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ORIGINAL RESEARCH PAPER

# Accelerated curing effects on performance of metakaolin concrete

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## ABSTRACT

The manufacturing of cement liberates the green-house gasses into atmosphere. To overcome this problem so many alternative materials has been invented by researchers to minimize addition of cement. The incorporation of these alternative materials as cementitious material in concrete enhances the attributes of concrete. In this scenario metakaolin gained momentum as a substitution to cement in concrete. Most of the researchers studied the performance of concrete incorporating metakaolin as cementitious material in normal curing conditions. There is a need for analysing the impact of accelerated curing on properties of concrete by incorporating metakaolin as cementitious material. The current construction industry needs high early strength for removal of form work in early ages. The accelerated curing is a method which provides high early strength. In this study, different proportions of metakaolin are added as partial alternative to cement and cured in accelerated curing tank for 3.5 h. The strength parameters test, durability test, and micro-structural parameter tests are performed on these samples. Further, micro-structural analysis has been carried out using SEM, and EDX tests. Results depicted the incorporation of 15% of metakaolin as substitute to cement amplifies the overall performance of concrete in accelerated curing regime.

## KEYWORDS

hot curing, metakaolin, strength, durability, SEM, EDX analysis

## 1. INTRODUCTION

Recent advances in the construction sector have dramatically increased the usage of concrete owing to its frequent usage and strength parameters. It has become the second-largest consumed material in the world next to water. As per IBEF (Indian Brand Equity Foundation) report 2021, the production of cement is estimated to increase by 18 per cent. The global demand for cement is expected to rise as a result of emerging economies' rapid infrastructure development [1]. Furthermore, according to a 2009 World Business Council for Sustainable Development report, global manufacture of cement is expected to touch 4,500 million tonnes by 2040 [2]. The cement manufacture produces both direct and indirect greenhouse gas emissions. The combustion of fossil fuels to heat the kiln generates $CO_2$ indirectly, whereas heating limestone emits $CO_2$ directly [3]. Reducing the emissions from the cement manufacturer or reducing the utilization of cement is an important challenge. Another option to reduce cement usage and concrete expenditures is to use industrial by-products as supplemental cementitious materials (SCMs) to substitute cement in concrete [4–6]. The repeatedly utilized SCMs are fly-ash, metakaolin, silica fume, lime stone, natural pozzolana; most of them are industrial by-products which require utilization. Metakaolin is rapidly gaining prominence as an SCM in concrete owing to its tiny particle size and large surface area, which allows it to respond fast and improve concrete performance. In blended concrete, SCM replace the cement to a major extent due to their strength gaining capacity, high resisting nature, durability and lower cost. In addition, the greenhouse gases are reduced, which are produced in the manufacturing of cement, resulting in a reduction of pollution. Concrete with binary, ternary and tetranary blending of SCM provides significant

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benefits over the mix containing only OPC. The partial substitute of cement by metakaolin not only reduces the production cost of concrete but also the addition of such SCM with cement has a beneficial effect on workability and permeability of concrete. The proportional use of a combination of different SCMs not only develops strength but also improves the overall durability of concrete [7, 8]. The utilization of silica fume as a substitute to cement amplifies the strength parameters. This is because of improvement in aggregate paste bond improvement and enhanced microstructure [8]. The incorporation of 10% of metakaolin as cement replacement material shows optimal amplification in durability and strength parameters [9]. The pozzolanic characteristic of metakaolin modifies the micro-structural properties of paste, improving the strength and durability [10, 11]. Metakaolin is obtained from various primary and secondary sources of kaolinite, mainly used in the manufacture of ceramics [12]. The impact of metakaolin on different types of concrete has been analysed by most of the researchers in water cured samples at standard temperature [13, 14]. Generally, the 28 days' water cured samples at a temperature of 20 °C are tested for standard compressive strength. This period is too long in today's concreting technology; also the maturity of concrete will depend on the both time and temperature of curing. The increasing the temperature will improve the strength of concrete in early ages [15]. There is a need to analyse the impact of accelerated curing on attributes of concrete incorporating metakaolin as cementitious material. The impact of metakaolin on performance of concrete in accelerated curing regime is done in the current experimental study. The reactivity of metakaolin depends on various factors with the curing temperature being one of the factors which accelerates its reactivity.

## 2. METHODOLOGICAL ANALYSIS

### 2.1. Materials utilized

The hydraulic cement of 43 grade having specific gravity of 3.1 has been utilized in this study as a binder. The natural sand confirming to zone-II, specific gravity as 2.65 is utilized as a fine aggregate. The coarse aggregate with utmost size 20 mm, specific gravity as 2.75 is used in mix. The metakaolin utilized in this study is procured from the ASTRRA chemicals, Chennai. In this study the cement has been substituted with the metakaolin in intervals of 5%, starting with 5% up to 25% substitution. Hence total six mixes were prepared for the current study. The super plasticizer PermaPlast PC 220 has been utilized in this study. The elemental composition particulars of metakaolin (Mk) are listed in Table 1 and the EDX analysis is shown in Fig. 1.

### 2.2. Concrete mix

The mix proportions of concrete are blended based on IS: 10262-2019 [16] standards and packing density method [10].

*Table 1.* Elemental configuration of metakaolin utilized in the study

| Element | Weight % | Atomic % |
|---|---|---|
| Si | 61.98 | 61.60 |
| Al | 35.57 | 36.80 |
| Ca | 0.23 | 0.26 |
| Mg | 0.25 | 0.29 |
| Ti | 1.96 | 1.14 |

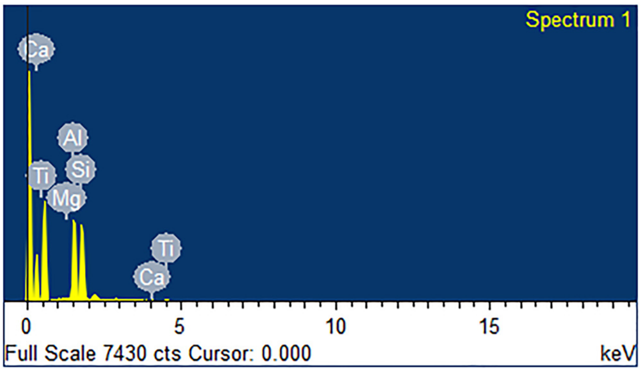


*Fig. 1.* EDX analysis of metakaolin powder used in the study

The mix particulars of concrete are illustrated in Table 2 considering different trial mixes. The cement content has been increased by decreasing the w/b ratio of concrete mix. Three water binder (w/b) ratios and different proportions of metakaolin are used in this study for knowing the impact of accelerated curing on performance of concrete incorporating metakaolin as cementitious material. The control mix denoted as CM, the mix containing 5% of metakaolin as replacement to cement denoted as MK5. Similarly, the mix MK10, MK15, MK20, and MK25 denotes that the mix containing 10%, 15%, 20%, and 25% of metakaolin as a SCM.

### 2.3. Methods

The strength, durability and micro-structural studies are conducted for all samples in triplicate. The concrete cubes of size 100 × 100 × 100 mm and beams of size 100 × 100 × 500 mm are casted after mixing of concrete. The cubes are dipped in curing tank with water at 100 °C for 3.5 h. The accelerated curing samples are collected and tested using compression testing machine as per IS: 516-1959 standards [17]. The beams after accelerated curing are collected and tested utilizing flexural strength testing machine as per the IS: 516-1959 standards [17]. The cubes are tested on triplicate for electrical resistivity test as per ASTM C 1202 standards [18]. The UPV test has been performed to assess the durability aspect of concrete by passing ultrasonic pulse velocity through cubes as per IS: 13311. After casting of the moulds, they are stored in the laboratory for 23 h before they may be kept in accelerated curing tank. The concrete samples are kept in accelerated curing tank for 3.5 h at 100 °C as per IS: 9013-1978 [19].

*Table 2.* Mix proportions (kg $m^{-3}$)

| Mix ID | Cement | Metakaolin (MK) | Sand | Coarse Aggregate |  |  | Water | Plasticizer |
|---|---|---|---|---|---|---|---|---|
|  |  |  |  | 20 mm | 12.5 mm | 10 mm |  |  |
| For 0.25 w/b ratio |  |  |  |  |  |  |  |  |
| C100 | 563.9 | 0 | 599.97 | 722.87 | 192.8 | 289.2 | 141.39 | 6.77 |
| MK5 | 536 | 28 | 587 | 707 | 188.8 | 283.2 | 141.39 | 6.77 |
| MK10 | 508 | 56 | 573 | 691 | 184 | 276 | 141.39 | 6.77 |
| MK15 | 479 | 85 | 559 | 673 | 179.6 | 269.4 | 141.39 | 6.77 |
| MK20 | 451 | 113 | 545 | 656 | 174.8 | 262.2 | 141.39 | 6.77 |
| MK25 | 423 | 141 | 532 | 641 | 170.4 | 255.6 | 141.39 | 6.77 |
| For 0.30 w/b ratio |  |  |  |  |  |  |  |  |
| C100 | 507 | 0 | 607 | 732 | 194.8 | 292.2 | 151.9 | 6.079 |
| MK5 | 481 | 26 | 587 | 707 | 188.8 | 283.2 | 151.9 | 6.079 |
| MK10 | 456 | 51 | 583 | 702 | 186.8 | 280.2 | 151.9 | 6.079 |
| MK15 | 431 | 76.05 | 570 | 686 | 182.8 | 274.2 | 151.9 | 6.079 |
| MK20 | 405 | 102 | 551 | 663 | 176.8 | 265.2 | 151.9 | 6.079 |
| MK25 | 380 | 127 | 546 | 658 | 175.2 | 262.8 | 151.9 | 6.079 |
| For 0.35 w/b ratio |  |  |  |  |  |  |  |  |
| C100 | 434.89 | 0 | 629.8 | 749.76 | 198.8 | 298.2 | 152.2 | 5.249 |
| MK5 | 413 | 21.87 | 618.8 | 745.6 | 198.4 | 297.6 | 152.2 | 5.249 |
| MK10 | 391.5 | 43.5 | 604 | 728 | 194 | 291 | 152.2 | 5.249 |
| MK15 | 369.75 | 65.25 | 592 | 713 | 190 | 285 | 152.2 | 5.249 |
| MK20 | 348 | 87 | 552 | 665 | 176.8 | 265.2 | 152.2 | 5.249 |
| MK25 | 326.25 | 108.75 | 547 | 660 | 175.6 | 263.4 | 152.2 | 5.249 |

## 3. RESULTS ANALYSIS

### 3.1. Compressive strength

The test results of varying concrete mixes are illustrated in Fig. 2. It is depicted in Fig. 2 that the accelerated curing enhances the compressive strength. The rise in temperature accelerated the reaction of hydration thus there is an early strength gain [20]. The incorporation of 5% of metakaolin as a SCM shows 18.75%, 15.9%, and 11.11% amplification in compressive strength of concrete for 0.25, 0.3, and 0.35 water cement ratios respectively. The incorporation of 10% of metakaolin as alternative to cement shows 35.41%, 27.3%, and 22.22% amplification in compressive strength of concrete for 0.25, 0.3, and 0.35 water cement ratios respectively. The incorporation of metakaolin as partial alternative to cement by 15% shows the optimum enhancement in strength of concrete. The 15% accumulation of metakaolin shows a maximum of 33.3%, 38.6%, and 45.8% enhancement in compressive strength for 0.35, 0.30, and 0.25 w/b ratios as compared to the normal mix. This is because of the pozzolanic reaction amongst metakaolin and portlandite, which results in the development of a C–S–H gel. It refines the microstructure of concrete as well as the bonding between the particles, resulting in increased concrete strength [21]. The incorporation of 20% of metakaolin as alternative to cement shows 39.58%, 31.8%, and 27.8% amplification in compressive strength of concrete for 0.25, 0.3, and 0.35 water cement ratios respectively. Similarly, the incorporation of 25% of metakaolin as alternative to cement shows 8.33%, 13.63%, and 18.75% augmentation in compressive strength for 0.25, 0.3, and 0.35 water cement ratios respectively. The diminution in strength is seen in the MK25 mix analogical to the MK15 Mix. This is because of the clinker dilution

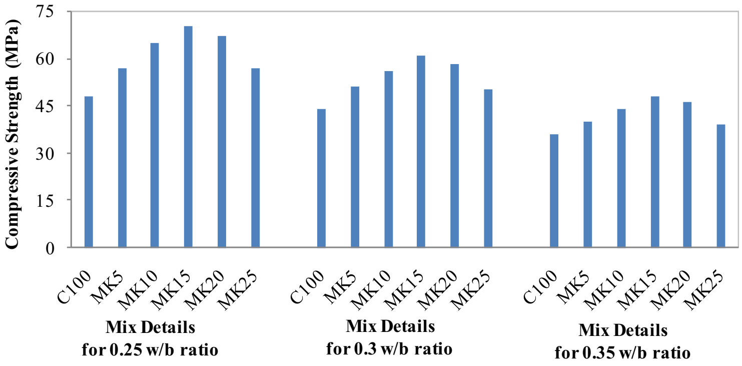


*Fig. 2.* Compressive strength test results

effect, which results from the replacement of higher proportions of cement by metakaolin. The available portlandite concentrations will decrease if cement replacements are higher [22]. It reduces the strength as compared to optimum mix MK15. The 0.25 w/b ratio mixes show amplified strength results as compared to 0.3 and 0.35 w/b ratios mixes. This is because of the lower w/b ratios diminish the air pores formed in concrete and improves the strength.

## 3.2. Flexural strength of concrete

The test was conducted for all w/b ratios for accelerated curing samples. The test results are depicted in Fig. 3. It depicts the flexural strength test results of varying concrete samples cured in accelerated curing tank for 3.5 h. From Fig. 3 it is identified that the incorporation of 5% of metakaolin as substitute shows 20, 17.39, and 11.62% enhancement in the flexural strength for 0.25, 0.30, and 0.35 w/b ratios. The incorporation of 10% of metakaolin displays 32, 26.08, and 22.1% amplification in the flexural strength results. The incorporation of 15% of metakaolin as alternative to cement shows 38, 32.6, and 27.9% amplification in the flexural strength for 0.25, 0.30, and 0.35 w/b ratios. The incorporation of 20% of metakaolin shows 36, 28.26, and 25.58% amplification in the flexural strength for 0.25, 0.3, and 0.35 w/b ratio. Similarly, the incorporation of 25% of metakaolin as alternative to cement shows 18, 15.21, and 10.46% amplification in the flexural strength for 0.25, 0.30, and 0.35 w/b ratios. The incorporation of 15% of metakaolin as alternative to cement shows desired amplification in the flexural strength of concrete cured in accelerated curing tank. This is because of higher pozzolanic activity of metakaolin in accelerated curing. An adequate amount of CSH gel has been developed by the addition of 15% of metakaolin in concrete. The 0.25 w/b ratio mixes show higher strength results as compared to 0.30 and 0.35 w/b ratio mixes. This is because of the lower w/b ratios diminishes the development of pores in concrete [23].

## 3.3. Electrical resistivity (ER)

This is a significant parameter for knowing the presence of micro-cracks and pores in concrete. The bulk electrical resistivity test has been conducted on 100 × 100 × 100 mm size cubes in accordance with ASTM C1202 [18] standards. Figure 4 depicts the ER test results of accelerated curing samples of different concrete mixes. It is depicted in Fig. 4 that the 0.25 w/b ratio mixes show enhanced electrical resistivity test results in comparison with other w/b ratios. The incorporation of 5% of metakaolin shows 11.4, 19.4, and 24.5% enhancement in electrical resistivity as compared to control mix for 0.35, 0.30, and 0.25 w/b ratio. The incorporation of 10% of metakaolin in concrete depicts 26.2, 28.15, and 35.45% amplification in electrical resistivity for 0.35, 0.30, and 0.35 w/b ratio as compared to control mix. Similarly, the incorporation of 15% of metakaolin displays desired results in electrical resistivity of concrete for 0.35, 0.30, and 0.25 w/b ratios. The incorporation of 15% of

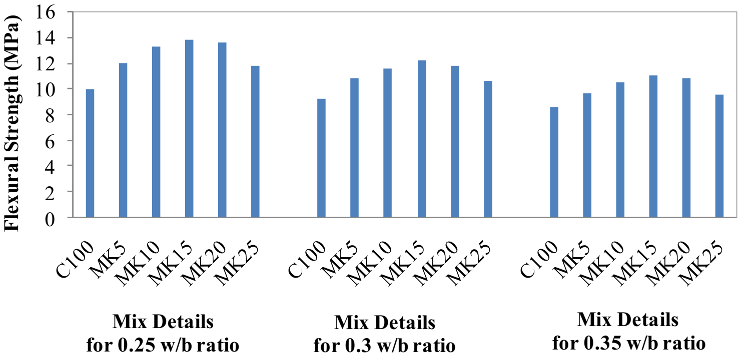


*Fig. 3.* Flexural strength test results

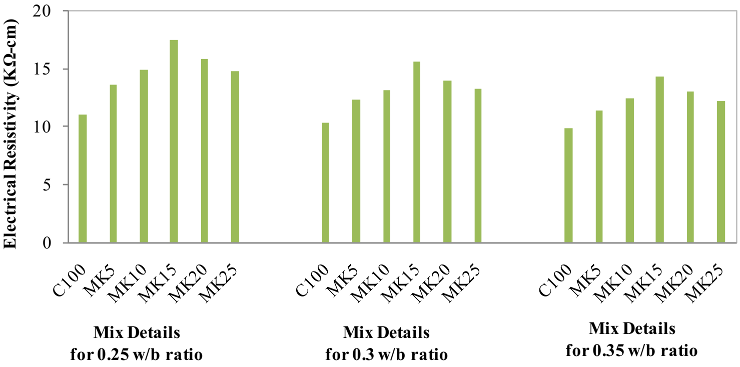


*Fig. 4.* Electrical resistivity results of accelerated curing samples

metakaolin shows a maximum of 45, 51.4, and 59% enhancement in electrical resistivity of concrete. The metakaolin accelerates the hydration process of cement due to its pozzolanic reactivity leading to development of C–S–H gel. It provides enhanced micro-structure with strong bonding thus improves the durability of concrete [24]. The addition of 20% of metakaolin shows 9.14, 10, and 9% decrease in the electrical resistivity of concrete as compared to the mix MK15 for 0.25, 0.3, and 0.35 w/b ratios. Similarly, the addition of 25% of metakaolin shows 15.43, 14.74, and 15.28% decrease in the electrical resistivity of concrete as compared to the mix MK15 for 0.25, 0.3, and 0.35 w/b ratios. The accumulation of high dosage of metakaolin slightly diminishes the electrical resistivity of concrete. At higher dosages dilution effect may occur in concrete for $Ca(OH)_2$ which lowers the durability.

## 3.4. Durability

UPV test is utilized to identify the integrity of concrete by measuring its speed and attenuation of an ultrasonic wave crossing the sample. The accelerated curing 100 × 100 × 100 mm cubes are utilized in this study. Figure 5 depicts the UPV test results of different mixes cured in accelerated curing tank at 100 °C for 3.5 h. The higher UPV values show the greater durability of concrete. The addition of metakaolin by 5% displays 15.15, 19.4, and 24.5% amplification in UPV in comparison with mix C100 for all w/b ratios. The incorporation of 10% of metakaolin as replacement shows 26.26, 28.15, and 35.4% amplification in UPV in comparison with control mix at 0.35, 0.30, and 0.25 w/b ratio. Similarly, with incorporation of 15% of metakaolin replacing cement renders 45.4, 51.4, and 59% amplification in UPV of concrete for all ratios as compared to standard mix. The 15% of metakaolin replacement gives desired amplification in UPV of concrete at 0.35, 0.30, and 0.25 w/b ratio. There is clear depiction that the metakaolin actively works as a pozzolanic material and forms secondary C–S–H gel in the short span of accelerated curing also. The metakaolin is a highly reactive material with small size particles. Due to having small size particles it may be evenly distributed in the concrete mix and develops secondary C–S–H gel by reacting with portlandite or $Ca(OH)_2$. The accumulation of metakaolin higher than 15% somewhat declines the UPV of concrete as compared to the mix MK15. At higher replacements the formation $Ca(OH)_2$ or portlandite it will minimize the pozzolanic activity.

Metakaolin is responsible for accelerated hydration of OPC, and its influence is visible within 24 h. Metakaolin combines with $Ca(OH)_2$ to produce additional cementing compounds, the materials responsible for holding concrete together [8]. The substitute up to 20%, the portlandite reacts, thereby developing the additional CSH phases due to which the cement paste undergoes subsequent densification thereby enhancing the durability and decreasing permeability.

## 3.5. Scanning electron microscope analysis

SEM test has been performed on accelerated curing samples for visualizing the micro-structure of hydrated cement paste. The accelerated curing samples are tested for compressive strength and broken pieces from the sample are collected. These samples are immersed in ethanol to stop further hydration process. Figure 6 depicts SEM test results of concrete and mixes. From Fig. 6(a) it is seen that the mix contains needle shaped material (ettriginte), which indicates the availability of $Ca(OH)_2$ in concrete mix containing 25% of metakaolin. A dense structure with minimum needle shaped material has been noticed in Fig. 6(b) for the mix containing 15% metakaolin as alternative to cement at 0.25 w/b ratio. It confirms that the addition of metakaolin will react with portlandite and forms secondary C–S–H gel in concrete matrix. Due to this the micro-structure of concrete improves which further enhances the durability and strength parameters. The accelerated curing of concrete samples is very effective in improving the overall performance of concrete mix containing metakaolin as cementitious material.

## 3.6. Energy dispersive X-ray analysis

This test has been conducted on control mix and mix containing 15% of metakaolin as cementitious material for learning the composition of the sample. The accelerated curing samples are utilized in this study. Figure 7 depicts the EDX results of control mix and the mix containing 15% of metakaolin as cementitious material. From Fig. 7 it is

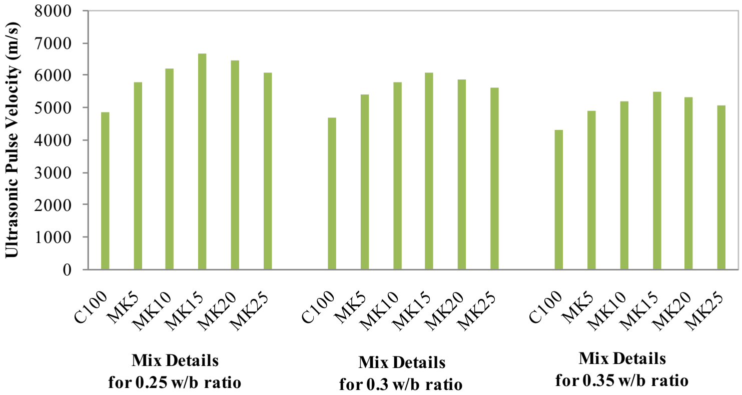
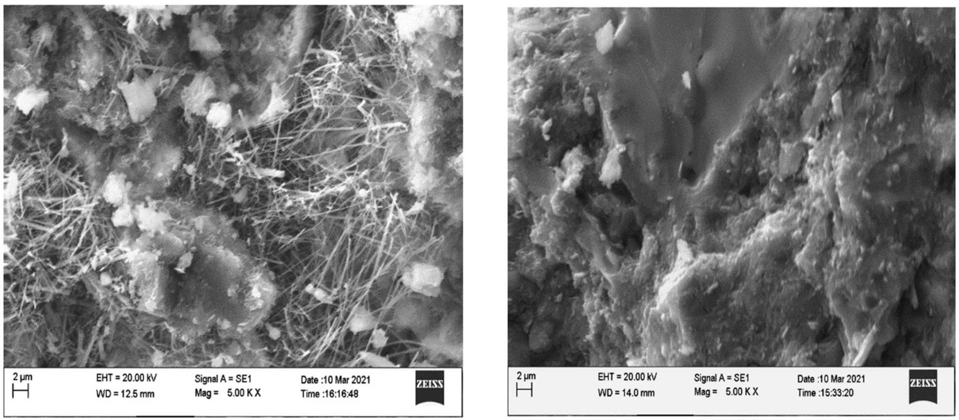
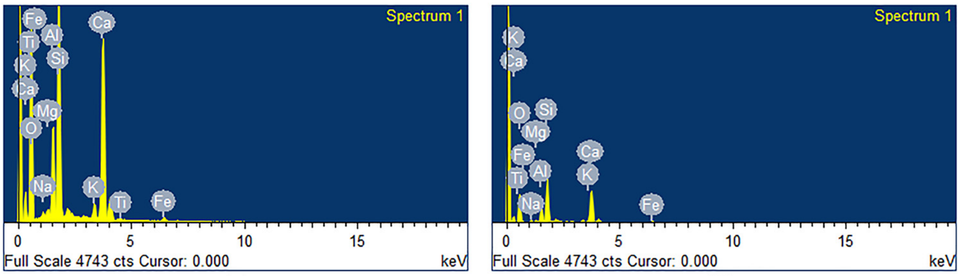


*Fig. 5.* UPV test results of accelerated curing test samples containing different proportions of metakaolin



(a) (b)

*Fig. 6.* SEM test results of (a) Concrete mix containing 25% of metakaolin, (b) Concrete mix containing 15% of metakaolin at 0.25 w/b ratio



(a) (b)

*Fig. 7.* EDX test results of (a) Control mix (b) Mix containing 15% of metakaolin as cement replacement at 0.25 w/b ratio

noticed that the amount of Ca is high in control mix as compared to mix containing 15% of metakaolin. It confirms that the $Ca(OH)_2$ present in concrete reacted with metakaolin and forms C–S–H gel; due to this strength and durability of concrete increase. A well-dispersed nanoparticle of metakaolin in cement/concrete enhances segregation resistance, fills the voids in the concrete matrix, accelerates hydration process, improves workability, consumes free Calcium hydroxide thereby amplifying the durability parameter of concrete.

## 4. CONCLUSIONS

- The impact of accelerated curing on performance of concrete incorporating metakaolin has been experimented in the current experimental study.
- The 15% accumulation of metakaolin shows a maximum of 33.3%, 38.6%, and 45.8% enhancement in compressive strength for 0.35, 0.30, and 0.25 w/b ratios as compared to the normal mix.
- The strength parameters display that 15% incorporation of metakaolin as alternative to cement amplifies the compressive strength for accelerated curing.
- The electrical resistivity, ultrasonic pulse velocity test results also confirms that 15% metakaolin is optimum for improving the overall performance of concrete.
- The accelerated curing is very effective for enhancing the performance of concrete utilizing metakaolin as cementitious material.
- The SEM and EDX test results confirms development of the C–S–H gel by reducing the availability of portlandite in concrete matrix.
- The test results depict that the utilization of 15% of metakaolin in concrete amplifies overall performance in accelerated curing regime.
- The amplification in strength of concrete is because of higher pozzolanic activity of metakaolin in accelerated curing condition. An adequate amount of C–S–H gel has been developed by the usage of 15% of metakaolin as a SCM, hence optimal enhancement in strength parameters has been noticed.

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