




## Influence of metakaolin and residual cement sludge from ready-mix concrete on strength and durability properties of concrete microstructure

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### ABSTRACT

Strength and durability characteristics of the concrete partially substituted with waste by-products have been reported. The optimal cement replacement percentage with cement sludge and metakaolin has been experimentally verified. The plausibility of replacing cement sludge and metakaolin with cement in concrete has been assessed based on test results. The percentage proportions of cement sludge were varied between 2% and 10%, increasing in 2% increments by weight of cement, with the optimal content identified as 4%. Subsequently, the metakaolin content was tested in the range of 3% to 15% by weight of cement, with the optimal proportion found to be 9% when combined with 4% cement sludge. An empirical model has been established between the 28-day compressive and flexural strength to assess the strength. The microstructure of cement sludge and metakaolin incorporated mix has been evaluated.

**Keywords:** Cement sludge; Metakaolin; Strength characteristics; Durability properties.

### 1. INTRODUCTION

In large-scale concrete production, ready-mix concrete (RMC) plays a crucial role. While RMC offers plentiful ads, the disposal of sludge produced from the cleaning process of RMC plants presents a substantial challenge for producers [1–3]. Each day, large volumes of cement sludge are produced in batching plants, concrete trucks, and mixing drums. RMC producers are facing a lot of problems in disposing of this wastewater a manner. Inappropriate clearance of this sludge can lead to severe environmental pollution due to its high ph content [4–8]. Cement sludge is an inevitable by-product of the RMC process, and disposing of waste cement sludge in water bodies can harm aquatic life, while disposal on land may contaminate groundwater, alter soil chemistry and inhibit plant growth; hence appropriate care should be taken in the disposal of this waste material [9–11].

The traditional disposal methods for cement sludge, such as dumping it at vacant lands, batching plant yards, construction sites, or landfills, are not only environmentally damaging but also contribute significantly to pollution [12, 13].

These practices increase cement production's overall carbon footprint, creating environmental and economic challenges in many developing and developed countries. Hence, improper disposal of this must be avoided to protect the environment. However, a more sustainable approach lies in recycling this waste material and integrating it into the concrete production process without being detrimental to its quality, which becomes an eco-friendly solution [14–16], ASTM C94 covers the requirements for materials, production, and delivery of ready-mixed concrete [17].

The process of recycling cement sludge for concrete production involves using the waste material as an alternative to cement. Cement sludge has high alkalinity, which can make it a suitable material for incorporation into concrete, provided it meets certain criteria [18–20]. Using sludge in concrete production helps address pressing environmental concerns such as waste management and pollution, while also offering substantial

economic advantages. Since the cement sludge production is unavoidable, one of the key benefits of recycling is the significant reduction in treatment costs for the concrete producers. Sludge does not require chemical pre-treatment, hence the overall cost for ready-mix concrete (RMC) producers is lowered, which is beneficial in a highly competitive industry [21–23].

In conjunction with cement sludge, metakaolin, a type of calcined clay mineral traditionally used in porcelain production, can also be employed as a cement replacement. Metakaolin is a pozzolanic material that reacts with the  $\text{Ca(OH)}_2$  in cement to form compounds towards enhance the properties [24–26]. When used as an alternative for Portland cement, metakaolin accelerates the hydration process, especially during the initial setting phase, and improves the concrete's overall durability. Incorporation of metakaolin not only improves the properties of concrete but also reduces the cost of production. The substitution of Portland cement with metakaolin lessens the environmental impact of concrete production by decreasing the reliance on energy-intensive cement manufacturing [27–31].

This study combines the use of dried cement sludge and metakaolin as cement replacement materials. It explores how such innovative practices not only create more sustainable and eco-friendly concrete but also lower production costs. By reusing waste by-products, the construction industry can contribute to a more sustainable future, creating high-performance, cost-effective building materials [32, 33]. This approach also supports the development of circular economy principles, where waste is minimised, and the lifecycle of materials is extended by increasing their strength and durability without detrimental to its quality.

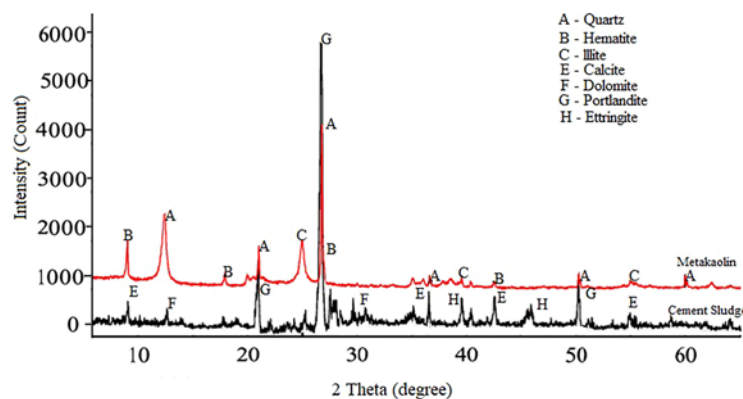
## 2. EXPERIMENTAL PROGRAMME

### 2.1. Materials

Cement sludge produced from the RMC was collected and oven dried at  $105^\circ\text{C}$  and repalcing cement. Metakaolin obtained from Supreme Industries Coimbatore was used in this study. Ordinary Portland cement of grade 53 endorsing the specifications of IS: 12269: 2013 has been used for the production of concrete [34]. Chemical properties of Metakaolin, cement sludge and Portland cement are shown in the Table 1.

**Table 1:** Chemical properties of Portland cement, cement sludge and Metakaolin.

CONSTITUENT	METAKAOLIN	CEMENT SLUDGE	OPC
CaO	16.54	31.33	64.40
$\text{SiO}_2$	52.27	13.75	21.53
$\text{Al}_2\text{O}_3$	27.29	9.28	5.95
$\text{Fe}_2\text{O}_3$	2.97	2.33	3.44
MgO	0.07	7.49	2.09
$\text{Na}_2\text{O}$	–	0.69	0.18
$\text{K}_2\text{O}$	0.39	1.31	0.13
LOI	0.47	22.83	0.54
Free CaO	–	0.49	0.46



**Figure 1:** XRD pattern of cement sludge and metakaolin.

In the present work, XRD analysis has been carried out for dry sludge and metakaolin. The most intense peaks were identified as quartz and hematite for the metakaolin. Besides peaks associated with clay mineral phases, such as illite also observed with a comparable angular extension in the XRD pattern. In the case of cement sludge, the most intense peaks were identified as portlandite and calcite. In addition, the peaks associated with hydrous calcium alumina sulfate phases, such as ettringite also found in the XRD pattern. Figure 1 shows the XRD pattern of cement sludge and metakaolin.

## 2.2. Mix proportion for the preparation of concrete

M<sub>20</sub> grade concrete was used with the mix ratio of 1:1.7: 3.19 showed in Table 2.

**Table 2:** Mix proportion of CC.

MIX ID	CEMENT SLUDGE (CSL) kg/m <sup>3</sup>	METAKAOLIN (MK) kg/m <sup>3</sup>	CEMENT kg/m <sup>3</sup>	FINE AGGREGATE kg/m <sup>3</sup>	COARSE AGGREGATE kg/m <sup>3</sup>	WATER kg/m <sup>3</sup>
CC	0	0	350	595	1116.5	175
CSL-2	7	0	343	595	1116.5	175
CSL-4	14	0	336	595	1116.5	175
CSL-6	21	0	329	595	1116.5	175
CSL-8	28	0	322	595	1116.5	175
CSL-10	35	0	315	595	1116.5	175
CSMK-3	10.5	10.5	329	595	1116.5	175
CSMK-6	21	21	308	595	1116.5	175
CSMK-9	31.5	31.5	287	595	1116.5	175
CSMK-12	42	42	266	595	1116.5	175
CSMK-15	52.5	52.5	245	595	1116.5	175

To water-to-binder ratio of 0.50 has been considered for the production of concrete. Potable water was used to prepare the control mix, and cement sludge (CSL) and metakaolin (MK) were added mixes. Mixes of the CSL and cement sludge metakaolin (CSMK) series were prepared. Concrete added with cement sludge is denoted as the CSL-series. In the CSL series of mixes, portland cement was replaced by the cement sludge in the percentage proportions of 2, 4, 6, 8 and 10 wt% of cement and specimens were named as CSL-2, CSL-4, CSL-6, CSL-8 and CSL-10, respectively.

Concrete incorporated with cement sludge and metakaolin CSMK-series consists of wt% of cement sludge and with different percentage proportions of metakaolin with cement. Metakaolin has been partially replaced at 3, 6, 9, 12, and 15 wt% of cement. CSMK-series mixes were designated as CSMK-3, CSMK-6, CSMK-9, CSMK-12, and CSMK-15.

## 2.3. Test methods

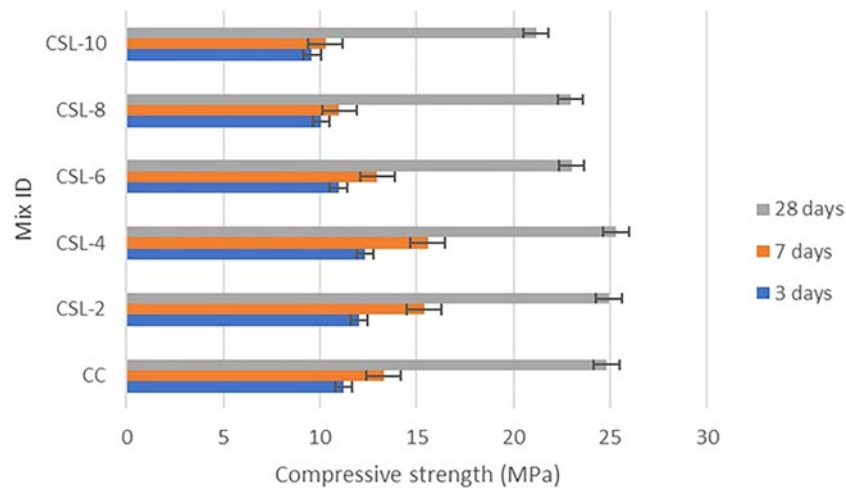
The mechanical properties of conventional, CSL, and CSMK mixes were investigated [35]. The experiments were executed according to the relevant Indian standards. 3, 7, and 28 days compressive strength has been calculated for the specimens of size 100 × 100 × 100 mm and 7 and 28 days flexural strength of prismatic samples of size 100 × 100 × 500 mm were tested. Furthermore, the durability of the concrete was evaluated by focusing on 3.5% acid resistance and the corrosion activity of steel. Samples were submerged in sulfuric acid, and after 30, 60, 90, and 120 days, the influence of acid immersion was assessed. Cylindrical specimens (150 × 300 mm) embedded with a steel rod have been used for corrosion testing. Half-cell potential test has been performed as per ASTM C 876 to study the corrosion activity of the steel [36].

## 3. RESULTS AND DISCUSSIONS

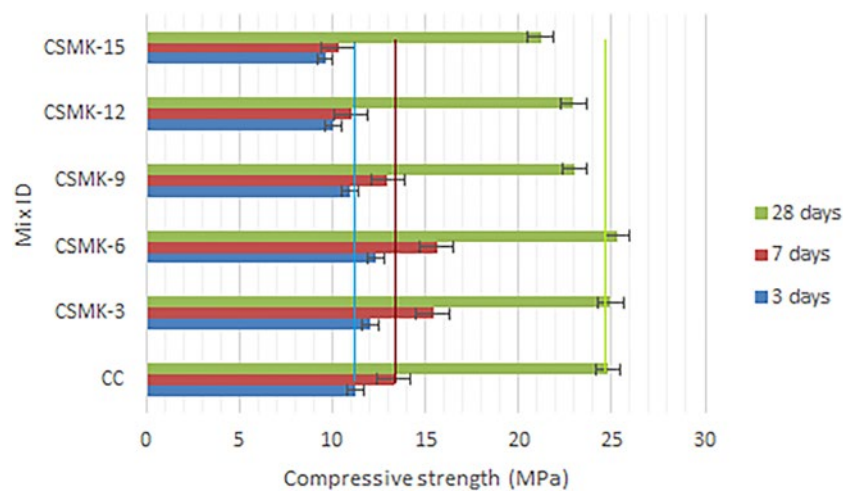
Effect of metakaolin and cement sludge has been explored and the outcome of the tests was considered for the evaluation.

### 3.1. Compressive strength variations of CSL mixes

Variations in compressive strength of CSL-series mixes at 3, 7 and 28 days are shown in Figure 2. It was evident that up to 4% cement can be replaced with sludge for CSL-series mixes. More than 4% replacement of cement



**Figure 2:** Compressive strength variations of CSL-series mixes.



**Figure 3:** Compressive strength variations of CSMK-series mixes.

with cement sludge would reduce the compressive strength. Hence, the optimal cement sludge replacement content can be considered as 4%.

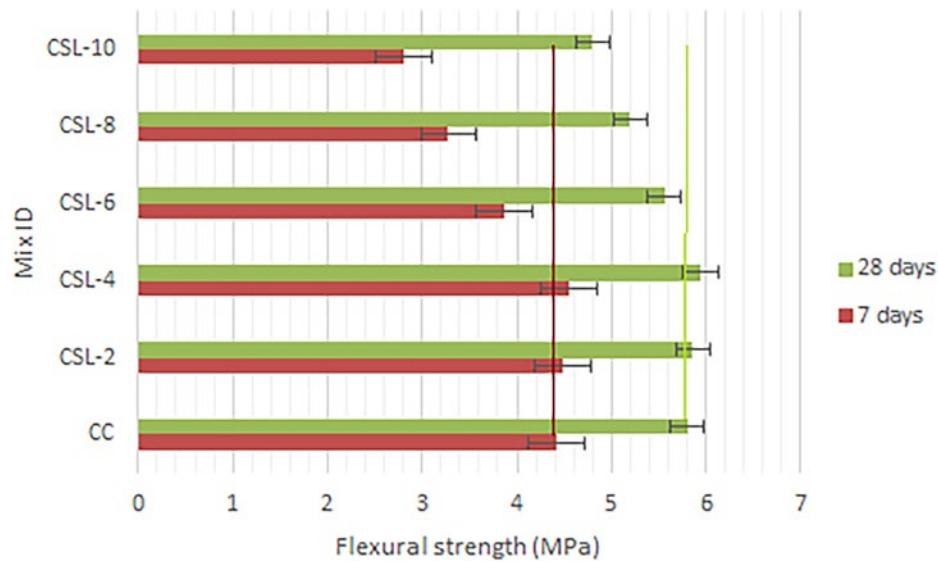
CSL-4 mixes gained 0.71%, 0.52%, and 0.63% higher strength than control mix at 3, 7 and 28 days, respectively. Only a minute increase in compressive strength was perceived (<1%) due to the pore filling effect of cement sludge in concrete, therefore, a minimal amount of cement sludge can be substituted. A drop in compressive strength was noted due to the presence of low Calcium oxide (CaO) content of sludge.

The CaO content of cement sludge is 31.33% compared with Portland cement. During the cleaning process, major Cao content is leached into to wash out water. Figure 3 shows the compressive strength variations of CSMK-series mixes. A prominent increase was observed in compressive strength of mixes CSMK-3, CSMK-6 and CSMK-9 with respect to the compressive strength of control concrete. Sample CSMK-9 encompassing 9% of sludge and 9% metakaolin obtained additional strength than CC mix.

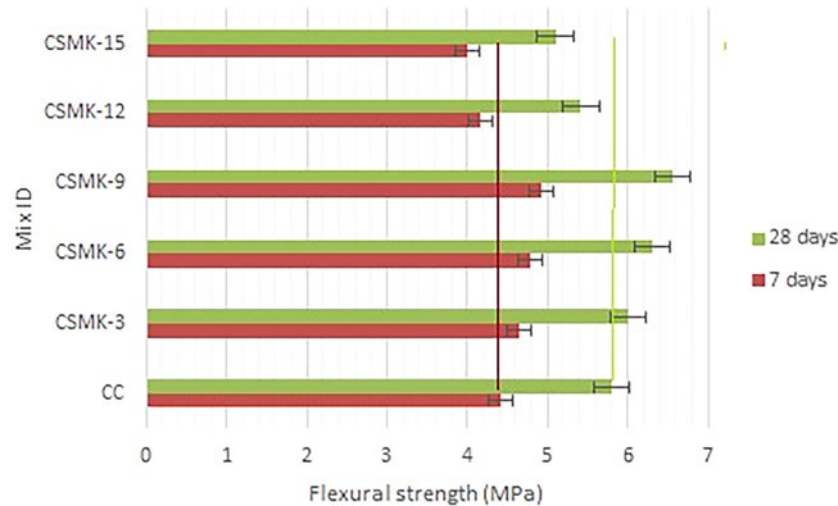
The compressive strength increase of CSMK-9 was found to be 10.86%, 11.23%, and 11.79% at 3, 7 and 28 days of curing, respectively. Presence of metakaolin enhanced the strength. Enhancement of compressive strength may be due to reaction of metakaolin with calcium oxide in cement that forms additional cementitious compounds upon cement hydration and tends to increase in the compressive strength.

### 3.1.1. Flexural strength

The flexural strength was assessed for the mixes of CSL and CSMK series. Two time intervals (7 and 28 days) were considered for the assessment. The flexural strength of the CC mix was compared with that of the



**Figure 4:** Flexural strength variations of CSL-series concrete prisms.



**Figure 5:** Flexural strength variations of CSMK-series concrete prisms.

cement sludge incorporated concrete mix. The test results indicate that up to 4% of cement can be replaced with cement sludge without any reduction in strength, but afterwards suddenly a sudden dip was observed in flexural strength. Flexural strength variations of concrete CSL-series mix at 7 and 28 days are shown in Figure 4.

Due to the densification effect of fines which limits the crack propagation through the cement matrix. The experimental results revealed that beyond 4% replacement of sludge in concrete reduces the strength. In case of mix CSL-4, it was witnessed that the flexural strength is marginally increased than the control concrete. Flexural strength increase of 0.39%, 0.51% have been found for the mix CSL-4 after 7 and 28 days than the control mix.

Higher replacements with cement sludge result in a reduction of flexural strength. This may be attributed towards less CaoCao content in the sludge. Only 31.33% Cao is present in cement sludge. During cleaning, water dissolves CaoCao present in cement. Hence, it was found less in the sludge. Besides, the higher LOI (22.83%) specifies the existence of more carbon particles. Carbon particles are porous and absorb more water, which results in lower strength. Flexural strength variations of CSMK-series mixes at 7 and 28 days are shown in Figure 5.

Mix combined with 4% cement sludge and 9% metakaolin (CSMK-9) gained higher strength than the control mix. The surface area of fine metakaolin particles is more and produces nuclei to improve the strength gaining process in the cement. Formation of C-S-H gel is limited and pores are more in control mix but in case of

metakaolin infused mix C-S-H gel formed in the cement matrix proliferate in to the voids. Formation of C-S-H gel enriches the strength properties of concrete [37].

### 3.1.2. Relationship between compressive and flexural strength

Compressive and flexural strength are two key indicators used for evaluating the quality of concrete. A mathematical model has been developed to predict flexural strength based on compressive strength. The data from the CSMK-series of mixes were taken into consideration for the prediction of strength, and by regression analysis, a correlation was established between the 28-day compressive and flexural strengths of CSMK mixes, as expressed in Eq. (1):

$$\epsilon_f = 0.0197 (\epsilon_c)^{1.1843} \text{ MPa} \quad (1)$$

Where  $\epsilon_f$  and  $\epsilon_c$  denote the 28-day flexural and compressive strengths (MPa) of CSMK mixes, respectively. The correlation coefficient of the regression equation indicates 91.3% confidence. Considering the dependable values, the error was found to be 8.7% which clearly evident the steadfastness of the developed model.

## 3.2. Durability of properties of CSL and CSMK mixes

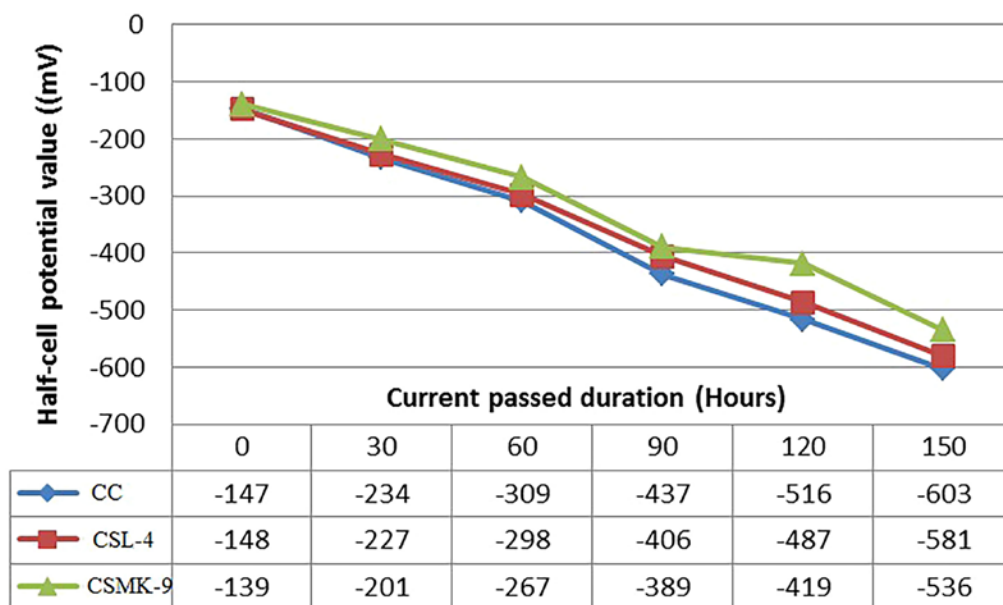
### 3.2.1. Probability of corrosion

To assess the chances of corrosion in reinforcement half-cell potential test has been conducted. The electrical potential difference has been measured in the test between a reference electrode fixed in the concrete and the embedded reinforcement. Readings taken from the test are indicative of the corrosion state of the reinforcement. By comparing the results with the standards, the probability of corrosion can be assessed.

A test was carried out for CC, CSL-4 and CSMK-9 mixes and based on the test outcomes, probability of the corrosion was assessed. The test revealed that chances of corrosion in sample CSL-4 is marginally less than CC mix. The pore filling effect of fines reduces the chloride penetration thereby reduces the probability of corrosion in sample CSL-4 and CSMK-9 (Table 3).

**Table 3:** Probability of corrosion (ASTM C-876).

SL.NO	HALF-CELL POTENTIAL (MV)	PROBABILITY OF CORROSION
1.	Lesser than -200	10% probability
2.	-200 to -350	Uncertain
3.	Higher than -350	90% probability



**Figure 6:** Half-cell potential values of CC, CSL-4 and CSMK mixes.



Figure 6 illustrates the half-cell potential values for control concrete, CSL-4, and CSMK-9 mixes. The analysis of these values shows that the probability of corrosion is less in the CSMK-9 mix than in other mixes. This higher corrosion confrontation is likely attributed to the addition of metakaolin. The fine particles of metakaolin reduces the porosity and leading to the formation of a denser microstructure. This denser structure acts as a barrier and minimizing the likelihood of corrosion.

In terms of Probability of corrosion, the incorporation of metakaolin also positively impacts the interfacial transition zone (ITZ) of the concrete [38]. By enriching the ITZ microstructure, metakaolin helps to reduce the number of pores in this critical region, which restricts penetration of chloride in samples. This decrease in chloride penetration significantly enhances the concrete's ability to resist corrosion, thereby improving the overall durability and longevity of the reinforced concrete structure [39, 40].

### 3.2.2. Resistance to acid attack

The investigation into the effect of CSMK mixes on 3.5%  $H_2SO_4$  confrontation focused on understanding how these materials influence the concrete's resistance against acidic environments. The results showed that sample CSMK-9, which contained Metakaolin, experienced a lower percentage of weight loss compared to the control concrete. This indicates that the presence of Metakaolin improves the concrete's resistance to sulfuric acid, making it more durable in such environments.

The higher resistance is due to the pozzolanic properties of Metakaolin. It is a silica-based material that undergoes a pozzolanic reaction with the calcium hydroxide (CH) in cement. Production of calcium silicate hydrate (C-S-H) improves the binding properties of concrete. Availability of C-S-H gives a denser and durable microstructure that is less susceptible to acid attack [41, 42].

Higher weight loss in the CC mix was observed because of the chemical reaction between calcium hydroxide (CH) and sulfuric acid. When sulfuric acid comes into contact with the concrete, it responds with the CH in the paste to form calcium sulfate ( $CaSO_4$ ), which is a soluble product [43, 44]. This reaction leads to the formation of a calcium sulfate layer on the surface of the concrete, which can be washed away over time, causing material loss. Furthermore, this reaction can also result in the breakdown of the bonds between the constituents, making the concrete more prone to deterioration and weakening.

Weight loss in samples due to the influence of  $H_2SO_4$  is shown in Figure 7. The presence of Metakaolin in CSMK-9 mitigates the leaching process by reducing the available calcium hydroxide, thereby limiting the extent of the acid attack. Metakaolin's pozzolanic activity leads to the formation of additional stable compounds that resist the corrosive effects of sulfuric acid, thus reducing the overall weight loss and enhancing the sulfuric acid resistance of the concrete [45–49].

### 3.2.3. Sem analysis of cc and csmk-9 mix

The microstructure analysis of the cement matrix reveals that the integration of metakaolin considerably improves its overall structure. Metakaolin, which is a dehydroxylated form of kaolin clay, acts as a pozzolanic material. It mainly reacts with  $Ca(OH)_2$  produced in cement hydration and formulates C-S-H gel [50, 51]. The C-S-H gel is the principal binding component imparts strength. Figure 8 shows the SEM image of CC and CSMK-9 mix.

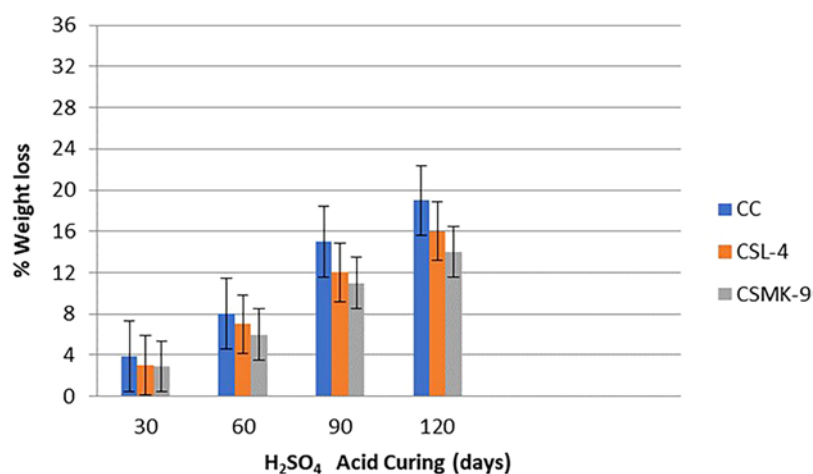
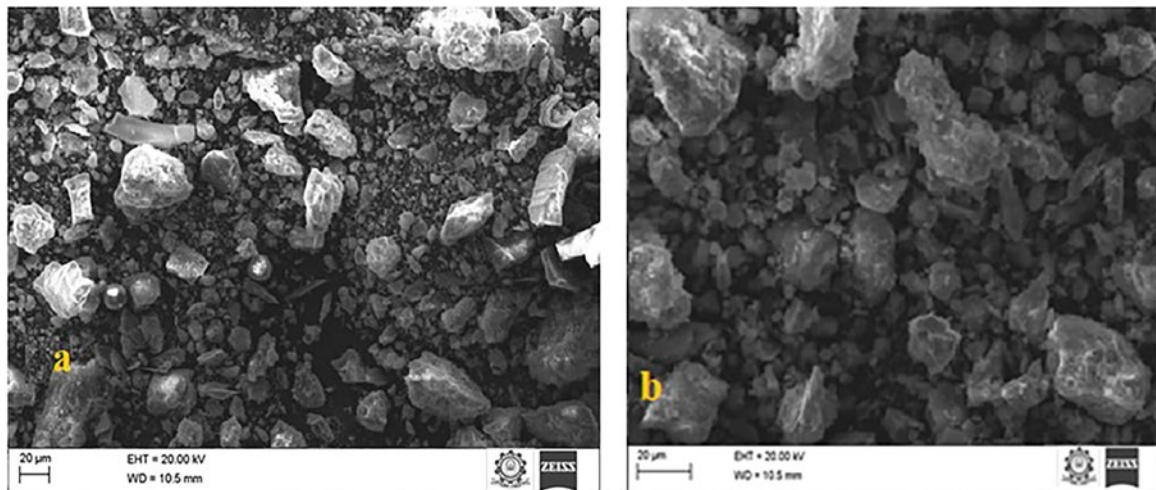


Figure 7: Weight loss due to  $H_2SO_4$ .



**Figure 8:** SEM image of CC and CSMK-9 mix.

The formation of C-S-H is crucial in filling the micro-pores within the cement matrix. These micro-pores are typically areas of weakness that can facilitate the ingress of moisture or other harmful agents, potentially leading to the formation and propagation of cracks. By filling these voids with C-S-H gel, metakaolin helps to make the cement structure denser, more compact, and less permeable. This improves the overall durability of the material, as it resists the penetration of water and other damaging substances.

Additionally, the enhanced microstructure reduces the potential for crack formation and propagation. The denser and more cohesive matrix is less prone to the stresses and forces that can cause fissures to develop. This increased resistance to cracking is directly tied to the pozzolanic effect of metakaolin, which not only strengthens the matrix but also helps to improve the long-term performance of the cementitious material by reducing shrinkage and increasing its mechanical properties.

#### 4. CONCLUSION

The experimental study on integrating cement sludge and Metakaolin into the cement matrix has revealed the following experimental findings:

- The durability and strength characteristics of the mix CSMK-9 are found on par with conventional mix for the replacement of 4% cement sludge and 9% metakaolin for cement.
- Only slight improvement in strength is possible if sludge alone replacing the cement. Addition of metakaolin with sludge would improve the pozzolanic properties of concrete.
- Metakaolin present with cement sludge amended the electrical resistivity and conferred dense microstructure and prolonged the corrosion activity of steel in concrete.
- The pozzolanic action of metakaolin with other constituents gives higher acid resistance to the mix CSMK-9 at all the ages.
- The presence of C-S-H compounds in the mix exhibit strength and improved performance of concrete against formation of cracks. The micro pore filling effect of cement sludge also strengthen the cement matrix against crack propagation.

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