Characterization of ternary blended self-compacting concrete exposed to sulphate environment

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ABSTRACT

A ternary blend comprising of 43 grade Ordinary Portland Cement (OPC), Fly Ash (FA) and Metakaolin (MK) was prepared for use in Self Compacting Concrete (SCC), and its optimum composition was determined, with respect to the compressive strength. Different SCC mixes were tested for workability parameters, as recommended by EFNARC and Bureau of Indian Standards (BIS) viz., Slump flow, T-50 time flow, V-funnel, L-box, U-box and J-ring. The concrete cubes of size 100mm were cast for all the mixes, and cured in Tap-water for determination of compressive strength and micro-structural changes at different ages.

The compressive strength and micro-structural changes of ternary blended SCC (TBSCC) were compared with a referral SCC mix (RSCC) i.e., SCC containing only OPC. The optimum TBSCC and RSCC were exposed to Ammonium Sulphate solution (4% and 16%) for different periods, after 28 days of water curing, and its compressive strength and micro-level changes were compared. The micro-structural changes due to FA and MK inclusion were observed by X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS).

Keywords: Self-compacting Concrete, Ordinary Portland Cement, Ternary Blend.

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1. INTRODUCTION

Durability is the main concern for concrete because it is highly susceptible to aggressive environments viz., acid and sulphate attacks. It is a serious issue due to the speed of damage of concrete structures worldwide. Ettringite is a very expansive compound which creates an internal pressure on the surrounding concrete. This leads to the formation of cracks, resulting in the loss of its mechanical properties. The sulphate ion responds to the hydrating cement, when the concrete specimens are exposed to its solutions, and generates expansive products that cause spalling and cracking [1, 2]. It is reported that a partial replacement of fly ash by metakaolin improves the mechanical properties, optimizes the microstructure, and reduces the level of damage due to sulphate attack [3].

The behaviour of every material is related to its microstructure. The micro-structural analysis is the best way to find the reason(s) behind the performance of concrete because it controls the properties and behaviour of concrete. The mineral data obtained from micro-structural study helps in interpreting the unique behaviour of concrete, and in finding the presence of other minor compound in the hardened concrete. MK mixed concrete showed improved micro-structural properties [4]. The SEM and EDS analyses of the specimens provide the details of the additional compounds formed. The bond between particles/constituents is improved with the curing time because of the formation of additional C-S-H gel due to pozzolanic activity [5]. Nehdi et al. reported that SCCs incorporating binary, ternary and quaternary mineral admixtures showed improved resistance to Ammonium Sulphate exposure; the formation of ettringite was identified by SEM and EDS analysis [6]. The XRD and SEM analysis confirms the formation of hydrated phases such as gismondine [CaAl2 Si2O8 .4(H2O)] and C–S–H in SCC for higher FA and MK contents. It is reported that formation of gismondine, due to dissolution of Si and Al present in MK and FA, may have contributed to the increased compressive strength of SCC because of addition of hydrated lime [7]. Paul Brown et al. reported that the micro-structural evidence supports the hypothesis that ettringite formation precedes that of thaumasite. Associated cracking due to expansion provides a means for ingress of carbon dioxide which supports thaumasite formation [8]. The incorporation of mineral admixtures stabilizes the concrete and makes it more durable due to improved pore structure [9, 10].

2. EXPERIMENTAL INVESTIGATION

2.1 Materials and their properties

For this research, a 43 grade OPC (Brand-MP Birla), conforming to IS: 8112-1989, was used. Rounded Natural Sand (NS) falling in Zone II of IS: 383-1970 was used. The other important properties of the NS are: Specific gravity- 2.65; Fineness modulus- 2.7; Bulk density- 1680 kg/m^3. The properties of 10 and 20 mm coarse aggregates are as follows: Specific gravity- 2.66 and 2.7; Water absorption- 1.0 and 0.9%;

Table 1: Chemical properties of OPC and mineral admixtures

Chemical Composition (%)	OPC	FA	MK
Silicon dioxide (SiO_2)	20.05	59.51	51.46
Calcium oxide (CaO)	62.95	5.0	36.05
Aluminum oxide (Al_2O_3)	5.28	20.34	2.21
Iron oxide (Fe_2O_3)	4.01	5.89	0.81
Magnesium oxide (MgO)	1.5	1.5	0.18
Potassium oxide (K_2O)	0.95	1.92	0.28
Sodium oxide (Na_2O)	0.24	0.61	0.74
Loss of ignition	3.12	0.35	0.96

Fineness modulus- 6.7 and 7.2; Bulk density- 1590 and 1560 kg/m³. The above values satisfy the requirements of IS: 383-1970. The FA (Class-F; Colour- grey; Specific gravity- 2.13), satisfying the requirements of IS: 3812-2000, was purchased from NTPC-Unchahar (UP). MK (Off-white colour) was procured from Kaolin Techniques Pvt. Ltd, Bhuj, Kutch, Gujarat. The specific gravity and bulk density of MK as reported by manufacturer are 2.5 and 0.9 gm/mm², respectively. A Naphthalene Sulphonate Based High Range Water Reducer (HRWR)/ Superplasticizer (Master Rheobuild @875) was used for maintaining the fresh properties as per the requirements. Table 1 presents the constituents of OPC and mineral admixtures.

2.2 Mix proportioning

The SCC mix of M25 grade was prepared using w/b ratio of 0.44, and the total binder content was kept constant (450 Kg/m³). The quantity of fine aggregate, coarse aggregate and dosage of superplastisizer were 890 Kg/m³, 750 Kg/m³ and 4.95 Kg/m³, respectively. The final mix proportion was 1: 1.98: 1.66 (Binder: Fine aggregate: Coarse aggregate).

2.2 Test procedure

For the experiments, 100 mm cubes of different mixes were prepared. The RSCC and TBSCC specimens were cured for 28 days in tap water. Thereafter, these were exposed to 4 and 16% Ammonium Sulphate solution for 180 days to study the durability and micro-structural properties.

The compressive strength of specimens, at different ages, was determined in accordance with IS: 516- 1959. To determine the change in weight, one specimen from each mix category was weighed before exposure to Ammonium Sulphate solution and tap-water. After the required exposure, the mass of specimens was found. The weight change of specimens at specified time intervals was calculated. The micro-structural analyses were conducted by using XRD, SEM and EDS.

3. RESULTS AND DISCUSSION

3.1 Preparation of TBSCC

For the optimization of doses of FA and MK, 25 different mixes were prepared for different replacement levels of OPC by FA, MK, and FA+MK. The replacement levels of OPC with FA was- 5, 10, 15 and 20%, by mass. The replacement level of OPC with MK was- 5, 10, 15 and 20%, by mass. For each replacement level of OPC with FA, replacement level of MK was also varied from 0-20%, at an interval of 5%. Total 150 cubes were cast and cured in tap water for 28 days and their compressive strengths was determined at 7 and 28 days. The optimum dose of FA and MK was found as 15 and 10%, respectively, with respect to the compressive strength. The TBSCC was prepared by using 75%OPC+15%FA+10%MK, as per the findings of Deep et. al [11].

3.2 Fresh and hardened properties

3.2.1 Fresh properties

The workability parameters of SCCs were found by performing different tests, and are included in Table 2. It was found that the workability of TBSCC improved in comparison to RSCC.

3.2.2 Hardened properties

The compressive strength of all mixes, exposed to different concentrations of Ammonium sulphate solution and tap water, were found at different intervals, and the results are represented in Table 3.

Table 2: Workability parameters of SCCs

Tests	Results	
	RSCC	**TBSCC**
Slump flow	670 mm	690 mm
T_{50} time	4.5 sec	3.0 sec
V- funnel	11.5 sec	10 sec
L-box (h_2/h_1)	0.85	0.94
U-box (h_2-h_1)	29 mm	20 mm
J-ring	9.5 mm	6 mm

Table 3: Compressive strength (N/mm²) of RSCC and TBSCC in different exposure conditions

Period (Days)	Exposure Condition					
	Tap water		4% $(NH_4)_2SO_4$ Solution		16%$(NH_4)_2SO_4$ Solution	
	RSCC	TBSCC	RSCC	TBSCC	RSCC	TBSCC
7	24.67	27.00	--	--	--	--
28	36.00	42.33	--	--	--	--
56	36.67	43.67	35.67	42.67	35.00	41.67
90	37.33	45.33	36.00	44.00	35.33	43.00
180	38.00	46.33	36.33	44.33	35.67	43.67

3.3 Durability properties and microstructural analysis

3.3.1 Compressive strength

The loss in RSCC's compressive strength after exposure to Ammonium Sulphate solution (4 and 16%) at 56, 90 and 180 days is 2.53%, 2.96%, 4.25% and 4.53%, 6.01%, 7.43%, respectively, with respect to those cured in tap water; while the respective improvements in TBSCC are 2.42%, 2.76%, 4.13% and 4.25%, 5.33%, 6.53%. Pera et al. [10] and Roy et al. [11] have also reported a similar pattern. The loss in compressive strength of both the SCCs are presented in Fig1.

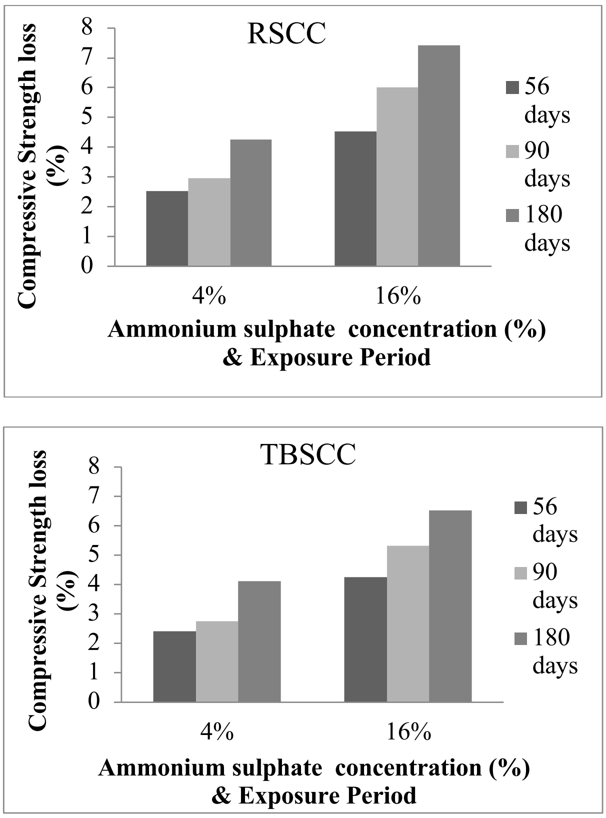


Figure 1: Compressive Strength loss of SCCs

3.3.2 Weight change

Figure 2 shows the variation in weight change of, RSCC and TBSCC samples in Ammonium Sulphate solution (4 and 16%) with age. Maximum weight change was found at 90 days, thereafter a decrement was observed. Most specimens underwent a continual increase in mass followed by a decreasing trend at advanced stages of the test. The former might be due to absorption and deposition of reaction products within the surface of specimens, while the latter might be linked to loss of surface and leaching to the surrounding solution. This result is similar to the findings of Roy et al. [8].

3.3.3 XRD, SEM and EDS Analysis

An XRD analysis of both the RSCC and TBSCC samples was conducted at 56 and 180 days, cured separately in Tap-water and Ammonium Sulphate solution (4 and 16%). Figures 3, 4 and 5 present the standard XRD results. Some of the significant crystalline phases identified are Quartz, Calcium Silicate Hydrate (C-S-H), Calcium Hydroxide (CH), Aluminum Sulphate, Monosulphate, Stratlingite, Potassium Aluminum sulphate hydrate and Ettringite. It is found that the TBSCC specimen exposed to the Sulphate solution have lower gypsum, ettringite and brucite intensity, which are mainly responsible for expansion and

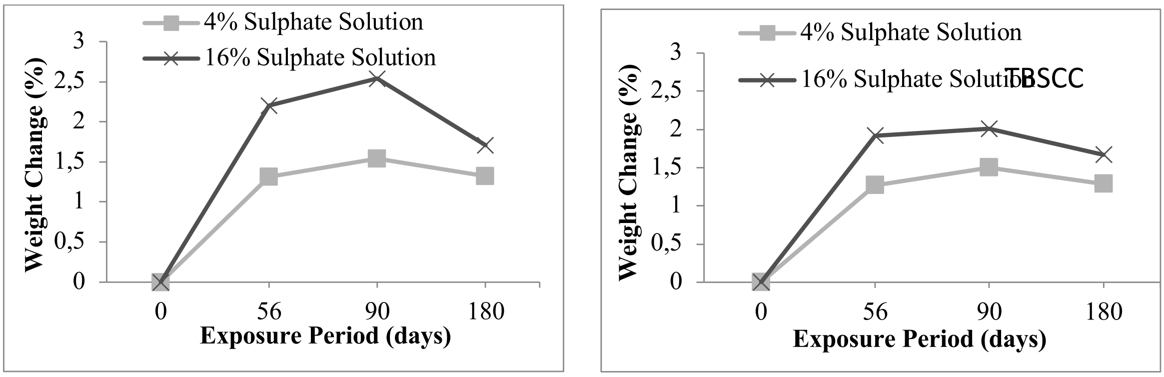


Figure 2: Weight change of SCCs

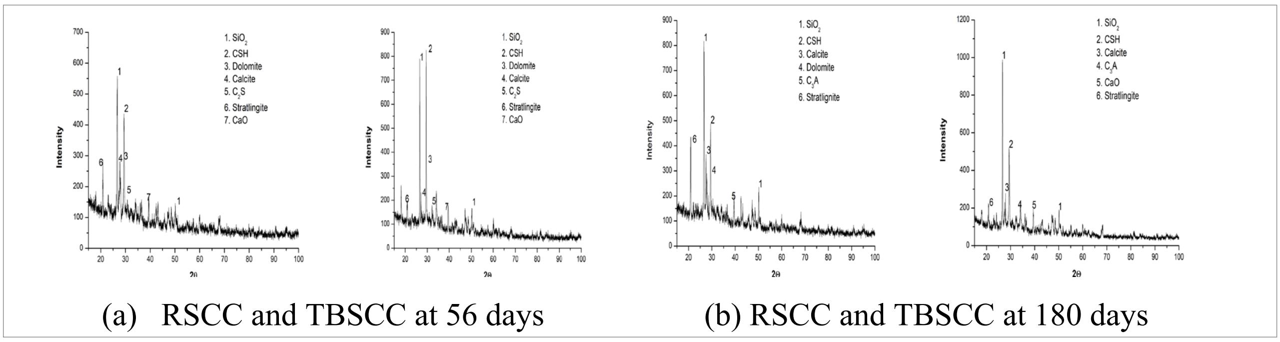


Figure 3: XRD traces of RSCC and TBSCC at 56 and 180 days tap water

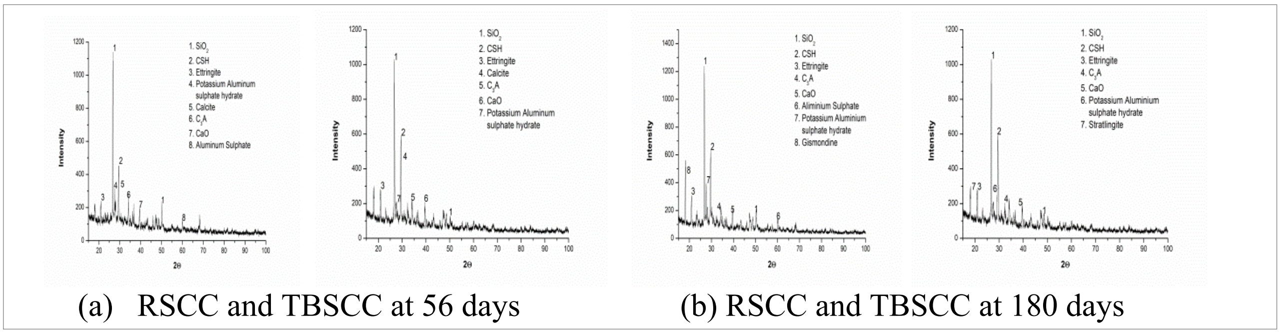


Figure 4: XRD traces of RSCC and TBSCC at 56 and 180 days Sulphate solution (4%)

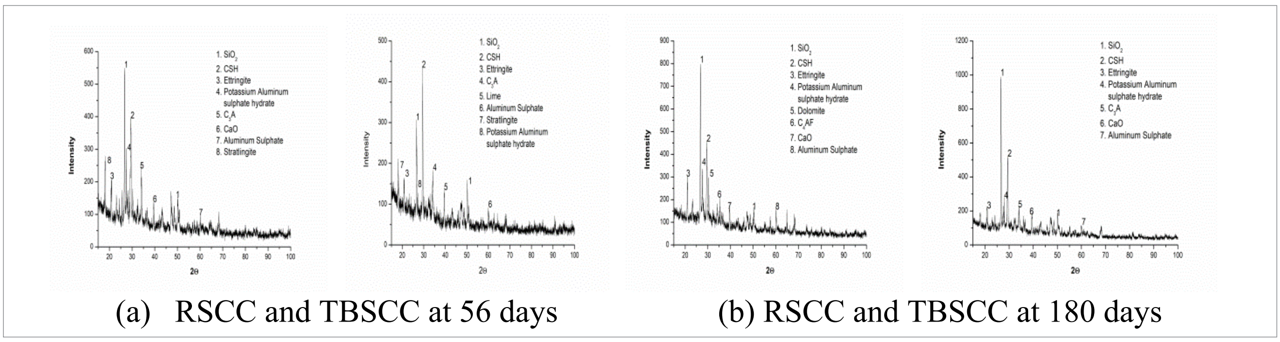


Figure 5: XRD traces of RSCC and TBSCC at 56 and 180 days Sulphate solution (16%)

cracking cracking of the concrete. In contrast to the TBSCC, higher peaks of ettringite are observed in RSCC after exposure to the Sulphate solution.

In order to validate the internal microstructure obtained by XRD, SEM, the EDS study on both the RSCC and TBSCC was performed. The morphological changes in the specimens after curing in Tap-water for 56 and 180 days are presented in Fig 6; while, the similar changes for the specimens exposed to the Ammonium Sulphate solution (4 and 16%) for 56 and 180 days are included in Figs 7 and 8. The corresponding EDS spectrum also verifies the formation of ettringite and Aluminum sulphate in the specimens exposed to Sulphate solution. In TBSCC specimen, the needle like crystals of ettringite were rarely seen; however, in RSCC specimen, these are clearly visible.

4. CONCLUSIONS

Followings are concluded from the present study,

- The workability of ternary blended SCC is improved in comparison to referral SCC.
- The loss in compressive strength of RSCC is more in comparison to the TBSCC and increases with the exposure period and strength of the Sulphate solution.
- The weight of SCCs increases up to 90 days, and then it decreases.
- XRD analysis reveals that Ettringite, Aluminum sulphate and Potassium Aluminum sulphate hydrate are formed in the specimens exposed to the Sulphate solution and these are more dominant in RSCC than TBSCC.
- SEM and EDS show the morphological and elemental composition of compounds formed and confirms the XRD results.

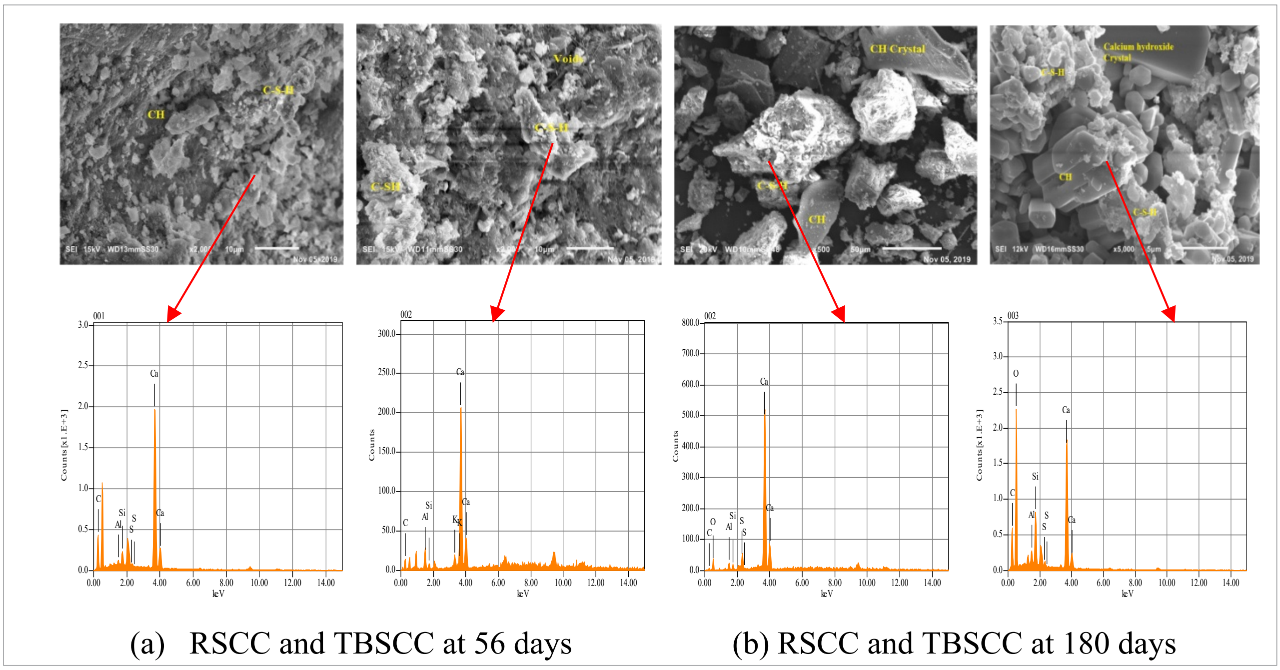


Figure 6: SEM and EDS of RSCC and TBSCC at 56 and 180 days tap water

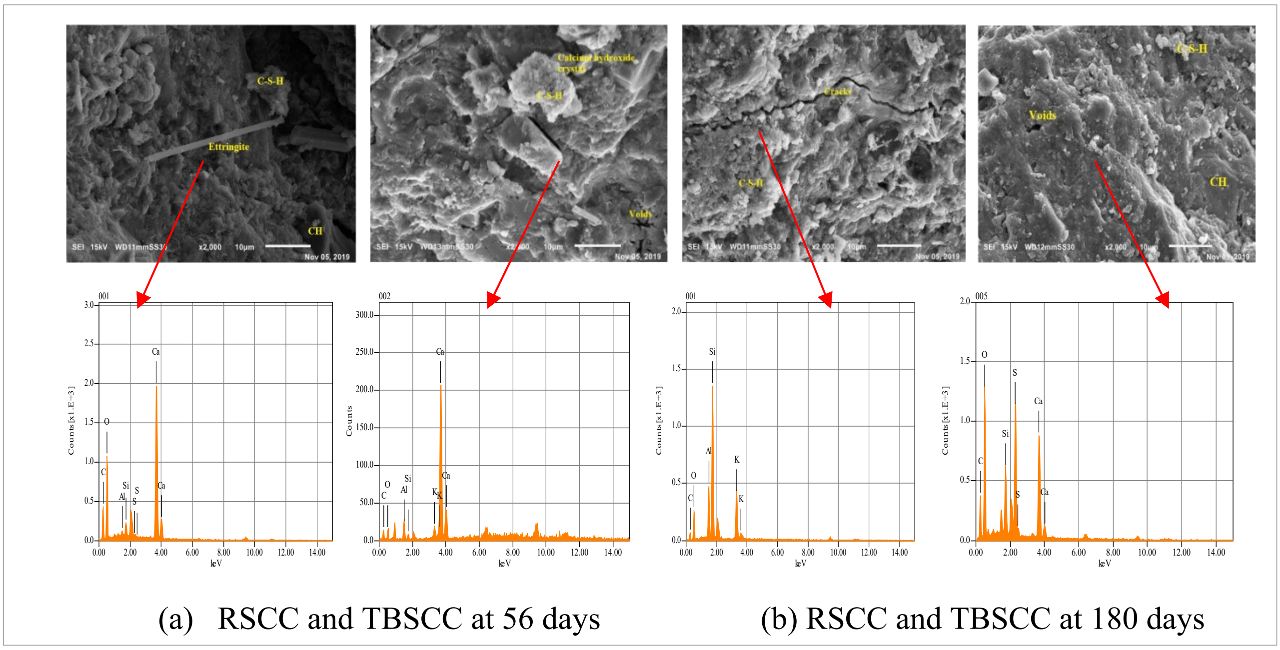
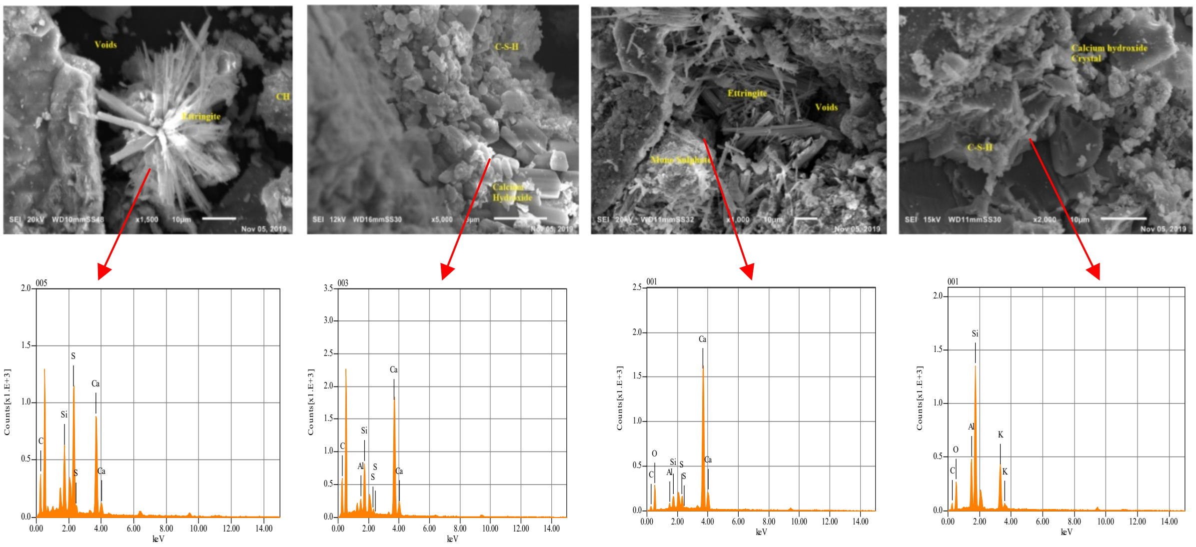


Figure 7: SEM and EDS of RSCC and TBSCC at 56 and 180 days Sulphate solution (4%)



(a) RSCC and TBSCC at 56 days (b) RSCC and TBSCC at 180 days

Figure 8: SEM and EDS of RSCC and TBSCC at 56 and 180 days Sulphate solution (16%)

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