as SO_4^{2-}).

3.2.4.4 Total Dissolved Solids (TDS)

For the determination of TDS of water following procedure has been followed

1. Filter water to remove suspended particles.
2. Evaporate 100 ml of water sample in a dish at 105°C until dry.
3. Weigh the dry residue.
4. Multiply by 10 to get TDS in mg/L.

3.2.4.5 Organic Matter Test

For the determination of organic matter in water following procedure has been followed

1. Add potassium permanganate (KMnO_4) and sulphuric acid to water.
2. Heat the solution for 10 minutes.
3. Observe colour change.

4. Titrate with oxalic acid for quantitative analysis if needed.

3.2.4.6 Suspended Solids

For the determination of suspended solids in water following procedure has been followed

1. Filter a known volume of water (e.g., 500 ml).
2. Dry the residue at 105°C.
3. Weigh the residue.
4. Calculate in mg/L.

3.2.4.7 Alkalinity Test

For the determination of the alkalinity of water following procedure has been followed

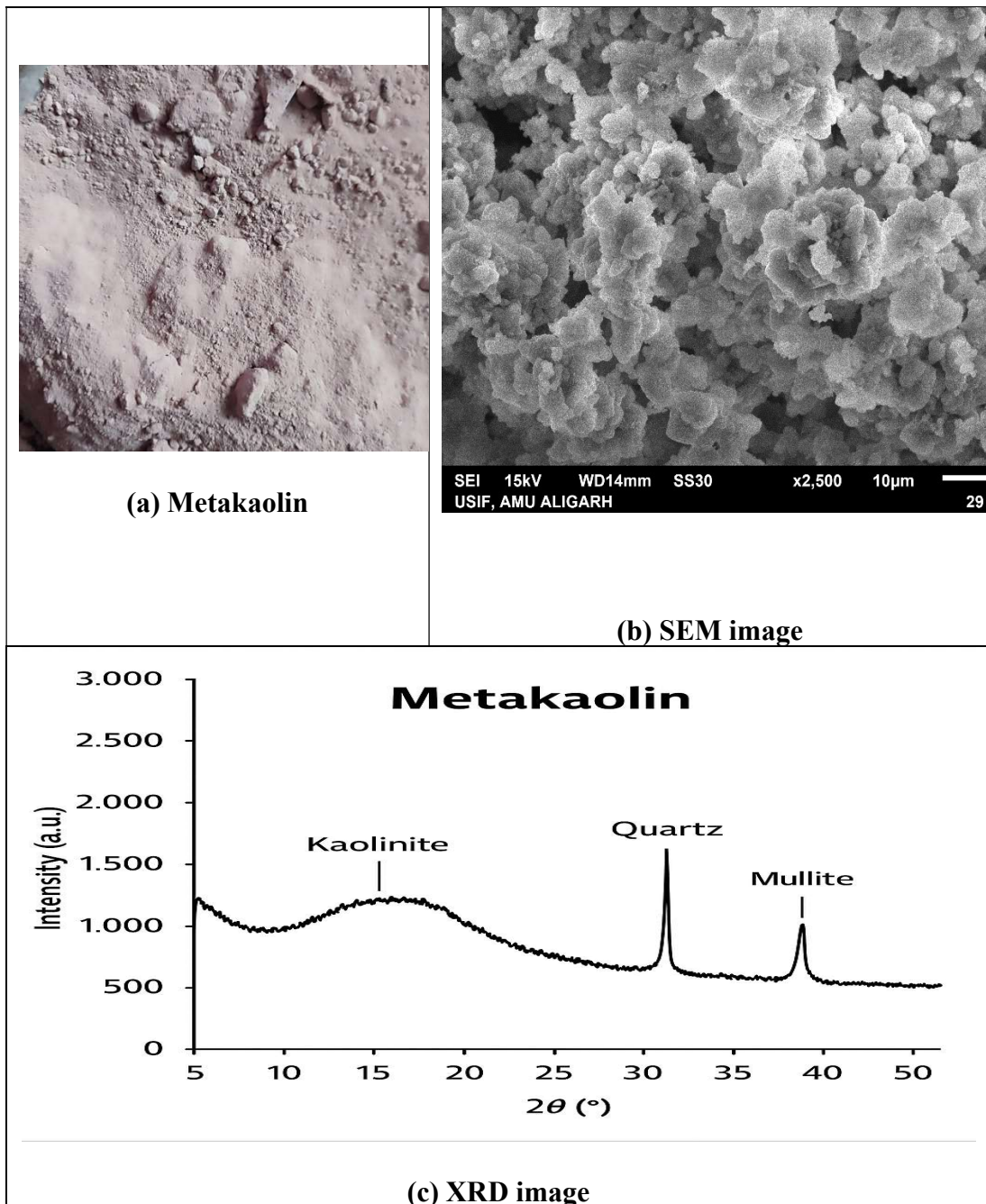
1. Take a known volume (100 ml) of water.
2. Add methyl orange indicator (colour turns yellow).
3. Titrate with standard HCl until the colour changes to orange-pink.
4. Calculate alkalinity as mg/L of CaCO₃.

Table 3.9: Properties of mixing water

Parameter	IS Code	Observed Value	Acceptable Limit	Remarks
pH Value	IS 3025 (Part 11)	7.9	6.0 – 8.5	Within Limits
Chloride (mg/L)	IS 3025 (Part 32)	150	≤ 200 (RCC)	Pass
Sulphates (mg/L)	IS 3025 (Part 24)	300	≤ 400	Pass
TDS (mg/L)	IS 3025 (Part 16)	1250	≤ 2000	Pass
Organic Matter (mg/L)	IS 3025 (Part 18)	85	≤ 200	Pass
Suspended Solids (mg/L)	IS 3025 (Part 17)	350	≤ 2000	Pass
Alkalinity (mg/L)	IS 3025 (Part 23)	110	≤ 120	Pass

3.2.5 Metakaolin (MK)

Metakaolin is a pozzolanic material used as a partial replacement of the binder to improve the mechanical and durability characteristics of concrete. The testing procedures are the same as those for cement. Figure 3.7 shows the images of MK, and Tables 3.10 and 3.11 show its physical properties and chemical composition.



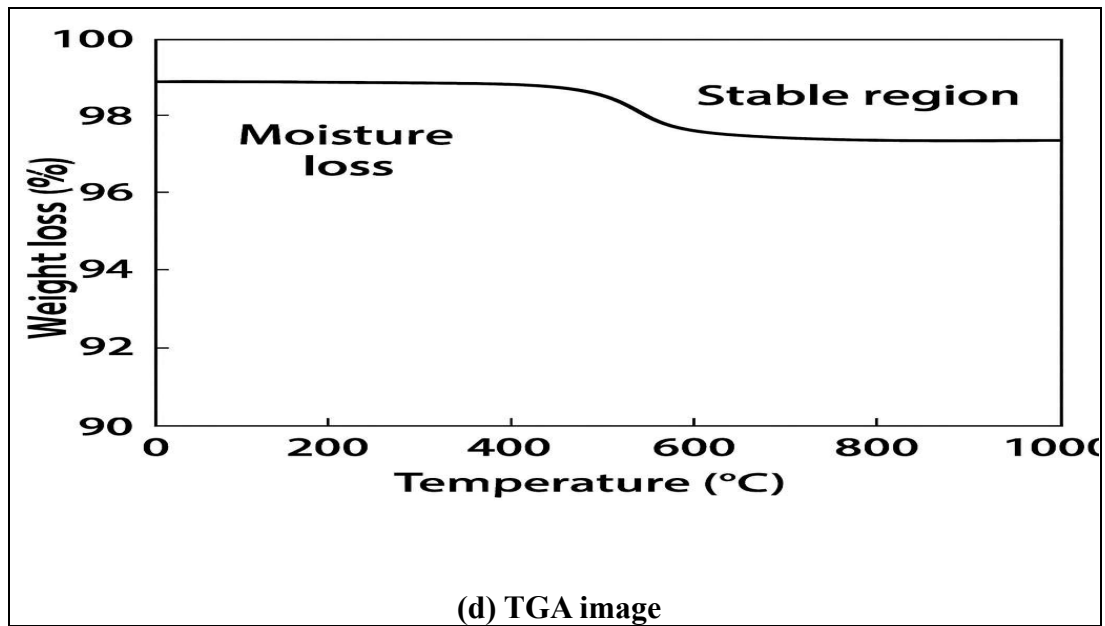


Figure 3.7: Metakaolin: (a) sample, (b) SEM image, (c) XRD pattern and (d) TGA curve

Table 3.10: Physical properties of metakaolin

Test	Value	IS/ASTM Code
Specific Gravity	2.6	IS 1727 / ASTM C618
Fineness (45 μm sieve)	2	ASTM C1447 / IS 1727
Bulk Density	300–450 kg/m^3 (depends on calcination)	—
Moisture Content	1%	IS 4031 (Part 2)

Table 3.11: Chemical composition of metakaolin

Oxide	Content (%)
SiO_2	52.1
Al_2O_3	41
Fe_2O_3	4.32
CaO	0.07
MgO	0.19
Na_2O	0.26
K_2O	0.63
SO_3	0.02

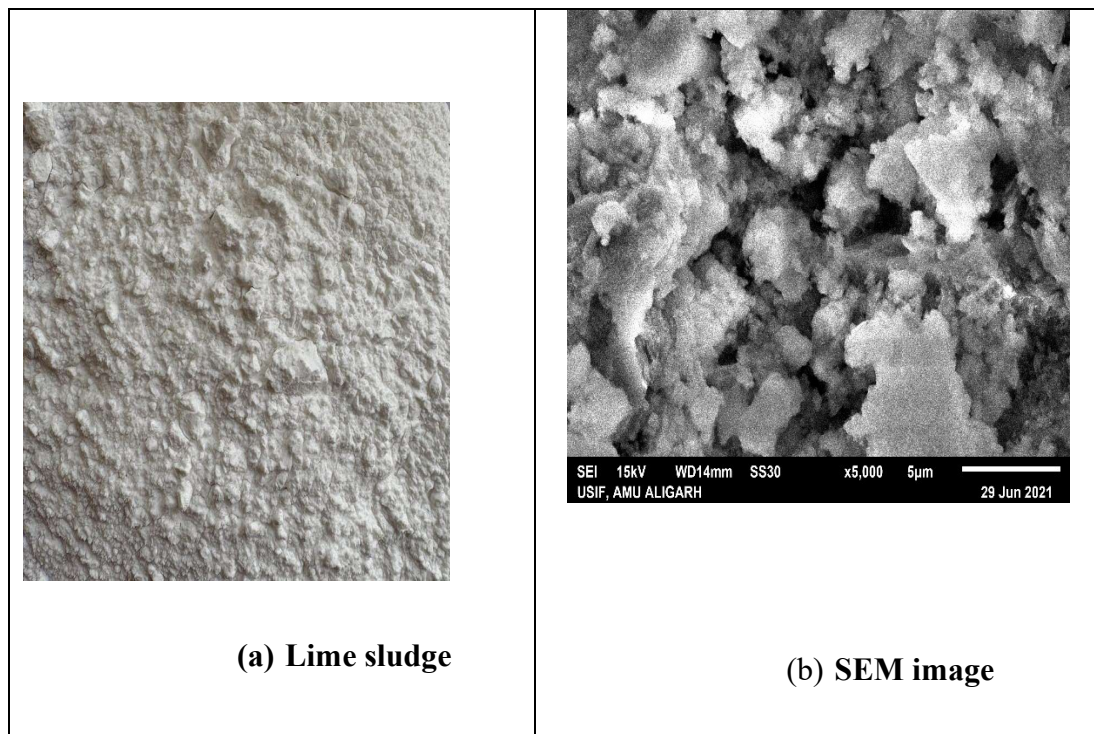
Loss on Ignition (LOI)	1.41
Others	4.6

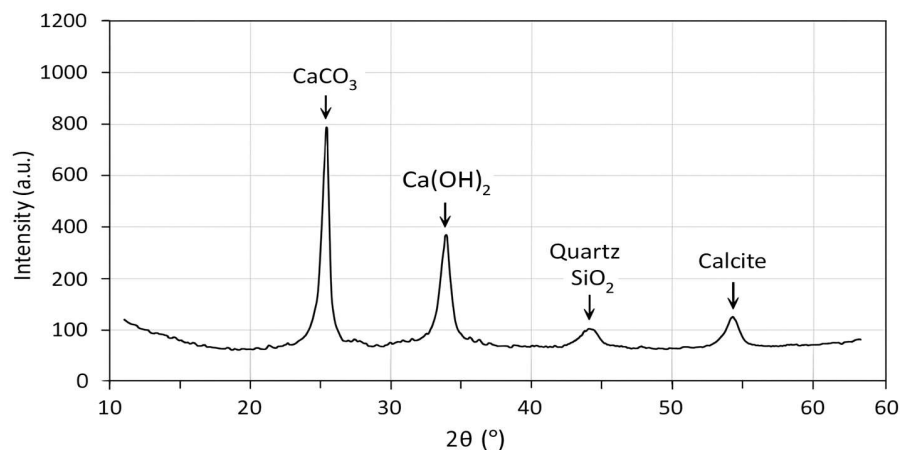
In this Experimental Study, Metakaolin is procured from TARA, Technology and Action for Rural Advancement, Qutub Institutional Area Delhi.

3.2.6 Lime Sludge (LS)

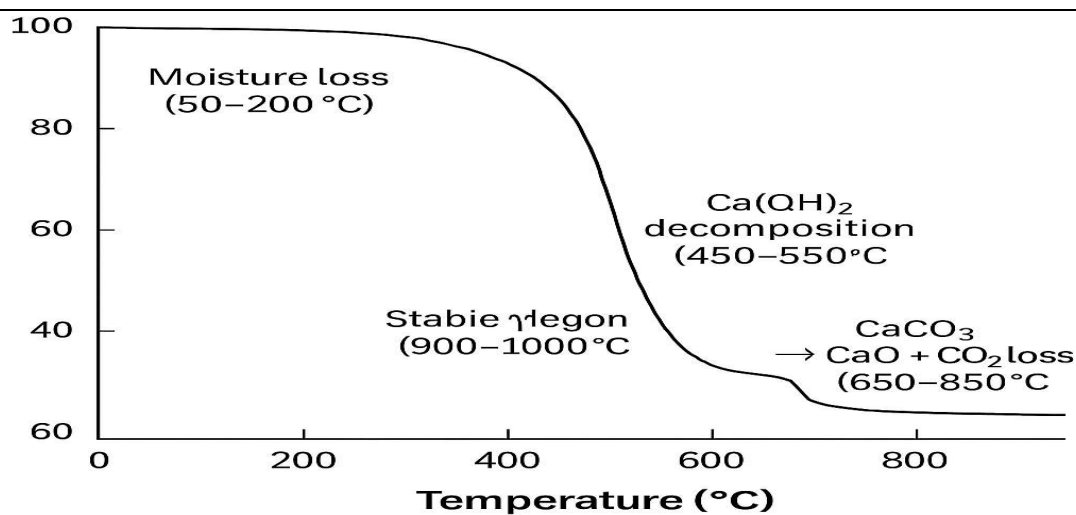
Lime sludge is a soft, powdery by-product of the paper industry. When processed, dried and ground into a fine powder, it can be used as a partial replacement for cement in concrete. The testing procedures are the same as those for cement. Figure 3.8 shows the sample of dried and ground LS, and Tables 3.12 and 3.13 show its physical properties and chemical composition.

For this experimental study, lime sludge was procured from Bilt Paper Industry, Yamunanagar, Haryana, located approximately 200 km from the laboratory.





(c) XRD image



(d) TGA image

Figure 3.8: Lime sludge: (a) sample, (b) SEM image, (c) XRD pattern and (d) TGA curve

Table 3.12: Physical properties of lime sludge

Property	Observed Value	Acceptable limit	IS Code
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Specific Gravity	2.30	2.2 – 2.4	IS 2720 (Part 3)
Moisture Content (%)	18%	10–30%	IS 2720 (Part 2)
Water Absorption (%)	40%	High absorption expected due to fine porous particles	IS 2720 (Part 3)
Bulk Density (kg/m³)	980 kg/m³	800–1100 kg/m ³	IS 2720 (Part 3)
Fineness (Passing 90 µm, %)	85%	≥ 75%	IS 4031 (Part 1)

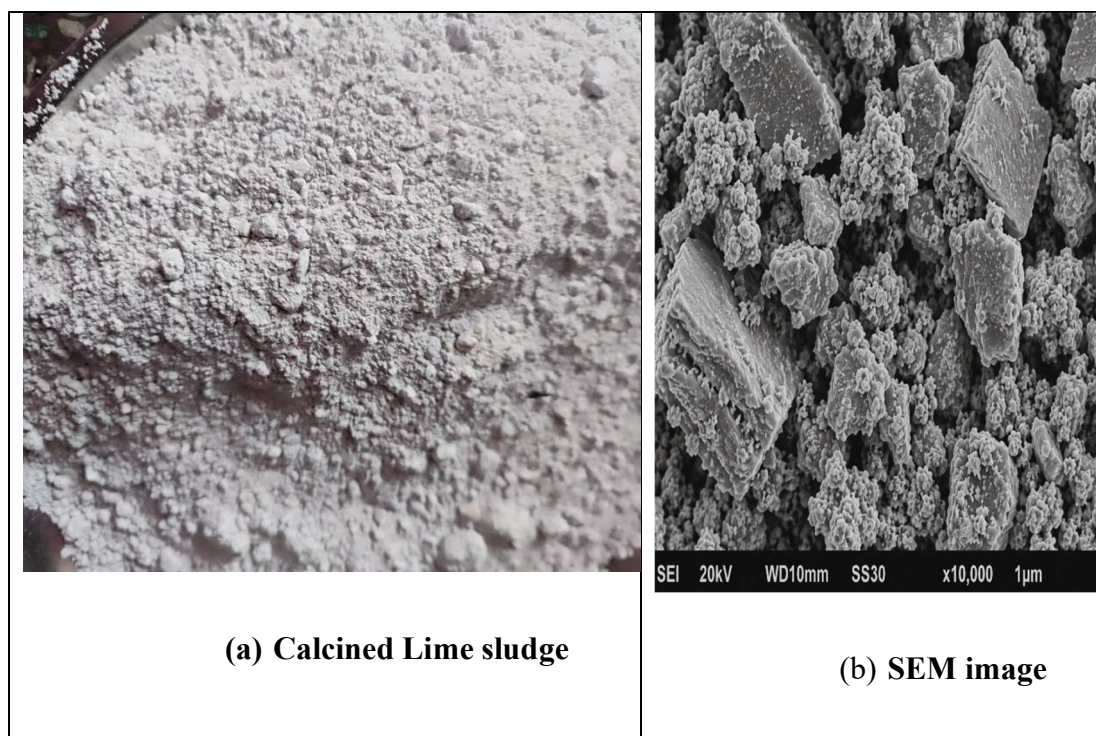
Table 3.13: Chemical composition of lime sludge

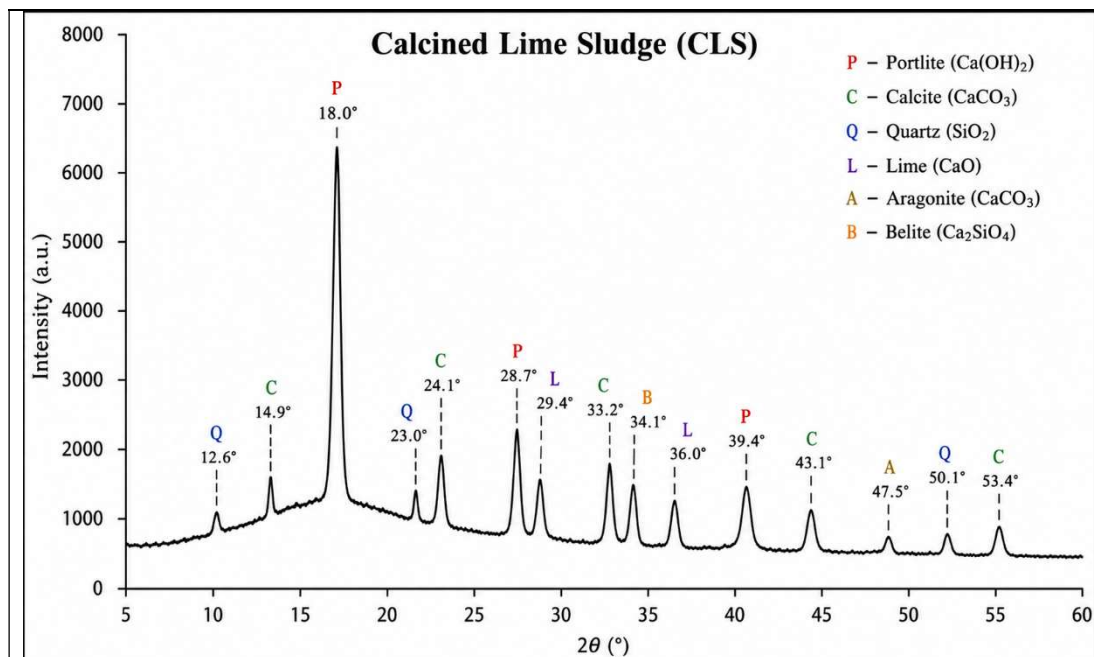
Oxide	Content (%)
SiO ₂	3
CaCO ₃	40
Al ₂ O ₃	2
Fe ₂ O ₃	1
CaO	41
MgO	1
Na ₂ O	0.5
SO ₃	0.5
Loss on Ignition (LOI)	21
Organic matter	5
Others	5

3.2.7 Calcined Lime Sludge (CLS)

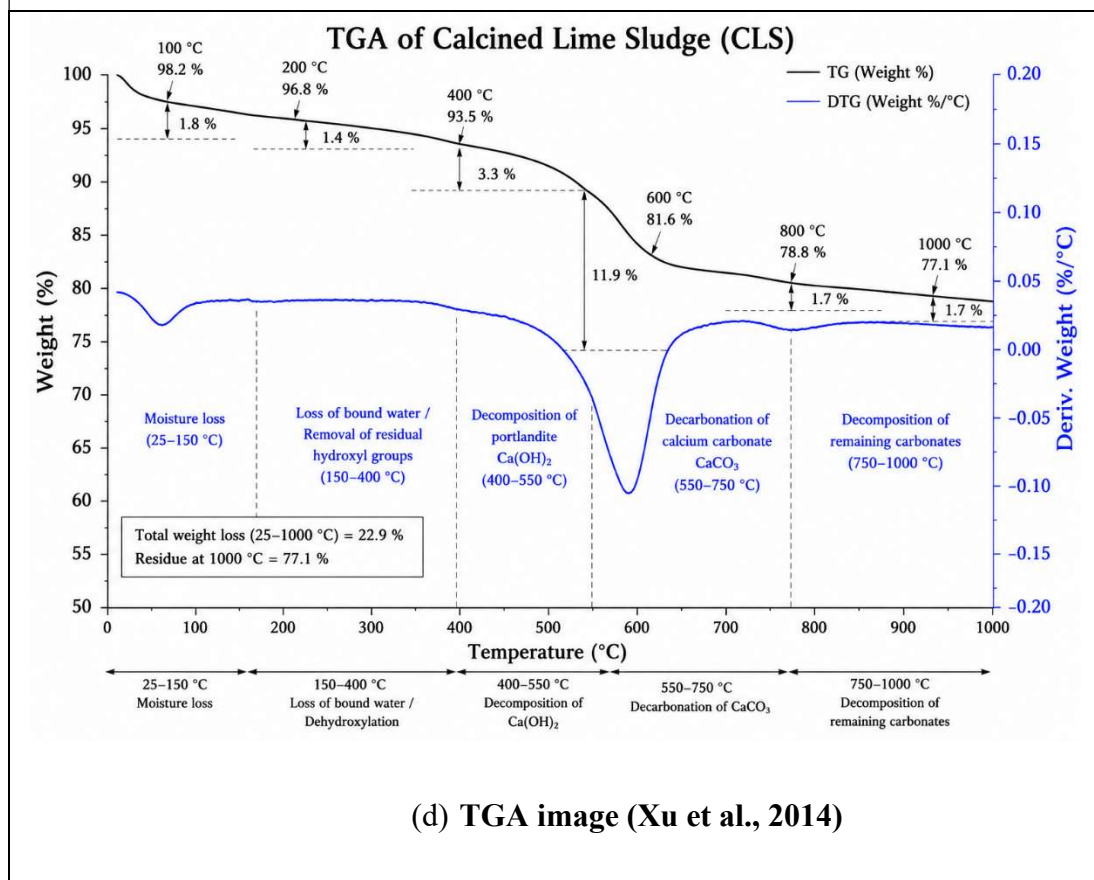
CLS was obtained by calcining the paper-mill lime sludge described in Section 3.2.6 in order to increase its reactivity. Calcination partially decomposes the CaCO₃ in the sludge to CaO, which hydrates to Ca(OH)₂ and can participate in cementitious and

pozzolanic reactions. Trial calcinations of the dried lime sludge were carried out in a muffle furnace at temperatures between 550 °C and 800 °C for 2 h, and the reactivity of the products was assessed by the strength activity index (SAI), the lime reactivity test, XRD and TGA (Chouksey et al., 2024). Calcination at 750 °C for 2 h followed by slow cooling gave the highest pozzolanic reactivity and was therefore adopted for producing the CLS used in the concrete programme (Chouksey et al., 2024). This choice is consistent with the material characterisation: the decarbonation of calcite occurs mainly in the 600–800 °C range (Table 3.28), and calcination at 750 °C reduced the loss on ignition from 21% (LS, Table 3.13) to 10.6% (CLS, Table 3.15) while increasing the CaO content from 41% to 65.6%. The process therefore produced a partially decarbonated material containing reactive CaO, without the over-burning and sintering associated with higher temperatures (S. K. Singh et al., 2020). The calcined lime sludge was ground in a ball mill and then characterised and used in the binder and concrete mixes. Figure 3.9 shows the sample of dried and ground CLS, and Tables 3.14 and 3.15 show its physical properties and chemical composition.





(c) XRD image



(d) TGA image (Xu et al., 2014)

Figure 3.9: Calcined lime sludge: (a) sample, (b) SEM image, (c) XRD pattern and (d) TGA curve

Table 3.14: Physical properties of calcined lime sludge

Property	Observed Value	Acceptable limit	IS Code
Specific Gravity	2.30	2.2 – 2.4	IS 2720 (Part 3)
Moisture Content (%)	14%	10–30%	IS 2720 (Part 2)
Water Absorption (%)	30%	High absorption expected due to fine porous particles	IS 2720 (Part 3)
Bulk Density (kg/m ³)	720 kg/m ³	800–1100 kg/m ³	IS 2720 (Part 3)
Fineness (Passing 90 µm, %)	95%	≥ 75%	IS 4031 (Part 1)

Table 3.15: Chemical composition of calcined lime sludge

Oxide	Content (%)
SiO ₂	9.5
Al ₂ O ₃	3.7
Fe ₂ O ₃	1.5
CaO	65.6
MgO	3.3
SO ₃	0.7
Na ₂ O	0.4
K ₂ O	0.5
TiO ₂	0.2
LOI	10.6
Others	4.0

3.2.8 Superplasticiser

A polycarboxylate ether (PCE) based superplasticiser, CemWet SP-3000, was used as the high-range water-reducing admixture. Its specific gravity, as declared by the manufacturer, lies in the range 1.08–1.25.

3.3 Mix Design for LS–MK Mixes (M25 Grade)

3.3.1 Mix Design without Superplasticiser

IS Standards Followed

1. IS 456:2000 – General Concrete Design
2. IS 10262:2019 – Mix Proportioning
3. IS 383:2016 – Aggregates
4. Table 3.16 shows the parameters adopted for the M25 mix design.

Table 3.16: Parameters for the M25 mix design

Parameter	Value
Grade of Concrete	M25
Target Mean Strength ($f_{ck} + 1.65 \times S$)	$25 + 1.65 \times 4 = 31.6$ MPa
Cement Type	OPC 43 Grade
Maximum Nominal Aggregate Size	20 mm
Workability	75–100 mm slump (no SP)
Exposure Condition	Severe
Method of Batching	Weight

Water-Cement Ratio (w/c)

$$w/c = 0.45$$

$$\text{Adopted W/C} = 0.45$$

For 75–100 mm slump and 20 mm aggregate

Estimated water = 186 kg/m³ (IS 10262 Table 4)

Adopted water = 180 kg/m³

Cement Content

$$\text{Water} = 180 \text{ kg/m}^3$$

$$\text{W/C} = 0.45$$

$$\text{Cement} = 180/0.45 = 400 \text{ kg/m}^3$$

As per IS 456:2000 maximum cement is 450 kg/m^3 therefore,

$$\text{Cement} = 400 \text{ kg/m}^3$$

$$\text{Therefore, water} = 400 \times 0.45 = 180 \text{ kg/m}^3 \text{ (without superplasticiser)}$$

Volume Calculations

Assume 2% air voids

Therefore,

$$\text{Total volume available for the constituents} = 1 - 0.02 = 0.98 \text{ m}^3$$

Without superplasticiser

$$\text{Volume of Cement} = \frac{400}{3.13 \times 1000} = 0.1278 \text{ m}^3$$

$$\text{Volume of Water} = \frac{180}{1000} = 0.18 \text{ m}^3$$

$$\text{Volume of aggregates} = 0.98 - 0.1278 - 0.18 = 0.6722 \text{ m}^3$$

based on 20 mm aggregate

$$\text{Coarse aggregate : fine aggregate (by absolute volume)} = 0.605 : 0.395$$

$$\text{Volume of coarse aggregates} = 0.605 \times 0.6722 = 0.4067 \text{ m}^3$$

$$\text{Therefore, coarse aggregate content} = 0.4067 \times 2.70 \times 1000 = 1098 \approx 1100 \text{ kg/m}^3$$

$$\text{Volume of fine aggregates} = 0.395 \times 0.6722 = 0.2655 \text{ m}^3$$

$$\text{Therefore, fine aggregate content} = 0.2655 \times 2.63 \times 1000 = 698 \approx 700 \text{ kg/m}^3$$

The coarse aggregate fraction of 0.605 of the total aggregate volume corresponds to the batched quantities of 1100 kg/m^3 of coarse and 700 kg/m^3 of fine aggregate. It lies within the range obtained from Table 5 of IS 10262:2019 (0.62 for 20 mm aggregate and Zone II sand at a w/c ratio of 0.50), after adjustment for the w/c ratio and the required workability.

3.3.2 Mix Design with Superplasticiser

With the superplasticiser, the water content was reduced by 18% while the w/c ratio of 0.45 was retained, as permitted in IS 10262:2019. The cement content was then recalculated from the reduced water content and checked against the minimum cement content of 320 kg/m^3 specified in IS 456:2000 for severe exposure.

$$\text{Therefore, water} = 180 - (180 \times 0.18) = 147.6 \text{ kg/m}^3$$

$$\text{Cement} = 147.6/0.45 = 328 \text{ kg/m}^3$$

$$\text{Volume of Cement} = \frac{328}{3.13 \times 1000} = 0.1048 \text{ m}^3$$

$$\text{Volume of Water} = \frac{147.6}{1000} = 0.148 \text{ m}^3$$

$$\text{Volume of aggregates} = 0.98 - 0.1048 - 0.1476 = 0.7276 \text{ m}^3$$

based on 20 mm aggregate

$$\text{coarse aggregates: fine aggregates} = 0.65: 0.35$$

$$\text{Volume of coarse aggregates} = 0.65 \times 0.7276 = 0.4729 \text{ m}^3$$

$$\text{Therefore, coarse aggregate content} = 0.4729 \times 2.70 \times 1000 = 1277 \text{ kg/m}^3$$

$$\text{Volume of fine aggregates} = 0.35 \times 0.7276 = 0.2547 \text{ m}^3$$

$$\text{Therefore, fine aggregate content} = 0.2547 \times 2.63 \times 1000 = 670 \text{ kg/m}^3$$

Note on the batched quantities: the quantities actually batched for the superplasticised M25 mixes (1241 kg/m³ of coarse and 651 kg/m³ of fine aggregate; Tables 3.17 and 3.19) were calculated with an aggregate volume of 0.7072 m³ owing to an arithmetic slip in the subtraction above. They are about 2.8% lower than the corrected values, so that the batched constituents occupy about 0.98 m³ instead of 1.00 m³, i.e., the actual binder content per cubic metre was about 2% higher than the nominal value. Because all the specimens were cast with the batched quantities, these values are retained in the mix proportion tables and in all subsequent analyses.

Tables 3.17, 3.18 and 3.19 show the mix proportions of the M25 control concrete and of the LS–MK mixes without and with superplasticiser, respectively.

Table 3.17: Mix proportions of the M25 control concrete

Without superplasticiser			
Material	Cement	Fine aggregates	Coarse aggregates
Quantity (kg/m ³)	400	700	1100
Proportion	1	1.75	2.75
With superplasticiser			
Quantity (kg/m ³)	328	651	1241
Proportion	1	1.98	3.78

Table 3.18: Mix proportions of LS–MK mixes without superplasticiser (kg/m³)

Mixes	OPC	MK	LS	Water	Fine Aggregate	Coarse Aggregate	w/b ratio
C100M0L0	400	0	0	180	700	1100	0.45
C95M0L5	380	0	20	180	700	1100	0.45
C90M0L10	360	0	40	180	700	1100	0.45
C85M0L15	340	0	60	180	700	1100	0.45
C95M5L0	380	20	0	180	700	1100	0.45
C90M5L5	360	20	20	180	700	1100	0.45
C85M5L10	340	20	40	180	700	1100	0.45
C80M5L15	320	20	60	180	700	1100	0.45
C90M10L0	360	40	0	180	700	1100	0.45
C85M10L5	340	40	20	180	700	1100	0.45
C80M10L10	320	40	40	180	700	1100	0.45
C75M10L15	300	40	60	180	700	1100	0.45
C85M15L0	340	60	0	180	700	1100	0.45
C80M15L5	320	60	20	180	700	1100	0.45
C75M15L10	300	60	40	180	700	1100	0.45
C70M15L15	280	60	60	180	700	1100	0.45

Table 3.19: Mix proportions of LS–MK mixes with superplasticiser (kg/m³)

Mixes	OPC	MK	LS	Water	Fine Aggregate	Coarse Aggregate	w/b ratio	SP* (%)
C100M0L0	328	0	0	147.6	651	1241	0.45	0.6
C95M0L5	311.6	0	16.4	147.6	651	1241	0.45	0.6
C90M0L10	295.2	0	32.8	147.6	651	1241	0.45	0.6
C85M0L15	278.8	0	49.2	147.6	651	1241	0.45	0.6
C95M5L0	311.6	16.4	0	147.6	651	1241	0.45	0.6

C90M5L5	295.2	16.4	16.4	147.6	651	1241	0.45	0.6
C85M5L10	278.8	16.4	32.8	147.6	651	1241	0.45	0.6
C80M5L15	262.4	16.4	49.2	147.6	651	1241	0.45	0.6
C90M10L0	295.2	32.8	0	147.6	651	1241	0.45	0.6
C85M10L5	278.8	32.8	16.4	147.6	651	1241	0.45	0.6
C80M10L10	262.4	32.8	32.8	147.6	651	1241	0.45	0.6
C75M10L15	246	32.8	49.2	147.6	651	1241	0.45	0.6
C85M15L0	278.8	49.2	0	147.6	651	1241	0.45	0.6
C80M15L5	262.4	49.2	16.4	147.6	651	1241	0.45	0.6
C75M15L10	246	49.2	32.8	147.6	651	1241	0.45	0.6
C70M15L15	229.6	49.2	49.2	147.6	651	1241	0.45	0.6

3.4 Mix Design for CLS–MK Mixes (M40 Grade)

3.4.1 Mix Design without Superplasticiser

IS Standards Followed

5. IS 456:2000 – General Concrete Design
6. IS 10262:2019 – Mix Proportioning
7. IS 383:2016 – Aggregates
8. Table 3.20 shows the parameters adopted for the M40 mix design.

Table 3.20: Parameters for the M40 mix design

Parameter	Value
Grade of Concrete	M40
Target Mean Strength ($f_{ck} + 1.65 \times S$)	$40 + 1.65 \times 5 = 48.25$ MPa
Cement Type	OPC 43 Grade
Maximum Nominal Aggregate Size	20 mm
Workability	75–100 mm slump (no SP)
Exposure Condition	Severe
Method of Batching	Weight

Water-Cement Ratio (w/c)

$$w/c = 0.40$$

Adopted W/C = 0.40

For 75–100 mm slump and 20 mm aggregate

Estimated water = 186 kg/m³ (IS 10262 Table 4)

Adopted water = 180 kg/m³

Cement Content

Water = 180 kg/m³

W/C = 0.40

Cement = $180/0.40 = 450 \text{ kg/m}^3$

As per IS 456:2000 maximum cement is 450 kg/m³ therefore,

Cement = 450 kg/m³

Therefore, water = $450 \times 0.40 = 180 \text{ kg/m}^3$ (without superplasticiser)

Volume Calculations

Assume 2% air voids

Therefore,

Total volume available for the constituents = $1 - 0.02 = 0.98 \text{ m}^3$

Without superplasticiser

Volume of Cement = $\frac{450}{3.13 \times 1000} = 0.144 \text{ m}^3$

Volume of Water = $\frac{180}{1000} = 0.18 \text{ m}^3$

Volume of aggregates = $0.98 - 0.144 - 0.18 = 0.656 \text{ m}^3$

based on 20 mm aggregate

coarse aggregates: fine aggregates = 0.65: 0.35

Volume of coarse aggregates = $0.65 \times 0.656 = 0.426 \text{ m}^3$

Therefore, coarse aggregate content = $0.426 \times 2.70 \times 1000 = 1150 \text{ kg/m}^3$

Volume of fine aggregates = $0.35 \times 0.656 = 0.2296 \text{ m}^3$

Therefore, fine aggregate content = $0.2296 \times 2.63 \times 1000 = 604 \text{ kg/m}^3$

3.4.2 Mix Design with Superplasticiser

With the superplasticiser, the water content was reduced by 20% while the w/c ratio of 0.40 was retained (IS 10262:2019); the recalculated cement content of 360 kg/m³ satisfies the minimum of 320 kg/m³ specified in IS 456:2000 for severe exposure.

Therefore, water = $180 - (180 \times 0.20) = 144 \text{ kg/m}^3$

Cement = $144/0.4 = 360 \text{ kg/m}^3$

Volume of Cement = $\frac{360}{3.13 \times 1000} = 0.1150 \text{ m}^3$

Volume of Water = $\frac{144}{1000} = 0.144 \text{ m}^3$

Volume of aggregates = $0.98 - 0.1150 - 0.144 = 0.721 \text{ m}^3$

based on 20 mm aggregate

coarse aggregates: fine aggregates = 0.65: 0.35

Volume of coarse aggregates = $0.65 \times 0.721 = 0.46865 \text{ m}^3$

Therefore, coarse aggregate content = $0.46865 \times 2.70 \times 1000 = 1265 \text{ kg/m}^3$

Volume of fine aggregates = $0.35 \times 0.721 = 0.25235 \text{ m}^3$

Therefore, fine aggregate content = $0.25235 \times 2.63 \times 1000 = 664 \text{ kg/m}^3$

Tables 3.21, 3.22 and 3.23 show the mix proportions of the M40 control concrete and of the CLS–MK mixes without and with superplasticiser, respectively.

Table 3.21: Mix proportions of the M40 control concrete

Without superplasticiser			
Material	Cement	Fine aggregates	Coarse aggregates
Quantity (kg/m ³)	450	604	1150
Proportion	1	1.34	2.56
With superplasticiser			
Quantity (kg/m ³)	360	664	1265
Proportion	1	1.84	3.54

Table 3.22: Mix proportions of CLS–MK mixes without superplasticiser (kg/m³)

Mixes	OPC	MK	CLS	Water	Fine	Coarse	w/b ratio
					Aggregate	Aggregate	
M0CL0	450	0	0	180	604	1150	0.4
M0CL5	427.5	0	22.5	180	604	1150	0.4
M0CL10	405	0	45	180	604	1150	0.4
M0CL15	382.5	0	67.5	180	604	1150	0.4

M0CL20	360	0	90	180	604	1150	0.4
M5CL0	427.5	22.5	0	180	604	1150	0.4
M10CL0	405	45	0	180	604	1150	0.4
M15CL0	382.5	67.5	0	180	604	1150	0.4
M20CL0	360	90	0	180	604	1150	0.4
M5CL5	405	22.5	22.5	180	604	1150	0.4
M5CL10	382.5	22.5	45	180	604	1150	0.4
M5CL15	360	22.5	67.5	180	604	1150	0.4
M5CL20	337.5	22.5	90	180	604	1150	0.4
M10CL5	382.5	45	22.5	180	604	1150	0.4
M10CL10	360	45	45	180	604	1150	0.4
M10CL15	337.5	45	67.5	180	604	1150	0.4
M10CL20	315	45	90	180	604	1150	0.4
M15CL5	360	67.5	22.5	180	604	1150	0.4
M15CL10	337.5	67.5	45	180	604	1150	0.4
M15CL15	315	67.5	67.5	180	604	1150	0.4
M15CL20	292.5	67.5	90	180	604	1150	0.4
M20CL5	337.5	90	22.5	180	604	1150	0.4
M20CL10	315	90	45	180	604	1150	0.4
M20CL15	292.5	90	67.5	180	604	1150	0.4
M20CL20	270	90	90	180	604	1150	0.4

Table 3.23: Mix proportions of CLS–MK mixes with superplasticiser (kg/m³)

Mixes	OPC	MK	CLS	Water	Fine Aggregate	Coarse Aggregate	w/b ratio	SP* (%)
M0CL0	360	0	0	144	664	1265	0.4	1
M0CL5	342	0	18	144	664	1265	0.4	1
M0CL10	324	0	36	144	664	1265	0.4	1

M0CL15	306	0	54	144	664	1265	0.4	1
M0CL20	288	0	72	144	664	1265	0.4	1
M5CL0	342	18	0	144	664	1265	0.4	1
M10CL0	324	36	0	144	664	1265	0.4	1
M15CL0	306	54	0	144	664	1265	0.4	1
M20CL0	288	72	0	144	664	1265	0.4	1
M5CL5	324	18	18	144	664	1265	0.4	1
M5CL10	306	18	36	144	664	1265	0.4	1
M5CL15	288	18	54	144	664	1265	0.4	1
M5CL20	270	18	72	144	664	1265	0.4	1
M10CL5	306	36	18	144	664	1265	0.4	1
M10CL10	288	36	36	144	664	1265	0.4	1
M10CL15	270	36	54	144	664	1265	0.4	1
M10CL20	252	36	72	144	664	1265	0.4	1
M15CL5	288	54	18	144	664	1265	0.4	1
M15CL10	270	54	36	144	664	1265	0.4	1
M15CL15	252	54	54	144	664	1265	0.4	1
M15CL20	234	54	72	144	664	1265	0.4	1
M20CL5	270	72	18	144	664	1265	0.4	1
M20CL10	252	72	36	144	664	1265	0.4	1
M20CL15	234	72	54	144	664	1265	0.4	1
M20CL20	216	72	72	144	664	1265	0.4	1

3.4.3 Mix Nomenclature

A uniform nomenclature is used for the mixes throughout this thesis. In the M25 LS–MK series (Phase I), a mix is designated CxMyLz, where x, y and z are the percentages of OPC, MK and LS in the binder by mass; for example, C80M10L10 contains 80% OPC, 10% MK and 10% LS, and C100M0L0 is the M25 control mix. In the M40

CLS–MK series (Phase II), a mix is designated MyCLz, where y and z are the percentages of MK and CLS and the remainder is OPC; for example, M15CL10 contains 15% MK, 10% CLS and 75% OPC, and M0CL0 is the M40 control mix. Where mixes of the two series are discussed together (Sections 4.17, 4.24 and 4.25), the prefixes M25- and M40- are added to indicate the grade, e.g., M25-C80M10L10 and M40-M15CL10.

3.5 Testing Procedures

3.5.1 Workability

To determine the consistency and ease of placement of freshly mixed concrete, the slump test is conducted as per IS 1199:1959. The apparatus comprises a standard frustum-shaped metal cone having a base diameter of 200 mm, a top opening of 100 mm, and a vertical height of 300 mm. The cone is placed on a non-absorbent surface and filled with fresh concrete in three equal layers. Each layer is uniformly tamped using a standard steel rod with a hemispherical tip, delivering 25 strokes per layer. After filling and levelling, the cone is carefully lifted vertically, allowing the concrete to slump under its own weight. The vertical drop, measured from the top of the cone to the highest point of the subsided concrete, is recorded in millimetres as the slump value. In this study, the slump test was extended to assess slump retention over time by recording measurements at 0, 20, 40, 60, and 80 minutes. This procedure aids in evaluating the concrete's stability and delayed workability in field applications where a delay in placement is expected.

3.5.2 Compressive Strength

The compressive strength test is fundamental in assessing the load-bearing capacity of concrete. In line with IS 516:1959, the test utilises cube specimens with dimensions of 150 × 150 × 150 mm. These specimens are cast and cured under standard water curing conditions and tested at 7, 28, and 90 days. At the designated age, the cube is placed centrally in a calibrated compression testing machine, with load applied axially at a controlled rate of 140 kg/cm² per minute until failure occurs. The maximum load sustained is recorded, and compressive strength is calculated by dividing this load by the cross-sectional area of the cube. This test confirms whether the mix meets the

required characteristic strength (25 MPa for the M25 series and 40 MPa for the M40 series) and helps to monitor the strength gain over the curing period.

3.5.3 Split Tensile Strength

Due to concrete's weak tensile behaviour, the split tensile test is used to indirectly measure its tensile resistance. As per IS 5816:1999, cylindrical specimens measuring 150 mm in diameter and 300 mm in length are prepared and cured under similar conditions to the cubes. For testing, the cylinder is laid horizontally in a compression testing machine. The load is applied vertically along its length, causing the cylinder to fail by splitting across the vertical diameter. The peak load at failure is recorded, and the tensile strength is determined using the standard formula that factors in the specimen's length and diameter. This test provides insight into the concrete's resistance to cracking under tensile stresses, crucial for structural members subjected to bending or axial tension.

3.5.4 Flexural Strength

Flexural strength is a measure of the ability of concrete to resist bending. The procedure adheres to IS 516:1959 and uses beam specimens of $100 \times 100 \times 500$ mm. After 7, 28 and 90 days of curing, the beams were subjected to two-point loading over a clear span of 400 mm. The specimen is placed on steel rollers, and the load is applied symmetrically at the third points of the span using a calibrated machine. As the load increases, the specimen experiences tensile stress at the bottom and compressive stress at the top. The load at which the beam fails is noted, and the flexural strength is computed using the modulus of rupture formula based on the span, width and depth of the specimen. Figure 3.12 shows the mixing, casting, curing and testing of the specimens prepared for this study, and Table 3.24 summarises the fresh, mechanical and durability tests carried out in this experimental programme.

3.5.5 Acid Attack Resistance Test

The acid attack resistance of the mixes was determined by immersing the specimens in a 5% HCl solution for 12 weeks in accordance with ASTM C267 (ASTM C267, 2020). Cylindrical specimens of 50 mm diameter and 100 mm height were cast and

cured for 28 days. The specimens were then surface-dried, and their dimensions and initial mass were recorded. They were transferred to the 5% HCl solution, which was covered with paraffin wax to avoid the loss of HCl to the atmosphere. Every week, the specimens were taken out of the solution, washed under tap water, surface-dried under a fan for at least half an hour and weighed to calculate the mass loss, and then immersed again in fresh 5% HCl solution; the solution was replaced every week to maintain a constant acidity. This procedure was repeated for 12 weeks. A higher mass loss indicates a lower acid resistance, and vice versa.

3.5.6 Rapid Chloride Ion Penetration Test

The chloride ion penetration of the mixes was determined in accordance with ASTM C1202. Cylinders of 100 mm diameter $\times$ 200 mm height were cast and cured for 28 and 56 days, after which they were cut into 50 mm thick discs using a diamond cutter. Each disc was vacuum-saturated to ensure uniform moisture conditions before testing. The saturated specimens were placed between two acrylic cells, one filled with 3% sodium chloride solution and the other with 0.3 N sodium hydroxide solution. A constant direct current potential of 60 ± 0.1 V was applied across the specimen for six hours, and the current passing through the specimen was recorded at 30-minute intervals. The total charge passed, expressed in coulombs, was calculated by integrating the current over the six-hour test period, and the chloride ion penetrability was classified on this basis. Lower charge values indicate higher resistance to chloride ingress, and vice versa (ASTM C1202, 2012).

Table 3.24: Summary of fresh and mechanical tests

Test Type	IS Code	Specimen Dimensions	Test Age	Units
Slump Test	IS 1199:1959	Slump cone (100/200 mm)	Fresh	mm
Compressive Strength	IS 516:1959	Cube – 150 $\times$ 150 $\times$ 150 mm	7, 28, 90	MPa
Split Tensile Strength	IS 5816:1999	Cylinder – 150 $\times$ 300 mm	7, 28, 90	MPa
Flexural Strength	IS 516:1959	Beam – 100 $\times$ 100 $\times$ 500 mm	7, 28, 90	MPa

Acid attack resistance test	ASTM C267	Cylinder – 50 × 100 mm	12 weeks (28 days cured specimens)	%
Rapid chloride ion penetration test	ASTM C1202	Disc – 100 mm dia. × 50 mm	6 h test (28- and 56-day cured specimens)	Coulomb

3.5.7 Specimen Replication and Treatment of Experimental Variability

The number of specimens tested for each property is summarised in Table 3.25. For each mix, the specimens for a given test series were cast from a single batch prepared under identical conditions, and replicate specimens were tested at each curing age. In accordance with IS 456:2000 (Clause 15.4) and IS 516, the strength of a mix at a given age is reported as the average of three specimens, and a set of results was accepted only when the individual values did not deviate by more than $\pm 15\%$ from the mean.

Table 3.25: Specimen replication scheme

Test	Specimen	Specimens per mix per age	Test age / duration
Slump	Fresh concrete (slump cone)	1 test per time interval	0, 20, 40, 60 and 80 min
Compressive strength (and density)	Cube, 150 mm	3	7, 28 and 90 days
Split tensile strength	Cylinder, 150 × 300 mm	3	7, 28 and 90 days
Flexural strength	Beam, 100 × 100 × 500 mm	3	7, 28 and 90 days
Water absorption	As per ASTM C642	3	28 days
Acid attack (5% HCl)	Cylinder, 50 × 100 mm	3	12 weeks after 28-day curing
RCPT	Disc, 100 mm dia. × 50 mm	3	28 and 56 days

3.5.8 Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) is a thermo-analytical technique used to measure the heat absorbed or released by a material as a function of temperature or time under a controlled heating or cooling programme. In cementitious materials, DSC and related calorimetric techniques are widely employed to investigate thermal behaviour, hydration products, dehydration reactions, phase transformations and the pozzolanic activity of cement, supplementary cementitious materials (SCMs) and hardened concrete. In this study, the heat flow and cumulative heat of selected mixes were recorded to evaluate the hydration kinetics (Section 4.17).

3.5.9 X-ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) is one of the most widely used analytical techniques for identifying the crystalline phases present in cementitious materials. It provides information on the mineralogical composition of hydrated cement paste, supplementary cementitious materials (SCMs) and the reaction products formed during hydration, and is useful for studying phase evolution and pozzolanic reactions in concrete containing industrial by-products such as calcined lime sludge and metakaolin. XRD is a non-destructive technique that identifies crystalline materials by measuring how X-rays are diffracted by the atomic planes of a crystal (Stutzman et al., 2016).

Principle

XRD is based on Bragg's Law:

$$n\lambda = 2d\sin \theta$$

where:

n = diffraction order (usually 1)

λ = X-ray wavelength

d = spacing between crystal planes

θ = diffraction angle

Samples taken from 28-day cured concrete specimens were ground to a powder passing a 75 μm sieve, and hydration was stopped using 99% pure isopropyl alcohol. The XRD patterns were recorded on a Malvern Panalytical diffractometer with a Cu radiation source over a 2θ range of up to 80° .

The peaks were identified using the PANalytical X'Pert HighScore software, and the Crystallography Open Database (COD) was used for peak matching and phase identification. Probable phases were assigned with the help of Table 3.26. It should be noted that C–S–H and C–A–S–H gels are poorly crystalline and do not produce distinct XRD reflections; their presence is therefore inferred indirectly (e.g., from the consumption of portlandite) rather than identified directly by XRD.

Table 3.26: Interpretation of XRD peaks

Approx. 2θ	Probable phase	Interpretation
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9–12°	Ettringite and/or gypsum	Possible sulfate-bearing hydration products
18.0°	Portlandite, $\text{Ca}(\text{OH})_2$	Cement hydration product
20.8–21.0°	Quartz	Secondary quartz reflection
26.6–26.8°	Quartz	Dominant crystalline peak
29.4–29.5°	Calcite, CaCO_3	Carbonation product or limestone-containing material
34.1°	Portlandite	Supporting reflection for $\text{Ca}(\text{OH})_2$
36.6°, 39.5°, 42.5°	Mainly quartz	Siliceous aggregate contribution
47.1°	Portlandite/calcite overlap	Requires reference-pattern fitting
50.2°, 55°, 60°, 68.3°	Mainly quartz	Higher-angle quartz reflections

3.5.10 Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) is a thermal characterization technique that measures the change in the mass of a material as it is heated under a controlled atmosphere (typically nitrogen, argon, or air). It is widely used to evaluate the **thermal stability, decomposition behavior, hydration products, and composition** of cementitious materials and concrete.

TGA was performed on samples from 28-day cured specimens, ground and sieved through a 75 μm sieve, with hydration stopped using 99% pure isopropyl alcohol. A sample of 10–30 mg was heated at a constant rate of 10 $^{\circ}\text{C}/\text{min}$ from room temperature to 1000 $^{\circ}\text{C}$ in a nitrogen atmosphere. Table 3.27 lists the mass-loss events and the associated phases in the different temperature ranges.

Table 3.27: Phase interpretation of TGA mass loss in different temperature ranges

Temperature ($^{\circ}\text{C}$)	Weight-loss event	Main phase
25–105	Evaporation of free moisture	Physically adsorbed water
105–200	Loss of bound water	C-S-H gel, Ettringite
200–350	Decomposition of AFm phases	Monosulfate, Hydrotalcite
400–500	Dehydroxylation	Portlandite ($\text{Ca}(\text{OH})_2$)

600–800	Decarbonation	Calcite (CaCO ₃)
>800	Stable residue	Silica, alumina, aggregates

3.5.11 Life Cycle Assessment (LCA)

A life cycle assessment (LCA) of concrete is a standardised methodology used to quantify the environmental impacts associated with concrete throughout its life cycle, from raw material extraction to end-of-life disposal or recycling. LCA is performed according to ISO 14040 and ISO 14044 and is widely used to evaluate the sustainability of conventional and low-carbon concrete (Monahan & Powell, 2011; Ulucan, 2024).

The primary goal of this LCA of concrete is to quantify the environmental impacts associated with the production of control and Blended concrete.

The following parameters were adopted for the life cycle assessment of the blended concrete:

1. System boundary: cradle to gate, up to the production of concrete (modules A1–A3; Figure 3.10)
2. Functional unit: 1 m³ of concrete
3. Impact assessment method: ReCiPe 2016 (commonly used for cementitious materials)
4. Database: ecoinvent 3.12

The major impact categories and their units are listed in Table 3.28.

Table 3.28: Impact categories considered in the life cycle assessment

Impact category	Unit
Global Warming Potential (GWP)	kg CO ₂ -eq
Acidification Potential (AP)	kg SO ₂ -eq
Eutrophication Potential (EP)	kg PO ₄ ³⁻ -eq
Ozone Depletion Potential (ODP)	kg CFC-11-eq
Water Consumption	m ³

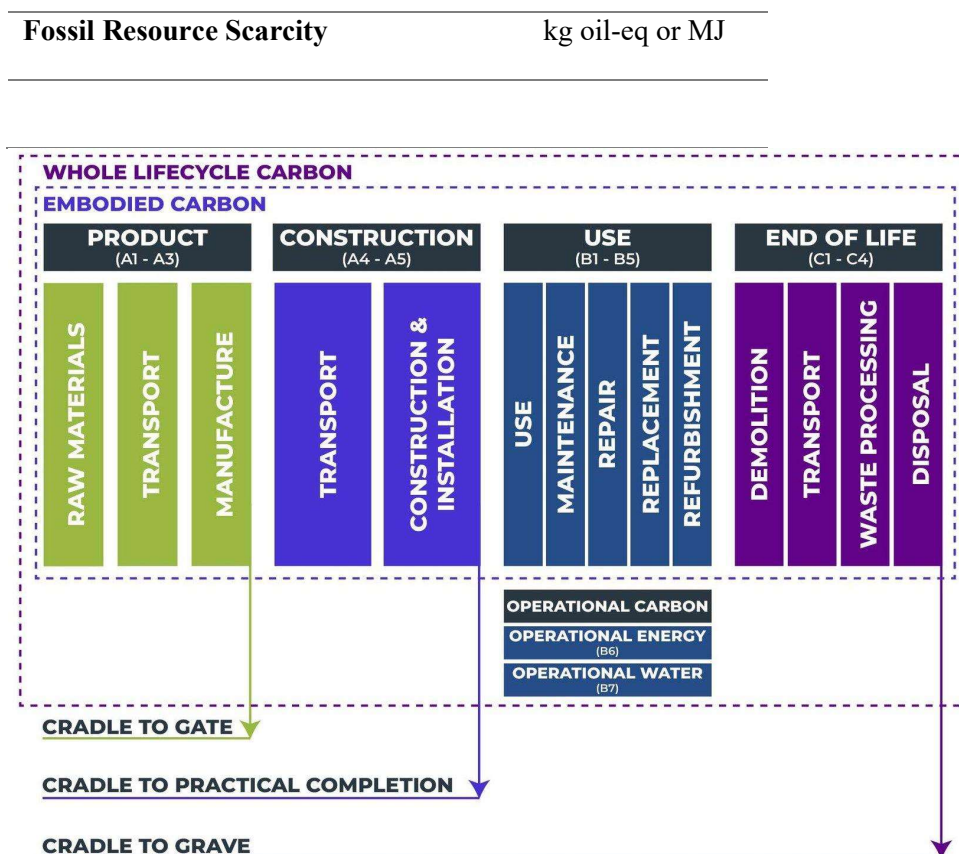


Figure 3.10: System boundaries for life cycle assessment

3.5.12 Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

Scanning electron microscopy (SEM) was used to examine the microstructure of the concrete specimens. SEM produces highly magnified images using a focused beam of accelerated electrons and reveals details such as the pore structure, microcracks, the morphology of hydration products and the interface between the cement paste and the aggregates. In this study, the SEM images were examined qualitatively; no quantitative image analysis of porosity or crack width was performed. SEM/EDS evaluation was conducted on cured specimens, at the curing ages stated in Sections 4.20 and 4.21. The concrete specimens were trimmed into 10 mm × 10 mm × 10 mm blocks, dried in an oven at 90 °C for 24 hours, cleaned with silicon carbide paper and kept in a desiccator to prevent carbonation. Before testing, the samples were sputter-coated with carbon. Figure 3.11 shows the SEM and EDS set-up at Kurukshetra University.

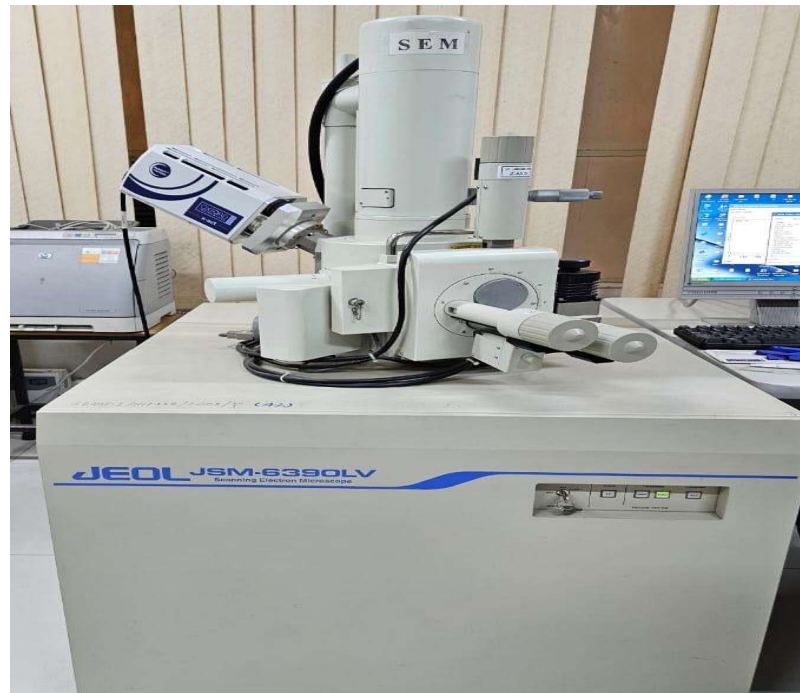


Figure 3.11: SEM and EDS set-up

3.5.13 Data Analysis and Optimisation

Regression analysis was performed using the SPSS software (2016 version) to examine the relationships between the physical properties (density and water absorption) and the mechanical properties of the LS–MK concrete. The complete 4×4 factorial matrix of the MK–LS mixes is equivalent to a Taguchi L16 (4^2) orthogonal array; the same 16 mixes were therefore analysed using the Taguchi method without additional experiments. Because each mix was tested as a single run in this analysis ($n = 1$), the signal-to-noise (S/N) ratio is a logarithmic transformation of the mean response and does not represent within-run noise; the Taguchi analysis is therefore used only to rank the main effects of MK and LS, with the $MK \times LS$ interaction pooled into the error term (Section 4.22.2). For the CLS–MK series, response surface methodology (RSM) with a full second-order (quadratic) model in MK content, CLS content and curing age was used to model and optimise the responses (Section 4.23).



(a) Mixing



(b) Casting



(C) Demoulding



(d) Curing

23

8



(e) Compressive strength testing



(f) Split tensile strength testing



(g) Flexural strength testing



(h) Water absorption

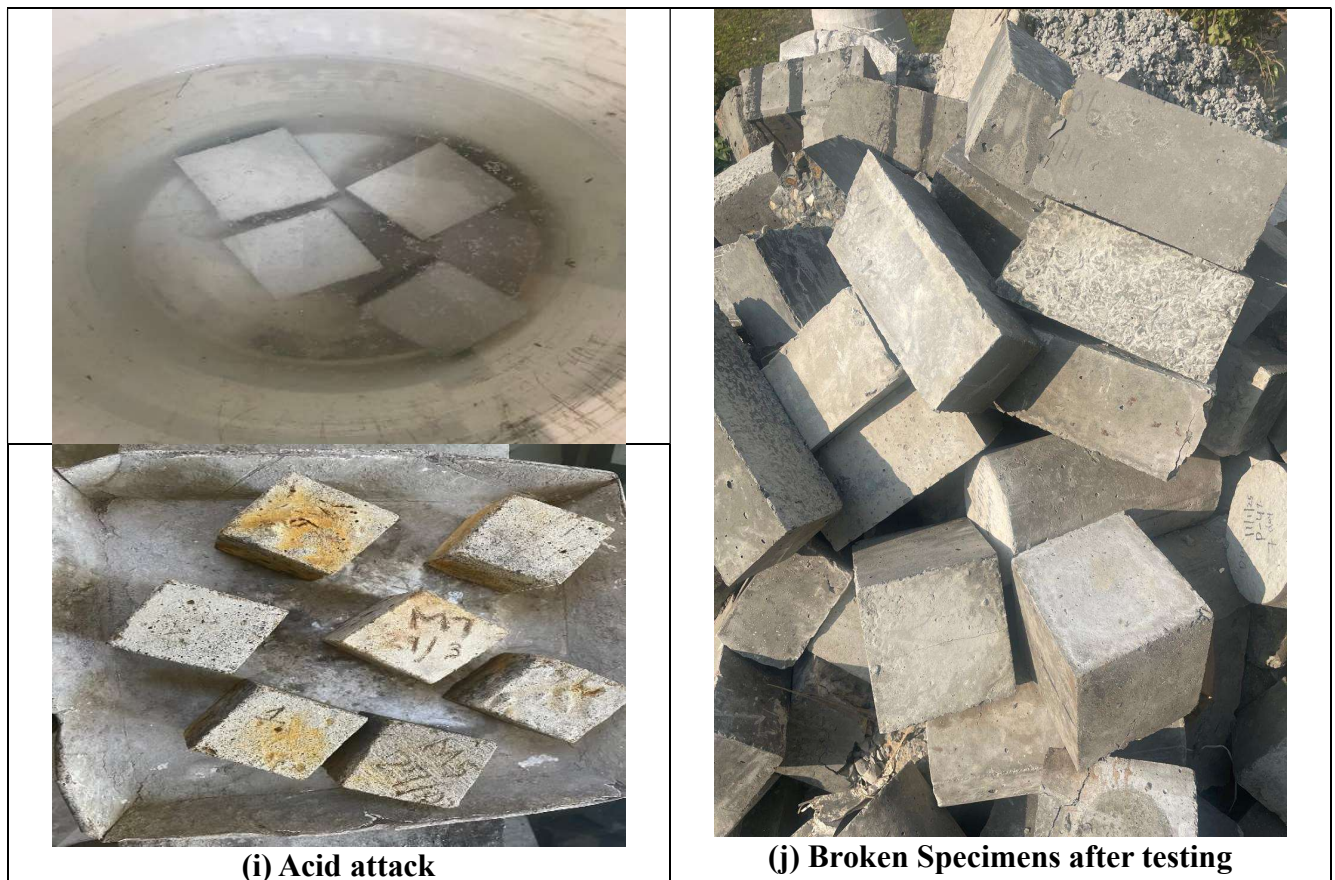


Figure 3.12: Mixing, casting, curing and testing of specimens

3.5.14 Testing Strategy

Workability was measured for both the mixes with and without superplasticiser, but all the mechanical, durability and microstructural tests were conducted only on the superplasticised mixes. The mixes without superplasticiser showed lower workability, poorer compaction and more voids, whereas the superplasticised mixes, which had the same w/c ratio but a lower water and binder content, achieved adequate workability, fewer voids and better compaction, which are essential for an accurate evaluation of hydration behaviour, strength and durability. Testing poorly compacted mixes would have produced unrepresentative strength and permeability results. Table 3.29 summarises the literature basis for this choice.

Table 3.29: Rationale for testing superplasticised mixes: effect of superplasticiser on concrete properties reported in the literature

Author/Source	Property	Explanation
(Iqbal et al., 2015)	Non-workable mixes give non-representative results	Mixes without SP typically have high water demand, poor flow, and honeycombing during casting. Such mixes cannot be compacted adequately, causing voids, artificially low strength, and misleading durability results.
(Praveenkumar & Sankarasubramanian, 2019)	Superplasticiser ensures uniform compaction & density	SP reduces water demand and increases flowability, ensuring uniform casting, proper compaction, and homogeneous microstructure, which is essential for tests like compressive strength, RCPT, acid attack, water absorption, SEM/EDS etc.
(Sabir et al., 2001; Snelson et al., 2008)	SP mixes allow evaluation of real performance of SCMs	Lime sludge and metakaolin show true pozzolanic and filler effects only when dispersion is adequate. Without SP, particle agglomeration occurs → false negative durability/strength outcomes.
(Nadeem et al., 2014)	Lower W/C ratio achieved by SP is essential for durability testing	Durability tests (acid attack, chloride penetration, sorptivity) require mixes of controlled low permeability. Non-SP mixes have higher W/C, leading to artificially high permeability → invalid comparison.
(Obla et al., 2016; Sumajouw et al., 2007)	SP mixes align with modern concrete standards & industry practice	All modern SCM-based concretes (MK, LS, FA, GGBS) use superplasticisers to meet workability without increasing water. Testing

		only SP mixes ensures standardized, practical, industry-ready evaluation.
(N. Gupta & Siddique, 2020)	Maintaining same water content avoids bias in mix comparison	Without SP, water must be increased to maintain workability → this would affect: hydration, pore structure, strength, durability, microstructure. To avoid bias, authors kept water constant and used SP.
(Antoni et al., 2012)	Ensures realistic evaluation of LS+MK synergy	LS (calcium-rich) + MK (aluminosilicate) form C–S–H and C–A–S–H gels. These reactions depend on proper mixing dispersion, achievable only with SP. Without SP, synergy is not measurable.
(ASTM C642-13, 2013)	Non-SP mixes violate rheological requirements of standard test methods	Standard procedures for strength, RCPT (ASTM C1202), acid attack (ASTM C267), water absorption (ASTM C642), SEM require void-free specimens, achievable only when slump is within workable range (≥ 75 mm).

CHAPTER 4: RESULTS AND DISCUSSION

This chapter presents and discusses the results of Phase I (LS–MK blends in M25 concrete) and Phase II (CLS–MK blends in M40 concrete) and compares them with the literature; Section 4.26 synthesises the macroscopic behaviour of the two series with the underlying physicochemical mechanisms. Unless stated otherwise, each strength value is the mean of three specimens (Section 3.5.7).

4.1 Workability of LS–MK Mixes without Superplasticiser

The workability of the fresh concrete was assessed through the slump and its retention over 80 minutes; a higher slump indicates higher workability (Abbass & Singh, 2022a, 2023b). Table 4.1 and Figure 4.1 show the slump values of the LS–MK mixes without superplasticiser. The initial slump ranged from 65 to 115 mm, and the slump at 80 minutes ranged from 51 to 93 mm. The control mix C100M0L0 (95 mm initially and 77 mm at 80 minutes) did not have the highest workability but occupied an intermediate position: the highest slump was recorded for C70M15L15 (115 mm initially and 93 mm at 80 minutes) and the lowest for C80M10L10 (65 mm initially and 51 mm at 80 minutes). The trends were not monotonic with the replacement level, which indicates that LS and MK influence workability through competing mechanisms. MK consists of very fine, plate-like particles with a high specific surface area that adsorb water and increase the water demand, and its early reactivity accelerates the loss of free water (Diaz-Loya et al., 2019; Stefanidou et al., 2017). LS consists predominantly of calcite particles of low reactivity that act as a filler; its porous particles absorb water (water absorption of 40%, Table 3.12), whereas the corresponding reduction in cement content lowers the early consumption of water by hydration (Maheswaran et al., 2011; Vashistha, Singh, et al., 2019). The lowest slump of C80M10L10 is consistent with its highest density (Table 4.5), indicating a dense, cohesive paste in which the water demand of MK and the absorption of LS reduce the free water available for flow. At the highest combined replacement (C70M15L15), the lower clinker content reduces the early consumption of water and the formation of early hydrates, which may account for the higher and better-retained slump;

inadequate dispersion of the fine particles at this dosage may also contribute (Liu et al., 2024; Venu et al., 2024). Since the particle size distribution and specific surface area of LS and MK were not measured in this study, these explanations remain qualitative.

Table 4.1: Slump (mm) of LS–MK mixes without superplasticiser

Time (minutes)	0	20	40	60	80
C100M0L0	95	91	86	83	77
C95M0L5	90	84	76	72	67
C90M0L10	85	82	76	73	67
C85M0L15	105	100	96	94	89
C90M5L0	100	94	87	84	81
C90M5L5	85	81	76	74	69
C85M5L10	90	87	82	80	77
C80M5L15	90	86	82	77	72
C90M10L0	85	80	76	73	67
C85M10L5	75	72	66	64	60
C80M10L10	65	63	60	57	51
C75M10L15	85	81	76	73	67
C85M15L0	95	92	86	83	79
C80M15L5	105	100	94	91	87
C75M15L10	105	101	96	94	90
C70M15L15	115	112	106	97	93

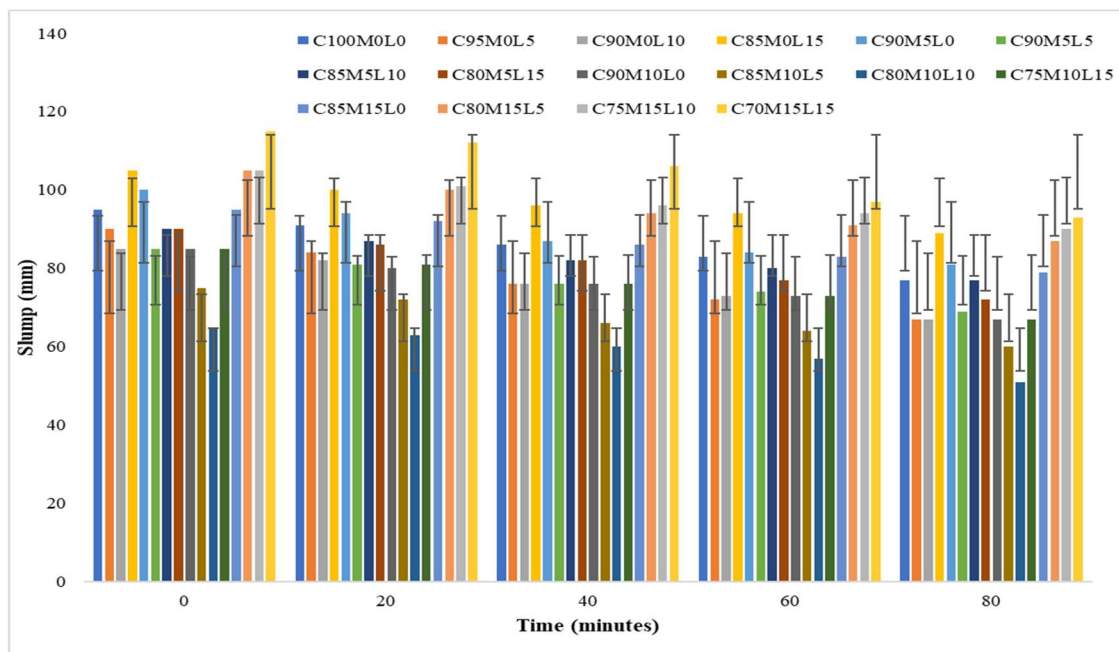


Figure 4.1: Slump retention of LS–MK mixes without superplasticiser

4.2 Workability of LS–MK Mixes with Superplasticiser

Table 4.2 and Figure 4.2 show the slump values of the LS–MK mixes with superplasticiser. The superplasticiser increased the initial slump of each mix by about 5 mm and the slump at 80 minutes by about 3 mm, while the relative ranking of the mixes remained unchanged. The slump at 80 minutes ranged from 54 mm (C80M10L10) to 96 mm (C70M15L15), and the control mix again occupied an intermediate position (100 mm initially and 80 mm at 80 minutes). The mechanisms discussed in Section 4.1 therefore apply equally to the superplasticised mixes (Al-Majidi et al., 2017; Suraneni & Weiss, 2017).

Table 4.2: Slump (mm) of LS–MK mixes with superplasticiser

Time (minutes)	0	20	40	60	80
C100M0L0	100	95	90	86	80
C95M0L5	95	88	80	75	70
C90M0L10	90	86	80	76	70
C85M0L15	110	104	100	97	92

C90M5L0	105	98	91	87	84
C90M5L5	90	85	80	77	72
C85M5L10	95	91	86	83	80
C80M5L15	95	90	86	80	75
C90M10L0	90	84	80	76	70
C85M10L5	80	76	70	67	63
C80M10L10	70	67	64	60	54
C75M10L15	90	85	80	76	70
C85M15L0	100	96	90	86	82
C80M15L5	110	104	98	94	90
C75M15L10	110	105	100	97	93
C70M15L15	120	116	110	100	96

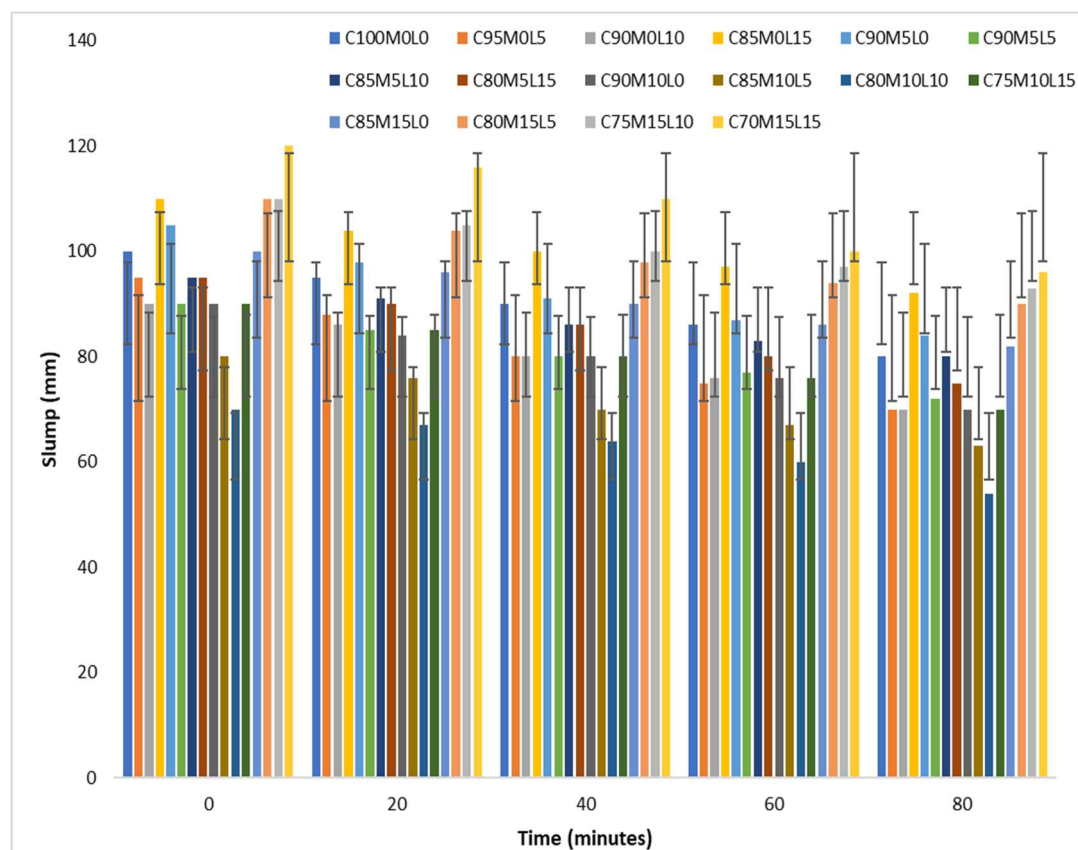


Figure 4.2: Slump retention of LS–MK mixes with superplasticiser

4.3 Workability of CLS–MK Mixes without Superplasticiser

Figure 4.3 and Table 4.3 show the slump values of CLS and MK mixes without superplasticiser, which measure the workability of the concrete mixes. The slump value ranged from 70 to 110 mm. The mix with 5 to 20% MK showed linearly decreasing workability with increasing MK content. The same pattern was observed when the cement was replaced with CLS. The lowest workability was achieved by the mix containing 20% MK and 20% CLS, corresponding to a 40% replacement of cement. The decrease in workability is due to the increased specific surface area of the MK and CLS, which increases the water demand in the concrete mixes for attaining flowability (Fernández et al., 2010; Murali et al., 2023; Venu et al., 2024). This behaviour contrasts with that of raw LS (Section 4.1), whose effect on slump was non-monotonic. Calcination converts part of the CaCO_3 in the sludge into CaO and produces finer particles (95% passing 90 μm for CLS against 85% for LS; Tables 3.12 and 3.14). The free CaO in CLS reacts rapidly with water to form $\text{Ca}(\text{OH})_2$, consuming mixing water, while the higher fineness of CLS increases its specific surface area. Together with the high water demand of the plate-like MK particles, these effects reduce the free water available for flow, so that the slump decreases progressively with increasing CLS and MK contents.

Table 4.3: Slump of CLS–MK mixes without superplasticiser

Mixes	Slump (mm)
M0CL0	110
M0CL5	105
M0CL10	100
M0CL15	95
M0CL20	90
M5CL0	105
M10CL0	100
M15CL0	95
M20CL0	90
M5CL5	100
M5CL10	95

M5CL15	90
M5CL20	85
M10CL5	95
M10CL10	90
M10CL15	85
M10CL20	80
M15CL5	90
M15CL10	85
M15CL15	80
M15CL20	75
M20CL5	85
M20CL10	80
M20CL15	75
M20CL20	70

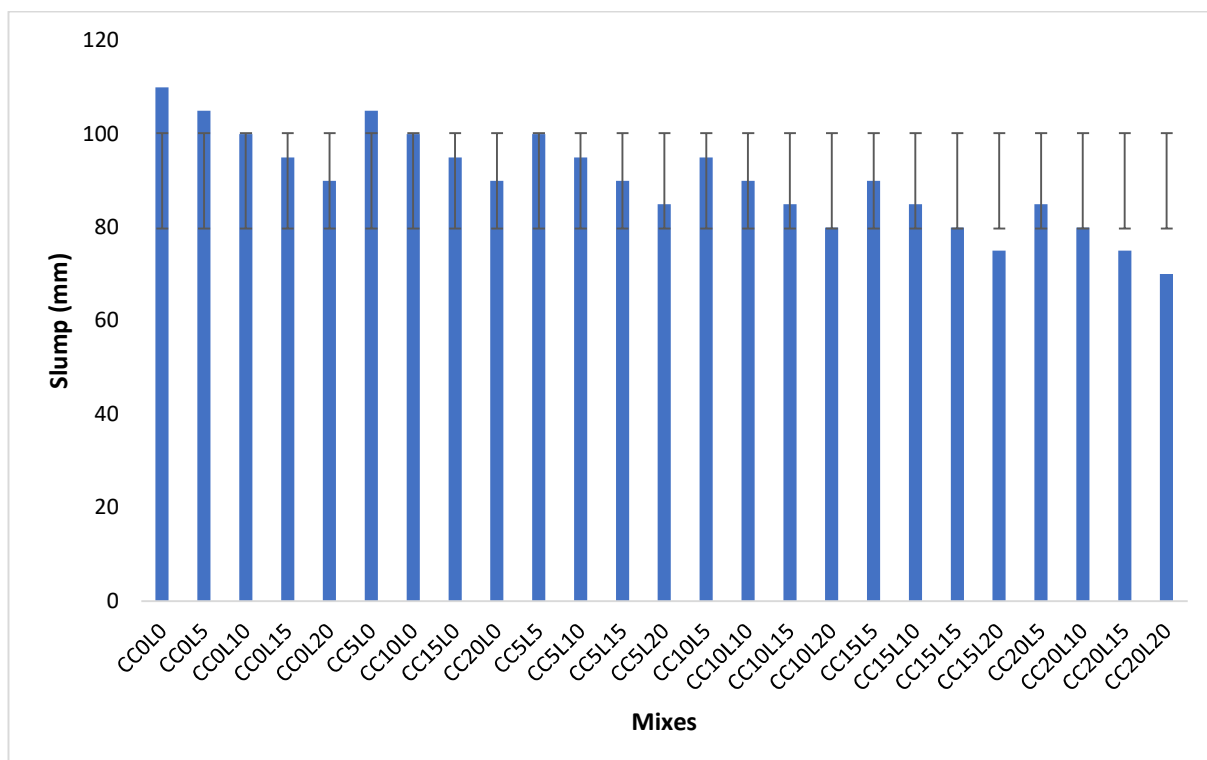


Figure 4.3: Slump of CLS–MK mixes without superplasticiser

4.4 Workability of CLS–MK Mixes with Superplasticiser

Figure 4.4 and Table 4.4 show the slump values for the mixes with superplasticiser, which measure their workability. The slump value ranged from 75 to 115mm. The mix with 5-20% MK showed a linear decrease in workability as MK content increased. The same pattern was observed when the cement was replaced with CLS. The lowest workability was achieved by the mix containing 20% MK and 20% CLS, corresponding to a 40% replacement of cement. The decrease in workability is due to the increased specific surface area of the MK and CLS, which increases the water demand in the concrete mixes for attaining flowability (Fernández et al., 2010; Murali et al., 2023; Venu et al., 2024).

Table 4.4: Slump of CLS–MK mixes with superplasticiser

Mixes	Slump (mm)
M0CL0	115
M0CL5	110
M0CL10	105
M0CL15	100
M0CL20	95
M5CL0	110
M10CL0	105
M15CL0	100
M20CL0	95
M5CL5	105
M5CL10	100
M5CL15	95
M5CL20	90
M10CL5	100
M10CL10	95
M10CL15	90

M10CL20	85
M15CL5	95
M15CL10	90
M15CL15	85
M15CL20	80
M20CL5	90
M20CL10	85
M20CL15	80
M20CL20	75

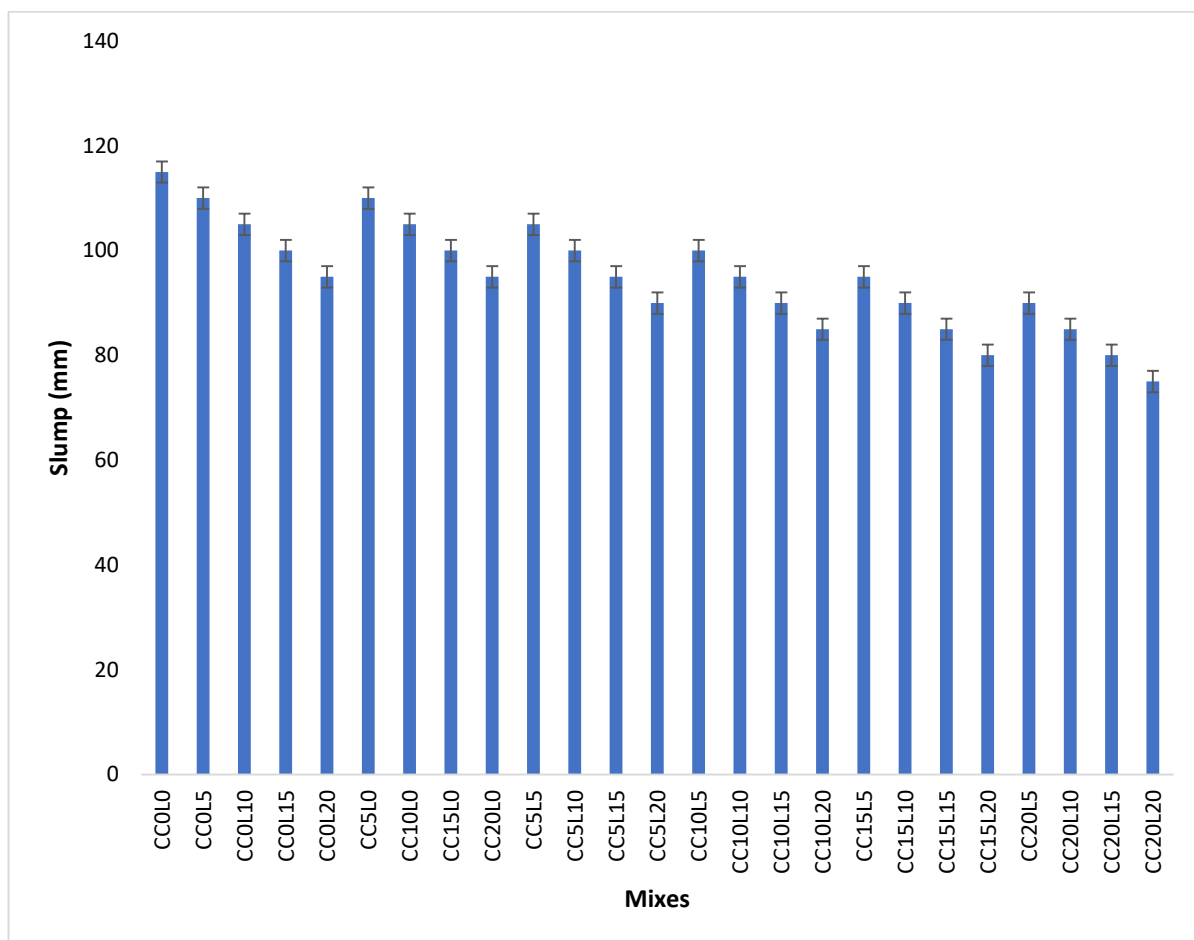


Figure 4.4: Slump of CLS-MK mixes with superplasticiser

4.5 Density of LS–MK Mixes without Superplasticiser

Table 4.5 and Figure 4.5 show the density of the LS–MK mixes without superplasticiser. The density ranged from 2300 to 2500 kg/m³; the control mix C100M0L0 had a density of 2310 kg/m³, which was lower than that of most LS–MK mixes. The mix C80M10L10 attained the highest density (2500 kg/m³), which is attributed to the combined filler effect of LS and the pozzolanic densification by MK, resulting in better particle packing and fewer voids (Barbosa & MacKenzie, 2003; El-Gamal, El-Hosiny, et al., 2017). The lowest density (2300 kg/m³) was recorded for C85M0L15, C80M15L5 and C70M15L15, which is attributed to the lower specific gravity of LS and MK compared with cement and to inadequate dispersion of the fine particles at high replacement levels (Al-Majidi et al., 2017; Suraneni & Weiss, 2017).

Table 4.5: Density of LS–MK mixes without superplasticiser

Mixes	Density (kg/m ³)
C100M0L0	2310
C95M0L5	2340
C90M0L10	2380
C85M0L15	2300
C95M5L0	2310
C90M5L5	2360
C85M5L10	2320
C80M5L15	2340
C90M10L0	2370
C85M10L5	2400
C80M10L10	2500
C75M10L15	2410
C85M15L0	2340

C80M15L5	2300
C75M15L10	2330
C70M15L15	2300

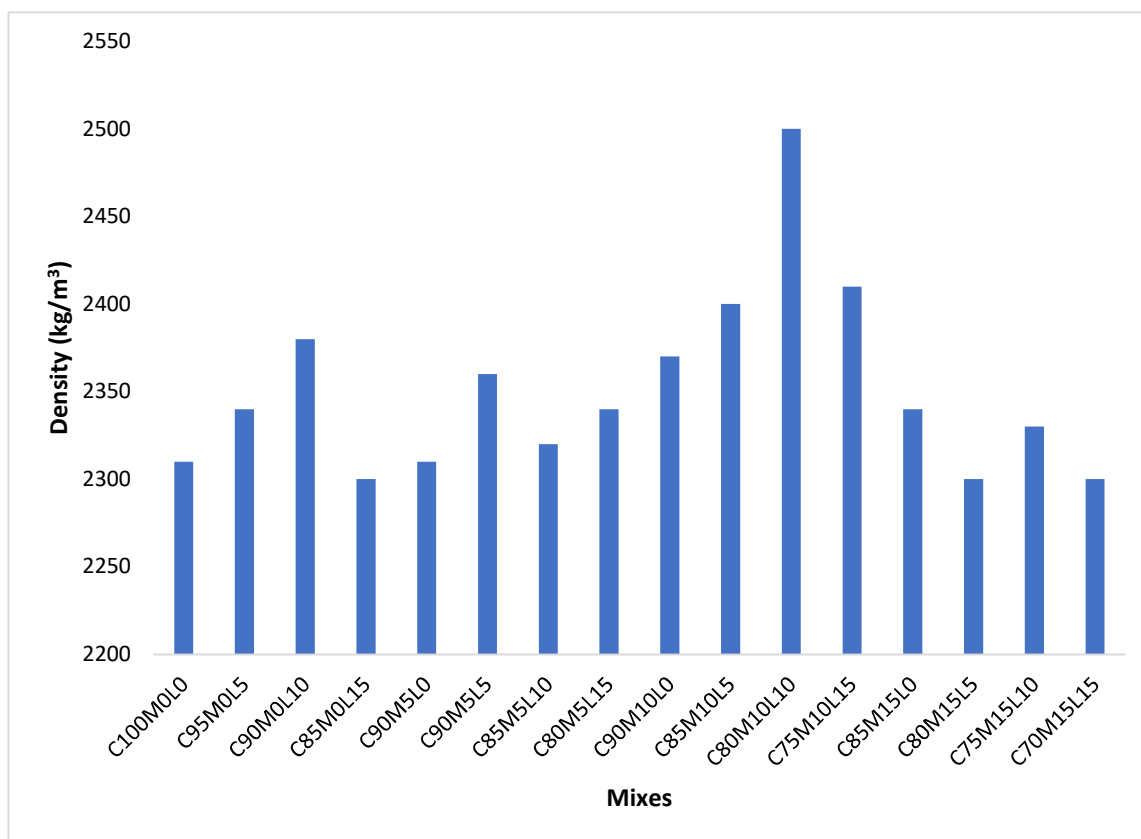


Figure 4.5: Density of LS–MK mixes without superplasticiser

4.6 Density of LS–MK Mixes with Superplasticiser

Table 4.6 and Figure 4.6 show the density of the LS–MK mixes with superplasticiser. The trends were the same as those without superplasticiser: the control mix had a density of 2301 kg/m³, the mix C80M10L10 attained the highest density (2490 kg/m³) and the mixes C85M0L15, C80M15L5 and C70M15L15 the lowest (2290 kg/m³), for the reasons discussed in Section 4.5.

Table 4.6: Density of LS–MK mixes with superplasticiser

Mixes	Density (kg/m³)
C100M0L0	2301
C95M0L5	2331
C90M0L10	2371
C85M0L15	2291
C95M5L0	2301
C90M5L5	2351
C85M5L10	2311
C80M5L15	2331
C90M10L0	2361
C85M10L5	2390
C80M10L10	2490
C75M10L15	2400
C85M15L0	2330
C80M15L5	2290
C75M15L10	2320
C70M15L15	2290

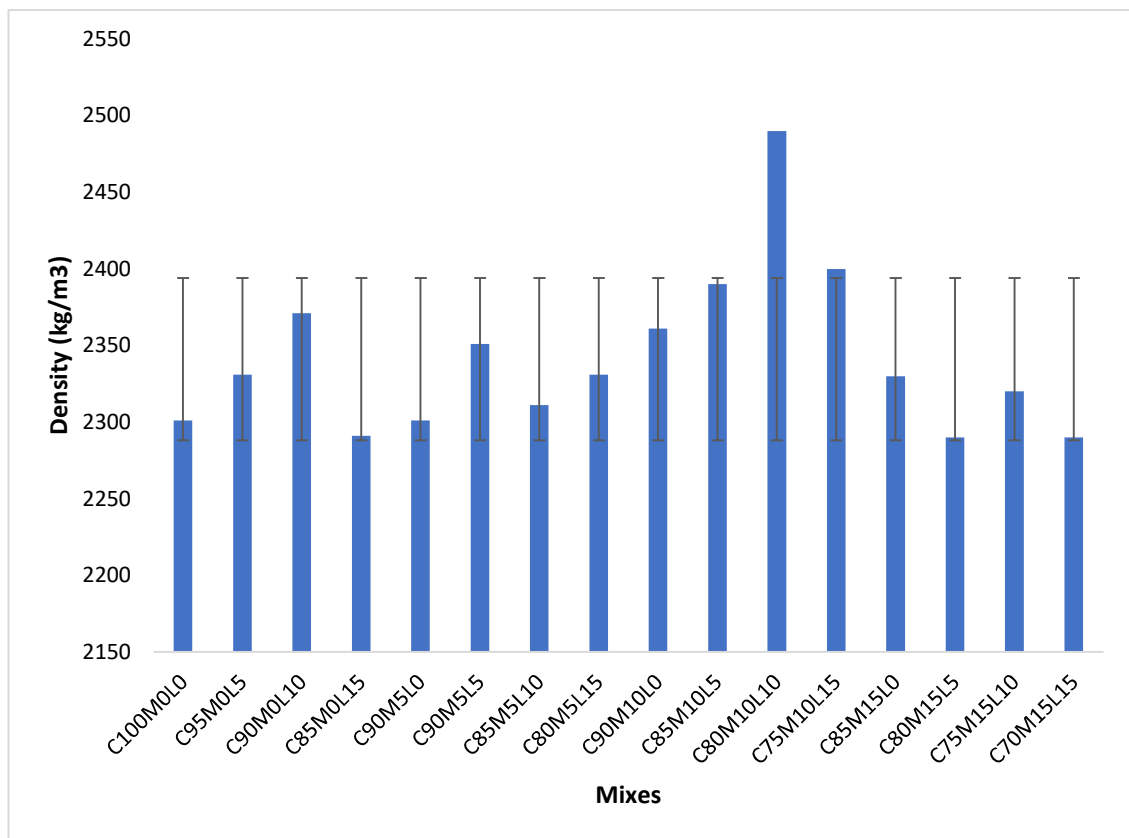


Figure 4.6: Density of LS–MK mixes with superplasticiser

4.7 Compressive Strength of LS–MK Mixes

The compressive strength of concrete represents its load-bearing capacity per unit area. Table 4.7 and Figure 4.7 show the compressive strength of the LS–MK mixes at 7, 28 and 90 days. The 7-day strength ranged from 21.7 to 25.3 MPa, the 28-day strength from 29.8 to 37.1 MPa and the 90-day strength from 34.5 to 44.9 MPa. The control mix C100M0L0 attained 22.5, 30.2 and 35.7 MPa at 7, 28 and 90 days, respectively, and was exceeded at all ages by every mix containing MK. The highest strength at all ages was attained by C80M10L10 (25.3, 37.1 and 44.9 MPa), i.e., about 12%, 23% and 26% higher than the control at 7, 28 and 90 days. The lowest strength at all ages was recorded for C85M0L15 (21.7, 29.8 and 34.5 MPa), which contains 15% LS without MK; this confirms that raw LS on its own acts mainly as a filler of limited reactivity, so that replacing 15% of the cement with LS alone dilutes the clinker

(Buruberri et al., 2015; Vashistha, Kumar, et al., 2019). Increasing the LS and MK contents beyond 10% each reduced the strength gradually, although C70M15L15 (32.8 MPa at 28 days) still exceeded the control. The improvement at moderate replacement is attributed to the pozzolanic reaction of MK with the portlandite released by cement hydration, to the filler and nucleation effects of the finely divided calcite in LS and possibly to the formation of carboaluminate phases from the reaction of calcite with the alumina supplied by MK, as reported for limestone–metakaolin systems (Antoni et al., 2012; Scrivener et al., 2018). At higher replacement levels, clinker dilution and inadequate dispersion of the fine particles reduce the amount of hydration products (Al-Majidi et al., 2017; Diaz-Loya et al., 2019; Suraneni & Weiss, 2017).

Table 4.7: Compressive strength of LS–MK mixes

Mixes	Compressive Strength (MPa)		
	7 days	28 days	90 days
C100M0L0	22.5	30.2	35.7
C95M0L5	22.8	31.5	36.2
C90M0L10	23.1	32	36.8
C85M0L15	21.7	29.8	34.5
C95M5L0	23.6	33.1	39.8
C90M5L5	24.2	34.7	41.5
C85M5L10	24.8	35.8	42.9
C80M5L15	23.9	33.9	40.6
C90M10L0	24.1	34.5	42.1
C85M10L5	24.9	36.2	43.8
C80M10L10	25.3	37.1	44.9
C75M10L15	24.5	35.1	42.5
C85M15L0	23.8	33.7	41.2
C80M15L5	24.3	34.9	42.7

C75M15L10	24	34.2	41.9
C70M15L15	23.2	32.8	40.1

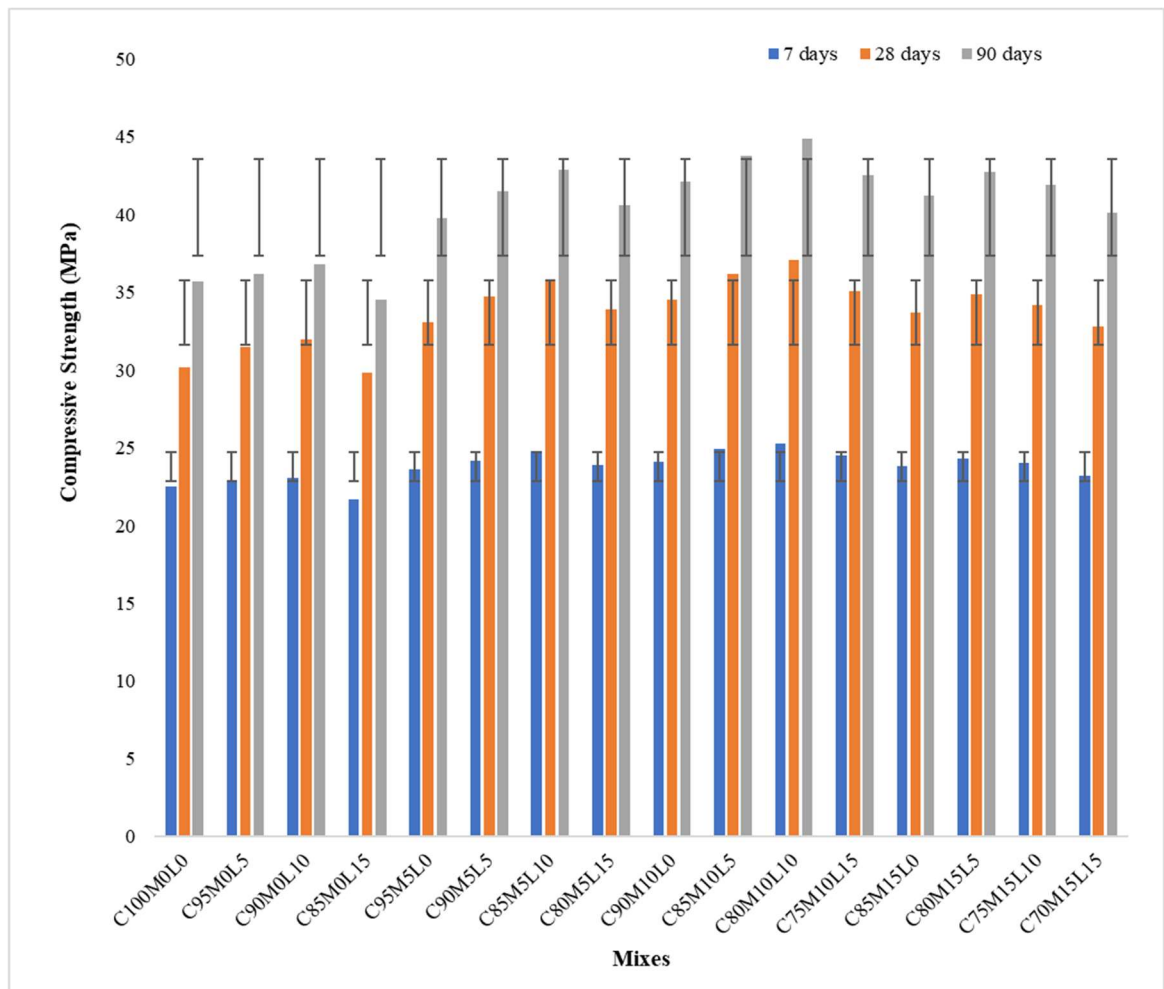


Figure 4.7: Compressive strength of LS–MK mixes

4.8 Compressive Strength of CLS–MK Mixes

Table 4.8 and Figure 4.8 show the compressive strength of the CLS–MK mixes at 7, 28 and 90 days. The compressive strength ranged from 25 to 37 MPa at 7 days, from 32 to 51 MPa at 28 days and from 34.8 to 55.2 MPa at 90 days. The control mix M0CL0 attained 32, 46 and 49 MPa at 7, 28 and 90 days, respectively (Table 4.8). The highest strength was attained by M15CL10 (15% MK and 10% CLS, i.e., 25% total replacement), with 37, 51 and 55.2 MPa, i.e., about 16%, 11% and 13% higher than

the control. The lowest strength was attained by M20CL20 (40% total replacement), owing to clinker dilution and to the higher specific surface area of MK and CLS, which increases the water demand and reduces compaction at a constant water content (Abbass & Singh, 2023a; Bhagath Singh & Subramaniam, 2019; Murali et al., 2023; M. Singh & Garg, 2006; S. K. Singh et al., 2020).

The superior strength of M15CL10 is consistent with a synergy between the calcium supplied by CLS and the reactive silica and alumina supplied by MK. The CaO in CLS hydrates to Ca(OH)_2 , which increases the portlandite available for the pozzolanic reaction of MK, and the residual calcite in CLS (loss on ignition of 10.6%, Table 3.15) can react with the alumina of MK to form hemi- and monocarboaluminate phases, as established for limestone–calcined clay systems (Antoni et al., 2012; Scrivener et al., 2018). The supporting evidence obtained in this study is, however, indirect: M15CL10 showed the lowest total TGA mass loss (Section 4.19), intermediate heat release in calorimetry (Section 4.17) and an XRD pattern with quartz, calcite and hydration-product reflections (Section 4.18). Because C–S–H, C–A–S–H and carboaluminate phases were not quantified directly (e.g., by quantitative XRD, NMR or FTIR), the proposed mechanism should be regarded as a plausible interpretation supported by the literature rather than as a demonstrated reaction pathway (Amar et al., 2024; Chong et al., 2025; Venu et al., 2024; Yang et al., 2024).

Table 4.8: Compressive strength of CLS–MK mixes

Mixes	Compressive strength (MPa)		
	7 days	28 days	90 days
M0CL0	32	46	49
M0CL5	34	47	50
M0CL10	35.2	47.2	50.1
M0CL15	34	46	49
M0CL20	29	43	46
M5CL0	34.2	47.3	50.2
M10CL0	35.4	48	51
M15CL0	36	49	51.7
M20CL0	32	44	47
M5CL5	34	47.8	50.2
M5CL10	35	48	50.9
M5CL15	31	44	46.9

M5CL20	30	41	45
M10CL5	34.5	48	51
M10CL10	36	49.2	52
M10CL15	34.8	47	50
M10CL20	32	42	44.8
M15CL5	36	49.4	51.8
M15CL10	37	51	55.2
M15CL15	35	48	50.8
M15CL20	30	41	44.4
M20CL5	33	42	45
M20CL10	31	39	43
M20CL15	27	34	37.2
M20CL20	25	32	34.8

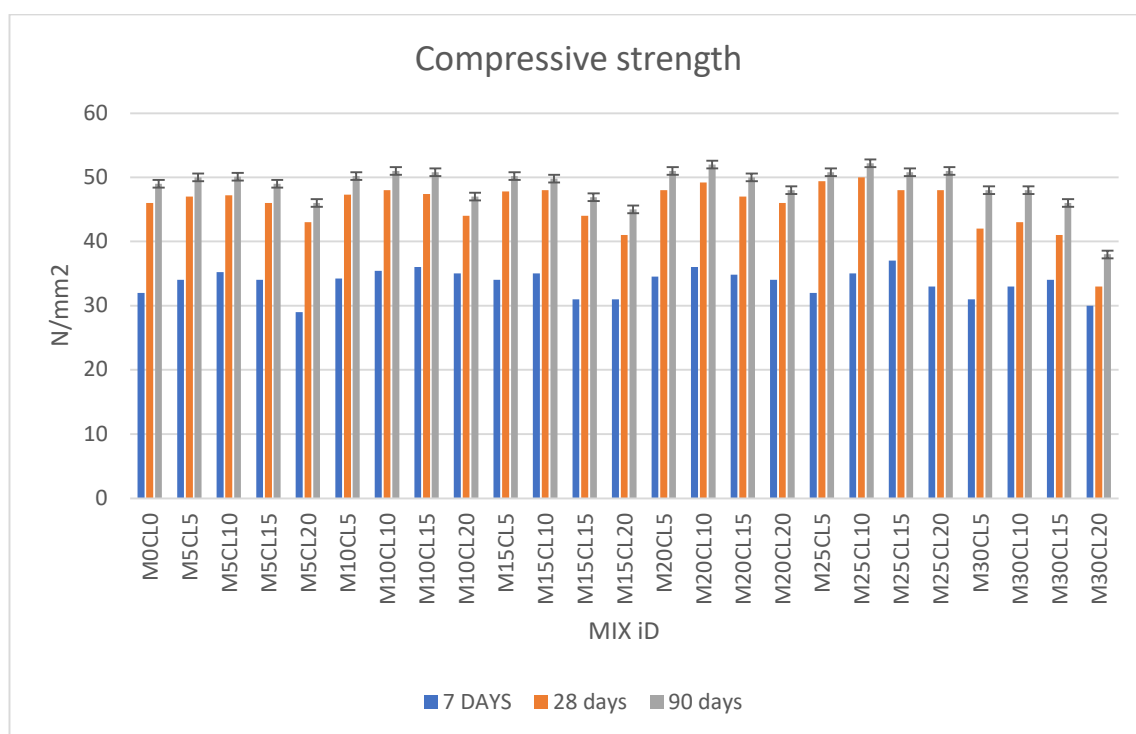


Figure 4.8: Compressive strength of CLS-MK mixes

4.9 Split Tensile Strength of LS–MK Mixes

Table 4.9 and Figure 4.9 show the split tensile strength of the LS–MK mixes. The 7-day split tensile strength ranged from 2.7 to 3.2 MPa, the 28-day strength from 4.1 to 4.7 MPa and the 90-day strength from 5.3 to 6.1 MPa. The control mix C100M0L0 (2.8, 4.1 and 5.4 MPa) was among the weakest mixes, and the lowest values were recorded for C85M0L15 (2.7, 4.1 and 5.3 MPa), which contains LS without MK, reflecting the limited reactivity of raw LS. The highest split tensile strength was attained by C80M10L10 (3.2, 4.7 and 6.1 MPa), i.e., about 13–15% higher than the control, and the strength decreased gradually when the LS and MK contents exceeded 10% each, although C70M15L15 still exceeded the control at all ages. The trends mirror those of compressive strength (Section 4.7) and are attributed to the same combination of pozzolanic, filler and nucleation effects at moderate replacement and clinker dilution at higher replacement (Al-Majidi et al., 2017; Barbosa & MacKenzie, 2003; El-Gamal, El-Hosiny, et al., 2017; Suraneni & Weiss, 2017).

Table 4.9: Split tensile strength of LS–MK mixes

<i>Mixes</i>	<i>Split Tensile Strength (MPa)</i>		
	<i>7 days</i>	<i>28 days</i>	<i>90 days</i>
<i>C100M0L0</i>	2.8	4.1	5.4
<i>C95M0L5</i>	2.8	4.2	5.5
<i>C90M0L10</i>	2.9	4.3	5.6
<i>C85M0L15</i>	2.7	4.1	5.3
<i>C95M5L0</i>	3.0	4.4	5.7
<i>C90M5L5</i>	3.0	4.5	5.9
<i>C85M5L10</i>	3.1	4.6	6.0
<i>C80M5L15</i>	3.0	4.5	5.8
<i>C90M10L0</i>	3.1	4.5	5.9
<i>C85M10L5</i>	3.1	4.6	6.0
<i>C80M10L10</i>	3.2	4.7	6.1

<i>C75M10L15</i>	3.1	4.5	5.9
<i>C85M15L0</i>	3.0	4.5	5.8
<i>C80M15L5</i>	3.1	4.6	6.0
<i>C75M15L10</i>	3.0	4.5	5.9
<i>C70M15L15</i>	3.0	4.4	5.7

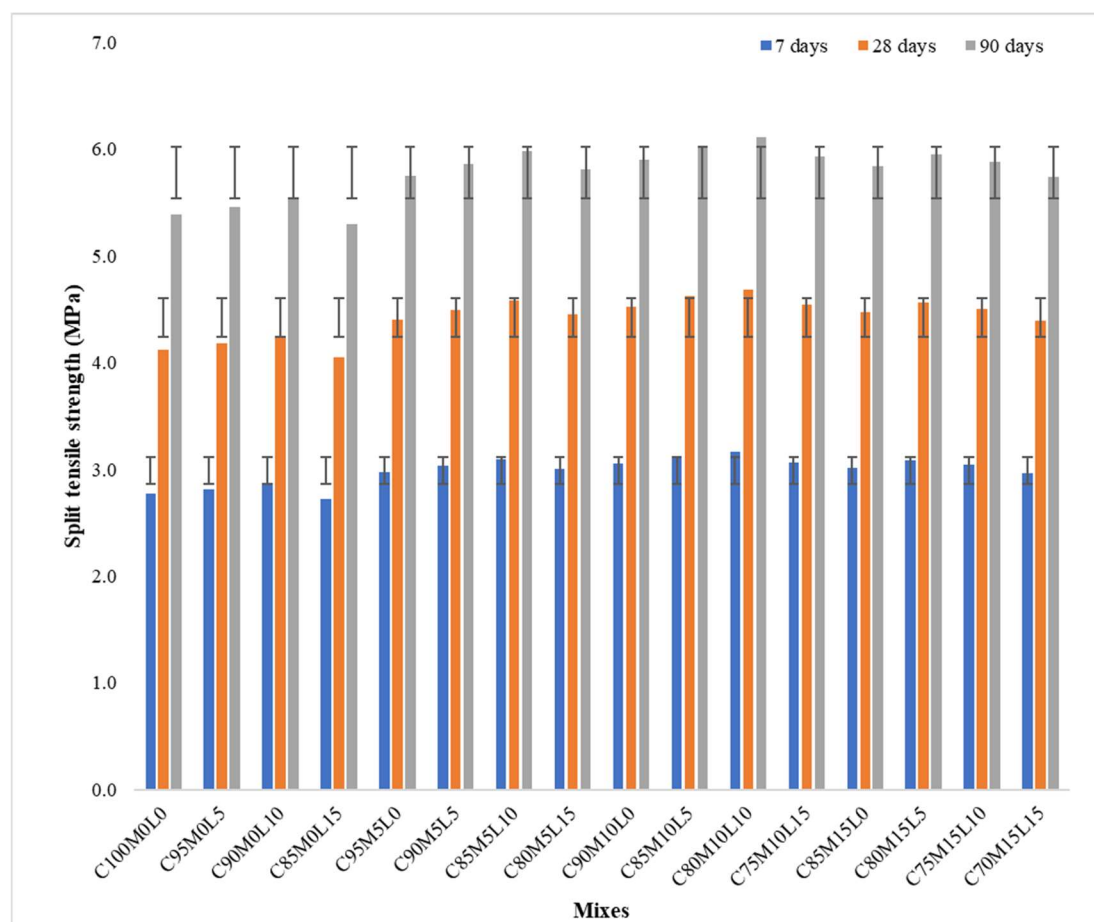


Figure 4.9: Split tensile strength of LS–MK mixes

4.10 Split Tensile Strength of CLS–MK Mixes

Figure 4.10 and Table 4.10 show the split tensile strength of the CLS and MK concrete mixes, which gives the measure of the ability of concrete to resist tension under

loading. The split tensile strength test was done after 7, 28 and 90 days of curing. It was observed that the tensile strength varied between 2.3 to 3.3 MPa at 7 days, 3.1 to 5.0 MPa at 28 days and 3.48 to 5.52 MPa at 90 days for MK and CLS mixes. The lowest tensile strength was attained by the mix with 20% MK and 20% CLS, which means a total replacement of cement by 40%. The decrease in tensile strength is due to the increased specific surface area of the MK and CLS, which leads to higher voids and lesser compaction in the concrete mixes (Bhagath Singh & Subramaniam, 2019; Buruberri et al., 2015; Cheah et al., 2016; Chouksey et al., 2025b, 2025a). The highest tensile strength was attained by the mix with 15% MK and 10% CLS, which means a total 25% replacement of cement. The control mix with no replacement of cement attained tensile strength 2.9, 4.5 and 4.9 MPa after 7, 28 and 90 days, which is about 11–14% lower than that of the mix with 15% MK and 10% CLS (3.3, 5.0 and 5.52 MPa). This is due to the high calcium content of CLS and high silica and alumina content of MK (Amar et al., 2024; Chong et al., 2025; Diaz-Loya et al., 2019; Mu et al., 2017; Venu et al., 2024; Yang et al., 2024).

Table 4.10: Split tensile strength of CLS–MK mixes

Mixes	Split tensile strength (MPa)		
	7 days	28 days	90 days
M0CL0	2.9	4.5	4.9
M0CL5	3.1	4.6	5
M0CL10	3.2	4.6	5.0
M0CL15	3.1	4.5	4.9
M0CL20	2.6	4.2	4.6
M5CL0	3.1	4.6	5.0
M10CL0	3.2	4.7	5.1
M15CL0	3.2	4.8	5.17
M20CL0	2.9	4.3	4.7
M5CL5	3.1	4.7	5.02
M5CL10	3.2	4.7	5.09
M5CL15	2.8	4.3	4.69
M5CL20	2.7	4.0	4.5
M10CL5	3.1	4.7	5.1
M10CL10	3.2	4.8	5.2
M10CL15	3.1	4.6	5

M10CL20	2.9	4.1	4.48
M15CL5	3.2	4.8	5.18
M15CL10	3.3	5.0	5.52
M15CL15	3.2	4.7	5.08
M15CL20	2.7	4.0	4.44
M20CL5	3.0	4.1	4.5
M20CL10	2.8	3.8	4.3
M20CL15	2.4	3.3	3.72
M20CL20	2.3	3.1	3.48

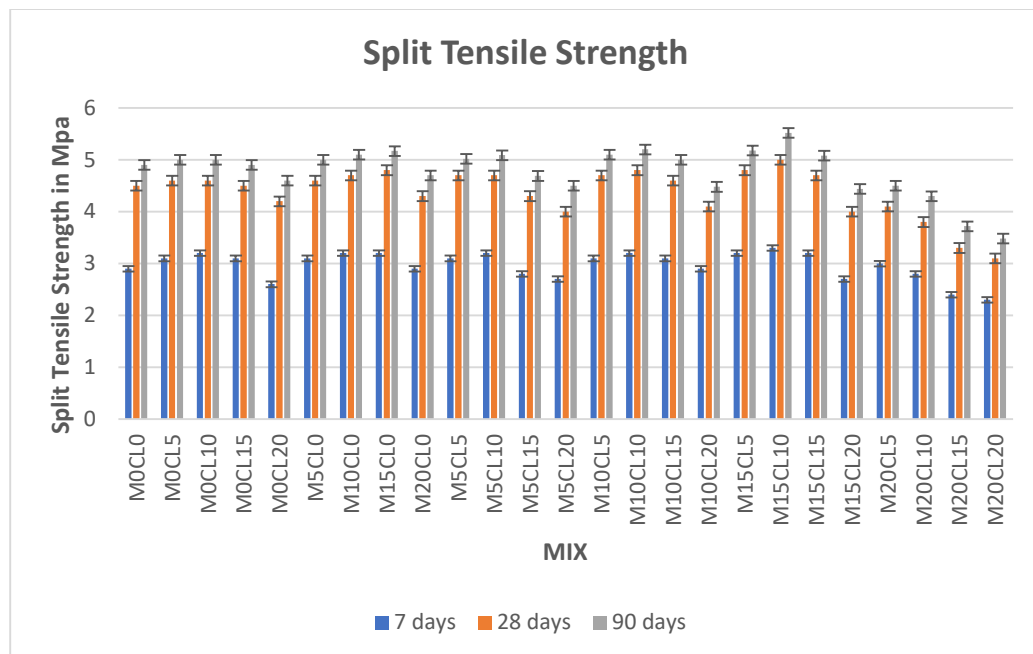


Figure 4.10: Split tensile strength of CLS-MK mixes

4.11 Flexural Strength of LS-MK Mixes

Table 4.11 and Figure 4.11 show the flexural strength of the LS-MK mixes. The 7-day flexural strength ranged from 2.9 to 3.4 MPa, the 28-day strength from 4.18 to 4.81 MPa and the 90-day strength from 5.4 to 6.3 MPa. The control mix C100M0L0 (3.0, 4.25 and 5.5 MPa) was among the weakest mixes, and the lowest values were recorded for C85M0L15 (2.9, 4.18 and 5.4 MPa), which contains LS without MK. The

highest flexural strength was attained by C80M10L10 (3.4, 4.81 and 6.3 MPa), i.e., about 13–15% higher than the control, and the strength decreased gradually when the LS and MK contents exceeded 10% each, although C70M15L15 still exceeded the control. As for compressive and split tensile strength, MK acted as the pozzolanic material while LS acted mainly as a filler, and the combination of the two at moderate levels produced an eco-friendly concrete without compromising strength (Al-Majidi et al., 2017; Barbosa & MacKenzie, 2003; El-Gamal, El-Hosiny, et al., 2017; Suraneni & Weiss, 2017).

Table 4.11: Flexural strength of LS–MK mixes

Mixes	Flexural Strength (MPa)		
	7 days	28 days	90 days
C100M0L0	3.0	4.25	5.5
C95M0L5	3.0	4.31	5.6
C90M0L10	3.1	4.38	5.7
C85M0L15	2.9	4.18	5.4
C95M5L0	3.2	4.53	5.9
C90M5L5	3.2	4.62	6.0
C85M5L10	3.3	4.71	6.1
C80M5L15	3.2	4.58	6.0
C90M10L0	3.3	4.65	6.0
C85M10L5	3.3	4.75	6.2
C80M10L10	3.4	4.81	6.3
C75M10L15	3.3	4.67	6.1
C85M15L0	3.2	4.6	6.0
C80M15L5	3.3	4.69	6.1
C75M15L10	3.2	4.63	6.0
C70M15L15	3.2	4.52	5.9

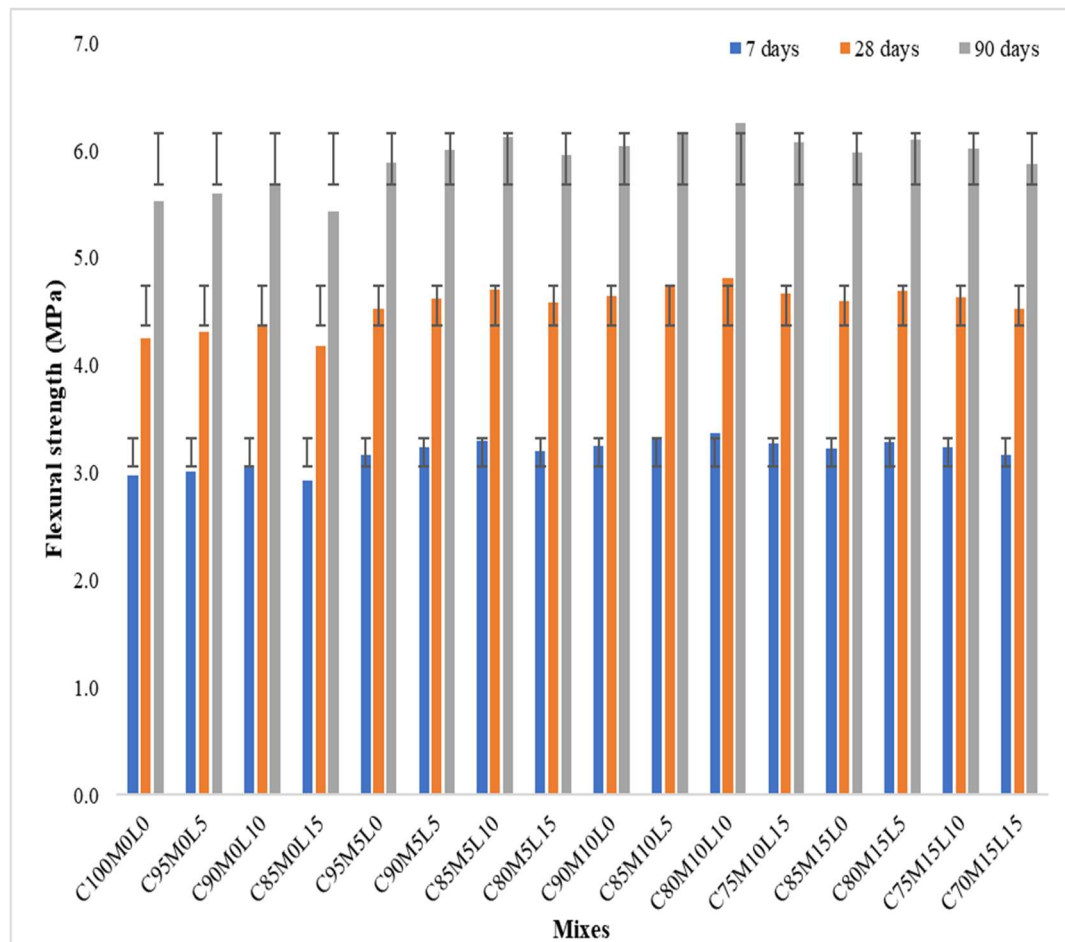


Figure 4.11: Flexural strength of LS–MK mixes

4.12 Flexural Strength of CLS–MK Mixes

Figure 4.12 and Table 4.12 show the flexural strength of the CLS and MK concrete mixes, which gives a measure of the ability of concrete to resist bending. The flexural strength test was done after 7, 28 and 90 days of curing. It was observed that the flexural strength varied between 2.5 to 3.7 MPa for 7 days, 3.52 to 5.61 for 28 days, and 4.18 to 6.63 for 90 days for MK and CLS mixes. The lowest flexural strength was achieved by the mix with 20% MK and 20% CLS, indicating a total replacement of cement by 40%. The decrease in flexural strength is due to the increased specific surface area of the MK and CLS, which leads to higher voids and lesser compaction

in the concrete mixes. The highest flexural strength was attained by the mix with 15% MK and 10% CLS, which means a total 25% replacement of cement. The control mix with no replacement of cement attained a flexural strength of 3.2, 5.06 and 5.88 MPa after 7, 28 and 90 days, which is less than the mix with 15% MK and 10% CLS. This is due to the high calcium content of CLS and high silica and alumina content of MK (Amar et al., 2024; Ambikakumari Sanalkumar & Yang, 2021; Chong et al., 2025; Diaz-Loya et al., 2019; Mu et al., 2017; Trindade et al., 2018; Venu et al., 2024; Yang et al., 2024).

Table 4.12: Flexural strength of CLS–MK mixes

Mixes	Flexural strength (MPa)		
	7 days	28 days	90 days
M0CL0	3.2	5.06	5.88
M0CL5	3.4	5.17	6
M0CL10	3.5	5.19	6.012
M0CL15	3.4	5.06	5.88
M0CL20	2.9	4.73	5.52
M5CL0	3.4	5.20	6.024
M10CL0	3.5	5.28	6.12
M15CL0	3.6	5.39	6.204
M20CL0	3.2	4.84	5.64
M5CL5	3.4	5.25	6.024
M5CL10	3.5	5.28	6.108
M5CL15	3.1	4.84	5.628
M5CL20	3.0	4.51	5.4
M10CL5	3.5	5.28	6.12
M10CL10	3.6	5.41	6.24
M10CL15	3.5	5.17	6
M10CL20	3.2	4.62	5.376
M15CL5	3.6	5.434	6.216
M15CL10	3.7	5.61	6.624
M15CL15	3.5	5.28	6.096
M15CL20	3.0	4.51	5.328
M20CL5	3.3	4.62	5.4
M20CL10	3.1	4.29	5.16
M20CL15	2.7	3.74	4.464

M20CL20

2.5

3.52

4.176

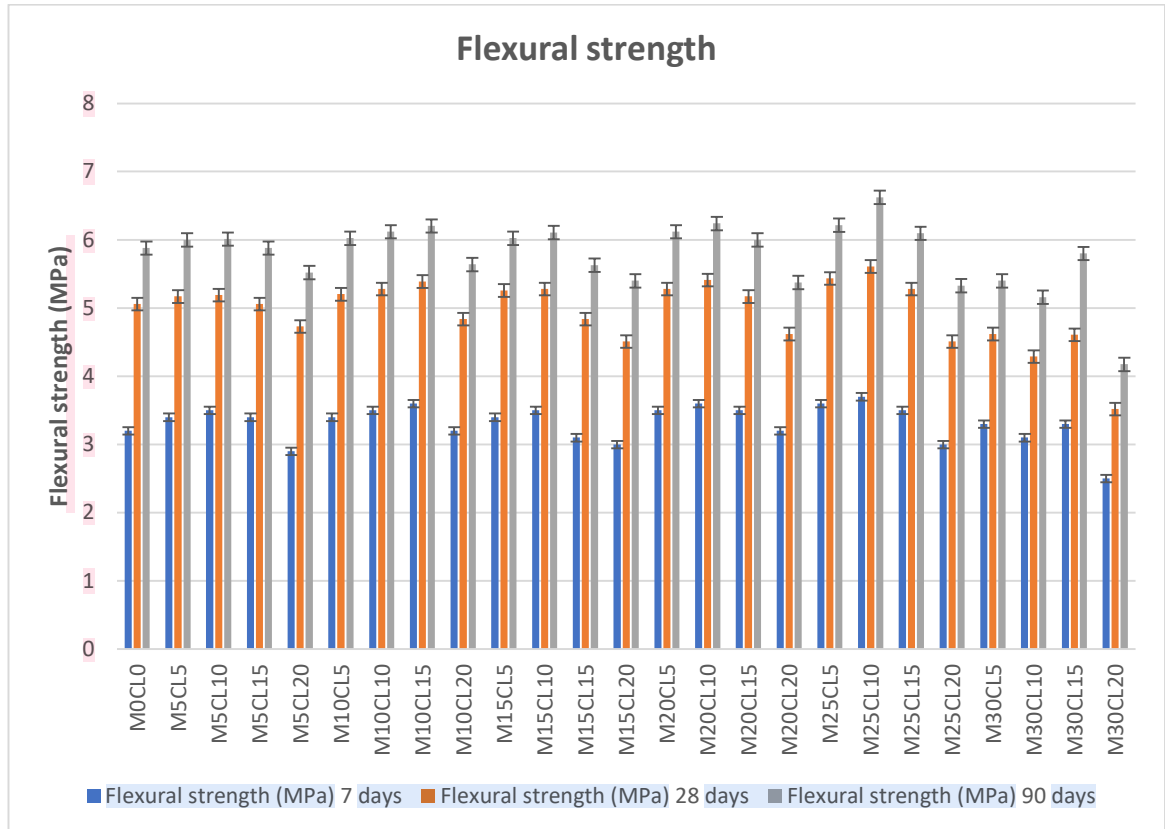


Figure 4.12: Flexural strength of CLS–MK mixes

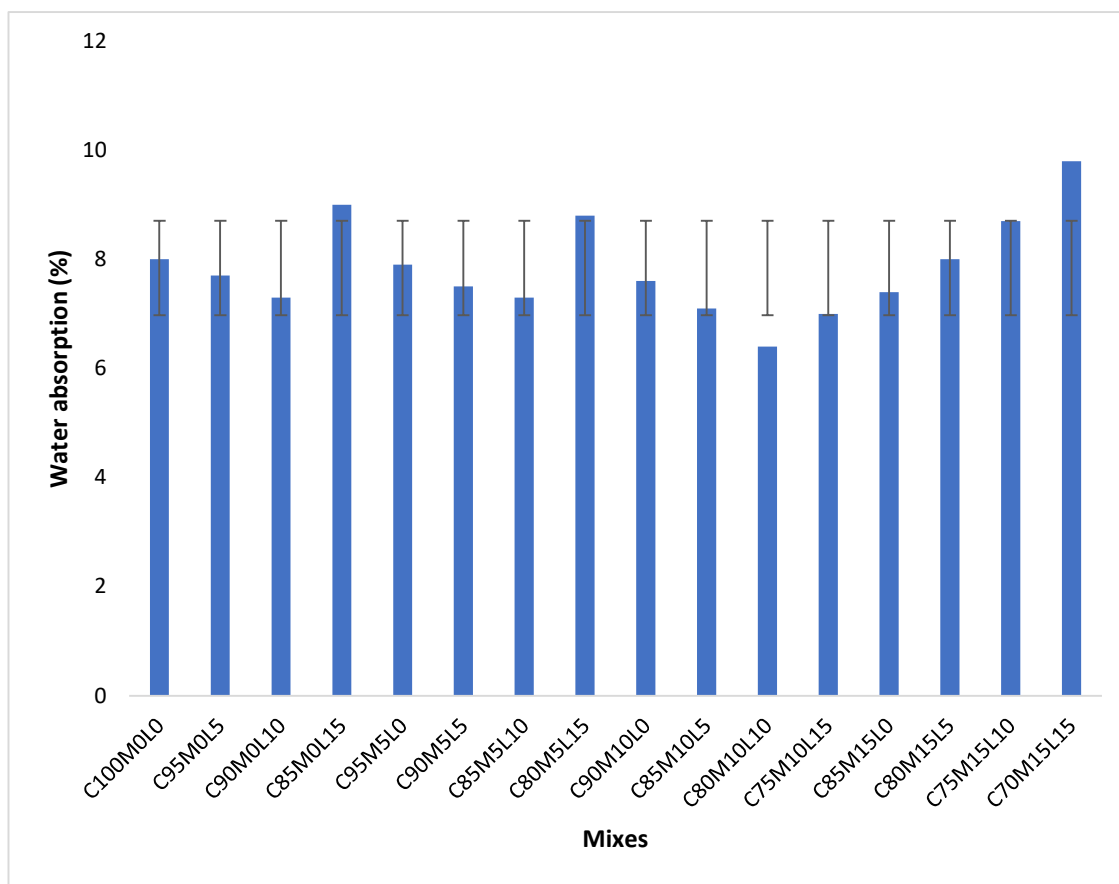
4.13 Water Absorption of LS–MK Mixes

The water absorption of concrete is a measure of the accessible pores that take up water during 24 hours of immersion; higher water absorption denotes a more porous, less dense mix. Table 4.13 and Figure 4.13 show the water absorption of the LS–MK mixes, which ranged from 6.4% to 9.8%. The control mix C100M0L0 absorbed 8.0%, and most mixes containing MK absorbed less. The lowest water absorption (6.4%, i.e., 20% lower than the control) was attained by C80M10L10, owing to better hydration, fewer voids and better compaction and bonding of the matrix (Barbosa & MacKenzie, 2003; El-Gamal, El-Hosiny, et al., 2017). The highest water absorption (9.8%) was recorded for C70M15L15, owing to clinker dilution and inadequate dispersion of the

LS and MK particles at this high replacement level (Al-Majidi et al., 2017; Suraneni & Weiss, 2017). The overall behaviour reflects the pozzolanic effect of MK, which produces denser mixes with fewer voids, and the filler effect of LS, which reduces the pore volume at moderate dosages.

Table 4.13: Water absorption of LS–MK mixes

Mixes	Water absorption (%)
C100M0L0	8
C95M0L5	7.7
C90M0L10	7.3
C85M0L15	9
C95M5L0	7.9
C90M5L5	7.5
C85M5L10	7.3
C80M5L15	8.8
C90M10L0	7.6
C85M10L5	7.1
C80M10L10	6.4
C75M10L15	7
C85M15L0	7.4
C80M15L5	8
C75M15L10	8.7
C70M15L15	9.8



SS

Figure 4.13: Water absorption of LS–MK mixes

4.14 Water Absorption of CLS–MK Mixes

Figure 4.14 and Table 4.14 show the water absorption of the mixes, which indicates the pore volume in the concrete mixes. The water absorption varied between 6 to 11.6%. The mix with 5 to 20% MK attained linearly decreasing water absorption up to 15% of MK, and beyond that, water absorption increased with the increase in the percentage of MK. The same pattern was observed when cement was replaced with CLS, but beyond 10%, water absorption increased with the percentage of CLS. The highest water absorption was observed in the mix containing 20% MK and 20% CLS, corresponding to a total replacement of cement by 40%. The increase in water absorption is due to the higher number of pores and a less dense matrix due to the

inclusion of a higher amount of MK and CLS (Ayub et al., 2014; Nana et al., 2024; Sabir et al., 2001).

Table 4.14: Water absorption of CLS–MK mixes

Mixes	Water absorption (%)
M0CL0	7.6
M0CL5	7.4
M0CL10	7.2
M0CL15	8.2
M0CL20	9.2
M5CL0	7.2
M10CL0	7
M15CL0	6.8
M20CL0	7.8
M5CL5	7.2
M5CL10	7.1
M5CL15	8
M5CL20	9
M10CL5	7.2
M10CL10	7
M10CL15	7.8
M10CL20	8
M15CL5	7
M15CL10	6
M15CL15	7.1
M15CL20	8.1
M20CL5	8.6
M20CL10	9.2

M20CL15	10.4
M20CL20	11.6

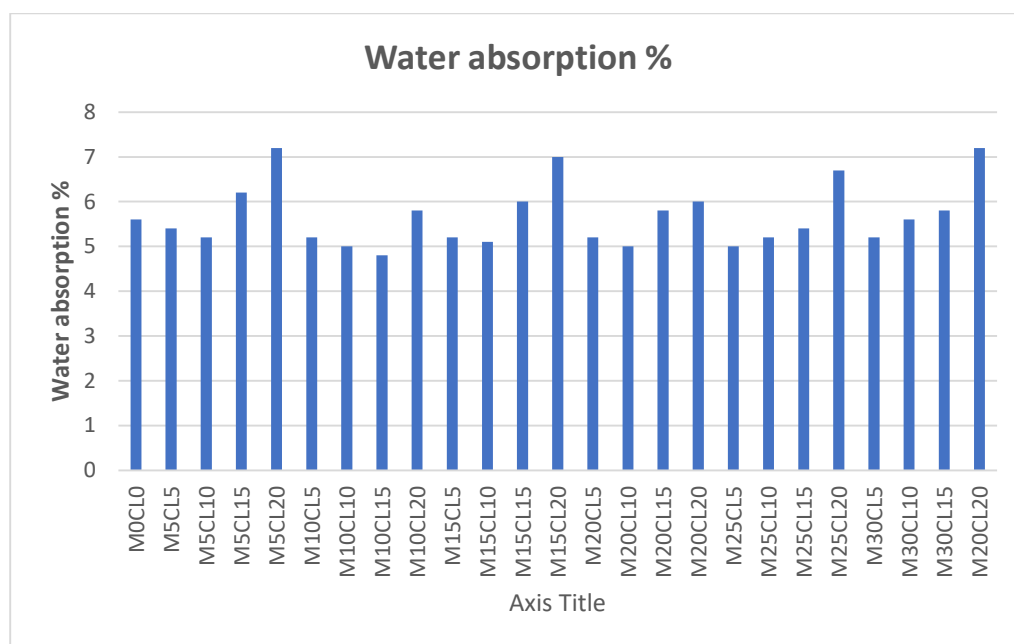


Figure 4.14: Water absorption of CLS–MK mixes

4.15 Acid Attack Resistance of the Mixes

Acid attack resistance was evaluated from the mass loss of specimens immersed in 5% HCl solution for 12 weeks. Table 4.15 and Figure 4.15 show the weekly mass loss of the LS–MK mixes. The control mix C100M0L0 lost 10% of its mass after 12 weeks. The mix C80M10L10 showed the highest acid resistance, with a mass loss of only 8% (20% lower than the control) and no measurable loss during the first five weeks, which is attributed to its dense, low-permeability matrix (lowest water absorption, Table 4.13) and to the consumption of acid-soluble portlandite by the pozzolanic reaction of MK (Abbass & Singh, 2023b, 2024; Barbosa & MacKenzie, 2003; El-Gamal, El-Hosiny, et al., 2017). It should be noted, however, that all the other LS–MK mixes lost more mass than the control (11–40%), so that the improvement in acid resistance is

specific to the optimum proportion rather than general. The mass loss increased sharply at high replacement levels, reaching 27%, 38% and 40% for C80M15L5, C75M15L10 and C70M15L15, respectively, owing to their higher porosity (Table 4.13) and the higher content of acid-soluble calcite contributed by LS (Abbass & Akhai, 2025a, 2025b; Al-Majidi et al., 2017; Suraneni & Weiss, 2017).

Table 4.15: Mass loss (%) of LS–MK mixes in 5% HCl solution

Time (weeks)	1	2	3	4	5	6	7	8	9	10	11	12
C100M0L0	0	0	0	0	3	4	4	5	6	8	9	10
C95M0L5	0	0	0	1	3	4	6	8	9	9	10	11
C90M0L10	0	0	1	3	3	5	7	8	9	10	11	12
C85M0L15	0	2	4	4	5	6	8	9	10	10	11	12
C95M5L0	0	0	1	2	4	6	7	8	9	9	12	13
C90M5L5	0	0	1	5	7	7	9	10	11	13	13	14
C85M5L10	0	1	3	7	8	8	10	11	11	12	14	14
C80M5L15	0	2	3	6	9	9	11	11	12	14	14	15
C90M10L0	0	1	2	4	7	8	9	9	10	12	12	13
C85M10L5	0	2	4	6	6	7	9	10	10	12	13	15
C80M10L10	0	0	0	0	0	1	2	3	5	7	7	8
C75M10L15	0	2	4	4	6	10	11	13	14	14	15	16
C85M15L0	0	2	5	6	7	7	8	13	13	15	16	17
C80M15L5	0	2	3	6	8	10	13	16	16	19	21	27
C75M15L10	0	3	4	7	8	11	14	17	18	23	32	38
C70M15L15	0	4	7	9	10	13	17	19	23	29	36	40

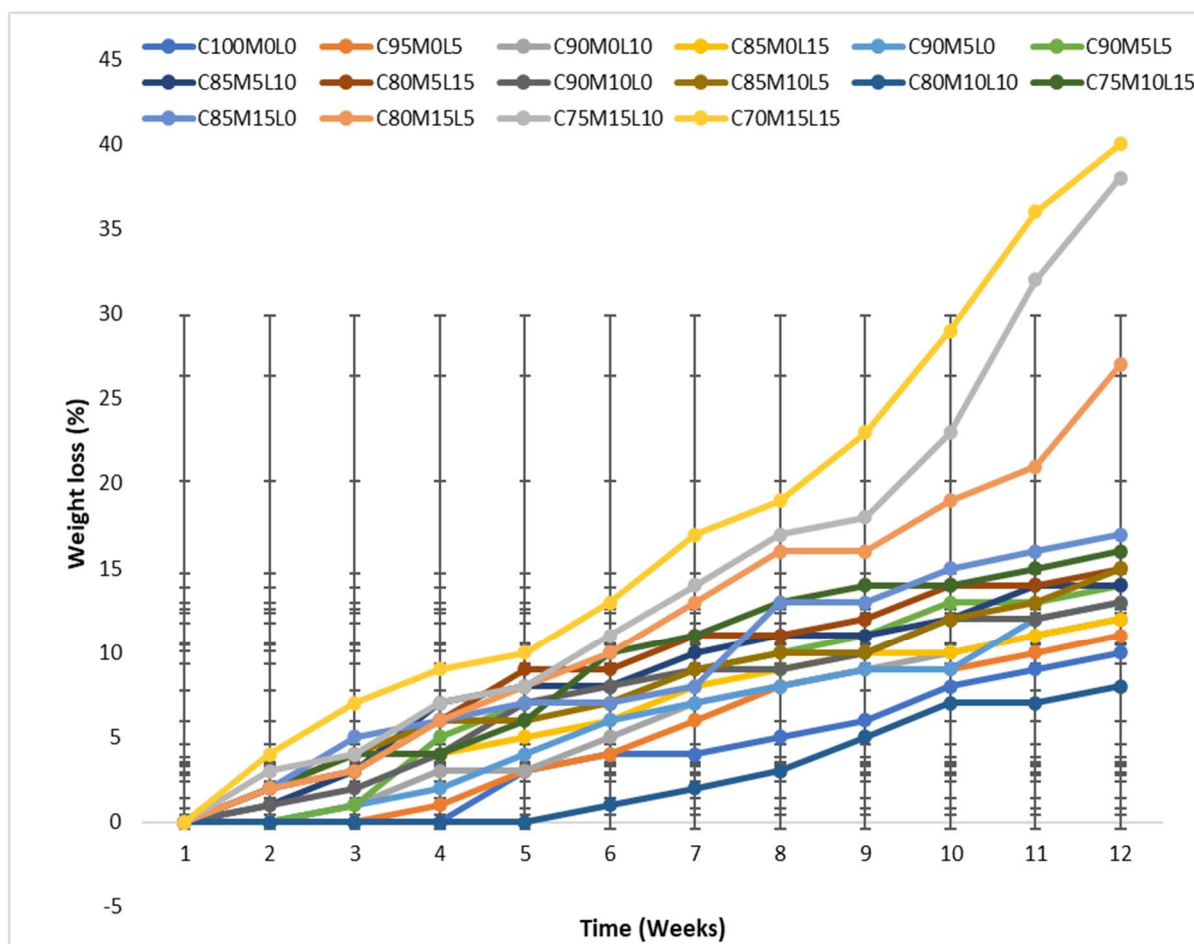


Figure 4.15: Mass loss of LS–MK mixes in 5% HCl solution

4.15.1 Acid Attack Resistance of CLS–MK Mixes

Table 4.16 and Figure 4.16 show the mass loss of the CLS–MK mixes in 5% HCl over 12 weeks. The control mix M0CL0 lost 11% of its mass. Only the mix M15CL5 showed a clearly lower mass loss (about 8%) than the control; it remained practically unaffected for the first five weeks, indicating the highest acid resistance among the CLS–MK mixes. Most of the other CLS–MK mixes lost the same as or more mass than the control (11–22%), so that the incorporation of CLS and MK did not generally improve acid resistance.

The improved resistance of M15CL5 can be explained by the pozzolanic consumption of $\text{Ca}(\text{OH})_2$ by MK, which removes the most acid-soluble hydration product, together with the formation of additional C–(A)–S–H gel, densification of the matrix and

reduced permeability, which slow the ingress of the acid (Abo-El-Enein et al., 2016; Kumar & Prasad, 2019). In the mixes with higher CLS contents, the CaO supplied by CLS hydrates to additional $\text{Ca}(\text{OH})_2$ and the residual CaCO_3 is itself acid-soluble; when this calcium is not fully consumed by the available MK, the matrix becomes more vulnerable to acid attack. Excessive total replacement also reduces the clinker available for hydration and produces a more porous matrix.

Table 4.16: Mass loss (%) of CLS–MK mixes in 5% HCl solution

Weeks	1	2	3	4	5	6	7	8	9	10	11	12
M0CL0	0	0	0	1	3	4	6	8	9	9	10	11
M5CL5	0	0	1	3	3	5	7	8	9	10	11	12
M5CL10	0	2	4	4	5	6	8	9	10	10	11	12
M5CL15	0	0	1	2	4	6	7	8	9	9	12	13
M5CL20	0	0	1	5	7	7	9	10	11	13	13	14
M10CL5	0	1	3	7	8	8	10	11	11	12	14	14
M10CL10	0	2	3	6	9	9	11	11	12	14	14	15
M10CL15	0	1	2	4	7	8	9	9	10	12	12	13
M10CL20	0	2	4	6	6	7	9	10	10	12	13	15
M15CL5	0	0	0	0	0	1	2	3	5	7	7	8
M15CL10	0	2	4	4	6	10	11	13	14	14	15	16
M15CL15	0	2	5	6	7	7	8	13	13	15	16	17
M15CL20	0	2	3	6	8	10	13	16	16	19	21	21
M20CL5	0	3	4	7	8	11	14	17	18	20	21	22
M20CL10	0	1	2	4	7	8	9	9	10	12	12	13
M20CL15	0	0	1	3	3	5	7	8	9	10	11	12
M20CL20	0	2	4	4	5	6	8	9	10	10	11	12
M25CL5	0	0	1	2	4	6	7	8	9	9	12	13
M25CL10	0	0	1	5	7	7	9	10	11	13	13	14
M25CL15	0	1	3	7	8	8	10	11	11	12	14	14

M25CL2 0	0	2	3	6	9	9	11	11	12	14	14	15
M30CL5	0	1	2	4	7	8	9	9	10	12	12	13
M30CL1 0	0	0	0	1	3	4	6	8	9	9	10	11
M30CL1 5	0	0	0	1	3	4	6	8	9	9	10	11
M30CL2 0	0	0	1	5	7	7	9	10	11	13	13	14

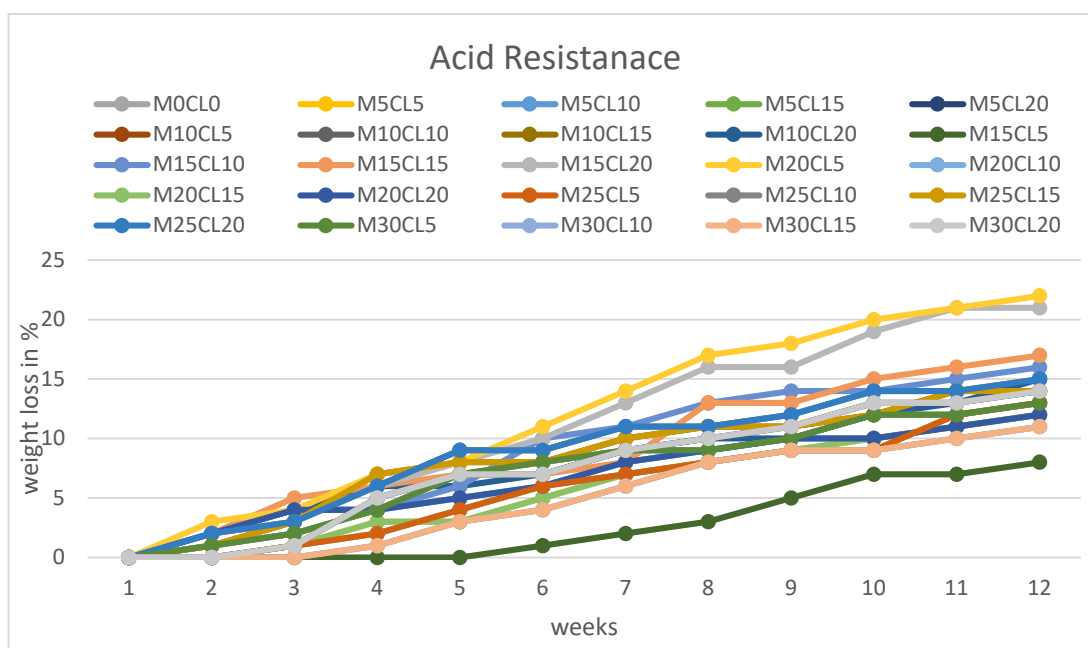


Figure 4.16: Mass loss of CLS–MK mixes in 5% HCl solution

4.16 Chloride Ion Penetration of LS–MK Mixes

The rapid chloride ion penetration test results of the LS–MK mixes are given in Table 4.17. The control mix passed a charge of 2,900 C at 28 days and 2,200 C at 56 days, corresponding to moderate chloride ion penetrability according to ASTM C1202 (ASTM C1202, 2012). The mixes containing only MK passed lower charges (1,000–1,700 C at 56 days), reflecting the pore refinement caused by the pozzolanic activity of MK, whereas the mixes containing only LS passed charges similar to that of the control (1,900–2,600 C), confirming the limited reactivity of raw LS. All the mixes containing both MK and LS passed lower charges than the control, and the mix

C80M10L10 showed the greatest improvement, with a charge of 800 C at 56 days (about 64% lower than the control), corresponding to very low chloride penetrability. This is attributed to the combined effect of pore refinement by MK and the filler effect of LS, and to the chloride-binding capacity of the additional aluminate phases formed in the presence of MK (Kumar & Prasad, 2019; Nadeem et al., 2014; Y. Wang et al., 2020). The mixes with LS and MK contents greater than 10% each showed slightly higher charges than C80M10L10 owing to reduced pore refinement and hydration (Bernal et al., 2012; Wong, 2022).

Table 4.17: Rapid chloride ion penetration of LS–MK mixes

Mixes	Charge passed (28-day) (C)	Charge passed (56-day) (C)	ASTM Class (56-days)
C100M0L0	2,900	2,200	Moderate (2000–4000)
C95M0L5	3,100	2,500	Moderate
C90M0L10	2,300	1,900	Low (1000–2000)
C85M0L15	3,200	2,600	Moderate
C95M5L0	2,200	1,700	Low
C90M5L5	1,900	1,400	Low
C85M5L10	1,700	1,200	Low
C80M5L15	2,100	1,600	Low
C90M10L0	1,800	1,200	Low
C85M10L5	1,500	1,000	Very Low (100–1000)
C80M10L10	1,250	800	Very Low
C75M10L15	1,500	1,100	Low to Very Low
C85M15L0	1,600	1,000	Very Low
C80M15L5	1,600	950	Very Low
C75M15L10	1,400	900	Very Low

C70M15L15	1,300	1,000	Very Low
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4.17 Differential Scanning Calorimetry (DSC)

Figures 4.17 and 4.18 show the heat flow and cumulative heat curves, and Table 4.18 compares the cumulative heat of selected mixes of both series. The heat flow curves indicate that moderate replacement levels significantly influence the hydration kinetics. The mixes **M40-M10CL10** and **M25-C80M10L10** exhibited the highest main hydration peaks, suggesting accelerated C_3S hydration through the nucleation and filler effects of the supplementary materials; sharp peaks are generally associated with rapid early formation of hydration products and higher early-age strength. In contrast, **M5CL5** showed a delayed and broader peak with the lowest maximum heat flow, reflecting slower hydration caused by clinker dilution and a greater reliance on later pozzolanic reactions. Although the early heat evolution was reduced, hydration continued over a longer period, which may benefit long-term microstructural refinement.

The **M15CL10** and **M15CL20** mixes display intermediate behaviour. Increasing the replacement level lowers the rate of clinker hydration but extends the hydration process, producing broader peaks and a more gradual decline in heat flow. Such behaviour is generally associated with reduced thermal cracking potential and continued later-age strength development.

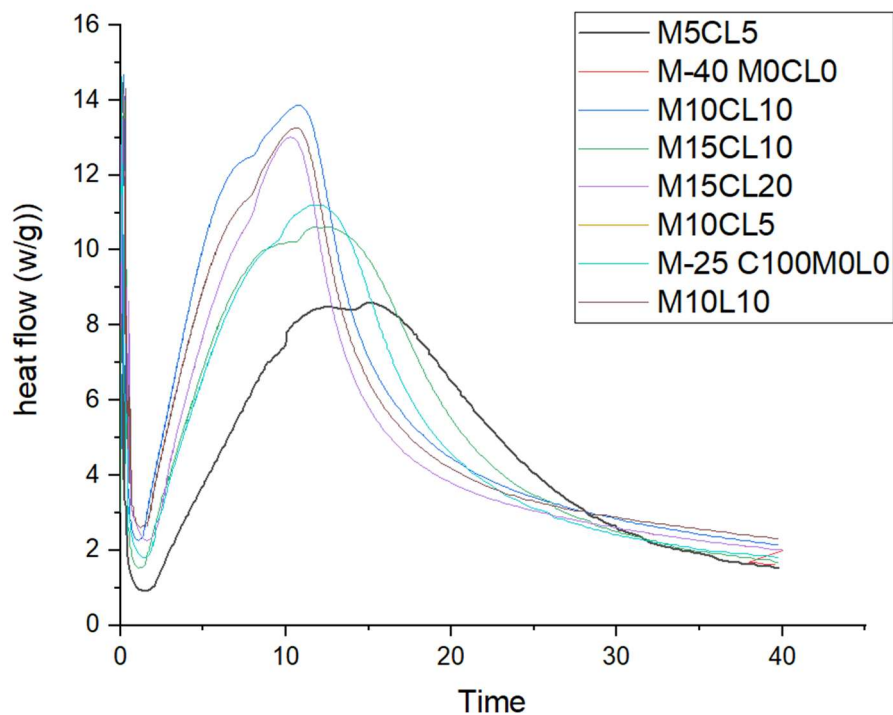


Figure 4.17: Heat flow curves of selected mixes

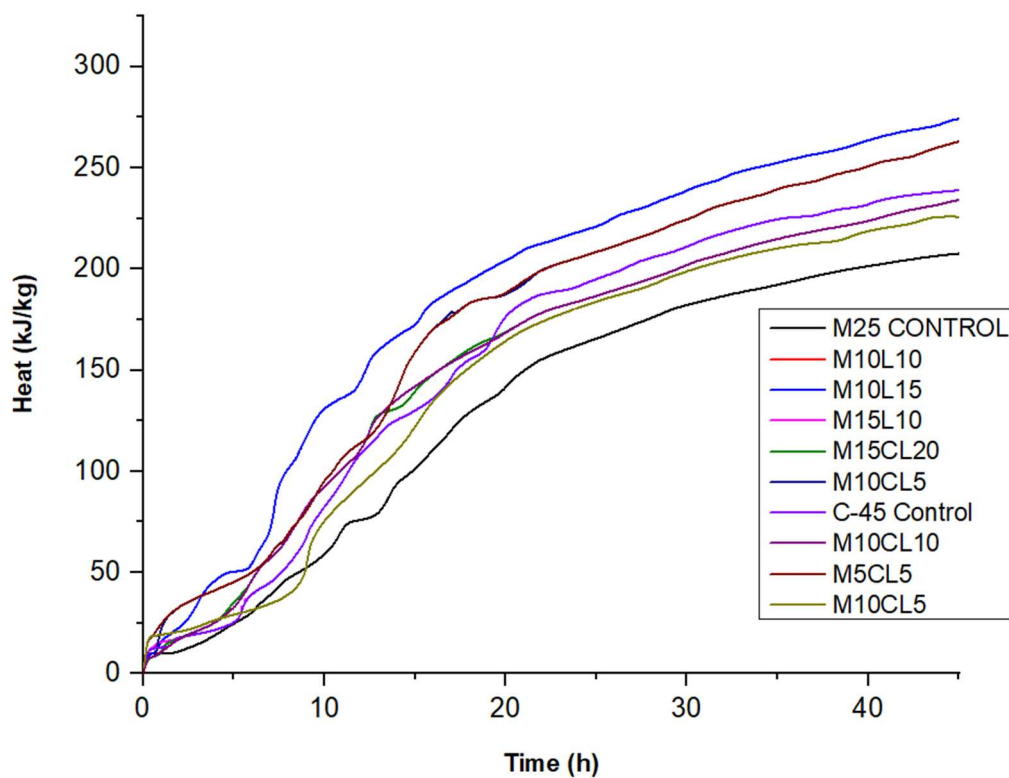


Figure 4.18: Cumulative heat of selected mixes

Table 4.18: Comparative cumulative heat of selected mixes

Mix	Heat at 12 h, approximate (kJ/kg)	Heat at 24 h, approximate (kJ/kg)	Heat at 45 h, approximate (kJ/kg)	Relative hydration response
M25-C100M0L0 (control)	75	158	207	Lowest cumulative heat
M25-C80M10L10	105–110	205	262	Very high and sustained hydration
M25-C75M10L15	140	220	273	Highest cumulative heat
M25-C75M15L10	105	190	238	High hydration
M40-M15CL20	125	180	230–233	Moderate-to-high hydration
M40-M10CL5 (dark blue curve)	110	181	232–235	Moderate-to-high hydration
M40-M0CL0 (control)	100	190	238–240	High hydration
M40-M10CL10	105	180	230–233	Moderate-to-high hydration
M40-M5CL5	110	205	262	Second-highest heat release
M40-M10CL5 (olive curve)	80–85	180	224–226	Moderate cumulative heat

The cumulative heat curves demonstrate that the incorporation of lime sludge, calcined lime sludge and metakaolin considerably altered the hydration behaviour of the cementitious systems. Among all the mixtures, M25-C75M10L15 exhibited the highest total heat release, reaching approximately 273 kJ/kg at 45 h. Its steep increase between approximately 6 and 15 h indicates a strong acceleration of hydration, which may be attributed to the nucleation and filler effects of the finely divided supplementary materials and the availability of reactive calcium and aluminosilicate phases.

M25-C80M10L10 and M40-M5CL5 produced the next-highest cumulative heat values, approximately 262 kJ/kg at 45 h. These mixtures appear to provide an effective balance between the retained clinker content and the stimulation of hydration by the supplementary constituents. In contrast, the M25 control exhibited the lowest cumulative heat, approximately 207 kJ/kg, indicating comparatively slower and less extensive hydration during the monitored period.

The mixtures containing greater quantities of calcined lime sludge, including M40-M15CL20 and M40-M10CL10, showed intermediate cumulative heat values. Their lower heat release relative to M25-C75M10L15 can be attributed partly to clinker dilution. However, the continued increase in heat beyond 24 h indicates that hydration and secondary pozzolanic reactions remained active. Such sustained reaction may contribute to later-age strength development and pore refinement, even where the initial heat evolution is lower.

Overall, the calorimetry results suggest that **moderate replacement (about 10% of each material, e.g., M40-M10CL10 and M25-C80M10L10)** provides a favourable balance between hydration acceleration and sustained reaction, whereas higher replacement levels reduce the maximum hydration rate but maintain ongoing hydration through secondary pozzolanic activity. Since calorimetry measures the overall heat release and cannot by itself distinguish clinker hydration from the pozzolanic reaction, these interpretations are indicative.

4.18 X-ray Diffraction (XRD) Analysis

X-ray diffraction patterns of the CLS–MK concrete mixtures M0CL0, M5CL5, M10CL10, M15CL10, M15CL15 and M15CL20 showed broadly similar mineralogical characteristics. The diffractograms were dominated by an intense reflection at approximately 26.6–26.8° 2 θ , accompanied by reflections near 20.9°, 36.6°, 39.5°, 42.5°, 50.2°, 60.0°, and 68.3°, which were attributed mainly to crystalline quartz originating from the siliceous aggregate. A peak near 29.4–29.5° was assigned to calcite, indicating the presence of limestone-bearing constituents and/or carbonation products. Weak reflections around 18.0° and 34.1° were consistent with portlandite formed during cement hydration, while minor low-angle features between approximately 9° and 12° may be associated with ettringite or gypsum. No major shift in the principal quartz peak was observed among the mixtures, indicating that the incorporated materials did not produce a substantial change in the dominant crystalline phase assemblage. Variations in peak intensity among the mixtures should be interpreted cautiously because they may also reflect differences in sample preparation, aggregate distribution, particle orientation, and counting statistics. The peak positions and probable phases of each mix are listed in Table 4.19. Because C–S–H and C–A–S–H are poorly crystalline, they cannot be identified directly from these patterns; the XRD results therefore confirm the crystalline phases present (quartz, calcite and portlandite) but provide only indirect information on gel formation, for example through the relative intensity of the portlandite reflections.

Table 4.19: XRD peak positions and probable phases of selected mixes

Sample	Major Peak Positions (2 θ , °)	Likely Phase
M0CL0	20.99, 26.76 , 36.66, 42.60, 50.22, 60.06, 68.24	Quartz dominant with hydrated cement phases
M5CL5	21.00, 26.78 , 36.68, 39.56, 45.92, 50.28, 60.10	Quartz, calcite
M10CL10	20.90, 26.66 , 36.58, 42.46, 50.18, 59.98, 68.34	Quartz with hydrated products
M15CL10	20.96, 26.74 , 29.46, 36.62, 50.20, 60.02, 68.20	Quartz, Calcite, Hydration products
M15CL15	20.86, 26.62 , 39.48, 42.42, 50.14, 59.96	Quartz dominant

M15CL20	20.92, 24.40, 26.72 , 29.46, 36.64, 50.22, 60.00	Quartz, Calcite and hydration phases
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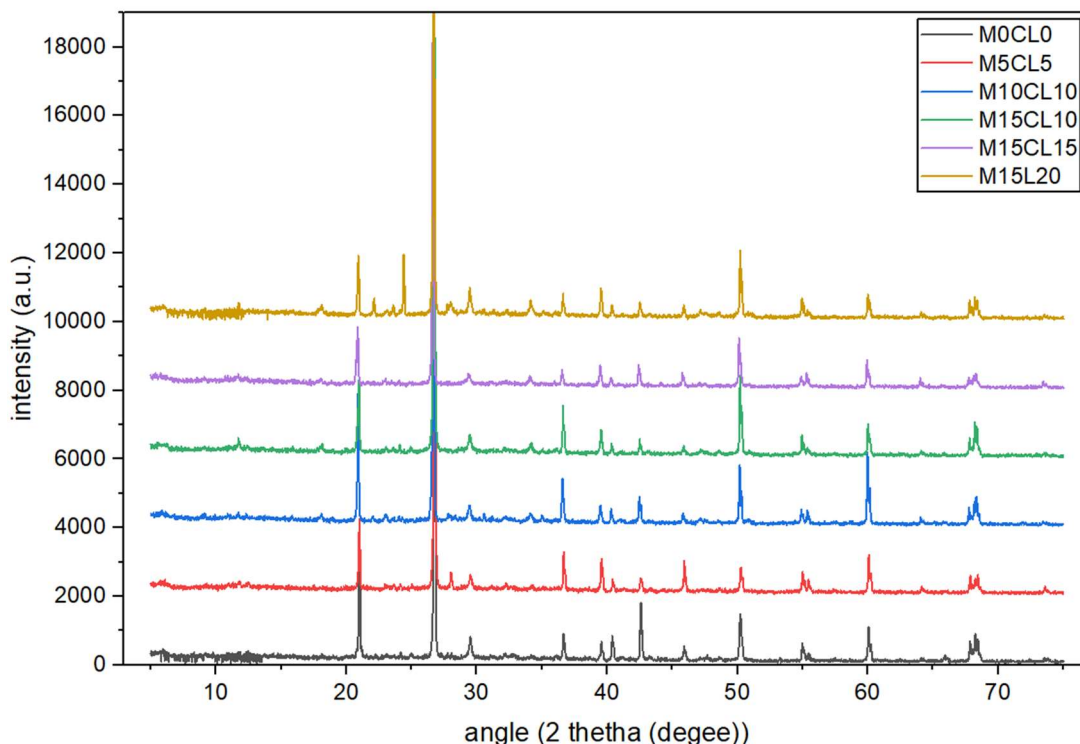


Figure 4.19: XRD patterns of selected mixes

4.19 Thermogravimetric Analysis (TGA)

Table 4.20 and Figure 4.20 present the TGA mass loss of selected CLS–MK mixes between room temperature and 1000 °C. The total mass loss ranged from 8.35% (M15CL10) to 11.11% (M15CL5). The total mass loss combines the loss of free and bound water from C–S–H, ettringite and AFm phases (approximately 105–350 °C), the dehydroxylation of portlandite (400–500 °C) and the decarbonation of calcite (600–800 °C) (Table 3.28), so a lower total loss does not by itself indicate a better-hydrated system. The lower total mass loss of M15CL10 is consistent with a smaller carbonate fraction and with the consumption of portlandite by the pozzolanic reaction of MK, which would favour a more thermally stable phase assemblage; conversely, the greater mass loss of M15CL5 is associated mainly with a larger carbonate-related fraction (Figure 4.21). A quantitative separation of the bound water, portlandite and

calcite contents (e.g., by the tangent method applied to the DTG curve) would be required to confirm these interpretations.

Table 4.20: TGA mass loss of selected CLS–MK mixes

Mix	Initial Weight (%)	Final Weight (%)	Total Weight Loss (%)
M0CL0	99.829	90.145	9.684
M5CL5	99.874	90.431	9.443
M10CL10	99.827	89.644	10.183
M15CL5	99.824	88.714	11.110
M15CL10	99.786	91.438	8.348
M15CL20	99.792	90.145	9.647

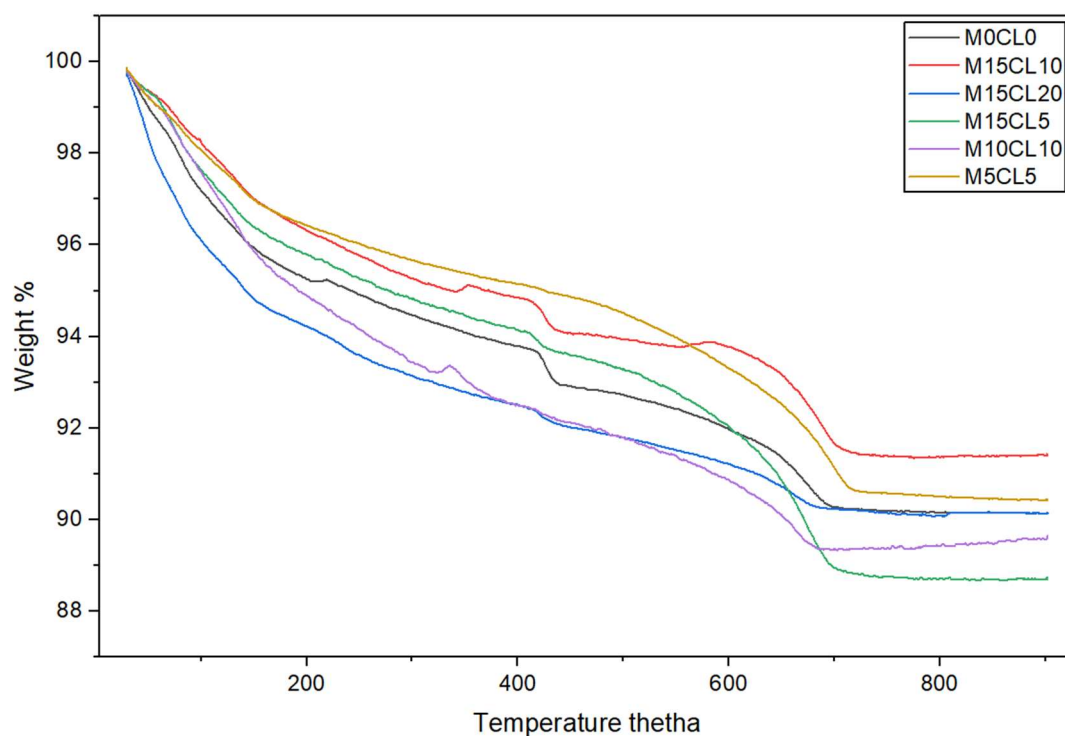


Figure 4.20: TGA curves of selected mixes

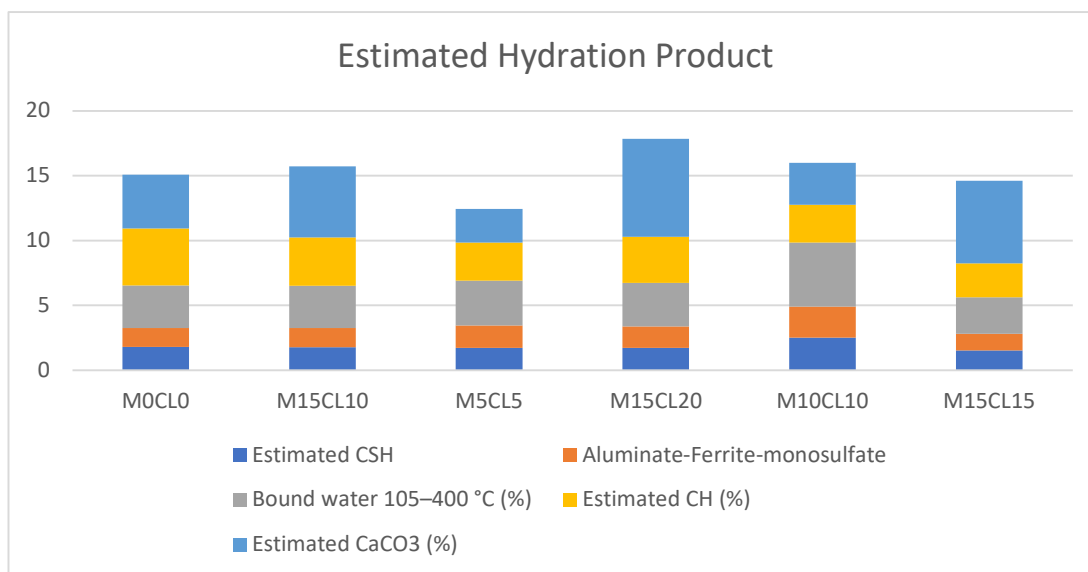


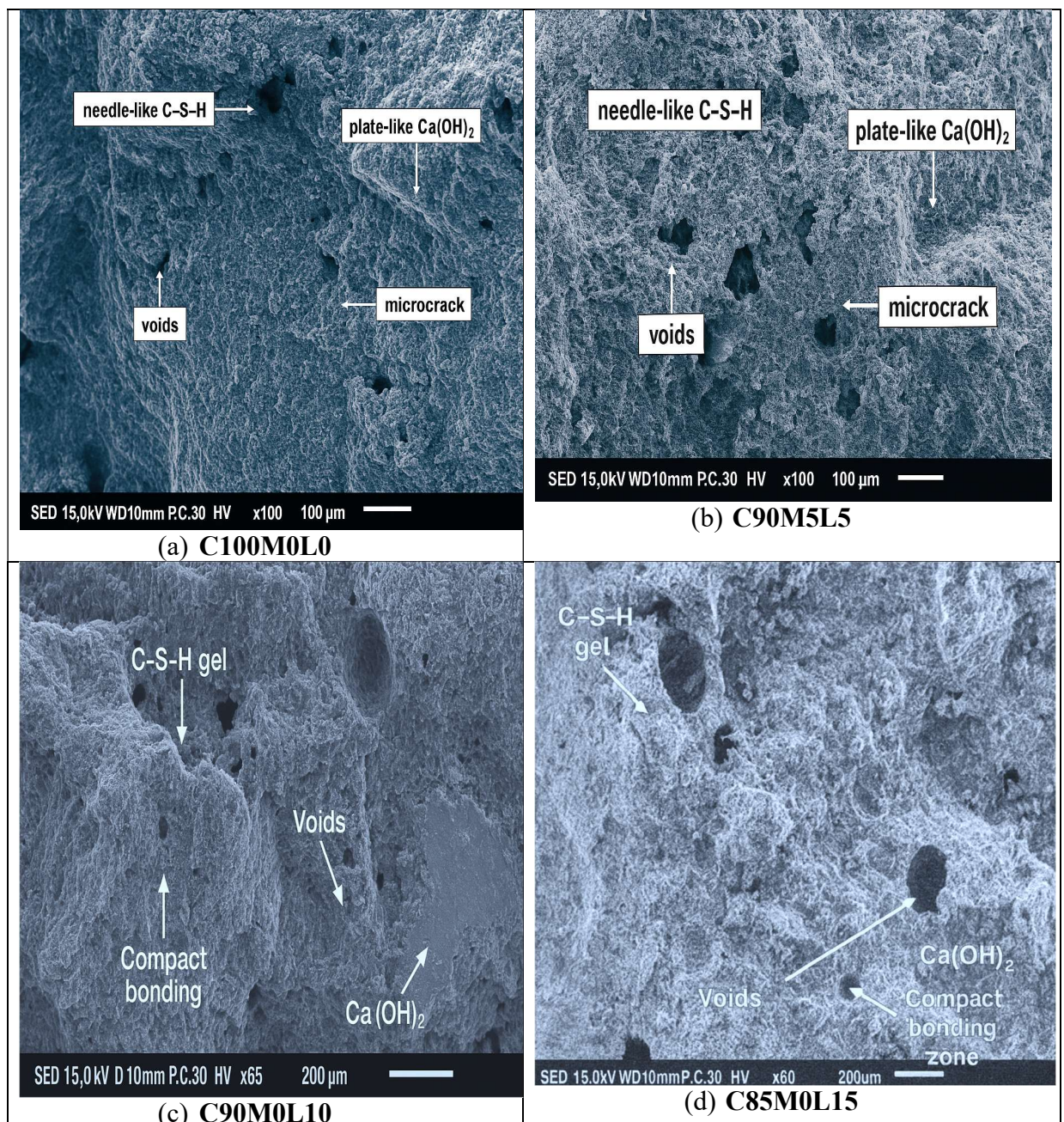
Figure 4.21: Estimated hydration products from TGA

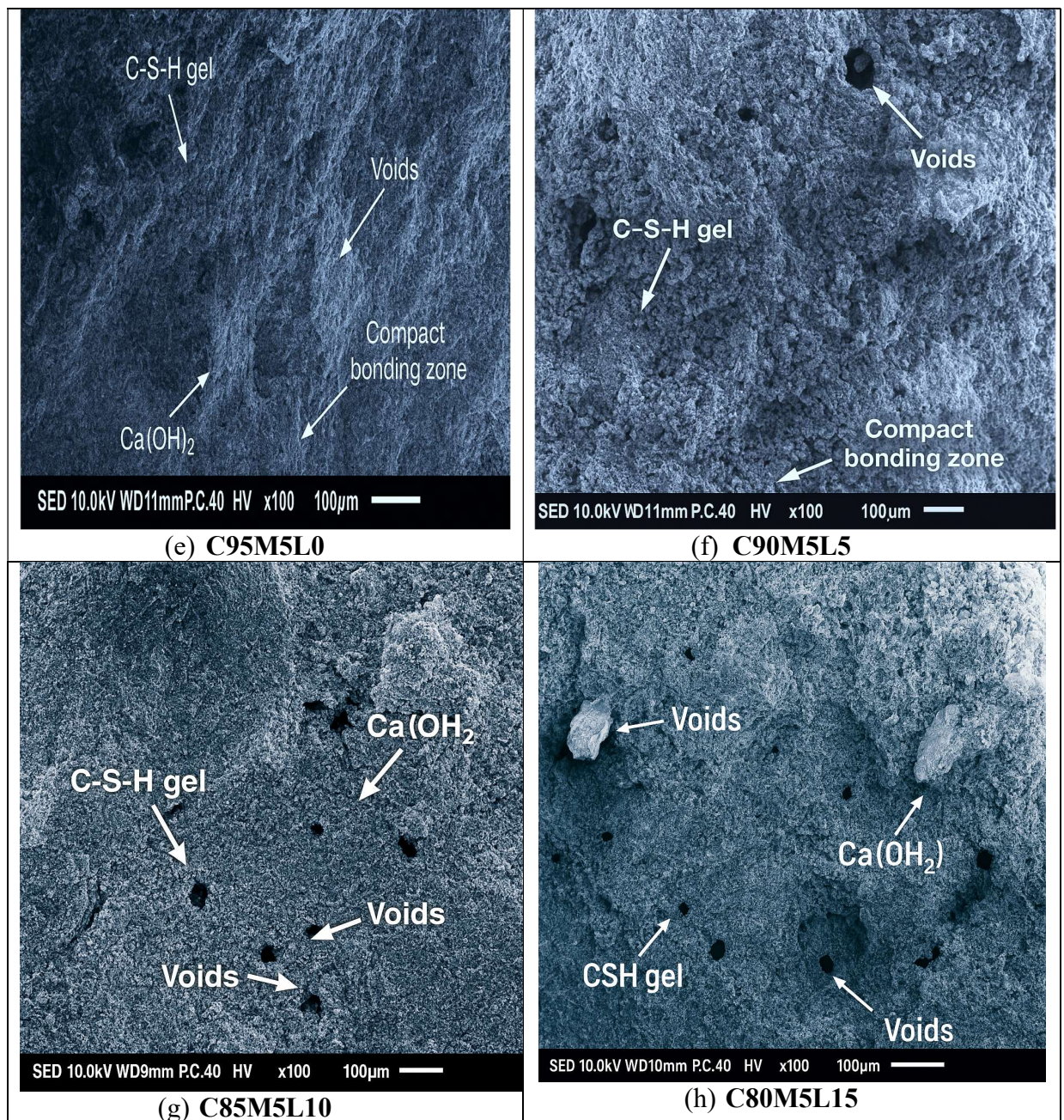
Considering the hydration-related region and the portlandite region together (Figure 4.21), M10CL10 shows the largest hydration-related mass loss, suggesting the most extensive formation of hydration products, whereas M5CL5 shows the largest estimated reduction in portlandite. M15CL10 exhibits the lowest overall mass loss, and M15CL5 the greatest carbonate-related fraction. These estimates are semi-quantitative and are interpreted together with the XRD and SEM/EDS results.

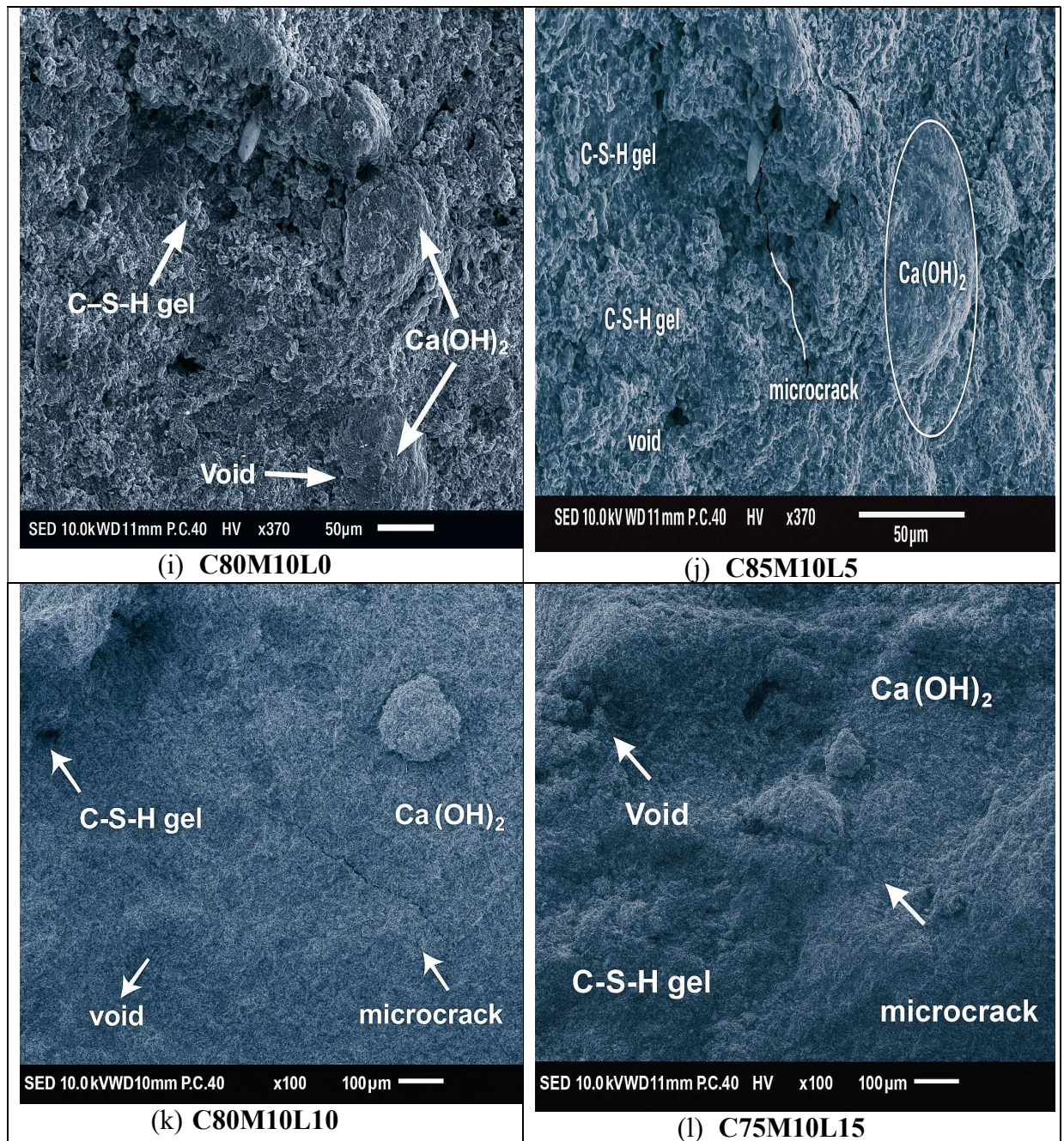
4.20 Scanning Electron Microscopy (SEM)

Figure 4.22 shows the SEM images of the 90-day cured LS–MK mixes. The images reveal the morphology of the hydrated paste, including voids, microcracks, plate-like crystals with the typical morphology of portlandite and dense, poorly crystalline gel regions characteristic of C–S–H. The control mix shows a relatively loose structure with plate-like portlandite crystals, microcracks and voids. The mix C80M10L10 shows fewer visible voids and microcracks and a more continuous gel matrix than the other mixes, whereas the mixes containing LS without MK show more voids and cracks. Beyond about 20% total replacement, the matrix appears more porous, with more microcracks. These observations are consistent with the pozzolanic action of MK and the filler effect of LS at moderate replacement levels (Aygörmez et al., 2020; Kakria et al., 2020; Kljajević et al., 2017; Maheswaran et al., 2015, 2016; S. K. Singh et al.,

2020). The identification of C-S-H and portlandite here is based on morphology and is supported by the EDS spot analyses (Section 4.21) rather than by direct crystallographic evidence, and the images were assessed qualitatively.







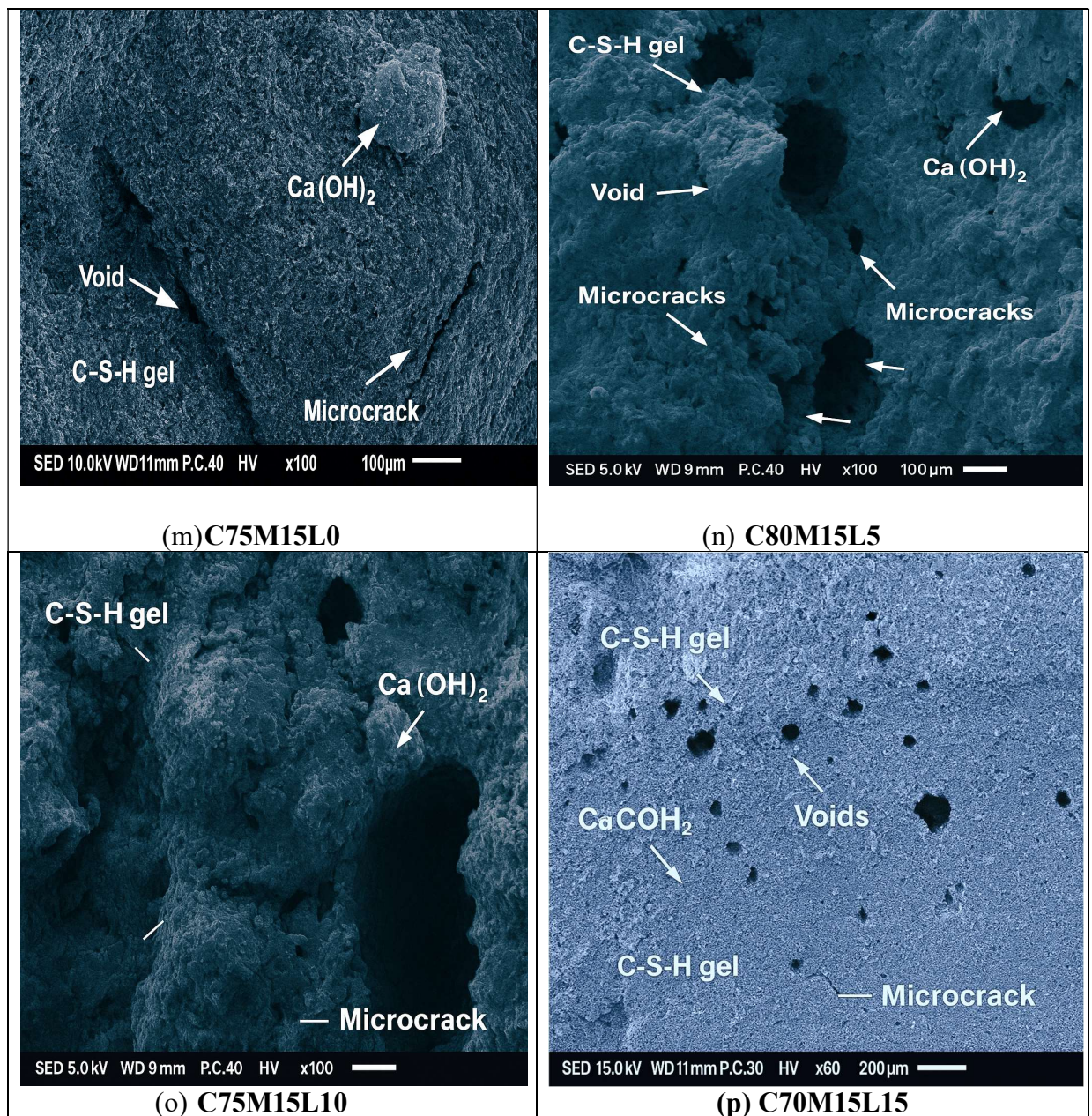


Figure 4.22: SEM images of 90-day cured LS-MK mixes

4.21 Energy Dispersive Spectroscopy (EDS)

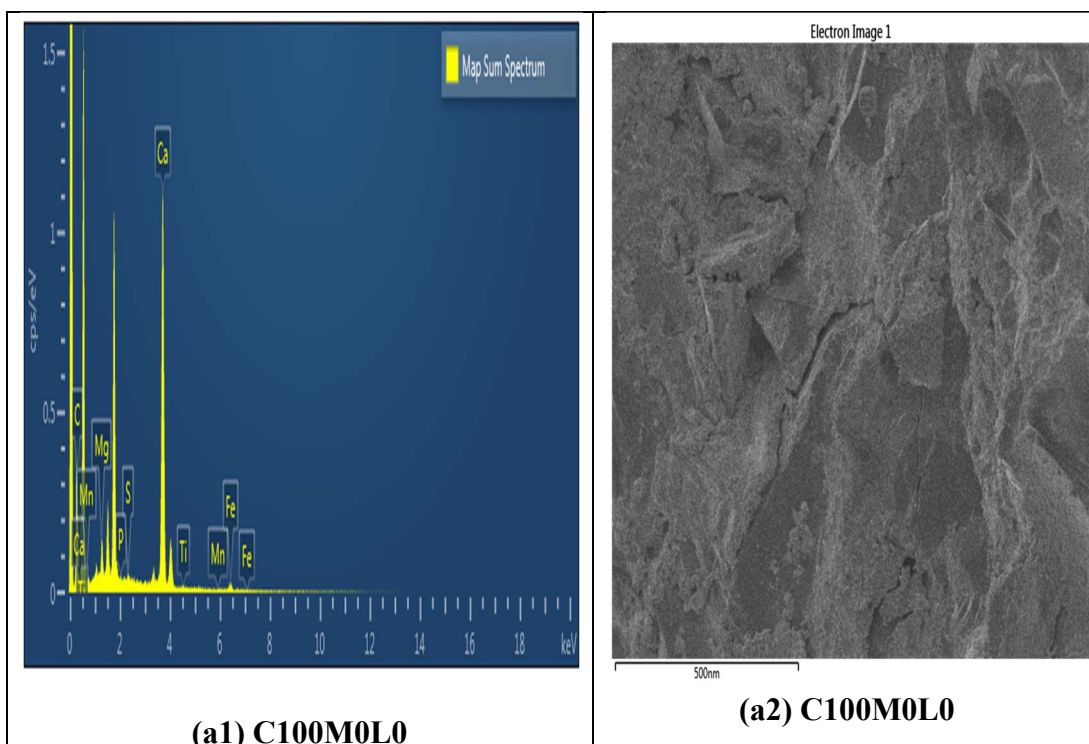
EDS spot analyses provide a semi-quantitative estimate of the elemental composition of the paste. Table 4.21 and Figure 4.23 present the EDS results of the LS-MK mixes, and Figure 4.24 shows those of selected CLS-MK mixes. The Ca/Si ratio ranged from 0.5 to 3.8 and the Si/Al ratio from 3.5 to 26.0. Ca/Si ratios of about 1.0–2.0 are typical

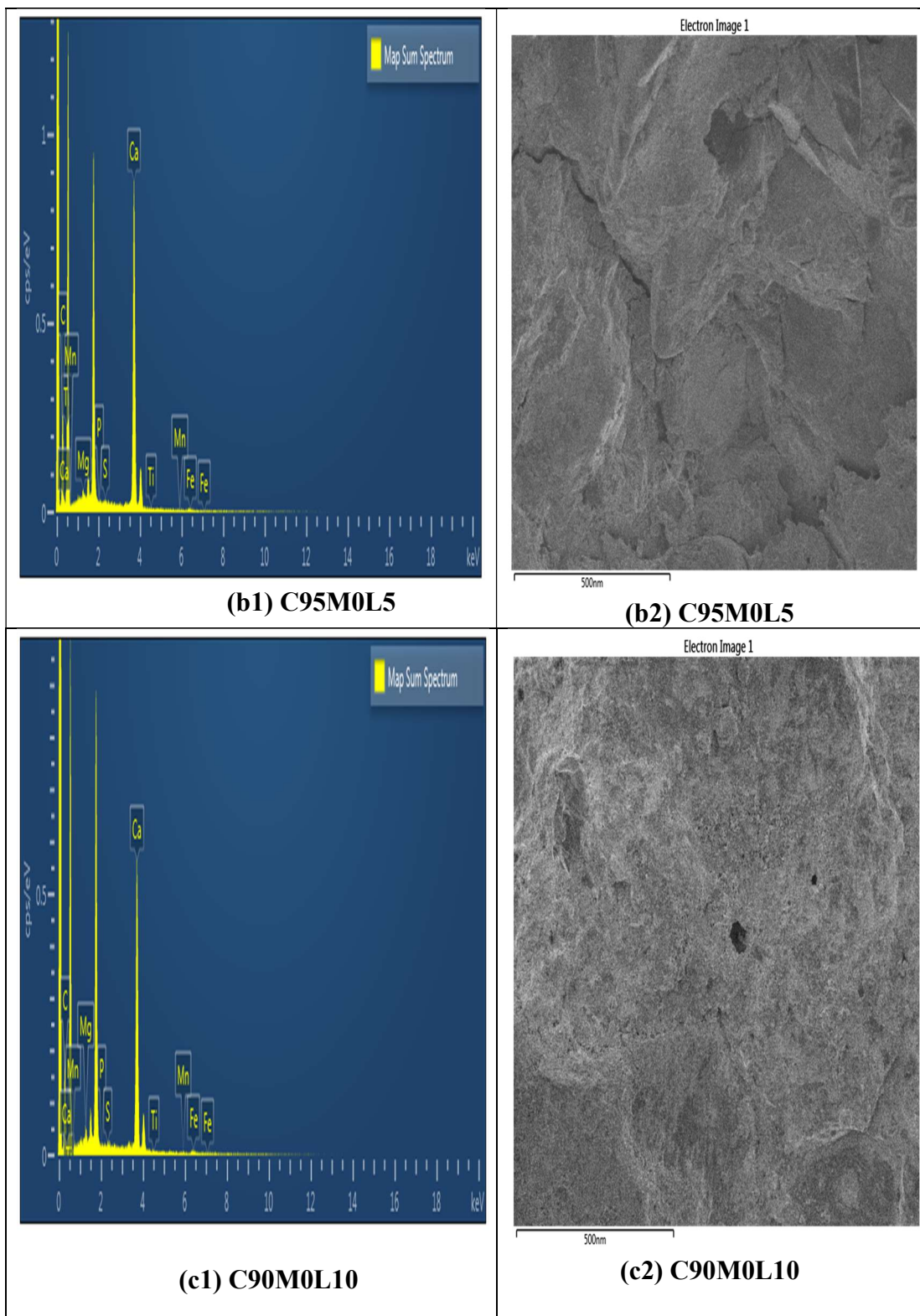
of C–S–H in blended cement pastes and are associated with the incorporation of silica into the gel through the pozzolanic reaction, whereas very high Ca/Si ratios indicate regions rich in portlandite or calcite. The control mix had a Ca/Si ratio of 2.1. The mix C80M10L10 had a Ca/Si ratio of 1.8 and the highest Si/Al ratio (26.0), which is consistent with a calcium silicate hydrate-dominated gel at the analysed spot. The mix C80M5L15, with a high LS content, showed the highest Ca/Si ratio (3.8), consistent with calcium-rich (calcite or portlandite) regions. However, the ratios scatter widely among the mixes and do not follow the replacement level systematically, because spot EDS on concrete is influenced by the local presence of aggregate (quartz contributes Si), unreacted LS (Ca) and MK (Al), and by surface roughness. The EDS results are therefore consistent with, but do not by themselves prove, the formation of additional C–S–H/C–A–S–H gel, and the relationship between the atomic ratios and strength should be regarded as qualitative (Aouan et al., 2023; Eckert et al., 2025; Gómez-Casero et al., 2022; Guo et al., 2024; Maheswaran et al., 2015).

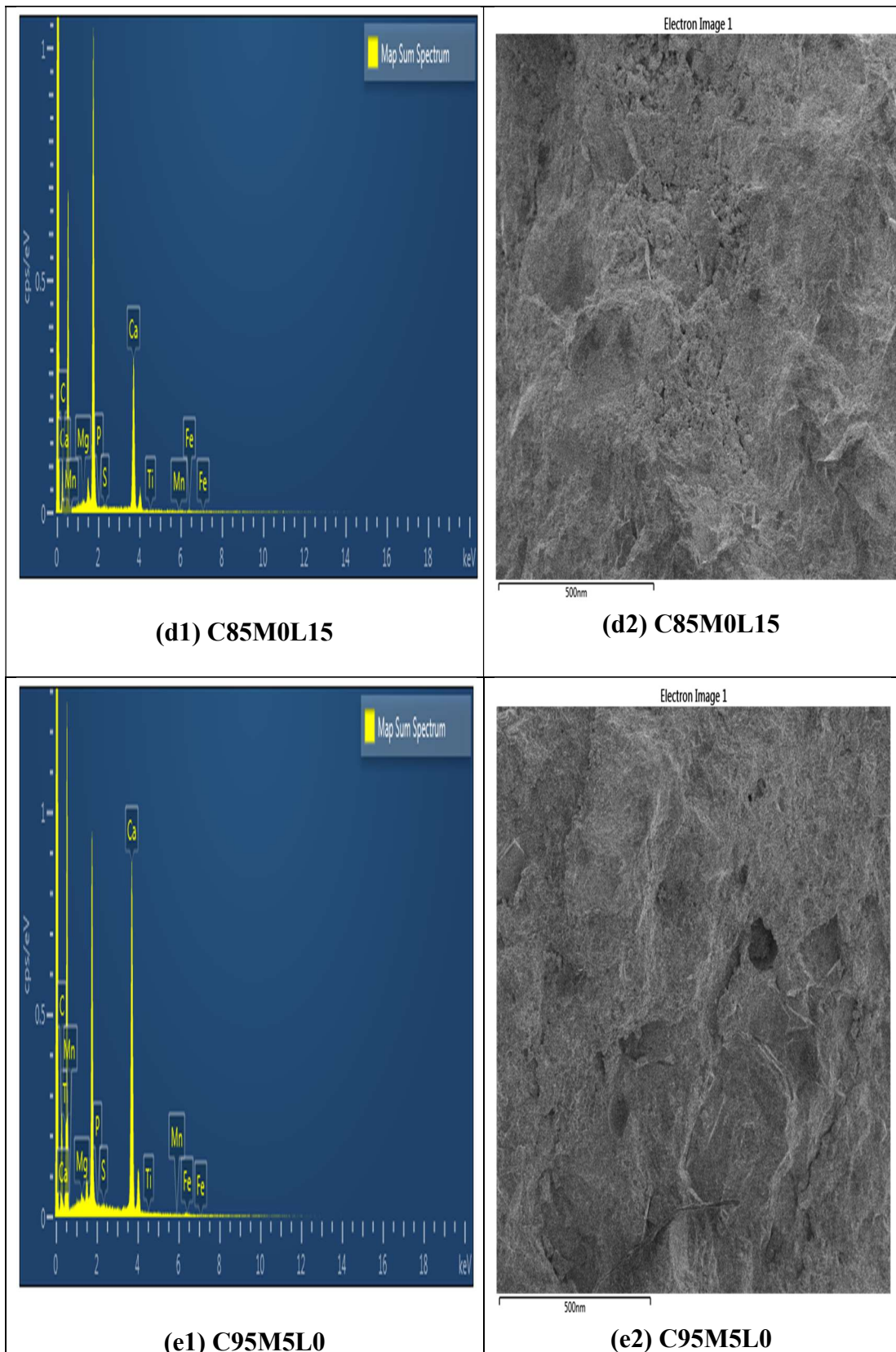
Table 4.21: EDS elemental composition (wt.%) and atomic ratios of LS–MK mixes

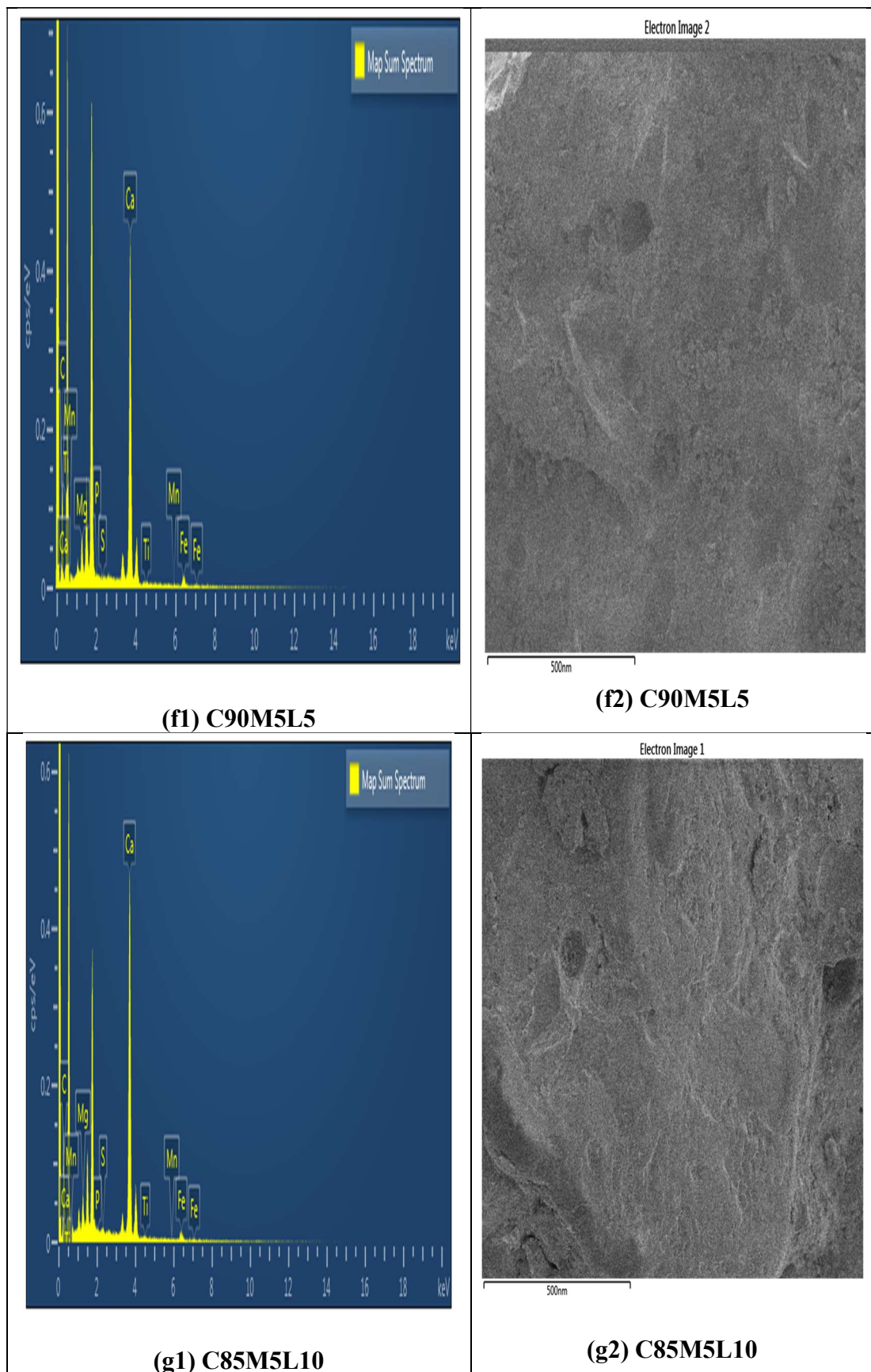
Element (%)	O	Ca	Si	C	N a	Al	Ti	K	M n	Fe	M g	Ca/ Si	Si/ Al
C100M0L0	56.8	20.14	9.8	9.26	0.28	1.81	0.32	0.53	0.35	0.48	0.23	2.1	5.4
C95M0L5	57.9	10.9	10.82	10	0.16	3.1	0.16	0.27	0.1	0.33	0.31	1.0	3.5
C90M0L10	50.23	22.23	20	3.99	0.23	1.16	0.1	0.1	0.17	0.99	0.8	1.1	17.2
C85M0L15	46.07	13.11	23.66	10.03	2.32	2.06	0.47	0.74	0.1	0.27	0.15	0.6	11.5
C95M5L0	50.43	20.11	12.81	8.74	0.47	2.43	0.13	1.39	0.12	2.27	1.11	1.6	5.3
C90M5L5	47	13	24	10	2.3	2	0.42	0.72	0.12	0.24	1.14	0.5	12.0
C85M5L10	54	23	11	10	0.06	0.9	0.27	0.14	0	0.32	0.2	2.1	12.2
C80M5L15	48.22	31.42	8.22	8.02	0.22	1.06	0.01	0.76	0	1.32	0.59	3.8	7.8
C90M10L0	55.35	17.04	18.92	0	0.56	2.73	0	1.15	0	2.81	1.43	0.9	6.9
C85M10L5	49.91	20.66	15.75	10.69	0.15	0.89	0.08	0.42	0	0.72	0.62	1.3	17.7

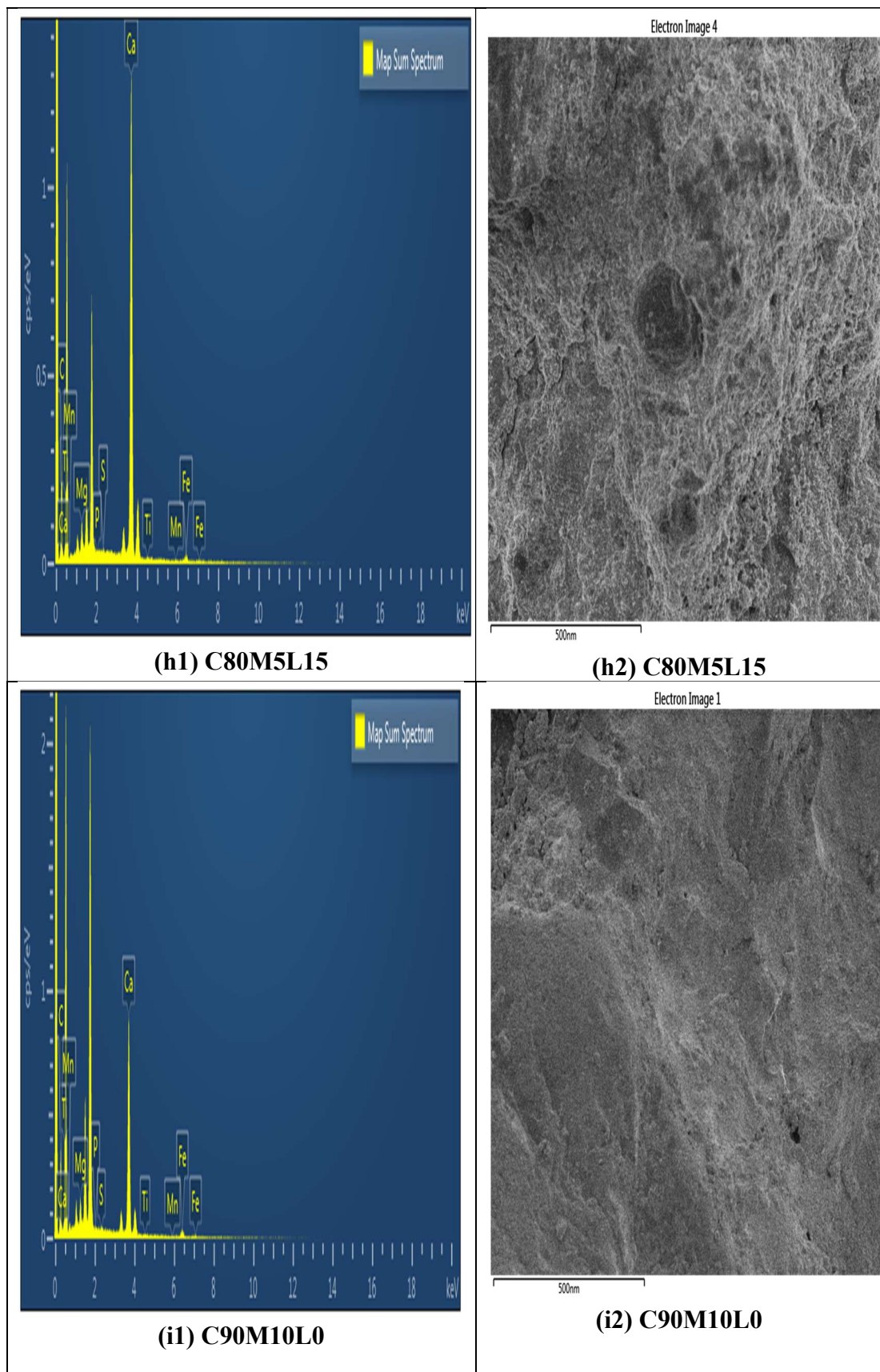
C80M1 0L10	50. 33	25. 58	14. 03	8.2	0. 08	0. 54	0. 03	0. 05	0. 05	0. 45	0. 3	1.8	26. 0
C75M1 0L15	50. 33	22. 33	20. 1	4.0 9	0. 13	1. 06	0	0	0. 07	0. 89	0. 6	1.1	19. 0
C85M1 5L0	48. 49	30. 65	9.0 3	6.7 3	0. 06	1. 53	0. 28	0. 4	0. 05	1. 24	1. 21	3.4	5.9
C80M1 5L5	57	20. 34	10	9.4 6	0. 08	1. 61	0. 12	0. 33	0. 15	0. 48	0. 43	2.0	6.2
C75M1 5L10	47. 04	25. 13	9.7 1	10. 13	0. 9	2. 36	0. 18	1. 05	0	2. 33	0. 96	2.6	4.1
C70M1 5L15	58	11	10. 92	10. 1	0. 06	3	0. 06	0. 17	0	0. 23	0. 21	1.0	3.6

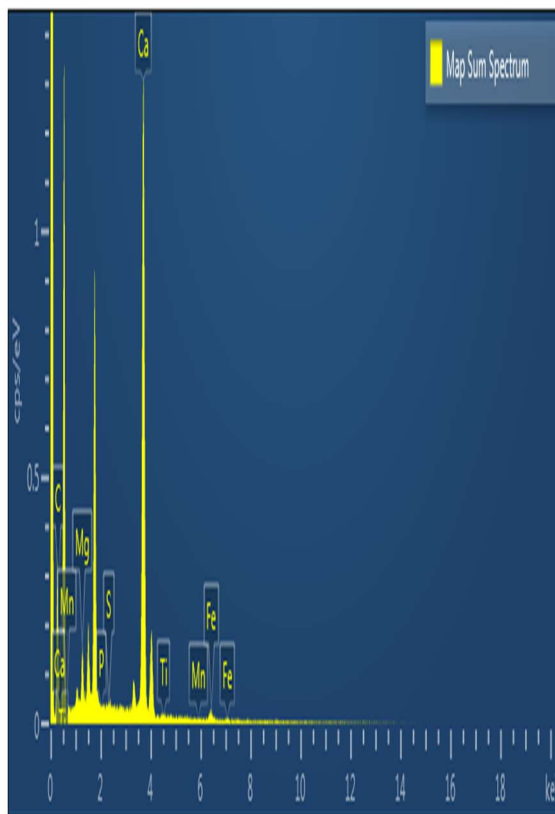




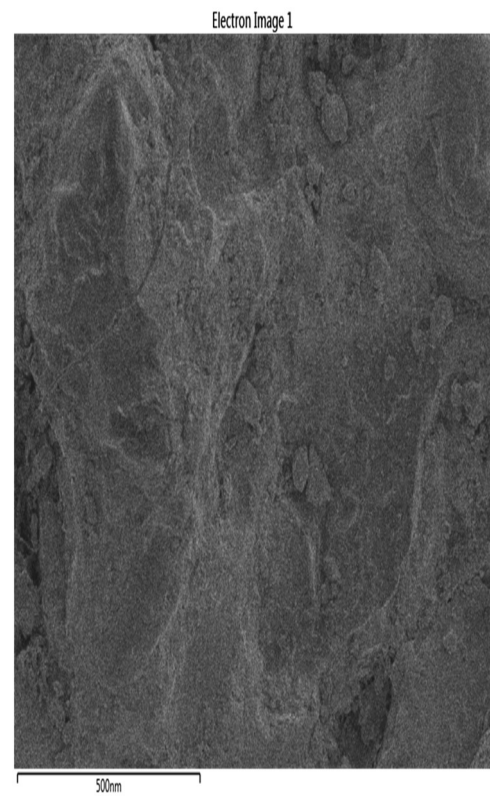




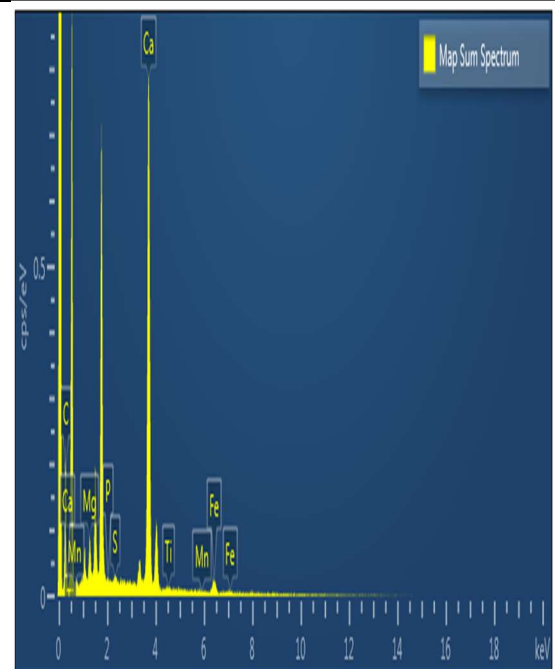




(j1) C85M10L5



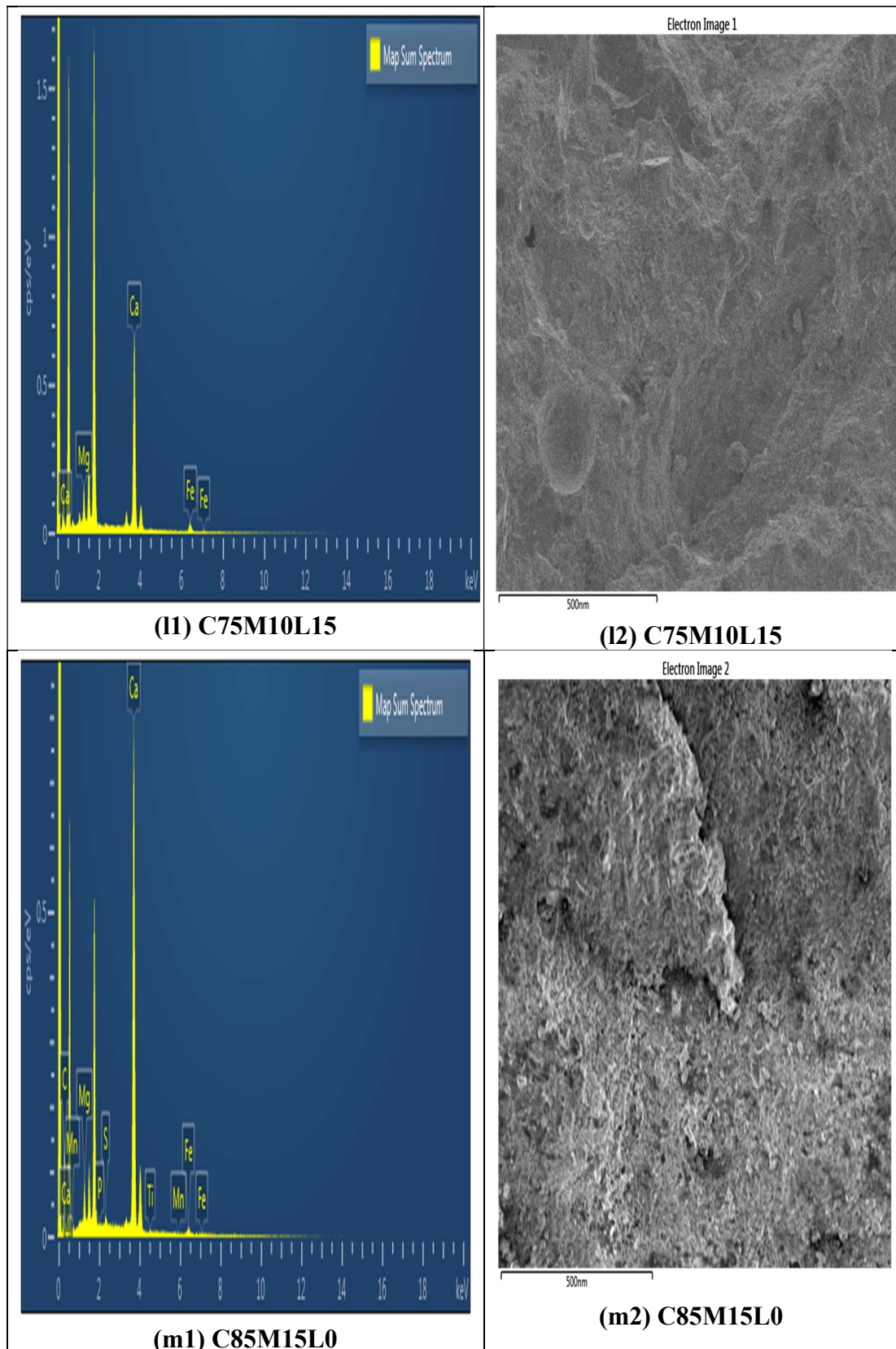
(j2) C85M10L5

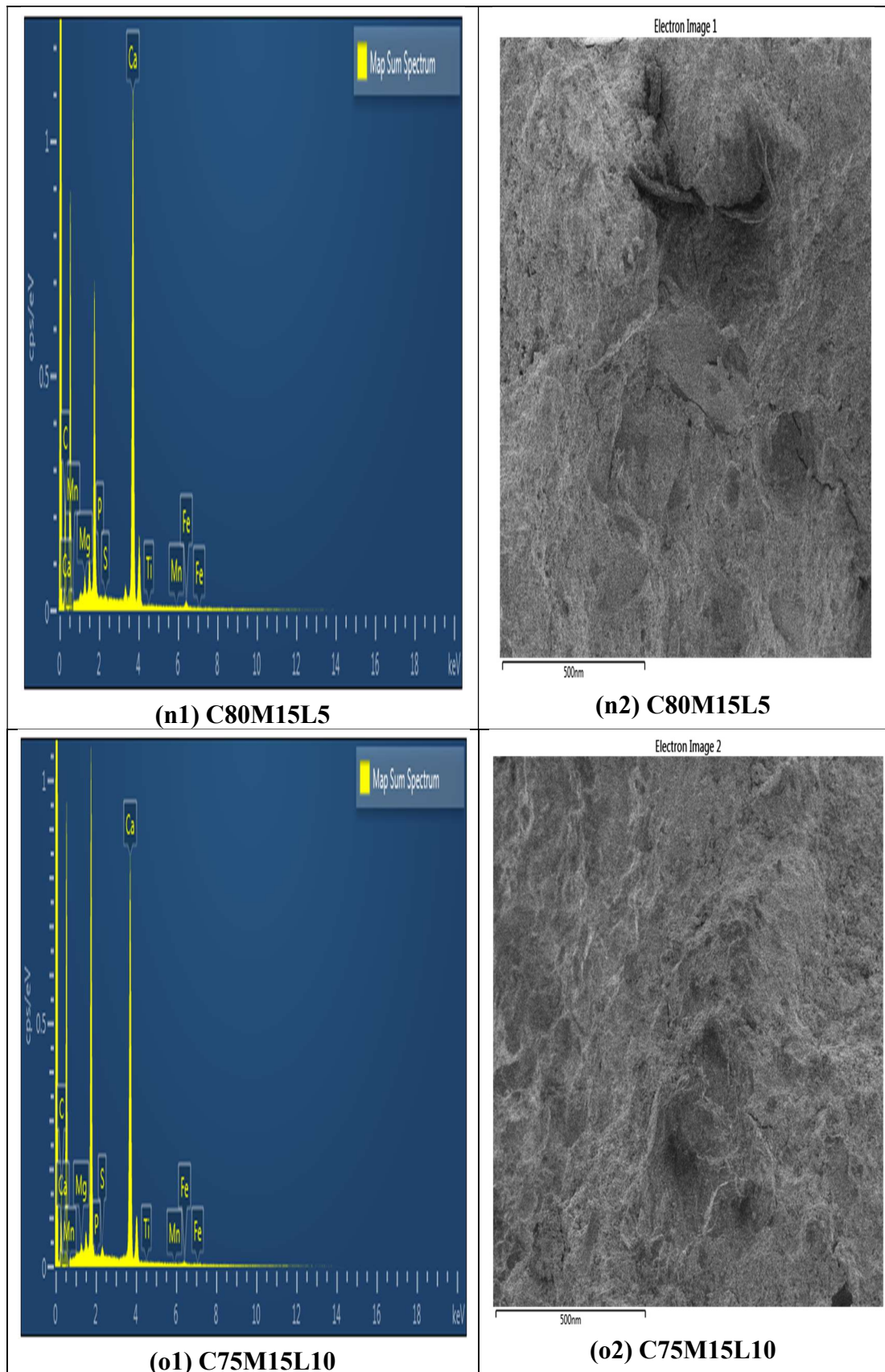


(k1) C80M10L10



(k2) C80M10L10





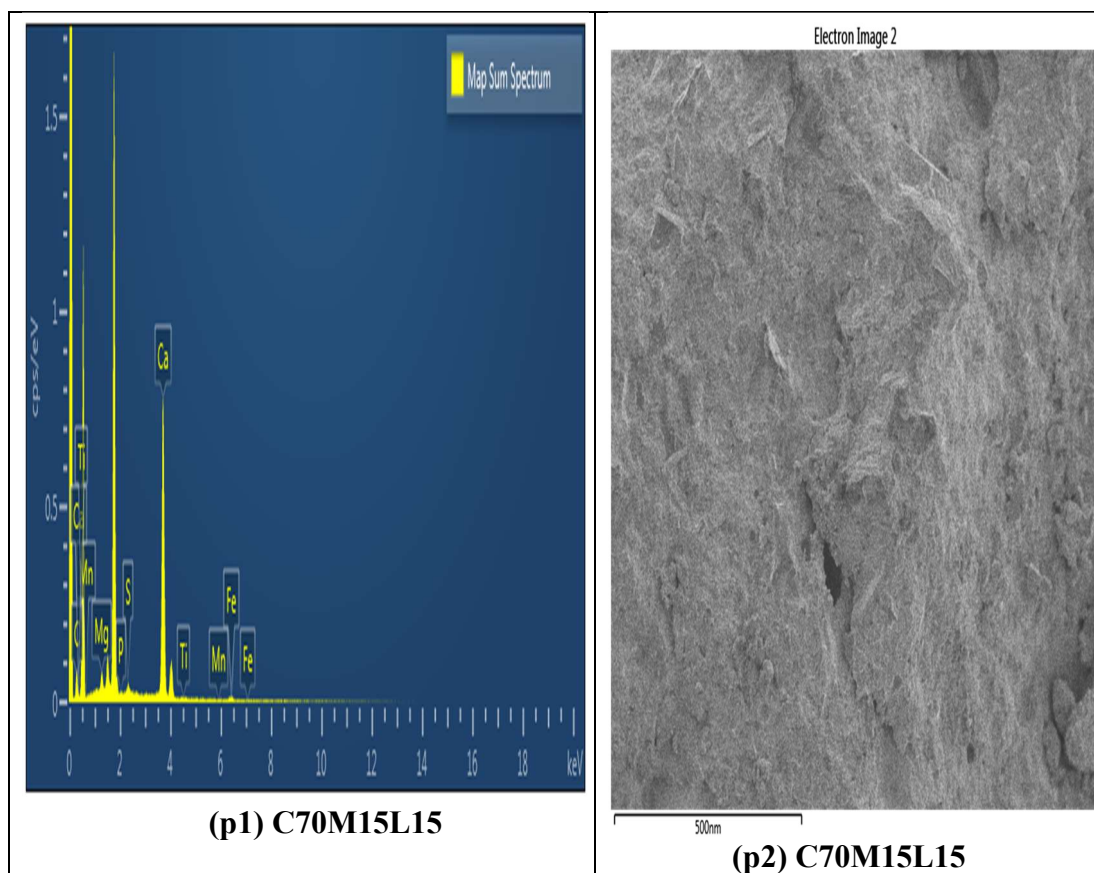
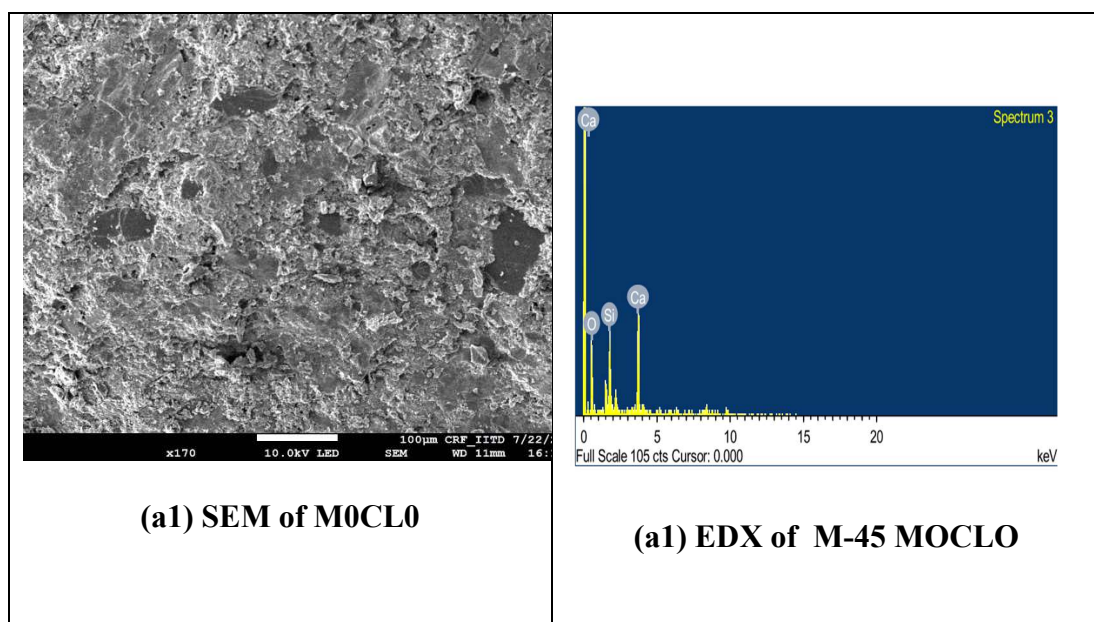
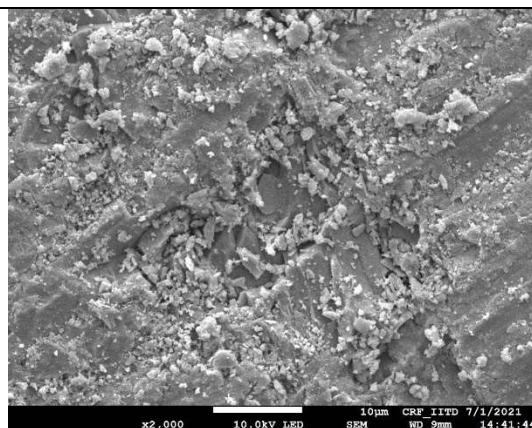
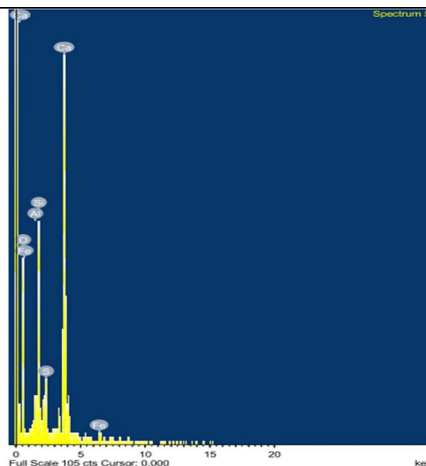


Figure 4.23: SEM micrographs and EDS spectra of LS-MK mixes

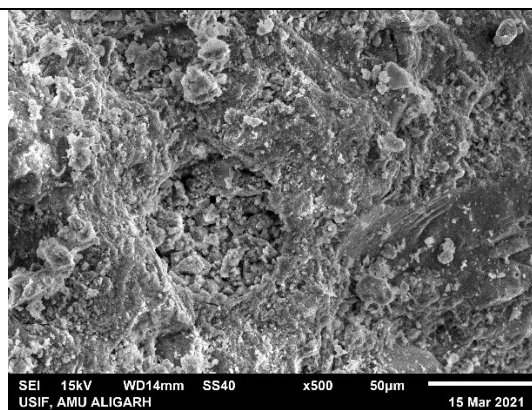




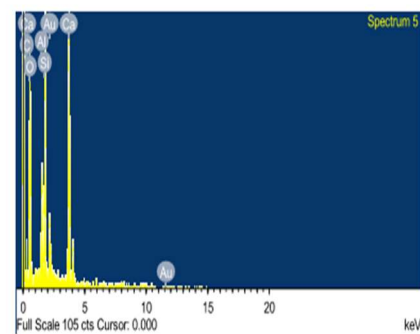
(b1) SEM of M5CL5



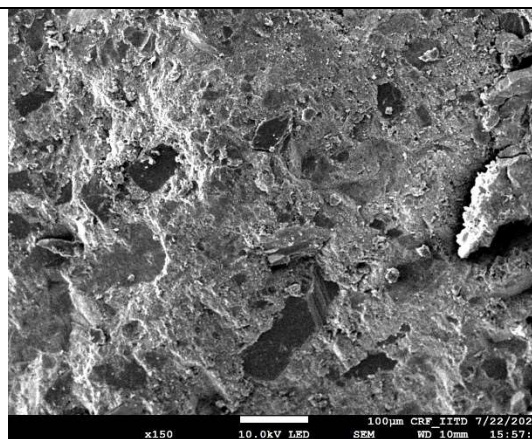
(b2) EDX of M5CL5



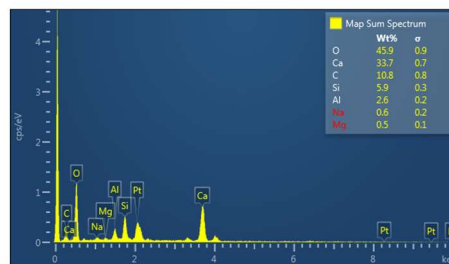
(c1) SEM of M10CL10



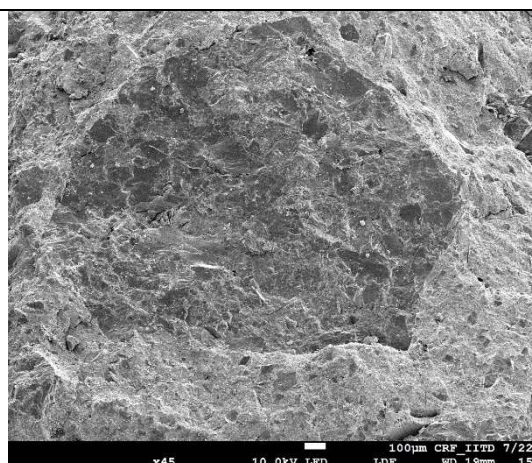
(c2) EDX of M10CL0



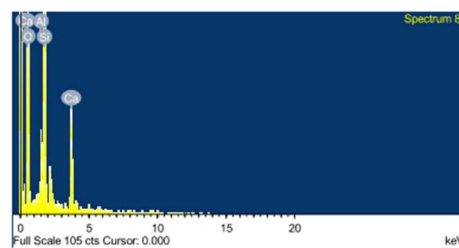
(d1) SEM of M15CL10



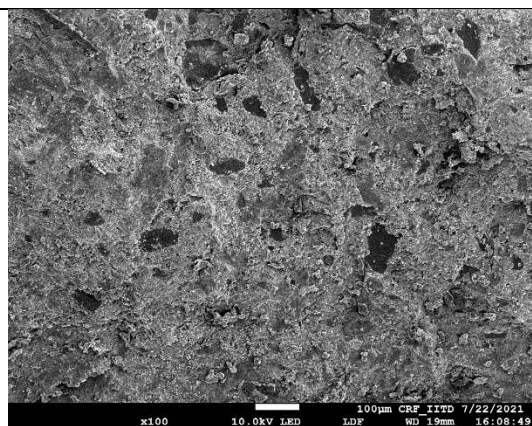
(d2) EDS of M15CL10



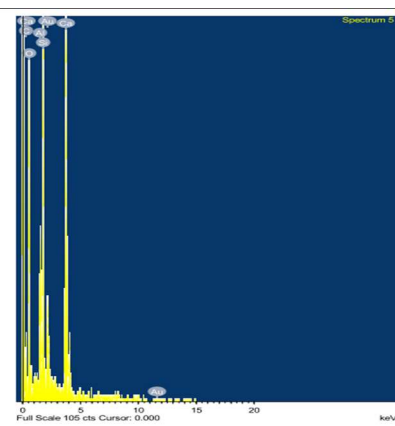
(e1) SEM of M15CL20



(e2) EDS of M15CL20



(f1) SEM of C-25 C100M0L0



(f2) SEM of C-25 C100M0L0

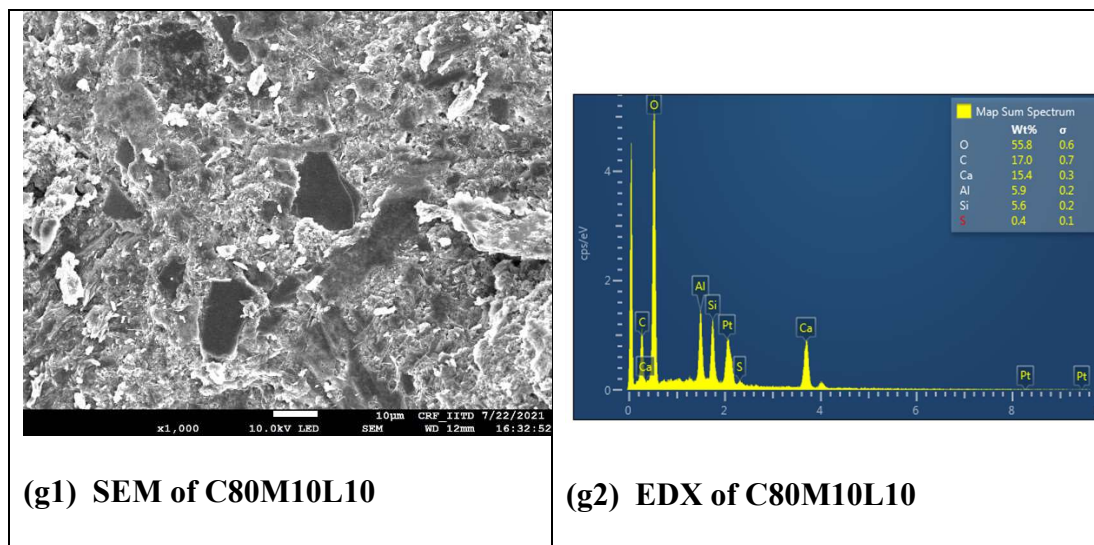


Figure 4.24: SEM micrographs and EDS spectra of CLS–MK mixes

4.22 Statistical Analysis of the Results

4.22.1 Regression Analysis using SPSS

To examine the relationships between the physical properties (density and water absorption) and the mechanical properties of the LS–MK concrete, linear regression analysis was performed in SPSS using the 28-day results of the 16 superplasticised mixes (Tables 4.6, 4.7, 4.9, 4.11 and 4.13).

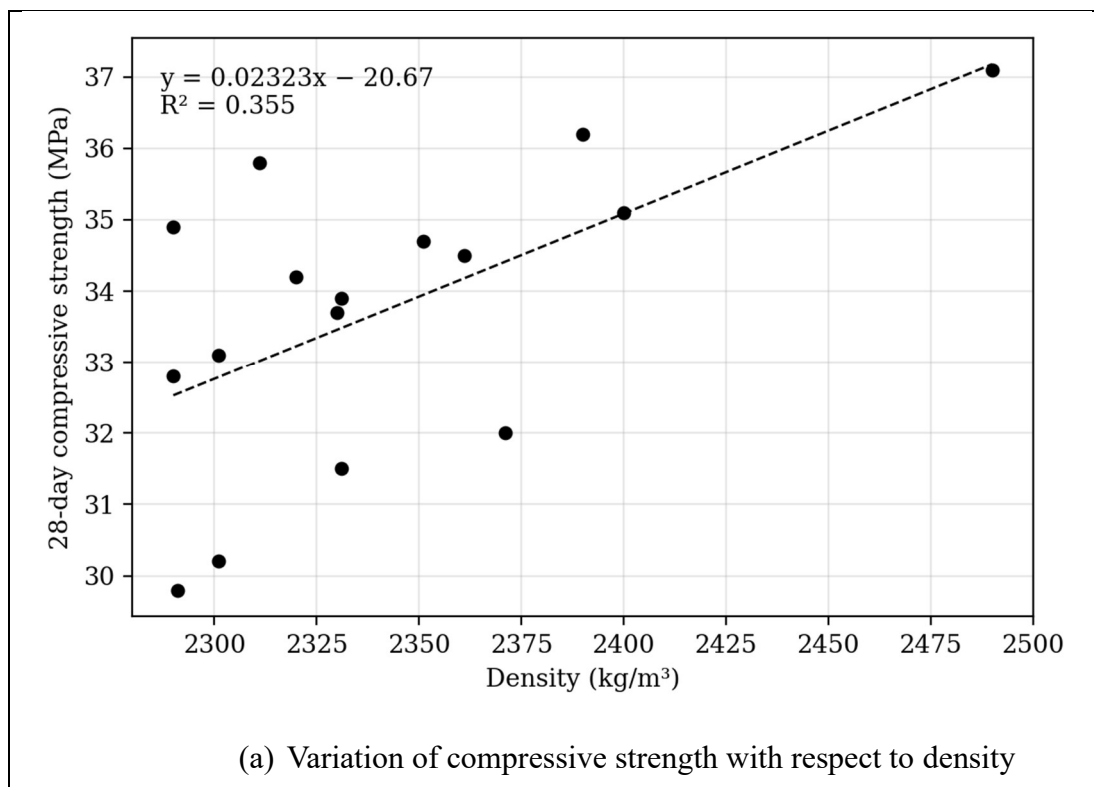
4.22.1.1 Correlation between Physical and Mechanical Characteristics

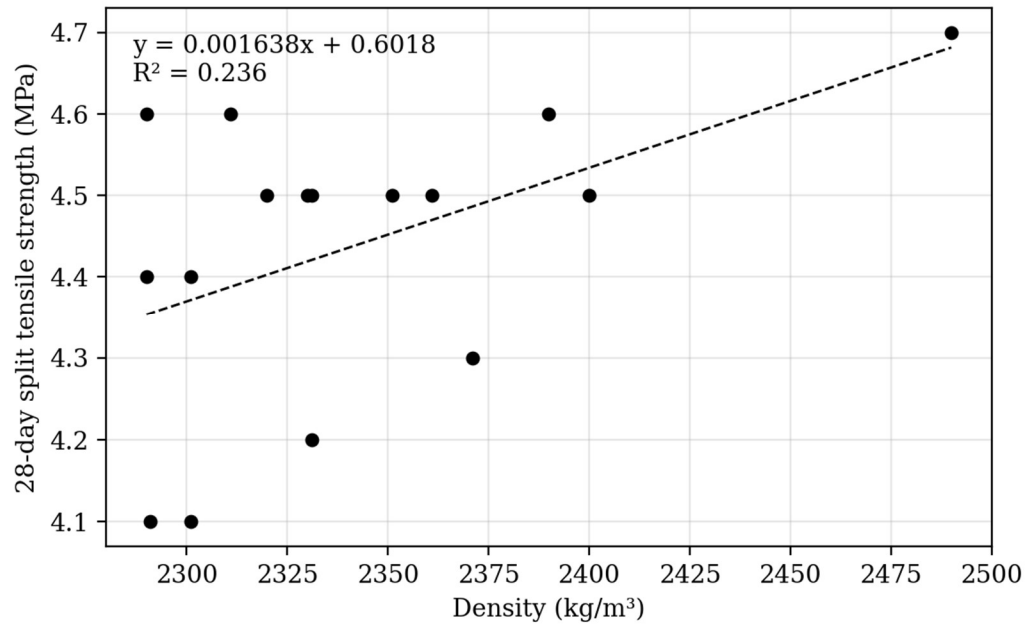
The relationships between density and the strength properties were positive across the blends. As the LS and MK contents increased up to 10% each, the density increased by about 8% (from 2301 to 2490 kg/m³), accompanied by increases of 22.8% in compressive strength and 14.6% in split tensile strength. This trend can be attributed to the reduced pore volume and denser particle packing, which restrict microcrack propagation under load (Bhagath Singh & Subramaniam, 2019; Kong et al., 2008; Koushkbaghi et al., 2019; Venu et al., 2024).

However, the regression analysis showed that these correlations are only moderate: density explained 24–36% of the variance of the 28-day strengths ($R^2 = 0.36, 0.24$ and 0.26 for compressive, split tensile and flexural strength), and water absorption explained 29% of the variance of compressive strength ($R^2 = 0.29$). A stronger inverse

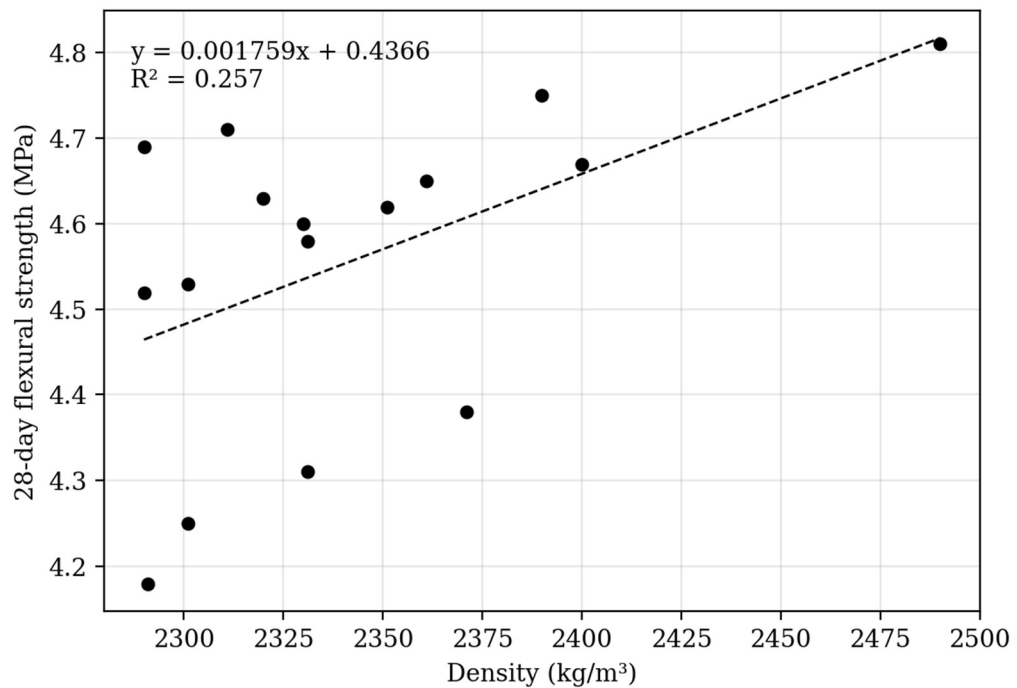
2

correlation was found between water absorption and density ($R^2 = 0.54$). These results indicate that density and water absorption are associated with strength but are weak predictors on their own; the reactivity of the binder, governed mainly by the MK content (Section 4.22.2), is the dominant variable. Figure 4.25 and Table 4.22 show the regression plots and the correlation analysis between the physical and mechanical properties of the LS–MK concrete.

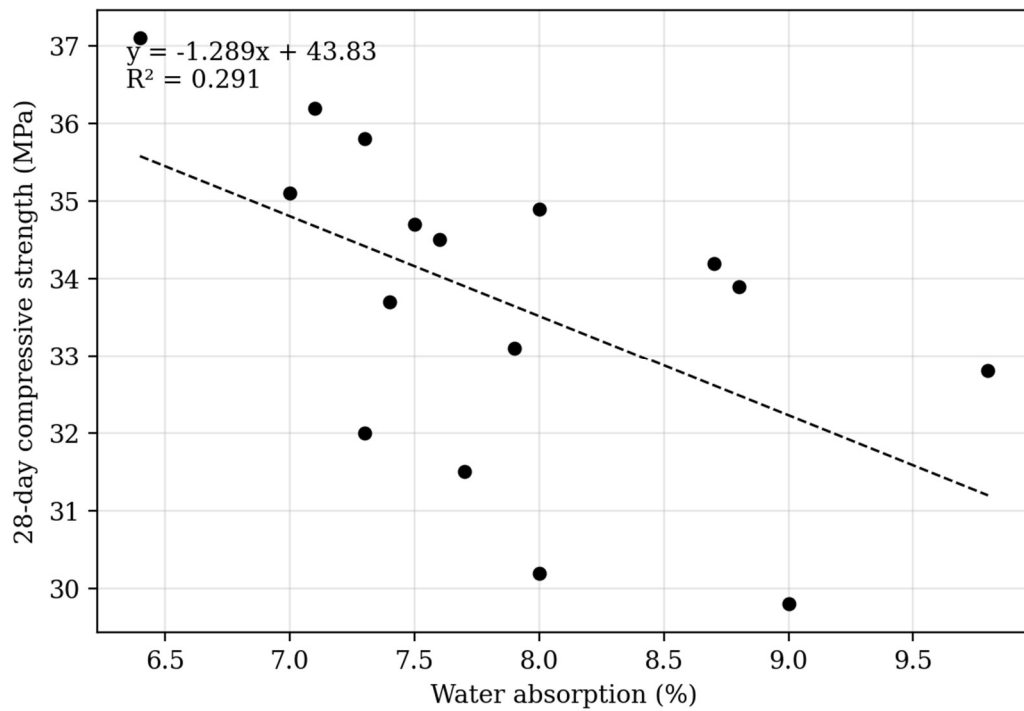




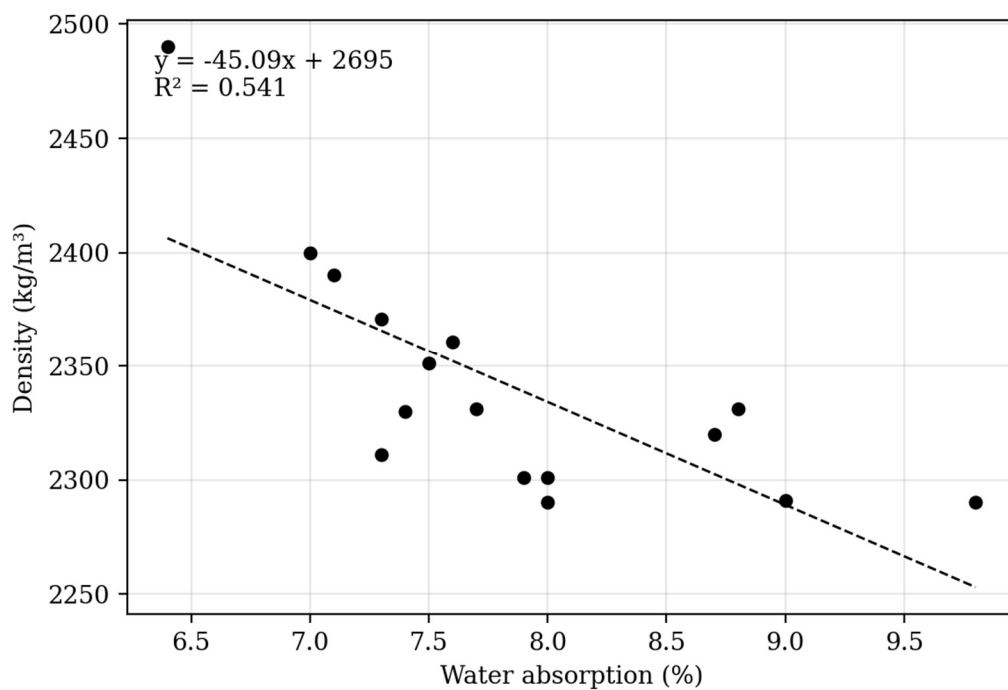
(b) Variation of split tensile strength with respect to density



(c) Variation of flexural strength with respect to density



(d) Variation of compressive strength with respect to water absorption



(e) Variation of density with respect to water absorption

Figure 4.25: Regression analysis of LS–MK concrete

Table 4.22: Correlation analysis between physical and mechanical properties of LS–MK concrete

Parameter correlation	Regression equation	R ²	p-value	Relationship
Density (ρ) vs. compressive strength (CS28)	$CS28 = 0.0232\rho - 20.67$	0.36	0.015	Moderate positive
Density (ρ) vs. split tensile strength (STS28)	$STS28 = 0.0016\rho + 0.60$	0.24	0.056	Weak to moderate positive
Density (ρ) vs. flexural strength (FS28)	$FS28 = 0.0018\rho + 0.44$	0.26	0.045	Weak to moderate positive
Water absorption (WA) vs. compressive strength (CS28)	$CS28 = -1.29WA + 43.83$	0.29	0.031	Weak to moderate negative
Water absorption (WA) vs. density (ρ)	$\rho = -45.09WA + 2694.83$	0.54	0.001	Moderately strong negative

4.22.2 Taguchi Analysis

To systematically quantify the relative influence of metakaolin (MK) and lime sludge (LS) on the mechanical and durability performance of the concrete mixes, the experimental data were re-analysed using the Taguchi method. The original experimental programme already comprised a full 4×4 factorial design with two factors—MK content and LS content—each varied at four levels (0, 5, 10 and 15%). This matrix is equivalent to an L-16 (4^2) orthogonal array, and therefore the same 16 mixes were directly adopted for Taguchi analysis without any additional experimental runs. The control factors and their levels are summarised in Table 4.23.

Table 4.23: Control factors and levels for the Taguchi analysis

Factor	Description	Level 1	Level 2	Level 3	Level 4
A	Metakaolin, MK (%)	0	5	10	15
B	Lime sludge, LS (%)	0	5	10	15

The 16 mixes (C100M0L0 to C70M15L15) already used in the experimental programme form the complete Taguchi design matrix, with each combination of MK–LS levels represented once. For Taguchi analysis, the 28-day mechanical properties - compressive strength, split tensile strength and flexural strength—and the key durability indicators - water absorption, rapid chloride penetration test (RCPT) charge at 56 days and 12-week weight loss in 5% HCl were treated as responses.

For mechanical properties where higher values are desirable, the “larger-the-better” criterion was adopted. For durability indicators where lower values are desirable (water absorption, chloride permeability and acid-induced weight loss), the “smaller-the-better” criterion was used.

Larger-the-better (mechanical properties) was applied to 28-day compressive strength, split tensile strength and flexural strength:

$$S/N = -10\log_{10}\left(\frac{1}{n}\sum_{i=1}^n \frac{1}{y_i^2}\right)$$

Smaller-the-better (durability properties) criteria was applied to water absorption, RCPT charge and 12-week weight loss in 5% HCl:

$$S/N = -10\log_{10}\left(\frac{1}{n}\sum_{i=1}^n y_i^2\right)$$

Here, y_i denotes the measured value and n is the number of observations per run. In this study, each mix was tested once for the Taguchi evaluation, so $n = 1$ for all responses.

With $n = 1$, the larger-the-better S/N ratio reduces to $20 \log_{10}(y)$ and the smaller-the-better ratio to $-20 \log_{10}(y)$. The S/N ratio is therefore a monotonic logarithmic transformation of the single measured response and contains no information on variability (noise); in this study it is used only as a convenient, scale-free metric for ranking the levels of MK and LS. Furthermore, because the L16 array is fully occupied by the two four-level factors, the 9 degrees of freedom of the MK $\times$ LS interaction are used as the error term in the ANOVA. The F-tests in Table 4.25 therefore test the main effects of MK and LS against the interaction, and the main-effect optima must be interpreted with caution wherever the interaction is large.

The S/N ratio for compressive strength increases sharply up to 10% MK and then decreases slightly at 15% MK. A similar trend is observed for LS, with the highest S/N at 10% LS. Thus, Taguchi analysis clearly identifies the combination MK = 10% and LS = 10% as the optimal region for maximising compressive strength, consistent with the experimental trends observed earlier.

For water absorption, a less negative S/N ratio indicates better durability (lower water absorption); the 10% MK level shows the maximum S/N ratio (−16.917 dB), and the 10% LS level also provides the best S/N ratio among the LS levels.

The combination MK10–LS10 therefore also emerges as the optimum for minimising water absorption, and the main-effect plots for water absorption peak at 10% MK and 10% LS. Tables 4.24 and 4.25 present the strength, durability and S/N ratios of the mixes and the ANOVA of the S/N ratios, respectively.

Table 4.24: Strength, durability and S/N ratios of the LS–MK mixes

Mix ID	MK %	LS %	Compressive Strength	S/N (dB)	Split Tensile	S/N (dB)	Flexural (MPa)	S/N (dB)	Water Absorption	S/N (dB)	RCPT (Coulombs)	S/N (dB)	Acid Loss (%)	S/N (dB)
C100M0L0	0	0	30.20	29.600	4.10	12.261	4.25	12.576	8.0	-18.062	2200	-66.848	10	-20.000
C95M0L5	0	5	31.50	29.966	4.20	12.465	4.31	12.689	7.7	-17.730	2500	-67.958	11	-20.828
C90M0L10	0	10	32.00	30.103	4.30	12.670	4.38	12.824	7.3	-17.266	1900	-65.575	12	-21.584
C85M0L15	0	15	29.80	29.484	4.10	12.261	4.18	12.427	9.0	-19.085	2600	-68.299	12	-21.584
C90M5L0	5	0	33.10	30.397	4.40	12.869	4.53	13.126	7.9	-17.953	1700	-64.609	13	-22.279
C90M5L5	5	5	34.70	30.807	4.50	13.073	4.62	13.299	7.5	-17.501	1400	-62.922	14	-22.923
C85M5L10	5	10	35.80	31.078	4.60	13.255	4.71	13.468	7.3	-17.266	1200	-61.584	14	-22.923
C80M5L15	5	15	33.90	30.604	4.50	13.073	4.58	13.214	8.8	-18.890	1500	-63.521	15	-23.522
C90M10L0	10	0	34.50	30.756	4.50	13.073	4.65	13.346	7.6	-17.616	1200	-61.584	13	-22.279
C85M10L5	10	5	36.20	31.174	4.60	13.255	4.75	13.534	7.1	-17.025	1000	-60.000	15	-23.522

C70M15L1 5	15	15	32.80	30.317	4.40	12.869	4.52	13.108	9.8	-19.825	1000	-60.000	40	-32.041
C75M15L1 0	15	10	34.20	30.681	4.50	13.073	4.63	13.311	8.7	-18.790	900	-59.084	38	-31.596
C80M15L5	15	5	34.90	30.857	4.60	13.255	4.69	13.423	8.0	-18.062	950	-59.554	27	-28.627
C85M15L0	15	0	33.70	30.553	4.50	13.073	4.60	13.258	7.4	-17.385	1000	-60.000	17	-24.609
C75M10L1 5	10	15	35.10	30.906	4.50	13.073	4.67	13.390	7.0	-16.902	1100	-60.828	16	-24.082
C80M10L1 0	10	10	37.10	31.387	4.70	13.446	4.81	13.646	6.4	-16.124	800	-57.958	8	-18.062

Table 4.25: ANOVA of S/N ratios for the LS–MK mixes (Taguchi analysis)

Response (S/N)	Source	DF	SS	MS	F	p-value	Contribution (%)
Compressive strength (28 d)	MK	3	3.4712	1.1571	74.19	<0.001	79.37
	LS	3	0.7620	0.2540	16.29	<0.001	17.42
	Error (MK × LS)	9	0.1404	0.0156	–	–	3.21
	Total	15	4.3736				100.00
Water absorption	MK	3	5.4026	1.8009	4.35	0.037	41.13
	LS	3	4.0066	1.3355	3.23	0.075	30.50
	Error (MK × LS)	9	3.7269	0.4141	–	–	28.37
	Total	15	13.1361				100.00
RCPT charge (56 d)	MK	3	144.7028	48.2343	97.66	<0.001	89.25
	LS	3	12.9808	4.3269	8.76	0.005	8.01
	Error (MK × LS)	9	4.4452	0.4939	–	–	2.74
	Total	15	162.1288				100.00
Acid mass loss (12 weeks)	MK	3	165.1285	55.0428	11.98	0.002	73.37
	LS	3	18.5711	6.1904	1.35	0.320	8.25

Response (S/N)	Source	DF	SS	MS	F	p-value	Contribution (%)
	Error (MK $\times$ LS)	9	41.3674	4.5964	—	—	18.38
	Total	15	225.0669				100.00

Table 4.25 presents the ANOVA of the S/N ratios, computed from the measured responses in Tables 4.7, 4.13, 4.15 and 4.17. MK is the dominant factor for all four responses, contributing 79.4% of the variation in compressive strength, 41.1% in water absorption, 89.3% in RCPT charge and 73.4% in acid-induced mass loss. The contribution of LS is statistically significant for compressive strength (17.4%) and RCPT charge (8.0%), marginal for water absorption (30.5%, $p \approx 0.08$) and not significant for acid mass loss (8.3%, $p \approx 0.32$). The pooled error term, i.e., the MK $\times$ LS interaction, is small for compressive strength (3.2%) and RCPT charge (2.7%) but large for water absorption (28.4%) and acid mass loss (18.4%), indicating that, for these durability responses, the effect of LS depends strongly on the MK content.

The main-effect analysis identifies 10% MK and 10% LS as the optimum levels for compressive strength and water absorption. For RCPT, the S/N ratio increases continuously with the MK content (optimum at 15% MK and 10% LS), while for acid mass loss the main effects favour low MK and LS levels because of the very high mass loss of the mixes combining 15% MK with high LS contents; this main-effect result is, however, dominated by the interaction, since the single best mix in acid resistance is C80M10L10 (8% mass loss). Considering all the responses together, C80M10L10 (MK 10% + LS 10%) is identified as the best overall mix: it has the highest compressive strength (37.1 MPa at 28 days), the highest split tensile and flexural strengths, the lowest water absorption (6.4%), the lowest RCPT charge (800 C) and the lowest acid mass loss (8%). Table 4.26 shows the regression models for the LS–MK concrete properties.

Table 4.26: Regression models for LS–MK concrete properties

Property	Quadratic Regression Equation	R ²	Interpretation
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Compressive Strength (MPa)	CS $= 29.7135$ $+ 1.03395MK$ $+ 0.53595LS$ $- 0.05325MK^2$ $- 0.03325LS^2$ $- 0.00356(MK \cdot LS)$	0.961	Strength increases strongly with MK and LS up to mid-range (around 10%). Negative MK^2 and LS^2 terms indicate a peak at MK10–LS10, after which excessive replacement reduces strength.
Water Absorption (%)	WA $= 8.3465$ $- 0.25745MK$ $- 0.18645LS$ $+ 0.01575MK^2$ $+ 0.01375LS^2$ $+ 0.00436(MK \cdot LS)$	0.546	WA decreases initially due to pore refinement (negative MK and LS coefficients). At high replacement levels, positive MK^2 and LS^2 terms cause WA to increase again. The low R^2 indicates that the model captures the general trend only qualitatively.
RCPT (Coulombs)	$RCPT$ $= 2391.0$ $- 201.925MK$ $- 60.675LS$ $+ 7.875MK^2$ $+ 4.125LS^2$ $- 0.66(MK \cdot LS)$	0.934	MK strongly reduces chloride permeability (large negative MK coefficient). LS provides additional but smaller reduction. Curvature terms correctly capture the minimum RCPT at MK10–LS10.
Acid Attack Loss (%)	AL $= 13.45 - 1.7225MK$ $- 0.0575LS$ $+ 0.1475MK^2$ $- 0.0075LS^2$ $+ 0.086(MK \cdot LS)$	0.811	Acid loss decreases up to MK10–LS10, then rises at higher replacements. Positive MK^2 term causes U-shaped behaviour. The positive interaction term indicates that a combined increase in MK and LS raises the mass loss.

The combined analysis of experimental results and Taguchi S/N ratios clearly identifies the mix with 80% cement, 10% metakaolin (MK) and 10% lime sludge (LS) (mix ID C80M10L10) as the optimum proportion. This blend consistently outperforms all other mixes in terms of both strength and durability. It develops the highest 28-day compressive strength (37.1 MPa),

alongside superior split tensile and flexural strengths, confirming its enhanced load-carrying capacity and crack resistance. From a durability perspective, the same mix exhibits the lowest water absorption (6.4%), the lowest rapid chloride penetration charge (800 C) and the lowest mass loss under 12-week exposure to 5% HCl, indicating a dense and refined pore structure with reduced permeability and improved acid resistance. Taguchi main-effect plots and S/N-based delta rankings further reinforce that 10% MK and 10% LS constitute the most favourable level combination, as the S/N ratios for compressive strength and water absorption peak at these levels. This optimum can be attributed to the synergistic interaction between the pozzolanic reactivity of MK and the filler–nucleation role of LS, which together promote secondary gel formation, pore refinement and improved microstructural integrity although at the cost of the lowest slump among the mixes (Section 4.1).

4.23 Response Surface Methodology (RSM) Analysis of the CLS–MK System

Response Surface Methodology (RSM) was used to determine the interaction effects of the selected process parameters and to identify the best combination that yields the maximum mechanical performance of the developed concrete. Three independent factors were examined in this study, namely: Metakaolin MK replacement, calcined lime sludge (CLS) replacement, and curing age. MK and CLS replacement levels were chosen in the range of 5% to 20%, with curing ages of 7, 28, and 90 days representing early, intermediate, and long-term strength development of concrete. Table 4.26 shows the selected factors and their levels for the RSM design.

Table 4.27: Factors and levels used in the RSM design

Factor	Parameter	Symbol	Low Level (-1)	Middle Level (0)	High Level (+1)
A	Metakaolin (%)	MK	5	10	15
B	Calcined Lime Sludge (%)	CLS	5	10	15
C	Curing Age (days)	Age	7	28	90

Design-Expert software was used to analyse the three-factor experiment and to investigate the interactions between the variables systematically. The experimental design consisted of 24 runs drawn from the CLS–MK matrix at three levels of each factor. The responses analysed were slump, compressive strength, split tensile strength, flexural strength and water absorption. Since slump is a property of fresh concrete and water absorption was measured at a single age, these two responses do not depend on curing age; their values are therefore repeated across the three ages in the matrix (**Table 4.28**), and curing age is excluded from their interpretation.

Table 4.28: RSM experimental matrix with measured responses

Run	MK (%)	CLS (%)	Age (days)	Slump (mm)	Compressive Strength (MPa)	Split Tensile Strength (MPa)	Flexural Strength (MPa)	Water Absorption (%)
1	15	5	7	95	36	3.24	3.60	7.0
2	10	5	90	100	51.0	5.10	6.12	7.2
3	10	15	28	90	47	4.61	5.17	7.8
4	5	15	28	95	44	4.31	4.84	8.0
5	10	5	28	100	48	4.70	5.28	7.2
6	10	5	7	100	34.5	3.11	3.45	7.2
7	15	10	7	90	37	3.33	3.70	6.0
8	10	15	90	90	50	5.00	6.00	7.8
9	5	10	28	100	48	4.70	5.28	7.1
10	5	15	90	95	46.9	4.69	5.63	8.0
11	5	5	90	105	50.2	5.02	6.02	7.2
12	15	15	28	85	48	4.70	5.28	7.1
13	5	15	7	95	31	2.79	3.10	8.0
14	5	10	7	100	35	3.15	3.50	7.1
15	5	10	90	100	50.9	5.09	6.11	7.1
16	15	15	7	85	35	3.15	3.50	7.1
17	10	10	7	95	36	3.24	3.60	7.0
18	10	15	7	90	34.8	3.13	3.48	7.8
19	10	10	90	95	52	5.20	6.24	7.0
20	15	5	28	95	49.4	4.84	5.43	7.0
21	15	5	90	95	51.8	5.18	6.22	7.0
22	15	10	28	90	51	5.00	5.61	6.0
23	15	10	90	90	55.2	5.52	6.63	6.0
24	5	5	28	105	47.8	4.68	5.26	7.2

The experimental data were fitted to a second-order polynomial regression model to establish the relationship between the process variables and the responses. RSM has a general quadratic regression model, which can be defined as follows:

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j$$

where Y represents the predicted value, β_0 is the constant term, β_i , β_{ii} , and β_{ij} are linear, quadratic, and interaction coefficients, respectively, and X_i and X_j are coded independent variables. The analysis of variance (ANOVA) was conducted to determine the effectiveness and statistical significance of the developed models. **The full quadratic model, with linear, two-factor interaction and squared terms of A (MK), B (CLS) and C (curing age), was fitted to each response using coded factors [A = (MK – 10)/5, B = (CLS – 10)/5, C = (Age – 48.5)/41.5]. Table 4.29 presents the ANOVA of the fitted quadratic models.**

Table 4.29: ANOVA of the quadratic RSM models

Response	Source	SS	df	MS	F-value	p-value
Compressive strength	Model	1247.6460	9	138.6273	333.26	<0.0001
	A – MK	35.9905	1	35.9905	86.52	<0.0001
	B – CLS	8.4356	1	8.4356	20.28	0.0005
	C – Age	1020.0961	1	1020.0961	2452.34	<0.0001
	AB	4.2587	1	4.2587	10.24	0.0064
	AC	0.9048	1	0.9048	2.18	0.1624
	BC	0.0811	1	0.0811	0.19	0.6656
	A ²	0.0324	1	0.0324	0.08	0.7844
	B ²	19.9024	1	19.9024	47.85	<0.0001
	C ²	367.0133	1	367.0133	882.31	<0.0001
	Residual	5.8236	14	0.4160	—	—
	Total	1253.4696	23			
Split tensile strength	Model	17.8374	9	1.9819	485.59	<0.0001
	A – MK	0.3429	1	0.3429	84.03	<0.0001
	B – CLS	0.0785	1	0.0785	19.23	0.0006
	C – Age	15.0318	1	15.0318	3682.89	<0.0001
	AB	0.0395	1	0.0395	9.68	0.0077
	AC	0.0123	1	0.0123	3.00	0.1050
	BC	0.0004	1	0.0004	0.11	0.7471
	A ²	0.0003	1	0.0003	0.06	0.8042
	B ²	0.1847	1	0.1847	45.25	<0.0001
	C ²	5.0939	1	5.0939	1248.03	<0.0001
	Residual	0.0571	14	0.0041	—	—
	Total	17.8945	23			
Flexural strength	Model	29.8376	9	3.3153	589.47	<0.0001
	A – MK	0.4636	1	0.4636	82.43	<0.0001
	B – CLS	0.1020	1	0.1020	18.14	0.0008
	C – Age	27.0685	1	27.0685	4812.87	<0.0001
	AB	0.0549	1	0.0549	9.76	0.0075
	AC	0.0246	1	0.0246	4.38	0.0550
	BC	0.0004	1	0.0004	0.06	0.8045
	A ²	0.0001	1	0.0001	0.02	0.8797
	B ²	0.2441	1	0.2441	43.41	<0.0001
	C ²	5.6630	1	5.6630	1006.90	<0.0001
	Residual	0.0787	14	0.0056	—	—
	Total	29.9163	23			
Water absorption	Model	7.1475	9	0.7942	28.60	<0.0001

5

Response	Source	SS	df	MS	F-value	p-value
	A – MK	2.0849	1	2.0849	75.09	<0.0001
	B – CLS	0.7994	1	0.7994	28.79	<0.0001
	C – Age	0.0022	1	0.0022	0.08	0.7839
	AB	0.2768	1	0.2768	9.97	0.0070
	AC	0.0017	1	0.0017	0.06	0.8086
	BC	0.0017	1	0.0017	0.06	0.8086
	A ²	0.3156	1	0.3156	11.37	0.0046
	B ²	2.2689	1	2.2689	81.71	<0.0001
	C ²	0.0009	1	0.0009	0.03	0.8575
	Residual	0.3887	14	0.0278	—	—
	Total	7.5362	23			

The ANOVA in **Table 4.29** shows that the quadratic models for compressive strength, split tensile strength, flexural strength and water absorption are highly significant ($p < 0.0001$). For the three strength responses, curing age (C and C²) is by far the most influential term, reflecting the progress of hydration and pozzolanic reaction with time, followed by MK content (A), CLS content (B and B²) and the MK $\times$ CLS interaction (AB); the interactions of age with MK and CLS (AC and BC) and the A² term are not significant ($p > 0.05$). For water absorption, A, B, AB, A² and B² are significant, whereas curing age has no effect because water absorption was determined at a single age. The significant B² and AB terms quantify the curvature and the synergy between MK and CLS, which a model containing only linear and interaction terms cannot represent. The slump data are described exactly by a linear function of MK and CLS (Slump = 95 – 5A – 5B in coded units, $R^2 = 1.00$), so slump is not included in the ANOVA; as expected for a fresh property, it is independent of curing age. Because the design contains no replicated runs, a lack-of-fit test could not be performed.

Table 4.30: Model summary statistics of the RSM models

Response	R ²	Adjusted R ²	Predicted R ²	Std. dev.	CV (%)	Adeq. precision
Compressive strength	0.9954	0.9924	0.9865	0.645	1.45	55.5
Split tensile strength	0.9968	0.9948	0.9906	0.064	1.48	63.9
Flexural strength	0.9974	0.9957	0.9920	0.075	1.51	70.5
Water absorption	0.9484	0.9153	0.8477	0.167	2.33	17.8

Response	R ²	Adjusted R ²	Predicted R ²	Std. dev.	CV (%)	Adeq. precision
Slump (linear model)	1.0000	1.0000	1.0000	0.000	0.00	—

The model summary statistics in Table 4.30 indicate that the quadratic models describe the data well: R² ranges from 0.948 (water absorption) to 0.997 (flexural strength), the adjusted and predicted R² values are in reasonable agreement (difference below 0.07), and the adequate precision of all models is well above 4, indicating an adequate signal-to-noise ratio for navigating the design space.

4.23.1 Regression Models Developed Using RSM

The regression equations of the quadratic models are given below in terms of the coded variables of metakaolin (A), calcined lime sludge (B) and curing age (C), showing the effects of these variables on slump, compressive strength, split tensile strength, flexural strength and water absorption.

Slump (mm)

$$Slump = 95 - 5A - 5B$$

Compressive Strength (MPa)

$$CS = 56.46 + 1.58A - 0.77B + 8.15C + 0.73AB + 0.32AC + 0.10BC - 0.08A^2 - 2.05B^2 - 12.02C^2$$

Split Tensile Strength (MPa)

$$STS = 5.680 + 0.154A - 0.074B + 0.989C + 0.071AB + 0.038AC + 0.007BC - 0.007A^2 - 0.197B^2 - 1.417C^2$$

Flexural Strength (MPa)

$$FS = 6.463 + 0.180A - 0.084B + 1.327C + 0.083AB + 0.053AC + 0.006BC - 0.005A^2 - 0.227B^2 - 1.494C^2$$

Water Absorption (%)

$$WA = 6.839 - 0.381A + 0.236B - 0.012C - 0.187AB - 0.014AC \\ - 0.014BC - 0.258A^2 + 0.692B^2 + 0.019C^2$$

In these equations the factors are in coded units, so the magnitudes of the coefficients indicate the relative influence of each term. Curing age has the largest positive linear effect on all three strengths, and the large negative C^2 coefficient reflects the decreasing rate of strength gain at later ages. MK (A) has a positive effect on strength and a negative (beneficial) effect on water absorption, while the negative B^2 coefficient for strength and the positive B^2 coefficient for water absorption indicate that CLS is beneficial up to an intermediate level (about 10%) and detrimental beyond it. The positive AB coefficients for strength and the negative AB coefficient for water absorption quantify the synergy between MK and CLS. Table 4.31 shows the optimisation results obtained from RSM.

Table 4.31: Optimisation results obtained from RSM

Factor / Response	Optimum Setting / Value	Predicted Value	Experimental Value	Error (%)
MK (%)	15	–	–	–
CLS (%)	10	–	–	–
Curing Age (days)	90	–	–	–
Slump (mm)	–	90	94	4.26
Compressive Strength (MPa)	–	54.40	54.6	0.36
Split Tensile Strength (MPa)	–	5.44	5.45	0.23
Flexural Strength (MPa)	–	6.52	6.54	0.25
Water Absorption (%)	–	6.19	6.12	1.20
Overall Desirability	0.95	–	–	–

The response surface methodology-based multi-response optimisation (maximising the three strengths and minimising water absorption) identified 15% MK, 10% CLS and a curing age of 90 days as the optimum. At these settings, the models predict a compressive strength of 54.4 MPa, a split tensile strength of 5.44 MPa and a flexural

strength of 6.52 MPa, with acceptable workability and water absorption. To test the reliability of the RSM models, confirmation experiments were carried out at the optimum conditions; the experimental results deviated from the predictions by less than 5% (less than 1.2% for the strength and water absorption responses), indicating that the models are adequate and predictive. The desirability of 0.95 indicates a favourable trade-off among mechanical strength, workability and durability. The quadratic models showed high R^2 values (0.95–0.997), confirming their adequacy. Table 4.32 compares the performance of selected CLS–MK mixes at 90 days (strengths) using the values of Tables 4.4, 4.8, 4.10, 4.12 and 4.14.

Table 4.32: Comparative performance of selected CLS–MK mixes at 90 days

Mix	MK (%)	CLS (%)	Slump with SP (mm)	Compressive strength (MPa)	Split tensile strength (MPa)	Flexural strength (MPa)	Water absorption (%)	Remarks
M0CL0	0	0	115	49.0	4.90	5.88	7.6	Control
M5CL0	5	0	110	50.2	5.00	6.02	7.2	Slight improvement
M0CL5	0	5	110	50.0	5.00	6.00	7.4	Slight improvement
M10CL0	10	0	105	51.0	5.10	6.12	7.0	Moderate improvement
M0CL10	0	10	105	50.1	5.00	6.01	7.2	Slight improvement
M10CL10	10	10	95	52.0	5.20	6.24	7.0	Good combined effect
M15CL10	15	10	90	55.2	5.52	6.62	6.0	Optimum

4.24 Life Cycle Assessment (LCA) of Different Mixes

A cradle-to-gate life cycle assessment (LCA) was carried out to evaluate the environmental performance of concrete mixtures containing calcined lime sludge (CLS) and metakaolin (MK). The assessment considered one cubic metre of concrete as the functional unit and included raw material production, lime sludge processing, transportation, and concrete manufacturing. Standard mix proportions and screening-level environmental coefficients were adopted where product-specific data were unavailable.

Portland cement production dominated the environmental profile of the control mixtures because of its high clinker content and energy-intensive manufacturing process. Consequently, progressive replacement of cement substantially lowered greenhouse gas emissions, fossil energy consumption, acidification, eutrophication, and photochemical ozone formation.

Within the M40 series, the control mixture exhibited the highest global warming potential (GWP) (295.48 kg CO₂-eq/m³). Incorporation of 5% calcined lime sludge and 5% metakaolin (M40-M5CL5) reduced the GWP by approximately 8.3%, while increasing the replacement level to 10% CLS and 10% MK (M40-M10CL10) produced a reduction of about 16.5%. The M40-M15CL10 mixture achieved a reduction of approximately 21%, whereas the highest replacement mixture (M40-M15CL20) exhibited the lowest environmental burden, reducing the GWP by approximately 28.5% relative to the control. These reductions are primarily attributed to the decreased cement requirement, which outweighs the environmental burden associated with calcining the lime sludge.

The M25 mixtures incorporating raw lime sludge exhibited even larger relative reductions. The mixture M25-C80M10L10 (10% MK + 10% LS), which was the experimentally optimum LS–MK mix, reduced the GWP by approximately 18% relative to the M25 control. An additional screening scenario with 15% MK and 20% LS (M25-M15L20, 35% total replacement), which lies outside the experimental LS–MK matrix (maximum 15% of each material) and was included only to illustrate the environmental potential of higher replacement, reduced the GWP by approximately 32%. Because raw sludge was assumed to require only collection, dewatering and handling, its embodied impacts were substantially lower than those of calcined sludge. Although transport impacts increased owing to the higher moisture content of raw sludge, the additional freight burden remained small compared with the avoided impacts of clinker production.

Overall, the results indicate that the simultaneous incorporation of lime sludge and metakaolin substantially improves the environmental sustainability of concrete. Among the experimentally tested mixtures, M40-M15CL20 showed the lowest environmental burden, although its strength (44.4 MPa at 90 days) was below that of the control; among the mixes that also improved mechanical performance, M40-

M15CL10 (21% lower GWP) and M25-C80M10L10 (18% lower GWP) offer the best balance between environmental and engineering performance.

Table 4.33: Calculated environmental impacts of the concrete mixes (per m³)

Mix	GWP-total (kg CO ₂ -eq)	ODP (kg CFC-11-eq)	AP (mol H ⁺ -eq)	EP-fw (kg P-eq)	POCP (kg NMVOC-eq)	ADP-fossil (MJ)
M40-M0CL0 (control)	295.477	1.19631E-06	1.153	0.008	0.705	1618.43
M40-M5CL5	271.079	1.19396E-06	1.055	0.007	0.653	1537.66
M40-M10CL10	246.681	1.19162E-06	0.957	0.007	0.601	1456.902
M40-M15CL10	233.363	1.18668E-06	0.905	0.006	0.572	1400.322
M40-M15CL20	211.203	1.19186E-06	0.814	0.006	0.526	1351.955
M40-M10CL5	257.761	1.18902E-06	1.003	0.007	0.624	1481.086
M25-C100M0L0 (control)	266.465	1.18938E-06	1.043	0.007	0.642	1463.74
M25-C80M10L10	218.021	1.16822E-06	0.855	0.006	0.537	1247.039
M25-M15L20*	181.567	1.15151E-06	0.714	0.005	0.457	1081.261

Table 4.34: Normalised environmental impacts of the concrete mixes

Mix	GWP- total normali zed	ODP norm alized	AP norm alize d	EP- terrestria l normaliz ed	POCP norma lized	ADP- fossil normali zed	WDP norm alized	GWP reducti on (%)
M40- M0CL0 (control)	1	1	1	1	1	1	1	0
M40- M5CL5	0.917	0.998	0.915	0.923	0.926	0.950	0.931	8.257
M40- M10CL1 0	0.834	0.996	0.830	0.846	0.852	0.900	0.863	16.514
M40- M15CL1 0	0.789	0.991	0.785	0.804	0.811	0.865	0.824	21.021
M40- M15CL2 0	0.714	0.996	0.705	0.735	0.745	0.835	0.767	28.521
M40- M10CL5	0.872	0.993	0.870	0.881	0.885	0.915	0.892	12.764
M25- C100M0 L0 (control)	1	1	1	1	1	1	1	0
M25- C80M10 L10	0.818	0.982	0.819	0.832	0.835	0.851	0.840	18.179
M25- M15L20 *	0.681	0.968	0.684	0.705	0.711	0.738	0.719	31.860

* Screening scenario (15% MK + 20% LS) outside the experimental LS–MK matrix; included only to illustrate the environmental potential of higher replacement.

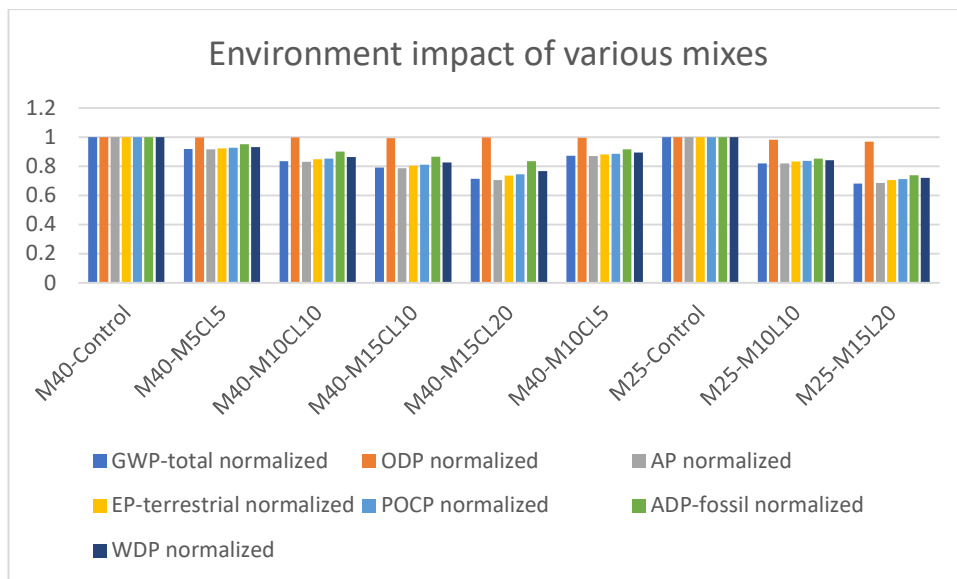


Figure 4.26: Environmental impact categories of various mixes

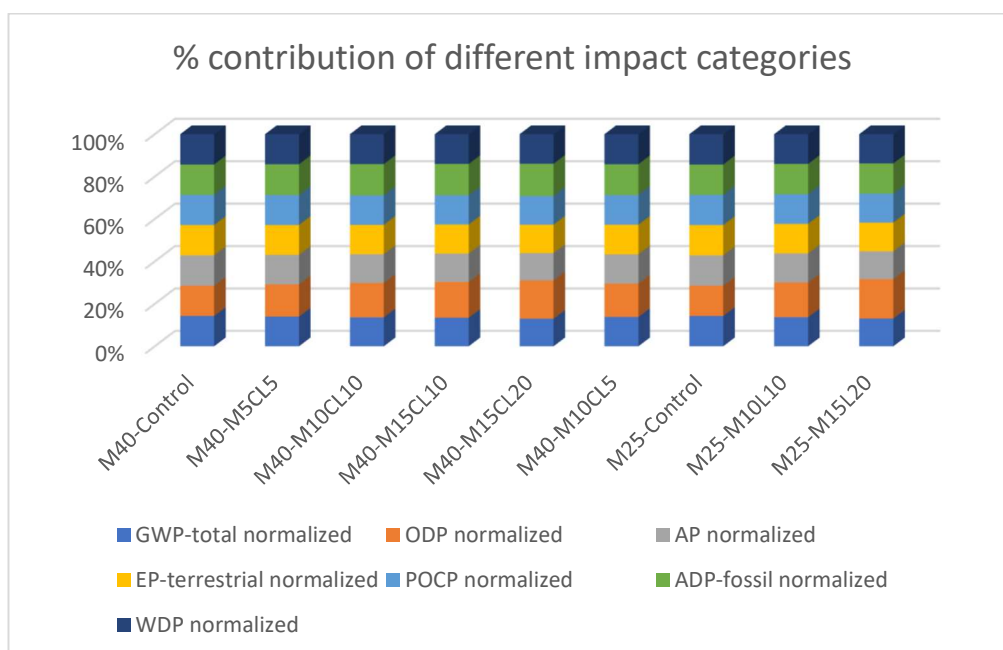


Figure 4.27: Percentage contribution of various impact categories

4.25 Cost Analysis

Table 4.35 lists the unit costs of the materials, and Table 4.36 presents the material cost of each concrete mix per cubic metre. The unit costs of lime sludge and calcined lime sludge include drying and grinding (and, for CLS, calcination) as well as transport from the paper mill. The cost of each mix is compared with the control mix of the

same grade: the M40 CLS–MK mixes with the M40 control (M0CL0, ₹5009.60/m³) and the M25 LS–MK mixes with the M25 control (C100M0L0, ₹4636.08/m³). All the mixes considered are the superplasticised mixes used for the hardened-concrete tests. Among the M40 mixes, M5CL5 increased the cost by 6.65%, M10CL5 by 13.47% and M10CL10 by 13.29%, while the highest increases were observed for M15CL10 (20.12%) and M15CL20 (19.76%). These increases arise primarily from the high unit cost of metakaolin (₹26/kg, about 3.7 times that of cement), which outweighs the savings from the reduced cement consumption.

For the M25 series, the optimum mix C80M10L10 increased the cost by 10.19% relative to the M25 control, C85M5L10 by 3.47% and C70M15L15 by 15.28%. Mixes with a low MK content are therefore almost cost-neutral, because lime sludge itself is inexpensive. Since C80M10L10 increased the 28-day compressive strength by 23%, reduced water absorption by 20% and the 56-day RCPT charge by 64%, and reduced the GWP by 18%, its cost per unit of compressive strength is about 10% lower than that of the control. For the M40 series, the cost increase of M15CL10 (20.1%) exceeds its strength gain (11% at 28 days), so this mix is justified mainly by its durability and environmental benefits.

Table 4.35: Unit cost of materials

Material	Unit	Unit Cost (₹)
Cement	kg	₹7.00
Lime sludge (processed, delivered)	kg	₹2.40
Metakaolin	kg	₹26.00
Fine Aggregate	kg	₹1.10
Coarse Aggregate	kg	₹1.20
Water	litre	₹0.05
Superplasticiser	kg	₹65.00
Calcined lime sludge (calcined, delivered)	kg	₹6.50
Transport assumptions (included in delivered LS/CLS prices)	Value	Unit
Distance	100	km
Truck transport rate	4	₹/tonne-km
Calculated transport cost	₹0.40	₹/kg

Table 4.36: Cost analysis of the concrete mixes

Mix	Cement	MK	LS / CLS	Aggregates (FA + CA)	Water + SP	Total (₹/m³)	Change vs. control of same grade (%)
M40-M0CL0 (control)	2520.00	0.00	0.00	2248.40	241.20	5009.60	0.00
M40-M5CL5	2268.00	468.00	117.00	2248.40	241.20	5342.60	+6.65
M40-M10CL5	2142.00	936.00	117.00	2248.40	241.20	5684.60	+13.47
M40-M10CL10	2016.00	936.00	234.00	2248.40	241.20	5675.60	+13.29
M40-M15CL10	1890.00	1404.00	234.00	2248.40	241.20	6017.60	+20.12
M40-M15CL20	1638.00	1404.00	468.00	2248.40	241.20	5999.60	+19.76
M25-C100M0L0 (control)	2296.00	0.00	0.00	2205.30	134.78	4636.08	0.00
M25-C85M5L10	1951.60	426.40	78.72	2205.30	134.78	4796.80	+3.47
M25-C80M10L10	1836.80	852.80	78.72	2205.30	134.78	5108.40	+10.19
M25-C70M15L15	1607.20	1279.20	118.08	2205.30	134.78	5344.56	+15.28

4.26 Synthesis of Results and Link between the LS–MK and CLS–MK Programmes

The two experimental programmes lead to a consistent picture of how a calcium-bearing paper-mill residue and a reactive aluminosilicate interact in concrete. In the M25 series, raw LS on its own reduced the strength at 15% replacement (C85M0L15) and had little effect on chloride resistance, confirming that its calcite is largely unreactive and acts mainly as a filler and nucleation surface. MK alone improved all the properties through its pozzolanic reaction. The combination of 10% LS and 10% MK gave the best overall performance, with the highest density and strength and the best resistance to water absorption, chloride penetration and acid attack, which is consistent with complementary filler/nucleation (LS) and pozzolanic (MK) effects and with the possible formation of carboaluminates from calcite and MK-derived alumina, as reported for limestone–calcined clay systems (Antoni et al., 2012; Scrivener et al., 2018).

In the M40 series, calcination converted part of the CaCO_3 in the sludge into reactive CaO. CLS alone improved the strength slightly up to 10% replacement but reduced the workability progressively, reflecting the rapid hydration of free lime and the higher fineness of CLS. The optimum shifted to a higher MK content (15% MK + 10% CLS), which is consistent with the larger amount of Ca(OH)_2 that must be consumed when CaO-rich CLS is used; at 20% CLS, the excess calcium and the reduced clinker content lowered the strength and increased the water absorption and acid mass loss. The RSM

models confirm a significant $MK \times CLS$ synergy (AB term) and an optimum CLS content of about 10% (negative B^2 term for strength).

The microstructural results (SEM/EDS, XRD, TGA and calorimetry) are consistent with these mechanisms, showing denser matrices, reduced portlandite and favourable Ca/Si ratios at the optimum proportions, but they provide indirect rather than conclusive evidence of the specific hydration products (Sections 4.17–4.21). Environmentally, both optimum mixes reduce the GWP by about 18–21%; the LS–MK optimum is also cost-effective per unit of strength, whereas the CLS–MK optimum is justified mainly by its durability and environmental benefits.

CHAPTER 5: CONCLUSIONS AND FUTURE SCOPE

5.1 General

This thesis investigated the use of paper-mill lime sludge, in raw (LS) and calcined (CLS) form, together with metakaolin (MK) as partial replacements for cement in M25 and M40 grade concrete. The principal conclusions are summarised below, separately for the two experimental programmes, followed by the statistical, environmental and economic findings.

5.2 Conclusions for M25 Concrete with LS and MK

1. The combined use of LS and MK improved the density, strength, durability and microstructure of M25 concrete at moderate replacement levels. The mix with 10% LS and 10% MK (C80M10L10) was the optimum, attaining compressive strengths about 12%, 23% and 26% higher than the control at 7, 28 and 90 days (37.1 MPa against 30.2 MPa at 28 days) and split tensile and flexural strengths about 13–15% higher than the control.
2. Raw LS alone acted mainly as a filler: at 15% replacement without MK (C85M0L15), it produced the lowest strength at all ages, whereas all the mixes containing MK exceeded the control.
3. Workability did not decrease monotonically with replacement: the slump was lowest for C80M10L10 (65 mm initially) and highest for C70M15L15 (115 mm), with the control mix in an intermediate position. All the mixes remained workable, and the superplasticiser increased the slump of every mix by about 5 mm without changing the ranking.
4. C80M10L10 showed the highest density (2490–2500 kg/m³), the lowest water absorption (6.4%, i.e., 20% lower than the control), the lowest 56-day RCPT charge (800 C, about 64% lower than the control, i.e., very low chloride penetrability) and the lowest 12-week mass loss in 5% HCl (8% against 10%). All the other LS–MK mixes showed a higher acid mass loss than the control, so the improvement in acid resistance is specific to the optimum proportion.
5. SEM images of C80M10L10 showed a denser, less cracked matrix, and EDS indicated a Ca/Si ratio of 1.8, consistent with a C–S–H-dominated gel. These

observations support, but do not by themselves prove, the formation of additional C–S–H/C–A–S–H gel through the LS–MK interaction.

5.3 Conclusions for M40 Concrete with CLS and MK

6. Calcination of lime sludge at 750 °C for 2 h followed by slow cooling gave the highest pozzolanic reactivity among the conditions examined, reducing the loss on ignition from 21% to 10.6% and increasing the CaO content from 41% to 65.6%.
7. The workability of the CLS–MK concrete decreased progressively with increasing MK and CLS contents, the slump ranging from 75 to 115 mm with superplasticiser, owing to the higher specific surface area of MK and CLS and the rapid hydration of the free lime in CLS.
8. The mix with 15% MK and 10% CLS (M15CL10) gave the highest compressive strength (37, 51 and 55.2 MPa at 7, 28 and 90 days, against 32, 46 and 49 MPa for the control) and the highest split tensile strength (5.52 MPa at 90 days).
9. Flexural strength also improved at moderate replacement levels and reached its maximum (6.62 MPa at 90 days) for M15CL10; beyond this level, the flexural strength decreased, down to 4.18 MPa at 90 days for M20CL20 (40% total replacement).
10. Water absorption was lowest (6.0%) for M15CL10, whereas higher replacement levels increased the porosity and water absorption (up to 11.6% for M20CL20).
11. In 5% HCl, only M15CL5 showed a clearly lower mass loss (8%) than the control (11%); most of the other CLS–MK mixes lost the same as or more mass than the control, indicating that the excess calcium supplied by CLS must be balanced by sufficient MK to improve acid resistance.
12. Quadratic response surface models fitted to the CLS–MK data ($R^2 = 0.95\text{--}0.997$) showed significant effects of MK, CLS, curing age and the MK $\times$ CLS interaction, and multi-response optimisation identified 15% MK and 10% CLS as the optimum (desirability 0.95), with confirmation results within 5% of the predictions.

5.4 Statistical, Environmental and Economic Conclusions

13. Regression analysis of the LS–MK data showed positive but moderate correlations of density with compressive, split tensile and flexural strength ($R^2 = 0.24\text{--}0.36$) and a stronger inverse correlation between water absorption and density ($R^2 = 0.54$). Density alone is therefore a weak predictor of strength; the reactivity of the binder, governed mainly by the MK content, is the dominant variable.
14. Taguchi analysis of the 4×4 factorial (L16) matrix showed that MK is the dominant factor, contributing 41–89% of the variation in compressive strength, water absorption, RCPT charge and acid mass loss, while LS contributed 8–31%. Since each mix was tested as a single run, the S/N ratios serve only to rank the main effects, and the $\text{MK} \times \text{LS}$ interaction forms the error term; this interaction is substantial for water absorption and acid mass loss.
15. The cradle-to-gate LCA showed that the optimum mixes reduce the global warming potential by about 18% (M25-C80M10L10) and 21% (M40-M15CL10) relative to their respective controls; higher replacement levels reduce the GWP further (by up to 28.5% for M40-M15CL20) but at the cost of strength.
16. Owing to the high price of MK, the optimum mixes increased the material cost by about 10.2% (M25-C80M10L10) and 20.1% (M40-M15CL10), whereas mixes with a low MK content were almost cost-neutral (e.g., +3.5% for M25-C85M5L10). For C80M10L10, the gain in strength exceeds the increase in cost, giving a lower cost per unit of compressive strength than the control.
17. Overall, LS–MK and CLS–MK blended concretes are technically feasible and more sustainable alternatives to conventional concrete. The blend of 10% LS + 10% MK is recommended for M25 concrete and the blend of 15% MK + 10% CLS for M40 concrete, contributing to waste valorisation, reduced clinker consumption and a lower carbon footprint.

5.5 Future Scope

Future work should first strengthen the statistical basis of the present findings by testing replicate batches of the key mixes and confirming the hydration products by quantitative phase analysis (Rietveld XRD, $^{29}\text{Si}/^{27}\text{Al}$ NMR and DTG-based

quantification of portlandite and carbonates). Future research on lime sludge–metakaolin blended concrete can be significantly expanded to address long-term performance, advanced characterization, and large-scale implementation. Extended durability studies under real environmental exposure, including carbonation, chloride ingress, freeze–thaw cycles, creep, and shrinkage, would provide deeper insights into service-life prediction. Further optimisation of the LS–MK and CLS–MK proportions using response surface methodology with replicated design points, artificial neural networks and other AI-based predictive models can enhance the precision of mix design and support performance forecasting for different grades of concrete. At the microstructural level, the use of sophisticated analytical techniques—such as X-ray microtomography, FTIR, NMR, and mercury intrusion porosimetry—may reveal detailed hydration mechanisms, pore structure evolution, and gel formation pathways. Structural application studies involving beams, columns, slabs, and fibre-reinforced variants can help determine load–deflection behaviour, crack propagation, toughness, and ductility improvements. The LS–MK binder system may also be evaluated in combination with other SCMs or nanomaterials to create hybrid, high-performance composites suitable for demanding environments. Large-scale field trials, life-cycle assessment studies, and economic viability analyses are essential to confirm sustainability benefits, cost savings, and practical feasibility of industrial adoption. Additionally, codal guidelines, standardised processing techniques, and quality-control measures for different types of lime sludge should be developed to ensure uniformity, safety, and performance reliability. Exploring the applicability of LS–MK blends in special concretes—including self-compacting concrete, high-performance concrete, 3D-printed materials, and precast products—can further expand their use in modern construction. Overall, the future work should focus on strengthening scientific understanding, improving predictive capability, and creating practical frameworks that support the widespread and sustainable utilisation of lime sludge–metakaolin blended concrete in the construction industry.

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LIST OF PUBLICATIONS

1. Chouksey, Arti, Nirendra Dev, and V. V. L. KantaRao. "Advancing sustainable concrete through lime sludge and metakaolin for reduced energy consumption and waste valorisation." *Scientific Reports* (2025).<https://doi.org/10.1038/s41598-025-28144-w>
2. Chouksey, A., Dev, N., & KantaRao, V. V. L. (2024). Effect of calcination condition and cooling method on reactivity of lime sludge. *Journal of Polymer and Composites*, 11(10), 36–48. <https://doi.org/10.37591/jopc.v11i10.133484>
3. Chouksey, Arti, Nirendra Dev, and V. V. L. KantaRao. "Combined effect of lime sludge (LS) and metakaolin (M) on the strength, physical parameters and microstructural properties of sustainable concrete." *AIP Advances* 15.11 (2025).<https://doi.org/10.1063/5.0289309>
4. Chouksey, A., Dev, N., & KantaRao, V. V. L. (2026, May). Advanced Low-Carbon Building Materials from Lime Sludge and Pozzolanic Additives: Review & Perspectives. In *Proceedings of the 2nd International Conference on Advanced Materials & Devices for Futuristic Applications-2024 (IC-AMDFA 2024)* (p. 215). Springer Nature.
5. Chouksey, A., Dev, N., & KantaRao, V. V. L. (2026, May). Metakaolin Blended with Lime Sludge in Concrete for Enhanced Performance and Eco-Efficient Infrastructure. In *Proceedings of the 2nd International Conference on Advanced Materials & Devices for Futuristic Applications-2024 (IC-AMDFA 2024)* (p. 131). Springer Nature.

RESPONSE TO EXAMINERS' COMMENTS

Ph.D. Thesis: “Mechanical and Microstructural Characterization of Lime Sludge–Metakaolin Blended Concrete Composite”

Candidate: Arti Chouksey (2K18/PHD/CE/27), Civil Engineering Department, Delhi Technological University

Supervisors: Prof. Nirendra Dev and Dr. V. V. L. Kanta Rao

All comments have been addressed in the revised thesis as follows:

Part A: Foreign Examiner

The responses to the minor revisions and to the chapter-wise observations of the foreign examiner are given in the table below.

No.	Examiner's comment	Response of the candidate	Location in revised thesis
F-1	Experimental repeatability and statistical variability: specify the number of specimens and independent batches and, where appropriate, provide SD or CoV.	A new Section 3.5.7 has been added. Table 3.25 now gives the number of specimens per mix per age for every test (three specimens for each strength test at each age, as required by IS 516 and IS 456:2000, Clause 15.4), together with the batching practice and the $\pm 15\%$ acceptance criterion. Each reported strength value represents the mean of three specimens. Since specimen-level raw readings were not retained separately, SD and CoV were not reconstructed retrospectively, Section 4 and the abstract now state that each strength value is the mean of three specimens.	Section 3.5.7; Tables 3.25; Chapter 4 introduction
F-2	Consistency and verification of experimental data; resolve the inconsistency in the 90-day compressive strength of the M40 control.	All numerical values in the text, abstract and conclusions were cross-checked against the tables. The 90-day strength of the M40 control (M0CL0) is 49 MPa as recorded in Table 4.8; the value of 59 MPa in the text and in the comparative table was a transcription error and has	Sections 4.7–4.16; Tables 4.8 and 4.32; Abstract; Chapter 5

No.	Examiner's comment	Response of the candidate	Location in revised thesis
		been corrected. The comparative performance table (Table 4.32) was rebuilt from Tables 4.4, 4.8, 4.10, 4.12 and 4.14 and now includes the optimum mix M15CL10. Strength ranges, percentage improvements and the identification of the weakest mixes in Sections 4.7–4.16 were also corrected to agree with the tables.	
F-3	Clarify the mix design, particularly the water reduction associated with the superplasticiser, and justify the selected calcination condition for lime sludge.	The mix design calculations have been verified and corrected (air-void term 1 – 0.02; aggregate volumes; coarse-to-fine aggregate fractions reconciled with the batched quantities). It is now stated that the water content was reduced by 18% (M25) and 20% (M40) with the superplasticiser while the w/c ratio was retained, as permitted by IS 10262:2019, and that the recalculated cement contents satisfy the IS 456:2000 minimum for severe exposure. An arithmetic slip in the aggregate volume of the superplasticised M25 mix is explained transparently together with its effect on the batched quantities. The calcination condition (750 °C for 2 h with slow cooling) is now justified by the reactivity study of the candidate (SAI, lime reactivity, XRD and TGA) and by the material characterisation (decarbonation range, reduction of LOI from 21% to 10.6%, increase of CaO from 41% to 65.6%).	Sections 3.2.7, 3.3 and 3.4

No.	Examiner's comment	Response of the candidate	Location in revised thesis
F-4	Justify the application of the Taguchi method to the 4×4 factorial LS–MK dataset, clarify the interpretation of S/N ratios with $n = 1$, and check and explain the formulation and optimisation of the RSM models.	A paragraph has been added explaining that the full 4×4 factorial equals an L16 (4^2) array, that with $n = 1$ the S/N ratio reduces to $\pm 20 \log_{10}(y)$ and therefore only ranks main effects without representing noise, and that the $MK \times LS$ interaction forms the error term. The ANOVA of the S/N ratios was recomputed from the measured data (Table 4.25), and the interpretation now distinguishes the responses for which the interaction is large. The RSM models were re-fitted as full quadratic models (linear, interaction and squared terms) in coded variables; the ANOVA, model statistics, regression equations and optimisation results (Tables 4.29–4.31) were recomputed accordingly. Slump, being a fresh property, is now modelled without curing age, and the absence of a lack-of-fit test (no replicated runs) is stated.	Sections 3.5.13, 4.22.2 and 4.23; Tables 4.25, 4.29–4.31
F-5	Strengthen and moderate the interpretation of SEM/EDS, XRD, TGA and thermal analysis; distinguish identified phases from proposed mechanisms (C–S–H/C–A–S–H, Ca/Si, Si/Al).	Sections 4.17–4.21 have been rewritten. The text now states that XRD identifies only crystalline phases (quartz, calcite, portlandite) and that C–S–H/C–A–S–H are inferred indirectly; that total TGA mass loss combines bound water, portlandite and carbonate decomposition and is not by itself a measure of hydration; that SEM morphology was assessed qualitatively; and that spot EDS ratios are semi-quantitative and influenced by aggregate and unreacted particles. Claims of direct “confirmation” of gel formation have been replaced by cautious wording, and the strength-development mechanism is presented as a literature-supported interpretation.	Sections 3.5.9, 3.5.12, 4.8, 4.17–4.21; Abstract
F-6	Overall presentation and editorial revision: terminology, grammar,	The thesis has been proofread throughout. A uniform mix nomenclature is defined in the new	Entire thesis

No.	Examiner's comment	Response of the candidate	Location in revised thesis
	typography, equations and units, mix nomenclature, figure/table numbering, captions, page formatting, cross-references and unresolved bookmark errors.	Section 3.4.3 and applied in all text, tables and figure labels. All headings, figure and table numbers, captions and in-text cross-references were renumbered and checked; the Table of Contents, List of Figures and List of Tables are now generated automatically from heading and caption styles, which removes the bookmark errors. Equations, units (e.g., kg/m ³ , m ³) and fonts were standardised, and front-matter page numbering is now continuous.	
F-7	Chapter 1: the scientific link between the LS–MK and CLS–MK investigations should be more explicitly established.	A paragraph in Section 1.12 now explains the two-phase design: Phase I (raw LS + MK, M25) and Phase II (calcined LS + MK, M40), linked by the hypothesis that a calcium source and a reactive aluminosilicate act synergistically and that calcination extends this synergy to higher-grade concrete. The link is repeated in the introduction of Chapter 3 and synthesised in the new Section 4.26.	Sections 1.12, 3 (introduction), 4.26
F-8	Chapter 2: the review would benefit from a more critical synthesis regarding sludge-source variability, calcination conditions, material reactivity and optimum replacement levels; claims concerning the absence of previous LS–MK investigations should be expressed cautiously and supported by comparison with closely related studies.	A new Section 2.6 critically discusses sludge-source variability, calcination conditions and reported optimum replacement levels, and Table 2.9 compares the most closely related studies (limestone–metakaolin, LC ³ , recycled lime sludge–calcined clay binders) with the present work. Statements of novelty throughout the thesis have been moderated (e.g., “to the best of the author’s knowledge”, “has received comparatively little attention”).	Sections 1 (introduction), 2.6–2.8; Table 2.9
F-9	Chapter 4: more critical synthesis linking macroscopic behaviour to physicochemical mechanisms; RSM models	A new Section 4.26 links workability, strength, durability and microstructure of both series to the underlying mechanisms. The RSM models now	Sections 4.23 and 4.26

No.	Examiner's comment	Response of the candidate	Location in revised thesis
	reported without explicit quadratic terms.	contain explicit quadratic terms (see F-4).	
F-10	Chapter 5: numerical claims and mechanistic interpretations should be verified and moderated.	Chapter 5 has been rewritten: every numerical value has been checked against Chapter 4, and the mechanistic statements are worded cautiously.	Chapter 5

Part B: Indian Examiner

The responses to the chapter-wise and general comments Indian Examiner of the Indian examiner are given in the table below.

No.	Examiner's comment	Response of the candidate	Location in revised thesis
I-1	Ch. 1, pp. 4–6, Sections 1.2–1.6: streamline to focus on LS and MK.	Sections 1.2–1.5 have been condensed, generic material removed and each section now ends with its relevance to LS and MK.	Sections 1.2–1.6
I-2	Section 1.9: lime sludge generation of 20–25 Mt/year inconsistent with Table 1.1.	Corrected: 20–25 Mt/year is India's paper production; dry sludge generation is 4.5–5 Mt/year in India and 16–20 Mt/year globally. The text now matches Table 1.1.	Section 1.9; Table 1.1
I-3	Section 1.13: objectives grammatically inconsistent; use a "To..." structure with measurable outcomes.	All five objectives were rewritten in a consistent "To..." form with measurable outcomes, retaining the approved scope.	Section 1.13
I-4	Section numbering and Table of Contents (1.12 used twice; 1.11–1.13 order).	Chapter 1 is now numbered 1.1–1.14 without duplication, and all sections throughout the thesis were renumbered; the TOC is generated automatically from the headings.	Chapter 1; TOC
I-5	Ch. 2: describe the literature search methodology.	A new Section 2.1 describes the databases, keywords, period and inclusion/exclusion criteria.	Section 2.1

No.	Examiner's comment	Response of the candidate	Location in revised thesis
I-6	Tables 2.2 and 2.3 duplicated; Table 2.2 should cover compressive, flexural and tensile strength.	Table 2.2 has been replaced with a new table on the effect of metakaolin on compressive, split tensile and flexural strength; Table 2.3 covers durability and microstructure only.	Section 2.2; Tables 2.2–2.3
I-7	MK and CC terminology used interchangeably.	The distinction is defined in Sections 1.10 and 2.3: MK is the high-purity calcined kaolin used experimentally; CC is used only for literature on calcined clays. All mixes, tables, figure captions and RSM symbols now use MK.	Sections 1.10, 2.3; Chapters 3–4
I-8	Research gaps 1, 2, 3 and 9 overlap.	The nine gaps have been consolidated into six distinct gaps.	Section 2.7
I-9	Section 2.5 missing from TOC.	Resolved by the automatic TOC (now Section 2.7, Identified Research Gaps).	TOC
I-10	Section 3.1: include source/supplier, grade and specifications of materials.	Table 3.1 added, giving the source, grade and specification of each material.	Section 3.1; Table 3.1
I-11	Two subsections numbered 3.2.1.2.	Subsections renumbered 3.2.1.1–3.2.1.6 (and 3.2.2.x–3.2.4.x).	Section 3.2
I-12	Table 3.4: heading “Result (Typical/Example)” unclear.	The column now reads “Measured value” (Table 3.5). The example values in the grading-zone table were replaced by the measured values computed from the sieve analysis (Zone II).	Tables 3.5–3.6
I-13	Section 3.3.1: 2% air void written $1 - 0.2$; aggregate calculations do not yield 1100 and 700 kg/m ³ .	Corrected to $1 - 0.02 = 0.98 \text{ m}^3$. The coarse-to-fine aggregate fraction (0.605:0.395 by absolute volume) that yields the batched 1100 and 700 kg/m ³ is now stated and justified with reference to IS 10262:2019; the superplasticised mix calculation was also corrected.	Sections 3.3.1–3.3.2, 3.4

No.	Examiner's comment	Response of the candidate	Location in revised thesis
I-14	Section 4.1: control does not have the highest workability; explain the contrasting effects of LS and MK.	Section 4.1 was rewritten: C70M15L15 has the highest and C80M10L10 the lowest slump, with the control intermediate. The contrasting effects are explained in terms of particle morphology, fineness, water demand and absorption, and clinker dilution.	Sections 4.1–4.2
I-15	Sections 4.7 and 4.8: cross-check values; C85M0L15 has the lowest strength; control 59 vs 49 MPa.	Corrected: C85M0L15 has the lowest strength at all ages; the strength ranges now agree with Table 4.7; the M40 control 90-day strength is 49 MPa.	Sections 4.7–4.8
I-16	Page 88: the explanation for the superior strength of 15% MK–10% CLS needs stronger scientific support.	The mechanism ($\text{CaO} \rightarrow \text{Ca(OH)}_2$ for pozzolanic reaction; calcite–alumina carboaluminate formation) is now supported by literature on limestone–calcined clay systems and by the TGA, calorimetry and XRD results, and is explicitly moderated as a plausible interpretation.	Section 4.8
I-17	Table 4.8 caption duplicates Table 4.7.	Caption corrected to “Compressive strength of CLS–MK mixes”.	Table 4.8
I-18	Sections 4.1 and 4.3: opposite workability trends for LS and CLS require explanation.	A technical explanation (calcination, free CaO hydration, fineness, water demand) has been added to Section 4.3.	Section 4.3
I-19	Sections 4.3 and 4.8: MK and CC used inconsistently.	All mix tables, captions and text now use MK (see I-7).	Chapters 3–4
I-20	Section 4.15.1: discussion contradicts Table 4.16; reference to 30% LS; explain acid-resistance mechanism.	Rewritten: only M15CL5 shows lower mass loss than the control (11%); most other mixes lose the same or more. The 30% reference was removed, and the mechanism (pozzolanic consumption of Ca(OH)_2 , C–(A)–S–H formation, densification, reduced	Section 4.15.1

No.	Examiner's comment	Response of the candidate	Location in revised thesis
		permeability, role of excess calcium from CLS) is explained.	
I-21	Table 4.35: improve format.	The cost table was rebuilt (Table 4.36) with mixes as rows and cost components as columns, each mix compared with the control of the same grade; the text was revised accordingly.	Section 4.25; Tables 4.35–4.36
I-22	Ch. 5: separate headings for M25 LS–MK and M40 CLS–MK; Conclusion 9 incomplete; check mix designation and GWP in Conclusion 17 and R^2 range in Abstract vs Conclusion 13.	Chapter 5 is reorganised under 5.1 General, 5.2 M25 LS–MK, 5.3 M40 CLS–MK, 5.4 Statistical, environmental and economic conclusions, and 5.5 Future scope. The incomplete flexural-strength conclusion was completed. Mix designations and GWP values now match Section 4.24 (the M25 15% MK + 20% LS case is identified as a screening scenario), and the R^2 values in the Abstract and Chapter 5 are identical and consistent with Table 4.22.	Chapter 5; Abstract
I-23	General: numbering and cross-references (1.10/1.11 duplicated; 2.5 missing; 3.2.1.2; 4.16.1.1; Figure 4.18/Table 4.18 references in 4.22.1).	All corrected; 4.16.1.1 is now 4.22.1.1, and Section 4.22.1 refers to Figure 4.25 and Table 4.22.	Throughout
I-24	Typographical and terminology errors (pages xi, xii, 77, 118, 135, 1).	All listed errors were corrected (chloride ion penetration, flexural strength, incorporating metakaolin, Correlation analysis, Ninety-day SEM images, superplasticiser, interfacial transition zone, Ca/Si ratios), and similar errors were corrected throughout.	Abstract; Chapters 3–4
I-25	Technical notation: “Ca by Si”, citation spacing, stray “s” after	Corrected throughout.	Throughout

No.	Examiner's comment	Response of the candidate	Location in revised thesis
	(Vashistha, Singh, et al., 2019).		
I-26	Formatting of TOC, List of Figures and List of Tables.	Regenerated as automatic Word fields with consistent styles, numbering, alignment and page numbers (right-click → Update Field after any further edit).	Front matter
I-27	References: consistency, DOI presentation; every cited source listed and every listed reference cited.	The reference list was cleaned in APA style: duplicate entries (a/b/c suffixes of the same paper) merged and the in-text citations updated, incomplete or irrelevant entries removed, missing ASTM C267 added, the XRD source corrected (Stutzman et al., 2016), DOIs added or corrected and presented uniformly, and the citations cross-checked against the list.	References

Part C: Additional Revisions

While addressing the comments on data consistency and statistical analysis, the following additional revisions were made; they are summarised in the table below.

No.	Examiner's comment	Response of the candidate	Location in revised thesis
A-1	Statistical re-check (in response to F-2 and F-4)	All statistical analyses were recomputed from the tabulated data. The correlation analysis (Table 4.22, Figure 4.25) now reports the values obtained from the data (moderate correlations of density with strength, $R^2 = 0.24\text{--}0.36$; water absorption–density $R^2 = 0.54$), and the related statements in the Abstract and Chapter 5 were revised.	Section 4.22.1; Table 4.22; Figure 4.25

No.	Examiner's comment	Response of the candidate	Location in revised thesis
A-2	Life cycle and cost assessment	Mix designations in the LCA tables were aligned with the experimental nomenclature, and the cost table was recomputed from the mix proportions and unit costs.	Sections 4.24–4.25

Regards

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