

# Effect of sulphate attack on strength of recycled aggregate concrete containing silica fume and hybrid fibres

Musa Aminu Albaji<sup>1</sup> and Pushpendra Kumar Sharma<sup>2\*</sup>

<sup>1</sup> Research Scholar, School of Civil Engineering,

Lovely Professional University, Phagwara, Punjab, India.

<sup>2</sup> Professor, School of Civil Engineering,

Lovely Professional University, Phagwara, Punjab 144411, India.

## Abstract

The environmental issues associated with the extraction of natural aggregates have necessitated the use of recycled aggregates for concrete production. However, the poor durability performance of recycled aggregate concrete (RAC) has limited its application in the aggressive environments. Therefore, this study aimed at improving the performance of RAC exposed to sulphate attack. In the experimentation, the natural coarse aggregates (NCAs) were substituted with coarse recycled concrete aggregate (RCA) at constant percentage of 75%, while ordinary Portland cement was partially substituted with silica fume (SF) by 8, 12 and 16% and hybrid of glass fibre (GF) and steel fibre (XSF) in the proportions of 0.5%GF-0.5%, 0.25%-0.75% and 0.75%-0.25% by volume fraction was added to each percentage substitution of cement with silica fume. The effect of sulphate attack was assessed by considering the change in compressive strength and loss in mass after exposed to the 4.0 percent magnesium sulphate ( $MgSO_4$ ) for the periods of 28, 56 and 120 days. Furthermore, FTIR and XRD techniques were conducted to study the microstructural characteristics. The experimental result revealed that 8% SF substitution with inclusion of hybrid of 0.25%GF and 0.75% exhibited a better resistance to sulphate attack than the normal concrete. Therefore, this combination can be used in civil engineering works especially when concreting in hostile environments.

**Keywords:** Glass fibre, recycled concrete aggregates, silica fume, steel fibre

## 1. Introduction

Concrete is considered as the most frequently utilized construction material in the world, thus accounts for around 5 to 7 percent of global carbon dioxide ( $CO_2$ ) emissions and consumes a significant amount of energy in the extraction of natural coarse aggregates (NCAs). Therefore, cement manufacture must emit no  $CO_2$  into the atmosphere in order to prevent global warming. Additionally, the over-exploitation of NCAs for concrete production results in the diminution of this non-renewable natural resource, posing severe environmental concerns. Recently, there has been much researches on the utilization of RCAs in the production of concrete [1-5].

Therefore, the use of RAC in construction can help to minimize the challenges of large amount of waste concrete and the associated environmental issues. Likewise, substituting recycled aggregate for virgin aggregate can help in minimizing the consumption of natural aggregates and consequently alleviate the natural resource shortage. However, the main issue with using this form of concrete is the poor bonding due to multiple interfacial transition zones (ITZs), which caused low strength and durability behaviours [6-8]. In addition, the inferior performance of RAC is linked to the production of cracks in the aggregate during processing, which makes the aggregate weaker and more susceptible to fluid permeation, diffusion, and absorption [9]. This could possibly be due to recycled aggregate containing weak ITZ due to residual cement mortar bonded to the aggregate, making recycled aggregate concrete more permeable than natural aggregate concrete [10].

External sulphate attacks have been identified as one of the most commonly stated causes of decreasing performance and shorter service life of concrete structures. External sulphate attack on concrete is common in structures exposed to high-sulphate-ion-content soils, groundwater, rivers, and seas, as well as industrial pollutants [11]. This mechanism causes ions to be transported into the interior via the pore structure, where they react with hydration products [12]. The decalcification of calcium silicate hydrate causes the paste to weaken due to the conversion of cement hydration products to hazardous compounds such as thaumasite, gypsum, and ettringite [13,14]. This has an impact on concrete softening, expansion, and cracking [12]. However, mineral additives such as silica fume, rice husk ash, fly ash etc. can be utilized to compensate for the poor performance of RAC. These additives increase the durability performance of RAC by filling the porous microstructure and lowering permeability. For examples, Sasanipour et al. [15] experimentally reported that inclusion of silica fume in RAC reduced pore spaces by minimizing the permeability of the RAC and thus, the durability parameters of RAC are improved. Also, Dimitriou et al. [16] experimentally found that the durability performance of RAC improved significantly by inclusion of fly ash and silica fume in the RAC, and with silica fume exhibited better resistance against sorptivity and rapid chloride penetration than fly ash addition.

Interestingly, fiber reinforcements are also considered to enhance the strength behaviour of RAC. The fibres like steel, polypropylene, glass, basalt fibres [17-22], and other forms of fiber-reinforcements have all been used in RAC. Experimental results revealed that, the inclusion of the fibres enhanced the compressive, splitting tensile, and flexural behavior of RAC. The improvement was more significant in split and flexural behavior than compressive strength, because of bridging of cracks by the fibre [23]. Koushkbaghi et al. [24] reported that inclusion of steel fibres in RAC showed a positive impact on resistance to acid attack. This is due to enhanced binder matrix integrity. Furthermore, the addition of rice husk ash (RHA) enhanced the bonding of fibres to the binder matrix, implying that mixing RHA with fibres enhanced the mechanical and durability performance of RAC [24]. However, the effect of sulphate attack on strength characteristics of RAC made with the combination silica fume and hybrid fibres is limited in the existing literature. Thus, this study fills this gap and presents the combined effects of silica fume and hybrid of Alkali-resistant glass fibre and crimped steel fibre on the sulphate attack resistance of RAC.

## 2. Experimental programmes

### 2.1 Research materials

Cement of grade 43 ordinary Portland cement (OPC) conformed to the Indian standard 269:2015 [25] and silica fume (SF) which satisfied the pozzolanic requirements of ASTM C618-2005 [26] were utilized in the experimentation. The specific gravity (S.G) of cement and silica fume is 3.15 and 2.81, respectively. Table 1 shows the percentage oxides of the OPC and SF. The experimental investigation used NCA and RCA with specific gravity of 2.84 and 2.34, respectively, with 60% of 10mm size and 40% of 20mm size. Furthermore, a river sharp sand that is free from any foreign matters was used in the study. The sand falls within zone II particle grading with a S.G of 2.78 and fineness modulus of 28.8 was employed in the experimental works. The fibres utilized in the study are; Alkali-resistant glass fibre and Xorex crimped steel fibre. The properties of the fibres are presented in Table 2.

**Table 1 Percentage of oxide compositions of ordinary Portland cement and silica fume**

Materials	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub> CaO	Fe <sub>2</sub> O <sub>3</sub> MgO	SO <sub>2</sub>	Alkalis	LOI
OPC	21.2	5.3	65.5	4.5	-	0.9
SF	95.1	0.02	0.03	1.2	0.10	0.23

**Table 2 Physical and mechanical characteristics of the fibres**

Property	Types of Fibres	
	Glass Fibre	Steel Fibre
Specific gravity	2.7	7.8
Tensile strength (MPa)	1400	510-1100
Elastic modulus (GPa)	72	210
Length (mm)	3050	
Diameter (mm)	0.038	1.14

### 2.2 Preparation of the concrete mixes

In this study, a grade of M25 concrete was employed and designed as per IS: 10262, (2009) [27]. Three series of mix combinations were considered in the study. The series I and II served as a control mixes. While in series III a natural coarse aggregate was partially substituted with coarse recycled aggregates at constant percentage of 75%. Further, cement was systematically replaced with silica fume by 8, 12 and 16% and each of these percentages a hybrid of glass (GF) and steel fibres (XSF) was added in the proportion of 0.5% GF-0.5% XSF, 0.25% GF-0.75% XSF and 0.75% GF-0.25% XSF by volume fraction of concrete to give nine mix combinations. Thus, eleven mixes were prepared and cast for the three series. Table 3 presents the materials mix proportions for the series.

**Table 3 Materials mix proportions for various mix combinations**

Family	Mix notations	Mix combinations	Materials in kg/m <sup>3</sup>							
			OPC	SF	FA	NCA	RCA	GF	XSF	
I	M1-0 (N-con.)	100% OPC + 100% NCA	433	-	618	1195	-	-	-	186
II	M2-0 (R-con.)	100% OPC + 25% NCA + 75% RCA	433	-	618	299	896	-	-	186

<b>M3-1</b>	92% OPC+8% SF+25% NCA + 10% RCA + 0.5% GF + 0.5% XSF	398.4	34.6	618	299	896	13.5	39.0	186
<b>M3-2</b>	92% OPC+8% SF+25% NCA + 75% RCA+ 0.25% GF + 0.75% XSF	398.4	34.6	618	299	896	6.75	58.5	186
<b>M3-3</b>	92% OPC+8% SF+25% NCA + 75% RCA + 0.75% GF + 0.25% XSF	398.4	34.6	618	299	896	20.25	19.5	186
<b>M3-4</b>	88% OPC+12% SF+25% NCA + 75% RCA+0.5% GF + 0.5% XSF	381.04	51.96	618	299	896	13.5	39.0	186
<b>Series III</b>	<b>M3-5</b> 88% OPC+12% SF+25% NCA + 75% RCA + 0.25% GF + 0.75% XSF	381.04	51.96	618	299	896	6.75	58.5	186
<b>M3-6</b>	88% OPC+12% SF+25% NCA + 75% RCA+ 0.75% GF+ 0.25% XSF	381.04	51.96	618	299	896	20.25	19.5	186
<b>M3-7</b>	84% OPC+16% SF+25% NCA + 75% RCA+ 0.5% GF + 0.5% XSF	363.7	69.3	618	299	896	13.5	39.0	186
<b>M3-8</b>	84% OPC+16% SF+25% NCA + 75% RCA + 0.25% GF + 0.75% XSF	363.7	69.3	618	299	896	6.75	58.5	186
<b>M3-9</b>	84% OPC+16% SF+25% NCA + 75% RCA + 0.75% GF + 0.25% XSF	363.7	69.3	618	299	896	20.25	19.5	186

**Note:** **N-con.** = Normal concrete, **R-con.** = Recycled aggregate concrete with zero addition of silica fume and fibres, **NCA**= Natural Coarse Aggregate, **OPC**= Ordinary Portland cement, **RCA**= Recycled Coarse Aggregate, **SF**= Silica Fume, **GF**= Glass Fibre and **XSF**= Xorex Steel Fibre.

### 2.3 Experimental methodology

As per ASTM C 452-(2002) [28], the compressive strength of the hardened specimens at 28, 56, and 120 days of immersion in a 4.0 percent solution of magnesium sulphate ( $MgSO_4$ ) was tested to determine the resistance to sulphate attack. The specimens were first cured in water for 28 days before being immersed in a 4.0 percent  $MgSO_4$  solution and the initial weight of the specimens was observed for the determination of loss in mass. Another set of the specimens were cured in water for the same durations for comparison and to calculate the strength change. The test on compressive strength was conducted using compression testing machined of 3000KN capacity as per the requirements of IS 516 – (1959) [29]. The specimens' loss in strength and mass were computed using the formulas in equations 1 and 2.

$$\text{Loss in strength (\%)} = \frac{\text{Change in strength after immersion in } MgSO_4}{\text{Original strength of specimen cured in water}} \times 100\% \quad (1)$$

$$\text{Loss in mass (\%)} = \frac{\text{Change in mass of specimen after immersion in } MgSO_4}{\text{Initial mass of specimen before immersion in } MgSO_4} \times 100\% \quad (2)$$



**Figure 1 Experimental setup for compression test**

In addition, characterization techniques such as X-ray Diffraction (XRD) and Fourier Transform Infrared (FTIR) were carried out on the best combination and the control specimen (normal concrete) to study the microstructural properties after 120 days exposure to sulphate solution. The sample was collected from the remnant of crushed concrete specimen after the compression test. The sample was grinded into powder form  $40\mu\text{m}$ . A Bruker D8 advance diffractometer with radiation ( $\lambda = 141.54$ ) was employed for the XRD analysis. The samples were scanned with a step of  $2\theta = 0.02^\circ$  over a range of  $10$  to  $90^\circ$ . A PerkinElmer Instrument with ATR Spectrum was employed to obtain FTIR spectra of the samples. The results were obtained using an FTIR spectrometer with a resolution of  $4\text{ cm}^{-1}$  and a range of  $4000$  to  $400\text{ cm}^{-1}$ .

### **3. Experimental Results and Discussion**

#### **3.1 Compressive strength due to sulphate attack**

The compressive strengths and loss in strength of the specimens after immersion in  $4.0\%$   $\text{MgSO}_4$  are depicted in **Fig. 2** and **3**, respectively. The loss in strength was found in comparison to companion specimens cured in water for 28, 56, and 120 days. **Fig. 2** shows that, the compressive strength reduces as the duration of exposure increases. The reason for the reduction of strength due to sulphate attack could be associated with the fact that sulphate ions react with calcium hydroxide and tricalcium aluminate in cement to form gypsum and ettringite, which resulted to loss of cohesion within the concrete matrix, and consequently, caused reduction in the strength [30]. In general, **R-conc.** exhibits a higher reduction in strengths at all ages compared to **N-conc. (control)** and all the mix combinations in **series III**. The reason could be due to the fact that RAC specimens have more voids and cracks, allowing sulphate ions to enter the RAC more easily than normal concrete and this resulted to the reduction in strength[31].

Furthermore, **Fig. 3** displays the loss in strength of the specimens due to sulphate attack for the periods of 28, 56 and 120 days. The inclusion of silica fume and a hybrid of glass and steel fibres in **series III** exhibited a lesser loss in strength at all ages and compensated for the negative effect of RAs in concrete. Among the mix combinations in **series III**, the mix notation **M3-2** exhibits the highest resistance due to sulphate attack than normal concrete having the lowest percentage loss in strength of 1.61%, 1.82% and 2.0% after 28, 56 and 120 days of exposure, respectively, compared to control (**N-conc.**) having 1.98%, 2.15% and 2.8% for 28, 56 and 120 days of exposure. This phenomenon could be associated with the consumption of calcium hydroxide by the silica fume, which produces a dense and less porous concrete matrix. On the other hand, the inclusion of the fibres in the mix bridged the cracks within the concrete mix; thus, the bonding zone is improved, which enhanced the strength behaviour of concrete [32].

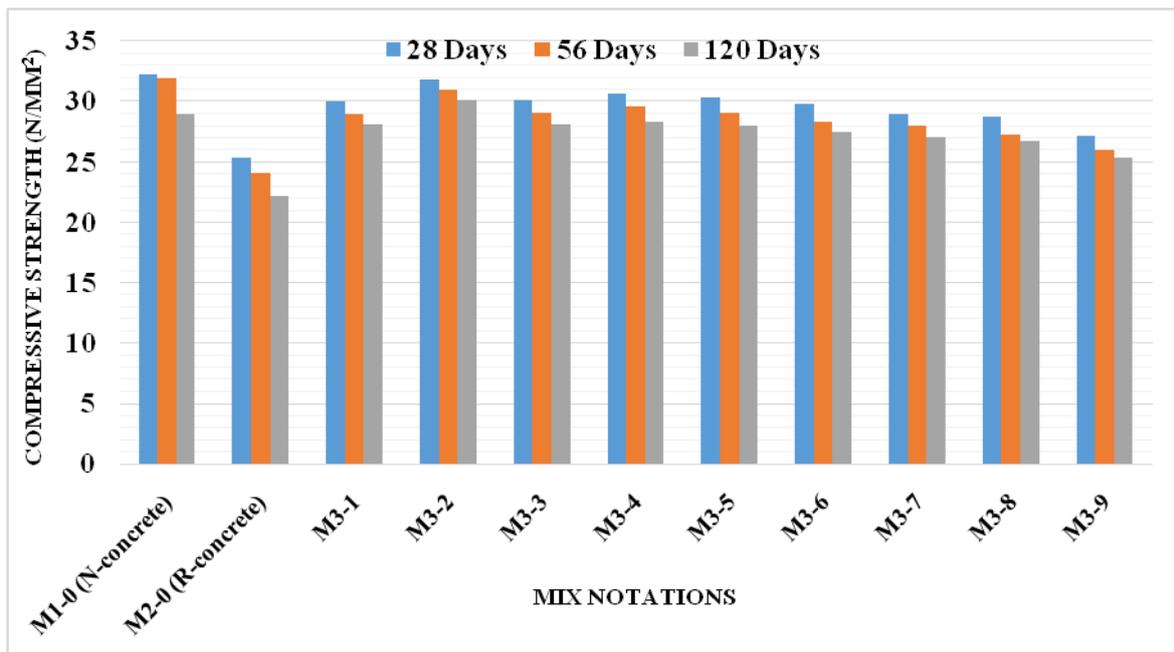


Fig. 2 Compressive strength of concrete specimens after immersion in 4.0% MgSO<sub>4</sub>

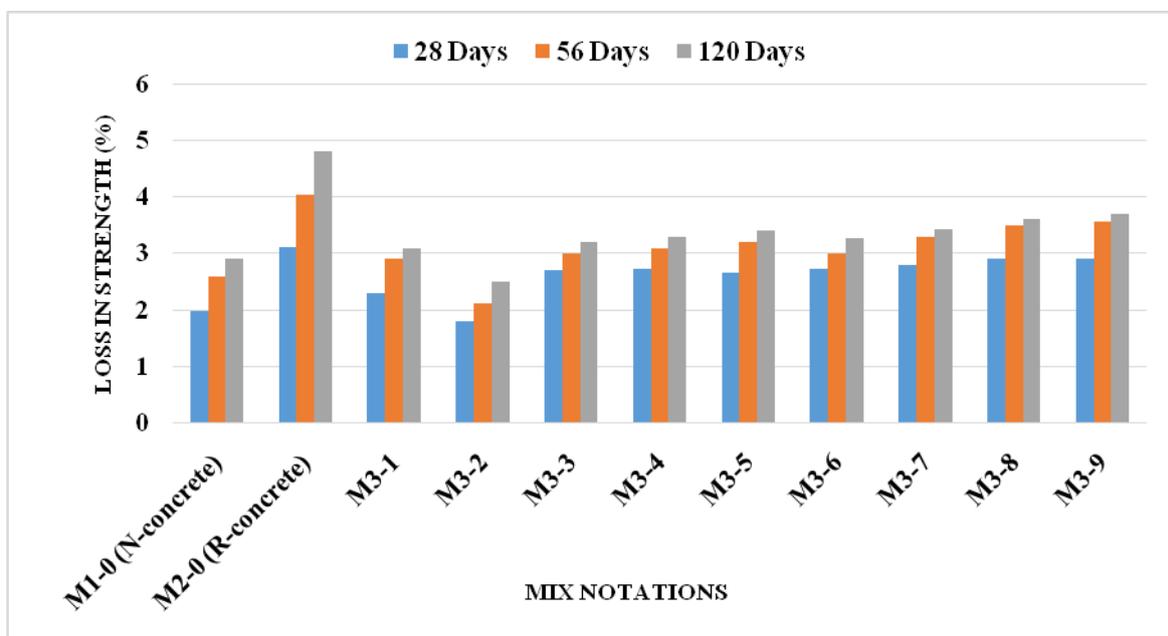


Fig. 3 Loss in strength due to MgSO<sub>4</sub> exposure.

### 3.2 Loss in mass due to sulphate attack

After 28, 56, and 120 days of immersion in the MgSO<sub>4</sub> solution, the resistance of concrete specimens to magnesium sulphate attack is measured in terms of mass loss. The percentage loss in mass loss is determined for each specimen based on the mass of 28-day water-cured specimens before immersion and after mass in a magnesium sulphate solution. It can be seen in Fig. 4 that the losses in mass for control (N-conc.) were observed as 1.7%, 1.9% and 2.4% after 28, 56 and 120 days of exposure, respectively. While R-conc. exhibits the highest rate of loss in mass by 2.18%, 2.78% and 3.5% after 28, 56 and 120 days, respectively. However, the incorporation of silica fume and hybrid fibres in series III reduced mass loss. Thus, among the mix combinations in series III the M3-4 mix notation had less mass losses of 1.62%, 1.77% and 1.9% in comparison to reference control (N-conc.). Therefore, this mix combination exhibits superior performance against mass loss due to MgSO<sub>4</sub> attack compared to the reference concrete (N-conc.). The foregoing results demonstrate that incorporating 8% SF and 0.25% GF-0.75% XSF into the RAC mixes reduce the adverse effect mass loss due to MgSO<sub>4</sub> attack.

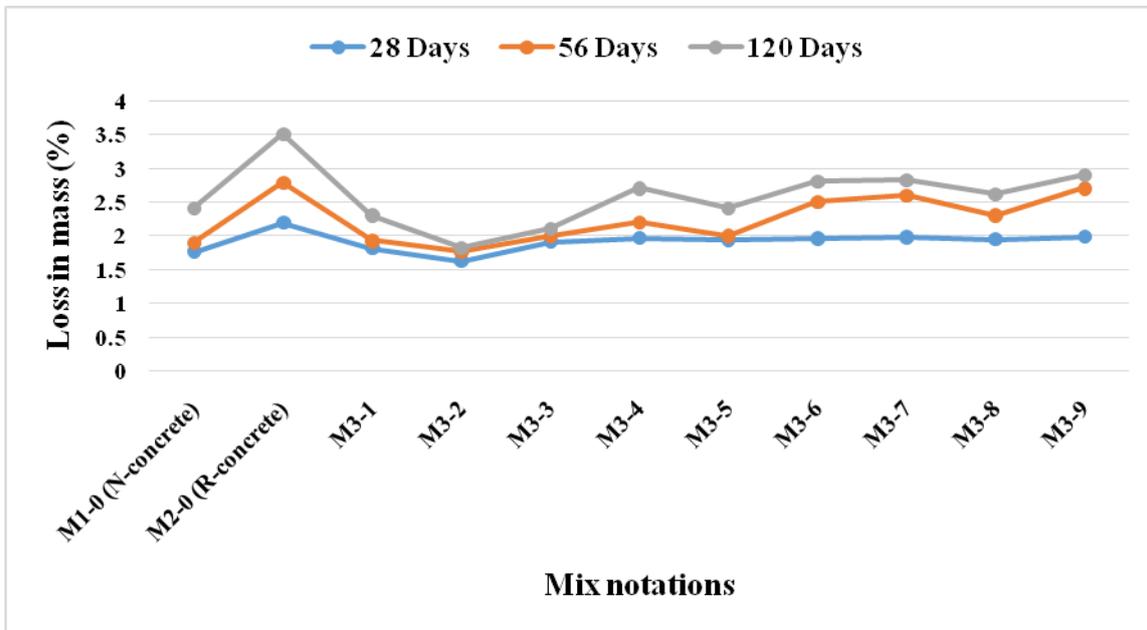


Fig. 4 Loss in mass due to sulphate attack

### 3.3 XRD analysis

The XRD analysis was conducted on the N- conc. (control) and the best combination in series III (i.e. M3-4) after 120 days of exposure to MgSO<sub>4</sub> solution. Fig. 5 depicts the XRD patterns of N-conc. and M3-4. According to Fig. 5, the peaks corresponding to portlandite, quartz, C-S-H, calcite, ettringite and gypsum were detected. It can be seen that the intensity of peak corresponding to SiO<sub>2</sub> (Quartz) at  $2\theta = 26.6^\circ$  in M3-2 was much higher than that of reference concrete (N-conc.). The intensity of the peaks associated to calcium hydroxide (portlandite) at  $2\theta = 34^\circ$  and  $2\theta = 18^\circ$  are higher in normal concrete than M3-2 specimen. This implies that the amount of portlandite in M3-2 is lower than that of normal concrete (N-conc.) which could be due to pozzolanic reaction [33]. Further, the strength of the peaks related to ettringite for M3-2 at  $2\theta = 22.9^\circ$  and  $2\theta = 31^\circ$  decreased compared to normal concrete. Also, the peaks at  $2\theta = 12.5^\circ$  prove the existence of the gypsum in all the specimens [34]. Additionally, the peaks at  $2\theta = 27.02^\circ$  and  $2\theta = 39.2^\circ$  are corresponding to C-S-H [35]. However, the strength of this peak is more in M3-2 specimen than normal concrete which is due to the pozzolanic reaction as a result of conversion of Ca(OH)<sub>2</sub> to C-S-H gel. Therefore, the resistance of M3-2 specimen against magnesium sulphate attack higher than that of normal concrete (control).

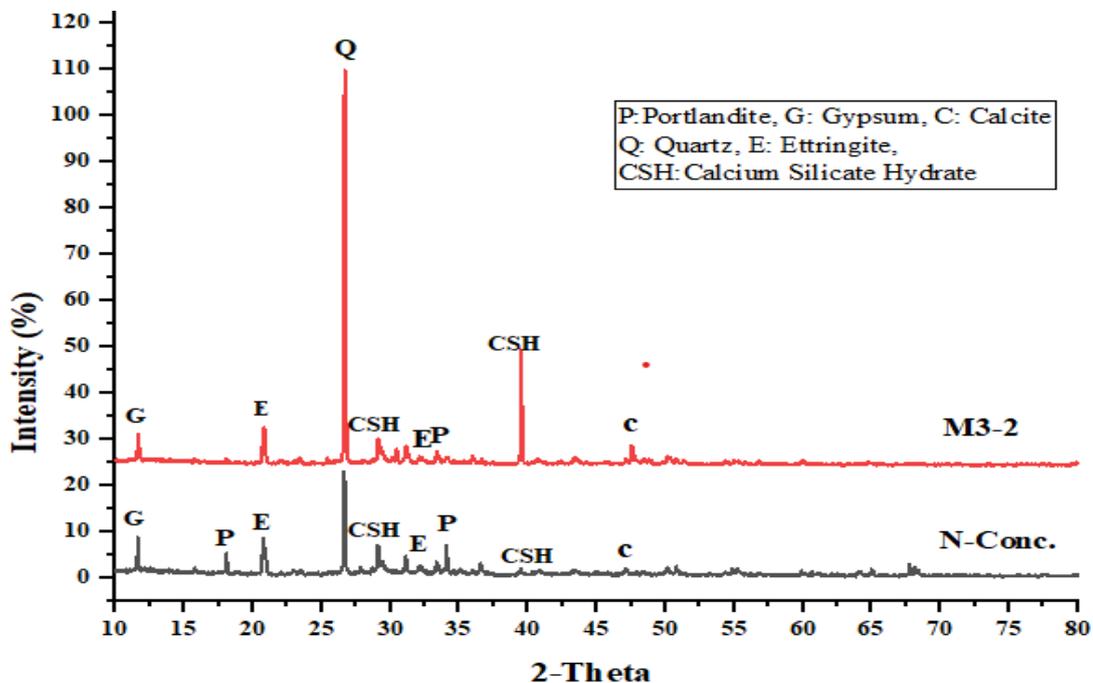


Fig. 5, XRD patterns of specimens after 120 days of magnesium sulphate exposure

### 3.4 FTIR analysis

The FTIR spectra for **N-concrete** (control) and **M3-2** concretes after 120 days of exposure to magnesium sulphate are given in **Fig. 6 a and b**, respectively. Generally, the peaks at  $3640\text{ cm}^{-1}$  and  $3630\text{ cm}^{-1}$  are assigned to the O-H stretching bond attributed to the presence of portlandite  $\text{Ca}(\text{OH})_2$ [33]. However, the strength intensity of the peak for **M3-2** is lower than that of **N-conc.** because of the pozzolanic reaction. Then, the peaks at  $964\text{ cm}^{-1}$  and  $970\text{ cm}^{-1}$  for normal concrete and **M3-2**, respectively are characterized to Si-O asymmetric stretching (tetrahedral silica) which are attributable to the presence of C-S-H. Likewise, the broad bands at  $1420\text{ cm}^{-1}$  and  $1417\text{ cm}^{-1}$  can be assigned to the C-O stretching and are associated with the presence of  $\text{CO}_3^{2-}$ . Moreover, the peaks at  $530\text{ cm}^{-1}$  and  $520\text{ cm}^{-1}$  are assigned to the presence of  $\text{SiO}_6$  bending (octahedral silica bend). These peaks must indicate thaumasite in the specimens; however, the strength intensity of this peak for **M3-2** is lower than that of **N-conc.** The peak at  $1100\text{ cm}^{-1}$  is due to the S-O stretching ( $\text{SO}_4^{2-}$ ) and also appearance of peak at  $850\text{ cm}^{-1}$  which is associated with  $\text{AlO}_6$  (octahedral alumina) in all the specimens confirms the presence of thaumasite and ettringite in the specimens[36]. This is line with XRD results, which also established the presence of thaumasite and ettringite in the specimens. As a result of the aforesaid results, it can be said that the existence of thaumasite and ettringite in the concrete had a negative impact on the strength of concrete specimens.

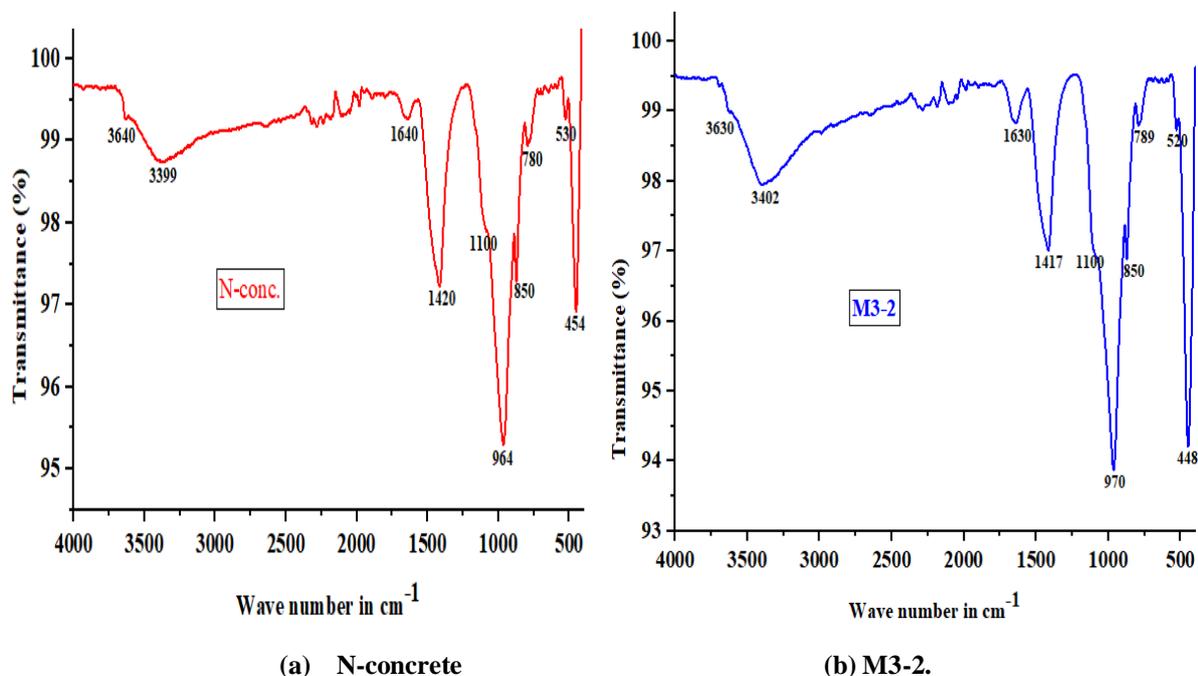


Fig. 5, FTIR spectra of specimens after 120 days of magnesium sulphate exposure

### 4. Conclusions

The influence of sulphate attack on the strength of RAC produced using a combination of silica fume and a hybrid of glass and steel fibres was investigated in this study. The following are the conclusions reached based on the finding of the study:

1. When the concrete specimens are exposed to 4.0%  $\text{MgSO}_4$  solution for the periods of 28, 56 and 120 days, the strength is reduced significantly as the duration of the exposure increases. However, the incorporation of silica fume and hybrid fibre compensated for the reduction in the strength especially mix combination with notation **M3-2** exhibited better performance than normal concrete at all ages.
2. The losses in strength due to sulphate attack for normal concrete (**N-conc.**) after 28, 56 and 120 days of exposure have been observed to be 1.98%, 2.15% and 2.8%, respectively. However, **M3-2** specimen exhibited lower values of 1.61%, 1.82%, and 2.0% for 28, 56 and 120 days.
3. It was discovered from the results of XRD and FITR analyses the existence of gypsum, ettringite and thaumasite in the specimens which could be the reason for the reduction in strength at all the durations of the exposure. However, the **M3-2** that exhibited less reduction in strength could be due to the consumption of  $\text{Ca}(\text{OH})_2$  by the active  $\text{SiO}_2$  in the silica fume to produce a dense C-S-H gel as a result of the pozzolanic reaction. This was substantiated by the results of XRD and FTIR analyses.
4. When 8% of silica fume is combined with a hybrid of glass and steel fibres (i.e. 0.25%GF and 0.75%XSF) in RAC, the durability performance of RAC against sulphate attack is improved significantly.

### Acknowledgement

The authors are thankful to the School of Civil Engineering and Centre of Instrumentation Facility at Lovely Professional University for providing a laboratory for the purpose of conducting this research.

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