Research Paper

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# Effects of Sulphuric Acid in Mixing And Curing Water on Strength of High-Performance Metakaolin Concrete

**Engineering**

**KEYWORDS :** Ordinary Portland cement, High-Performance concrete, Metakaolin, Compressive strength, Split tensile strength, Sulphuric acid

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## ABSTRACT

*This paper presents the results of an experimental investigation carried out to study the effect of aggressive chemical environment on High performance concrete with metakaolin in which Ordinary Portland cement is partially replaced by 20% of metakaolin by weight and aggressive chemical environment is simulated by subjecting the concrete to different concentrations of Sulphuric acid ($H_2SO_4$) in deionised water during mixing and curing. Compressive strengths and split tensile strengths were determined at 7, 28 and 90 days. The results indicate that the compressive strength and split tensile strength decrease with the decrease in concentration of Sulphuric acid when compared with concrete without Sulphuric acid in mixing and curing water.*

### Introduction

Concrete is a widely used construction material around the world, and its properties have been undergoing changes through technological advancement. Nima Farzadni et al. (2011) say that with a fast population growth and a higher demand for housing and infrastructure, accompanied by recent developments in civil engineering, such as high-rise buildings and long-span bridges, higher compressive strength concrete is needed [7]. Currently, high-performance concrete is used in massive volumes due to its technical and economic advantages. Such materials are characterized by improved mechanical and durability properties resulting from the use of chemical and mineral admixtures as well as specialized production processes.

Metakaolin is a high quality Pozzolanic material, which is blended with Portland cement in order to improve the durability of concrete and mortars; it removes chemically reactive calcium hydroxide from the hardened cement paste. Metakaolin reduces the porosity, densifies, thickness of interfacial zone, this improving the adhesion between the hardened cement paste and particles of sand or aggregates.

Bellmann and J. Stark studied the role of calcium hydroxide in the formation of thaumasite. P.K.Mehta et.al. studied sulphate attack on concrete can lead to expansion, strength loss and ultimately to disintegration[4]. This behavior is due to a reaction of hardened cement paste with sulphate ions from the environment to ettringite, gypsum and thaumasite [5]. Kohler et. al,studied the effect of ettringite on thaumasite formation [8].

Arunakanthi et.al., studied the effect of Hydrochloric acid in mixing and curing water on strength of High-Performance Metakaolin concrete [1] and the effect of calcium choride in mixing and curing water on strength of High-Performance Metakaolin concrete [2]. The present investigation presents the effect of $H_2SO_4$ on strength properties of HPC with and without metakaolin for various concentrations of $H_2SO_4$.

### Experimental program

#### Materials

#### Cement

Ordinary Portland (53 grade) cement of Ultratech brand was used. It was tested as per Indian Standards Specifications IS: 8112-1989. Its properties are specificgravity 3.1, normalconsistency of 33%, fineness of 5%, initial setting time is 105 minutes and final setting time is 350 minutes.

#### Fine aggregate

The locally available natural river sand was used as fine aggregate. It was tested as per Indian Standard Specification IS: 383-1970. Its fineness modulous is 2.69 and specific gravity is 2.7.

#### Metakaolin

Metakaolin obtained from KOAT manufacturing company, Vadodara, Gujarat, India is used in this investigation. The properties are Bulk Density (Gms / Ltr) 300 to 340, Average Particle Size 1.5 – 2.5 micron, Residue (> 45 micron) (max. %) 0.5 – 2%, Moisture content ≤ 1%, Specific Surface Area BET (m2/gm) 12 – 18.

#### Super-plasticizer

GLENIUM B233 is the super-plasticizer of BASF company. The properties are Aspect: Light brown liquid, Relative Density: 1.08 ± 0.01 at 25°C, PH: >6, Chloride ion content: < 0.2%

#### Variables studied:

Concrete mix: The mix ratio of cement: sand: corse aggregate is 1:0.76:1.8 with water/ binder ratio as 0.3. The dosage of superplasticizer is 1% by weight of cement. 20 % of Cement was replaced with of Metakaolin.

Mixing and curing environment: Four different concentrations of $H_2SO_4$ (50 mg/L, 100 mg/L, 400 mg/L and 800 mg/L) were adopted during the mixing in the deionised water and cured in same condition.

Exposure period: Specimens were tested periodically after the specified curing periods of 7, 28 and 90 days.

Size of specimens: 150mm x150mm x150mm size of cubes for compressive strength test and 150mm dia- 300mm height of cylinders for split tensile strength test.

Samples for XRD and SEM testing: The cubes with maximum concentration of $H_2SO_4$ after 90 days testing are collected and grinded and sieved under 40 micron sieve. This powder sample is sent for XRD testing and the broken pieces of cube samples are sent for SEM testing. A total of 135 cubes and 135 cylinders were cast in the laboratory. After 72 hours, all the specimens were demoulded and cured in water in a curing tank at room temperature. After specific exposure period, specimens were tested for compressive strength, split tensile strength in accordance with test procedure IS 516: 1959 [3].

### Results and discussion

#### Effect of $H_2SO_4$ on compressive strength

The effect of $H_2SO_4$ concentration on the compressive strength and split tensile strength of HPC with and without metakaolin is presented in table: 1, 2, 3 and 4 and figures 1, 2, 3 and 4. Continuous decrease in compressive strength and splittensile strength is observed with the increase in concentration of $H_2SO_4$ for both HPCs. If the difference is less than 10% the change is considered to be negligible and if the difference is more than 10% the change is considered to be significant. It is observed that there is significant decrease in both compressive strength and split tensile strength of both HPC's from 100 mg/L concentration of $H_2SO_4$. The percentage change in compressive strength and splittensile strength of HPC with and without metakaolin increased with the increase in concentration of $H_2SO_4$. The XRD and SEM results show the formation of Calcium silicate hydrate

(C-S-H gel) and Portlandite ($Ca(OH)_2$) in both samples without $H_2SO_4$. But the intensities of C-S-H gel and Portlandite differ in two samples. The XRD patterns corresponding to HPC without metakaolin and HPC with metakaolin are shown in figures 3 and 5. By analyzing the XRD patterns of samples of two HPC's, the formation of C-S-H gel and Portlandite are formed at angles as shown in table 5 and 6. The intensity of C-S-H gel is more and the intensity of Portlandite is less for HPC with metakaolin when compared with HPC without metakaolin. This may be the cause for the strengths of HPC with metakaolin to be high under aggressive environment. The XRD tests are conducted for the two samples after 90 days with 800 mg/L concentration (maximum) of $H_2SO_4$. It is observed that inaddition to the compounds formed above, a compound named Thaumasite **($Ca_3Si(SO_4)_2(OH)_6.9H_2O$)** is formed in both HPC's with $H_2SO_4$. So the decrease in strengths may be due to the formation of Thaumasite. The angles at which the compounds are formed is shown in the table 4. The XRD patterns are shown in figures 5 and 6 respectively. The compound Thaumasite is hexagonal and dipyramidal in structure. This is fibrous and looks like crystals made up of fibers. This has very brittle fracture producing small conchoidal fragments. This is of massive and fibrous form.

The probable chemical reaction upon the hydration of cement with mixing water containing $H_2SO_4$ concentration is

$$2H_2SO_4 + SiO_2 + 3CaO + 10H_2O \qquad Ca_3Si(SO_4)_2(OH)_6.9H_2O$$

In tables and graphs CHPC is HPC without metakaolin and MHPC is HPC with metakaolin

**Table 1: Compressive strength in N/mm2 for different concentrations of $H_2SO_4$**

| S No | Dosage of $H_2SO_4$ mg/L | Compressive strength (N/mm²) | | | | | |
|---|---|---|---|---|---|---|---|
| | | 7 days | | 28days | | 90 days | |
| | | CHPC | MHPC | CHPC | MHPC | CHPC | MHPC |
| 1 | 0 | 39. 5 | 60.0 | 58.0 | 78.0 | 63. 5 | 80.0 |
| 2 | 50 | 36. 8 | 57.7 | 52. 9 | 73.9 | 57.7 | 74.6 |
| 3 | 100 | 29. 6 | 48.2 | 39.8 | 59.7 | 39.1 | 59.6 |
| 4 | 400 | 24.9 | 45.2 | 34.7 | 55.9 | 34.0 | 54.1 |
| 5 | 800 | 21.6 | 42.9 | 29.6 | 52.6 | 28.8 | 50.5 |

**Table 2 : %age change in compressive strength for different concentrations of $H_2SO_4$**

| S No | Dosage of $H_2SO_4$ mg/L | Percentage change in Compressive strength | | | | | |
|---|---|---|---|---|---|---|---|
| | | 7 days | | 28days | | 90 days | |
| | | CHPC | MHPC | CHPC | MHPC | CHPC | MHPC |
| 1 | 0 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 2 | 50 | -6.05 | -5.32 | -3.92 | -8.81 | -7.45 | -5.26 |
| 3 | 100 | -24.50 | -23.23 | -19.62 | -31.26 | -28.24 | -23.45 |
| 4 | 400 | -36.45 | -29.53 | -24.65 | -40.24 | -34.61 | -28.32 |
| 5 | 800 | -44.89 | -34.63 | -28.34 | -48.93 | -38.66 | -32.63 |

**Table 3: Split tensile strength in N/mm2 for different concentrations of $H_2SO_4$**

| S No | Dosage of $H_2SO_4$ mg/L | Split tensile strength (N/mm²) | | | | | |
|---|---|---|---|---|---|---|---|
| | | 7 days | | 28days | | 90 days | |
| | | CHPC | MHPC | CHPC | MHPC | CHPC | MHPC |
| 1 | 0 | 4.19 | 5.3 | 5.35 | 6.05 | 5.67 | 5.75 |
| 2 | 50 | 3.93 | 5.08 | 4.86 | 5.7 | 5.15 | 5.24 |
| 3 | 100 | 3.15 | 4.24 | 3.68 | 4.63 | 3.49 | 3.88 |
| 4 | 400 | 2.64 | 3.98 | 3.18 | 4.32 | 3.04 | 3.54 |
| 5 | 800 | 2.31 | 3.77 | 2.73 | 4.06 | 2.57 | 3.3 |

**Table 4 : %age change in split tensile strength for different concentrations of $H_2SO_4$**

| S No | Dosage of $H_2SO_4$ mg/L | Percentage change in Split tensile strength | | | | | |
|---|---|---|---|---|---|---|---|
| | | 7 days | | 28days | | 90 days | |
| | | CHPC | MHPC | CHPC | MHPC | CHPC | MHPC |
| 1 | 0 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 2 | 50 | -6.24 | -4.01 | -9.02 | -5.62 | -9.05 | -6.81 |
| 3 | 100 | -24.81 | -20.01 | -31.27 | -23.48 | -38.42 | -25.73 |
| 4 | 400 | -36.82 | -24.89 | -40.42 | -28.56 | -46.43 | -32.45 |
| 5 | 800 | -44.95 | -28.84 | -48.94 | -32.82 | -54.63 | -36.92 |

**Table 5: Compounds and their intensities for samples of HPC's without $H_2SO_4$**

| SNO | Angle in degrees | Compound | Intensity | |
|---|---|---|---|---|
| | | | CHPC | MHPC |
| 1 | 12.58 | C-S-H Gel | 76 | 132 |
| 2 | 17.94 | Portlandite | 266 | 66 |
| 3 | 21.18 | C-S-H Gel | 32 | 86 |
| 4 | 24 | Portlandite | 110 | 72 |
| 5 | 34.34 | C-S-H Gel | 124 | 154 |
| 6 | 37.3 | C-S-H Gel | 34 | 72 |
| 7 | 42.24 | Portlandite | 64 | 38 |
| 8 | 50.76 | C-S-H gel | 90 | 120 |

**Table 6:Compounds and their intensities for samples of HPC's with $H_2SO_4$**

| SNO | Angle in degrees | Compound | Intensity | |
|---|---|---|---|---|
| | | | CHPC | MHPC |
| 1 | 10.34 | C-S-H gel | 60 | 84 |
| 2 | 11.04 | Thaumasite | 74 | 54 |
| 3 | 16.16 | C-S-H gel | 36 | 58 |
| 4 | 21.08 | Thaumasite | 160 | 88 |
| 5 | 31.6 | C-S-H gel | 66 | 96 |
| 6 | 35.3 | Portlandite | 94 | 70 |
| 7 | 41.42 | C-S-H gel | 54 | 44 |
| 8 | 42.74 | C-S-H gel | 42 | 60 |
| 9 | 46.02 | Thaumasite | 68 | 46 |
| 10 | 50.38 | Thaumasite | 114 | 62 |
| 11 | 53.36 | Portlandite | 60 | 32 |
| 12 | 53.46 | Portlandite | 62 | 40 |

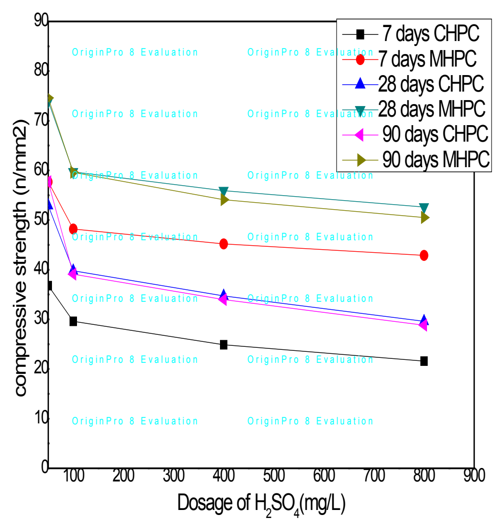


**Fig 1: compressive strengths**

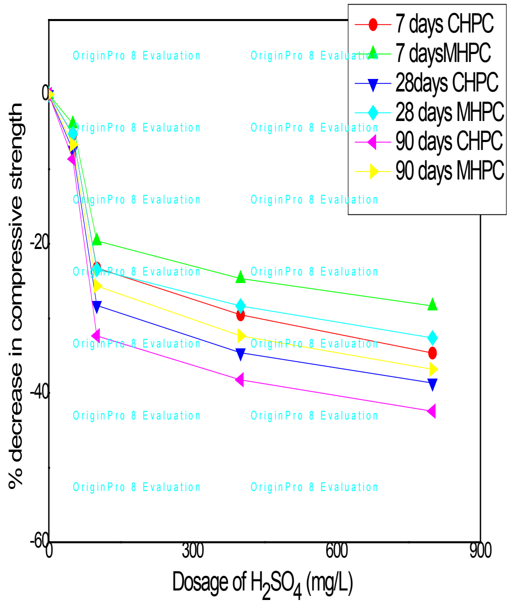


Fig 2: percentage decrease of compressive strengths

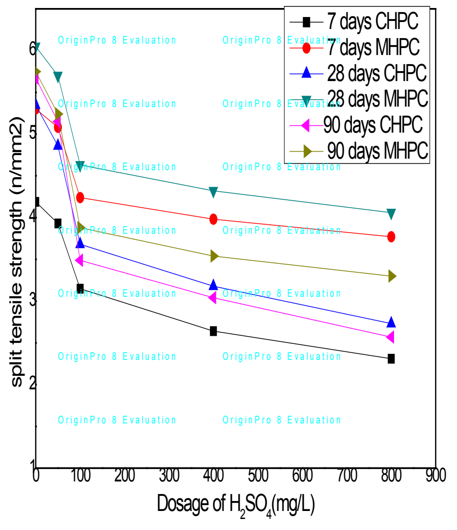


Fig 3: splittensile strengths

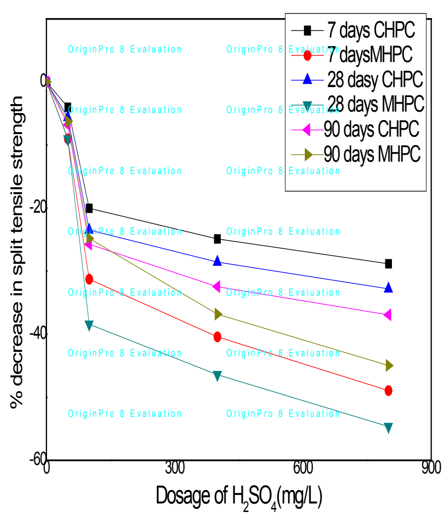


Fig 4: percentage decrease of splittensile strengths

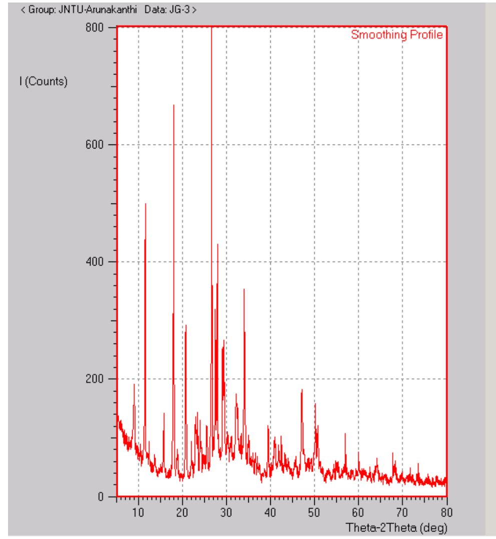


**Figure 5: XRD pattern of HPC i) without Metakaolin**

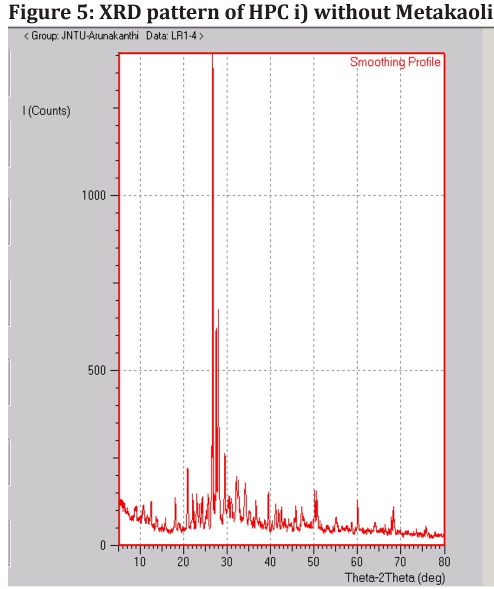


**Figure 5: XRD pattern of HPC ii) with Metakaolin**

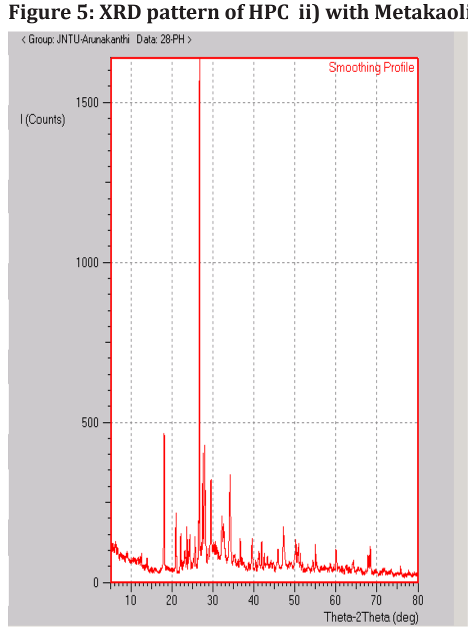


**Figure 6: XRD pattern of HPC i) without Metakaolin +**

**H2SO4**

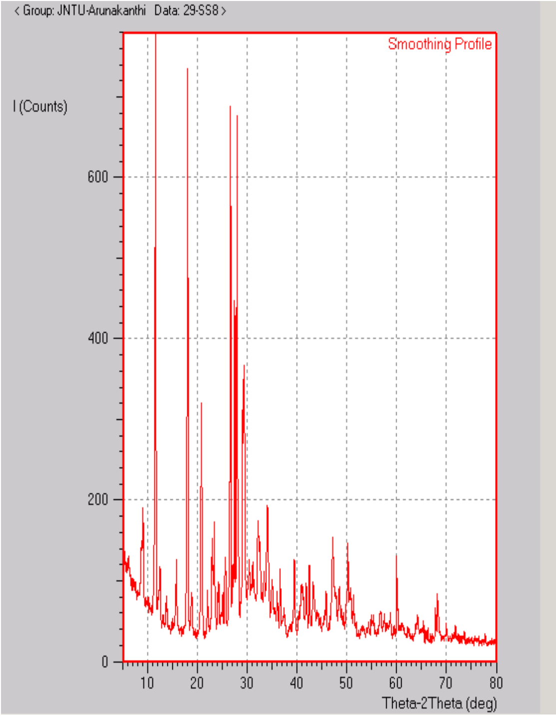


**Figure 6: XRD pattern of HPC ii) with Metakaolin + H2SO4**

## Conclusions

Based on the above results of the investigation conducted on High-performance concrete with partial replacement of cement by 20% metakaolin and subjected to various concentrations of $H_2SO_4$, the following conclusions can be drawn:

1. Compressive strength and split tensile strength of HPC increased with the replacement of cement by 20% metakaolin. And the stengths decreased with the increase in concentration of $H_2SO_4$ in mixing and curing water.
2. Compressive strength and split tensile strength increase as the curing period decreases for later ages of curing i.e., 7 days, 28 days and 90 days for 20% metakaolin and for all concentrations of $H_2SO_4$.
3. From XRD studies it is concluded that the formation of C-S-H gel with more intensity and Portlandite with less intensity may be responsible for more strengths of HPC with metakaolin.
4. From XRD studies it is concluded that the formation of compound Thaumasite may be responsible for decrease in strengths with the increase in concentration of $H_2SO_4$.

## REFERENCE

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