



Research Article

# Durability of concrete exposed to combined freeze-thaw, sulfate, and acid attacks after two years

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**Abstract:** This study investigated the frost resistance of concrete exposed to sulfate and acid attacks after two years. The cement content was selected as 300 kg/m<sup>3</sup>, 350 kg/m<sup>3</sup>, 400 kg/m<sup>3</sup>, 450 kg/m<sup>3</sup> and 500 kg/m<sup>3</sup> in this study. 100 mm cubic specimens were prepared for experiments. After the specimens were cured in the water at 20 ± 2 °C for 28 days, they were kept in the laboratory conditions at 20 ± 2 °C for 23 months+2 days. Then, these samples were subjected to freeze-thaw cycles after being exposed to 5% sodium sulfate, 5% magnesium sulfate, 1% sulfuric acid, and 2% sulfuric acid for four days. Thus, the samples were exposed to the four different combined attacks. Lastly, the mechanical properties, weight change, and relative dynamic modulus of elasticity of these specimens were determined. Furthermore, the SEM and EDS analyses were carried out on samples. This study found that the highest compressive strength, the highest ultrasonic pulse velocity, and the lowest weight loss were the samples with 500 kg/m<sup>3</sup> cement content subjected to combined freeze-thaw and 1% acid attack.

**Keywords:** Concrete, freeze-thaw, acid attack, sulfate attack.

## 1. Introduction

Durability is a significant factor affecting the service life of reinforced concrete structures. The service life of these structures is affected by whether the durability effects have been damaged. The durability effects are corrosion, alkali-aggregate reactions, sulfate, carbonation, etc. The reinforced concrete structures are exposed to the freeze-thaw effect for the long term. Due to this, scientists have examined the effect of the freeze-thaw effect on concrete in the long term (Aygörmez 2021; Duran et al. 2014; Grabiec 1999; Kilic and Gok 2021; Kumar 1991; Li et al. 2016; Marczevska and Piasta 2018; Mohr et al. 2000; Omran et al. 2017; Powers 1967; TANYILDIZI 2018; Tanyildizi et al. 2020; Tanyildizi and Şahin 2017; Won et al. 2010). When the air temperature falls below zero, water in the cracks and pores in concrete has been freezing. The freezing water increases its volume by about 9% (Powers 1967). Because of the increasing freezing, the internal pressure in the concrete increases (Marczevska and Piasta 2018). The depth and width of the crack increases. In addition, new network cracks are formed by combining old cracks. Due to these cracks, the strength of concrete decreases. Furthermore, it can happen to the disintegration of concrete (TANYILDIZI 2018). Another of the durability effects is the sulfate attack. Scientists have also investigated the sulfate attack in the long term (Akyuncu et al. 2019; Hou, Li, et al. 2014; Hou, Ma, et al. 2014; Ma 2013; Ma et al. 2013; Mahmoud et al. 2013; Marchand, Odler, and Skalny 2001; Niu et al. 2015; Tanyildizi 2019, 2016, 2018; Zhang et al. 2017). Chemical reactions and physical events occur due to the sulfate effect on concrete. Due to this chemical reaction, new substances such as gypsum and ettringite form (Marchand et al. 2001). The ettringite causes the expansion in concrete.

Furthermore, it causes fracture formation, cracking, and flaking off the surface. These events decrease the strength of concrete (Hou, Li, et al. 2014; Hou, Ma, et al. 2014; Ma 2013; Ma et al. 2013; Niu et al. 2015; Tanyildizi 2018; Zhang et al. 2017). Also, many studies in recent years have been carried out on geopolymer concrete exposed to sulfate attack and freeze-thaw effects. Kumar et al. (Kumar et al., 2022) examined the properties of geopolymer concrete and conventional concrete exposed to sulfate attack, seawater, freeze-thaw, and acid attack. They stated that geopolymer concrete was more resistant to durability than conventional concrete. Bingöl et al. (Bingöl et al. 2020) studied the resistance of the blast furnace slag-based geopolymer mortar to the acid effect. They stated that there was an increase in the compressive strength of the mortars exposed to the acid effect.

Many researchers have researched the combined durability of concrete in recent years (Jiang et al. 2015; Kosior-Kazberuk and Berkowski 2017; Li et al. 2018; Liu et al. 2018; Niu et al. 2015; Piasta, Marczewska, and Jaworska 2015; Wang and Niu 2016; Xia et al. 2023; Yu et al. 2008). Niu et al. (Niu et al. 2015) examined the properties of shotcrete exposed to the combined sulfate attack and drying–wetting. They found that the macro cracks are the predominant factor in the sulfate resistance of shotcrete. Jiang et al. (Jiang et al. 2015) researched the durability properties of concrete exposed to both freeze-thaw and sulfate attacks. They said that the deterioration of concrete exposed to freeze-thaw accelerates the process because of the expansion products produced from the sulfate solution. Wang et al. (Li et al. 2018) studied the freeze-thaw and sulfate resistance of concrete containing coarse recycled concrete aggregates. They used high-volume and low-volume fly ash in concrete.

They said that the magnesium sulfate limited concrete deterioration during the freeze-thaw cycles, but then, the concrete degradation accelerated. Piasta et al. (Piasta et al. 2015) examined the properties of non-air-entrained and air-entrained mortars exposed to both sulfate and freeze-thaw. They found that the sulfate effect notably reduced the freezing and thawing resistance of the air-entrained mortar. Yu et al. (Yu et al. 2008) studied the freeze-thaw durability of traditional concrete, high-strength concrete, and high-strength concrete containing steel fiber under external flexural stress exposed to the chemical solution attack. They said that the stress accelerates the damage process. Wang and Niu (Wang and Niu 2016) studied the freeze-thaw and sulfate resistance of shotcrete containing steel fiber. They found that steel fiber increased the durability of shotcrete subjected to both freeze-thaw and sulfate attacks. Liu et al. (Liu et al. 2018) examined the properties of cement paste exposed to the combined freezing/thawing and calcium leaching.

They said that the level of degradation of cement pastes exposed to freeze-thaw is closely related to the proportion of big pores. Kosior-Kazberuk and Berkowski (Kosior-Kazberuk and Berkowski 2017) investigated the durability properties of concrete exposed to freeze-thaw, sodium chloride, and flexural load. They found that the combined freeze-thaw and flexural load accelerated the damage in concrete. Xia et al. (Xia et al. 2023) examined the properties of concrete containing hybrid fiber subjected to combined freeze-thaw and salt attack. They mentioned that the use of hybrid fiber in concrete could increase the freeze-thaw resistance of concrete. Su et al. (Su et al. 2022) studied the effect of carbonation cure on the strength properties of concrete exposed to combined freeze-thaw and sulfate attack. They stated that carbonation increased the resistance of concrete exposed to freeze-thaw and sulfate attack.

This study investigated the mechanical properties of concrete exposed to the combined actions of freeze-thaw cycles, acid attack, and sulfate attack after two years of curing.

## 2. Materials and methods

### 2.1. Materials

In all experiments of this study, it was used CEM I 42.5 R as cement. The properties of cement used in experiments are shown in Table 1. The diameter aggregate of 16 mm was used in this current study. The aggregates were divided into 0-7 mm crushed aggregate and 7-16 mm stream aggregates. The different cement contents (300, 350, 400, 450, and 500 kg/m<sup>3</sup>) were used in this study. The mixture proportions were given in Table 2.

**Table 1.** The chemical properties of the cement

	%
CaO	62.94
Al <sub>2</sub> O <sub>3</sub>	5.62
Fe <sub>2</sub> O <sub>3</sub>	3.24
SiO <sub>2</sub>	21.12
MgO	2.73
LOI	1.42
Specific gravity (g/cm <sup>3</sup> )	3.10
Specific surface area (cm <sup>2</sup> /g)	3430

**Table 2.** The mixture ratios.

	Cement (kg/m <sup>3</sup> )	W/C	Super plasticizer (kg/m <sup>3</sup> )	Aggregates, 0-3 mm (kg/m <sup>3</sup> )	Aggregates, 7-16 mm (kg/m <sup>3</sup> )
PS1	300	0.47	6.6	1120.32	883.54
PS2	350	0.47	5.8	1061.22	836.92
PS3	400	0.47	5.5	1001.32	789.68
PS4	450	0.47	2.4	945.85	745.94
PS5	500	0.47	0.4	888.65	700.83

## 2.2. Curing procedures

In this study, 100x100x100 mm specimens were produced using the mixing ratios in Table 2. The curing procedure of concrete specimens had two stages. In the first stage, the specimens were cured in water at 20±2°C for 28 days after demolding. In the second stage, they were cured in laboratory conditions at 20±2 °C for 23 months+2 days. Thus, the concrete samples were cured for two years.

## 2.3. Durability procedures

In the current study, the durability procedures were performed in two stages. In the first stage, the samples were exposed to two different sulfate solutions (5 % NaSO<sub>4</sub> and 5 % MgSO<sub>4</sub>) and two different sulfuric acid solutions (1% H<sub>2</sub>SO<sub>4</sub> and 2% H<sub>2</sub>SO<sub>4</sub>) for four days (Jiang et al. 2015; Li et al. 2018; Niu, Jiang, and Fei 2013). In the second stage, the specimens were subjected to freeze-thaw according to TS EN 15177 (TSE CEN/TR 15177 2012). After they were frozen at -20 ±2 °C for seven hours, they were thawed at 20±2°C for 5 hours. This cycle was repeated 56 times. Freeze-thaw equipment used in experiments is shown in Photo 1. Thus, all samples were subjected to a combined durability effect. In the final stage of the durability process, the strength properties and the microstructure of specimens were determined.



Figure 1. Freeze-thaw test cabin.

### 3. Results

#### 3.1. The weight loss

Measuring the weight change of samples exposed to an aggressive environment such as sulfuric acid is a generally accepted method (Powers 1967). In the current study, the weight changes of samples subjected to the combined freeze-thaw cycles and sulfate attacks were investigated. Eq. 1 was used to determine the weight change of the samples.

$$\text{The weight change} = \frac{100 \cdot (W_{\text{first}} - W_{\text{last}})}{W_{\text{first}}} \quad (1)$$

where,

$W_{\text{first}}$  = The weight of samples before the durability attacks.

$W_{\text{last}}$  = The weight of samples after the durability attacks.

The weight loss results of concrete exposed to sulfate attacks are given in Figure 2.

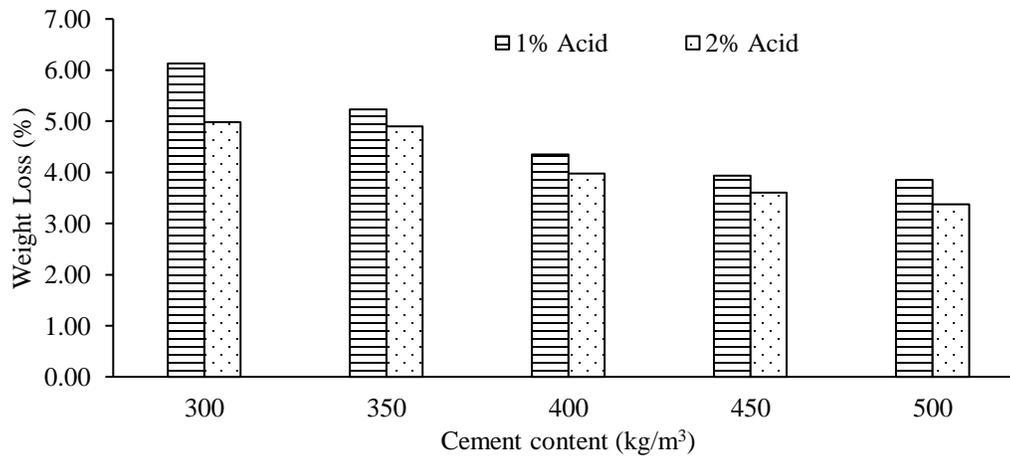
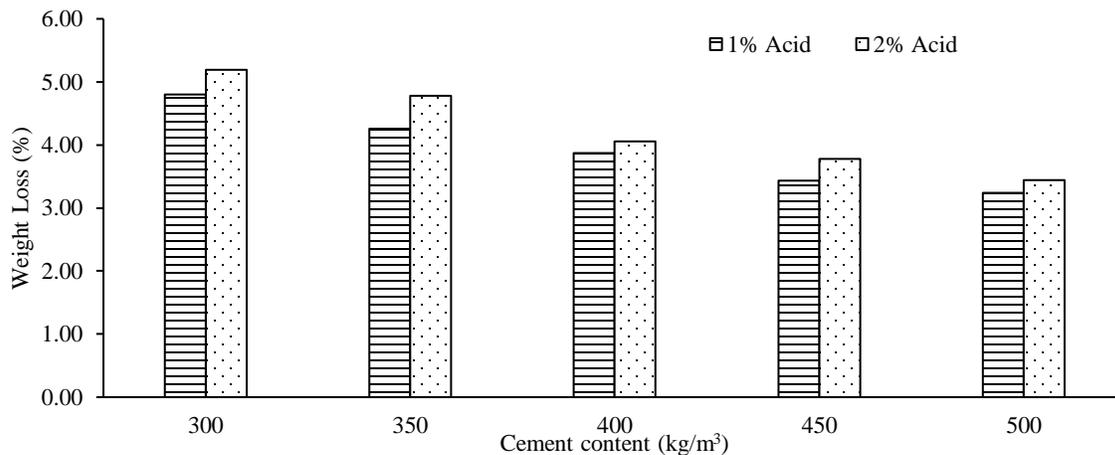


Figure 2. The weight loss of samples subjected to freeze-thaw after magnesium and sodium sulfate attack.

It can be seen from Fig. 1 that the weight loss decreased with increasing the cement content of concrete subjected to sulfate attack. When the cement content was increased, the strength of the concrete was increased, and the gaps were decreased. This prevented entering more sulfate water into the concrete and causing further damage. Furthermore, it can be seen from Fig. 1

that the concretes exposed to sodium sulfate perform better than those subjected to magnesium sulfate. The weight losses of samples containing 300 kg/m<sup>3</sup> cement content exposed to magnesium and sodium sulfate were 6.13% and 4.98%, respectively. The weight losses of specimens with 350 kg/m<sup>3</sup> cement content subjected to magnesium and sodium sulfate were 5.23 % and 4.90%, respectively. The weight losses of specimens with 400 kg/m<sup>3</sup> cement content exposed to magnesium and sodium sulfate were obtained as 4.35% and 3.98%, respectively. The weight loss of specimens containing 450 kg/m<sup>3</sup> cement content subjected to magnesium and sodium sulfate were 3.94% and 3.60%, respectively.

The weight losses of specimens with 500 kg/m<sup>3</sup> cement content exposed to magnesium and sodium sulfate were calculated as 3.85% and 3.38%, respectively. In this study, it was found that magnesium sulfate attack harmed concrete more than sodium sulfate attack. The magnesium sulfate had a more devastating effect on concrete. This destructive effect can be explained in two stages. After the Ca ions in C-S-H (Calcium silicate hydrate) are replaced by Mg ions, M-S-H (Magnesium silicate hydrate) is formed in the first stage (Miao et al. 2002). The M-S-H causes loss of weight and strength. In the second stage, the ettringite and gypsum are formed because of sulfate. This also causes weight and strength loss in concrete. Since these two stages come together, magnesium sulfate has a more destructive effect on the weight loss of concrete (Jiang et al. 2015). This study found that the magnesium sulfate attack has a destructive effect on concrete, similar to the literature (Miao et al. 2002; Mu et al. 2001). The weight changes of samples subjected to both the acid attack and freeze-thaw are given in Figure 2.



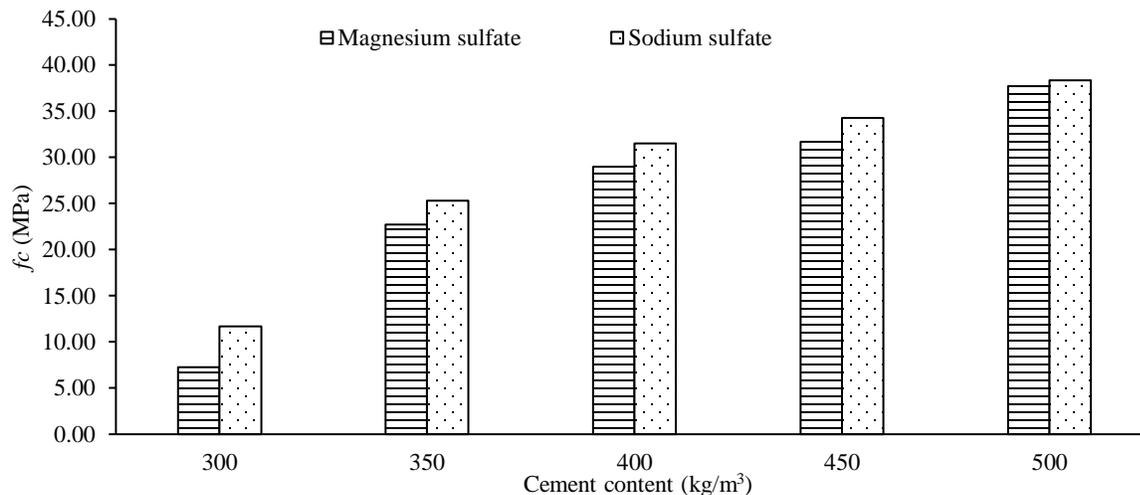
**Figure 3.** The results of weight loss of samples subjected to freeze-thaw after acid attack

Figure 3 shows that the weight loss decreased with increasing the cement content of the concrete exposed to acid attack. Furthermore, it can be seen from Fig. 2 that the increases in the percentage of acid in the solution increased the weight loss in the samples. The weight losses of specimens with 300 kg/m<sup>3</sup> cement content subjected to 1 % and 2 % acid attacks were found as 4.80% and 5.19%, respectively. The weight losses of specimens containing 350 kg/m<sup>3</sup> cement content exposed to 1 % and 2 % acid attacks were 4.26% and 4.78%, respectively.

The weight losses of specimens with 400 kg/m<sup>3</sup> cement content subjected to 1 % and 2 % acid attacks were obtained as 3.87 % and 4.06%, respectively. The weight losses of specimens containing 450 kg/m<sup>3</sup> cement content exposed to 1 % and 2 % acid attacks were 3.43% and 3.78%, respectively. The weight loss of specimens with 500 kg/m<sup>3</sup> cement content subjected to 1 % and 2 % acid attacks were 3.24% and 3.44%, respectively. The weight reduction is due to the dissolution of the hydration products (Ghrici, Kenai, and Meziane 2006). The weight loss in concrete is due to the degradation of hydration products (Lotfy, Hossain, and Lachemi 2016). In this case, it causes both the loss of weight and the loss of compressive strength of concrete exposed to freeze-thaw and acid attack.

### 3.2. Compressive strength

In this current section, the compressive strength ( $f_c$ ) of long-term cured specimens subjected to freeze-thaw cycles after being exposed to 5% sodium sulfate attack, 5% magnesium sulfate attack, 1% acid attack, and 2% acid attack for four days were examined. The results of  $f_c$  are given in Figure. 4.



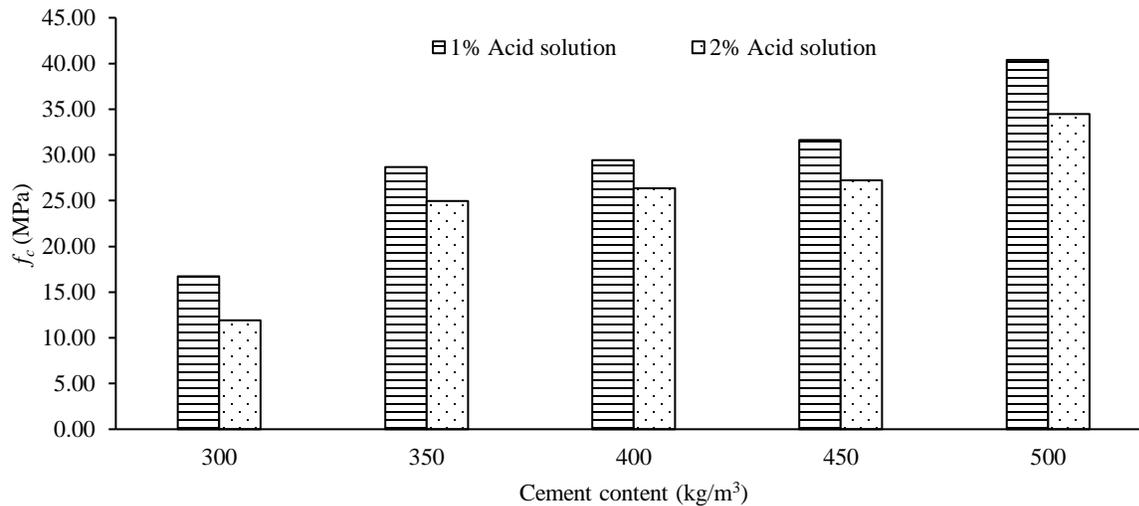
**Figure 4.** The  $f_c$  results of specimens subjected to freeze-thaw after magnesium and sodium sulfate attack.

As can be seen from Figure. 4, the sulfate attack resistance of the concrete increased with the increase of the cement content. Furthermore, the most significant decrease in  $f_c$  was obtained from specimens subjected to magnesium sulfate attack. While the  $f_c$  of specimens with 300 kg/m<sup>3</sup> cement content subjected to sodium sulfate attack was 11.68 MPa, the  $f_c$  of specimens subjected to magnesium sulfate attack was 7.25 MPa. There was a difference of 37.93 % between specimens exposed to sodium sulfate and magnesium sulfate attack. The  $f_c$  results of samples with 350 kg/m<sup>3</sup> subjected to sodium sulfate and magnesium sulfate attack were found 25.31 MPa and 22.72 MPa, respectively. The difference between sodium sulfate and magnesium sulfate attack was 10.23%. The  $f_c$  of specimens containing 400 kg/m<sup>3</sup> subjected to the sodium sulfate and magnesium sulfate attack was obtained at 31.53 MPa and 28.98 MPa, respectively. The  $f_c$  of the samples exposed to the sodium sulfate attack was found to be 8.09 % higher than the specimens exposed to the magnesium sulfate attack. The  $f_c$  of specimens with 450 kg/m<sup>3</sup> subjected to sodium sulfate and magnesium sulfate attack was found 34.28 MPa and 31.67 MPa, respectively. The 7.61% change in  $f_c$  was obtained between both sulfate attacks.

The  $f_c$  of specimens containing 500 kg/m<sup>3</sup> subjected to sodium sulfate and magnesium sulfate attack was obtained to be 38.35 MPa and 37.75 MPa, respectively. The difference between sodium sulfate and magnesium sulfate attack was 1.56 %. When these results were examined, the percentage difference between magnesium sulfate and sodium sulfate attack for  $f_c$  decreased as cement content increased. In the literature, it was stated that the concrete subjected to the sodium sulfate attack would have less strength loss (Miao et al. 2002; Mu et al. 2001; Niu et al. 2013). It mentioned that this was due to a positive effect. The sodium sulfate penetrates pores in concrete. Then, the concentration of the solution in these pores increases. So, it causes to fall the freezing point of water (Ghrici et al. 2006).

Therefore, a lower drop in strength occurs when exposed to freeze-thaw after sodium sulfate. Ozbay et al. (Özbay et al. 2013) investigated the effects on ECC (Engineering Cementitious Composite) exposed to both sulfate attack and freeze-thaw. They stated that crack widths of samples exposed to both sulfate attack and freeze-thaw increased. Nehdi and Bassuoni (Nehdi and Bassuoni 2008) studied the properties of self-consolidating concrete (SCC) exposed to both sulfate attack and freeze-thaw. They expressed that SCC could fail when subjected to both sulfate attack and freeze-thaw. Wang and Petru (Wang and Petru 2019) investigated the fiber-reinforced polymer concrete subjected to sulfate solutions and freeze-thaw cycling. They

stated that the durability of the fiber-reinforced polymer concrete interface could be improved with the silane coupling agent. The  $f_c$  results of samples subjected to freeze-thaw and acid attack are given in Figure. 5.



**Figure 5.** The results of  $f_c$  of samples subjected to freeze-thaw after acid attack.

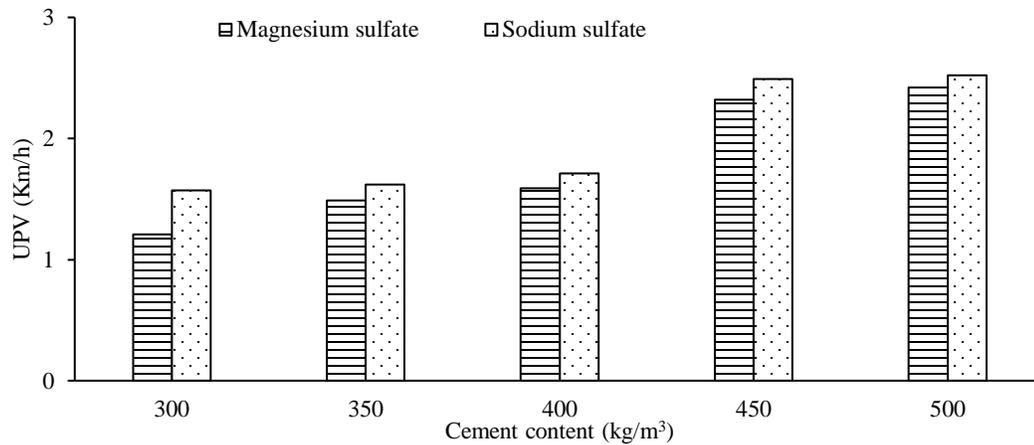
While the  $f_c$  of samples with  $300 \text{ kg/m}^3$  cement content subjected to the 1% acid attack was 16.75 MPa, the  $f_c$  of samples subjected to the 2% acid attack was 11.90 MPa. There was a difference of 28.96 % between samples exposed to 1% and 2% acid attacks. The  $f_c$  of specimens with  $350 \text{ kg/m}^3$  exposed to 1% and 2% acid attack was 28.65 MPa and 24.99 MPa, respectively. The percentage difference between these two acid attacks was 12.77%. The  $f_c$  of samples containing  $400 \text{ kg/m}^3$  subjected to 1% and 2% acid attack was obtained to be 29.42 MPa and 26.33 MPa, respectively. The difference between 1% and 2% acid attacks was found as 10.5 %. The  $f_c$  of samples with  $450 \text{ kg/m}^3$  exposed to 1% and 2% acid attack was 31.62 MPa and 27.21 MPa, respectively. The percentage difference between these two acid attacks was obtained as 13.95 %. The  $f_c$  of samples containing  $500 \text{ kg/m}^3$  subjected to 1% and 2% acid attack was obtained as 40.39 MPa and 34.48 MPa, respectively. The difference between 1% and 2% acid attacks was found as 14.63 %. When the concrete is exposed to acid or acidic water, the deterioration and neutralization of concrete increase (Marcos-Meson et al. 2019). The deterioration of concrete exposed to acid attack can occur in two ways:

- When acid solutions react with calcium hydroxide, it occurs calcium sulfate, and water (Omrane et al. 2017). The speed of formation of these products depends on the porosity of the concrete, the concentration of the acid, and the pH of the solution (Li, Leung, and Xi 2009; Mohseni, Tang, and Cui 2017). Thus, the volume of the solids is significantly increased. Due to this expansion, cracks and breaks occur in the concrete (Kawai, Yamaji, and Shinmi 2005).
- The other is the dissolution of the aggregate in the acid. If such an aggregate is in concrete, it will increase the porosity in the concrete. Thus, this event will simplify ionic transport (Beddoe and Dorner 2005; Marcos-Meson et al. 2019).

Sun and Wu (Sun and Wu 2013) investigated the properties of geopolymer mortar subjected to freeze-thaw and acid attack. They found that the mortar was resistant to freezing-thawing and attacking the acid. Leiva et al. (Leiva et al. 2019) examined the durability resistance of concrete with bottom ash aggregate exposed to acid attack, sulfate attack, and freeze-thaw. They found that there was a significant decrease in the  $f_c$  of concrete subjected to acid attack. Joorbchian (Joorabchian 2010) tried the properties of concrete containing limestone and metakaolin exposed to acid attack. The different acid concentrations were prepared in the experiments. He mentioned that the deterioration of the concrete was more when the acid concentration increased. In this study, similar results were obtained. Also, there was an important decrease in the  $f_c$  strength of the samples subjected to freeze-thaw and acid attack.

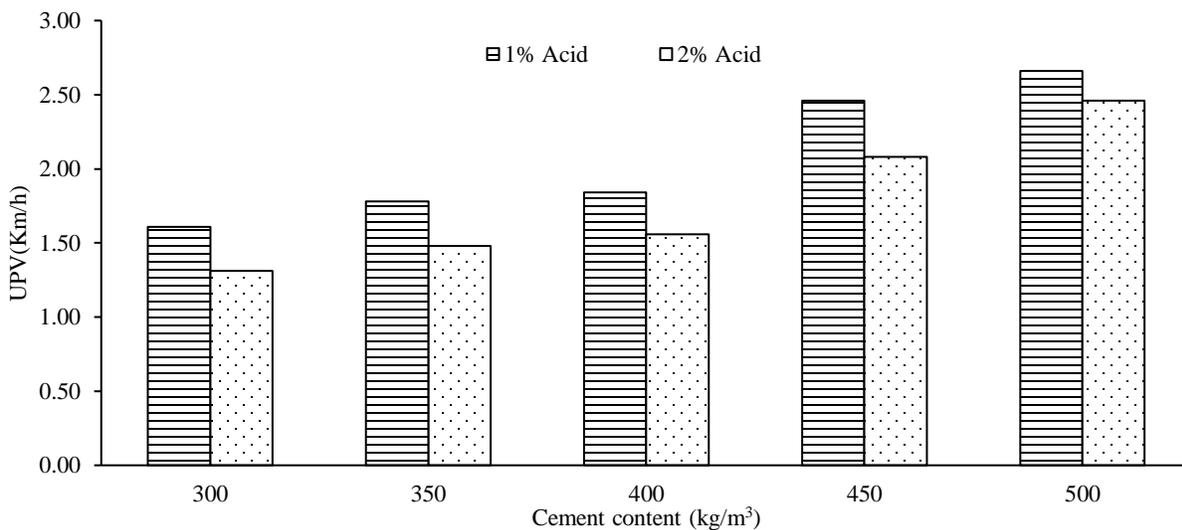
### 3.3. Ultrasonic pulse velocity

In this current study, the ultrasonic pulse velocity (UPV) results of concrete subjected to freeze-thaw cycles after being subjected to 5% sodium sulfate and 5% magnesium sulfate solution for four days were given in Figure. 6.



**Figure 6.** The results of UPV of samples subjected to freeze-thaw after magnesium and sodium sulfate attack.

Similar to  $f_c$  strength and weight loss, the best results of UPV were obtained from the samples exposed to sodium sulfate attack. The UPV results of samples subjected to sodium sulfate attack were 1.57, 1.62, 1.71, 2.49, and 2.52 km/h for 300, 350, 400, 450, and 500 kg/m<sup>3</sup>, respectively. The UPV results of samples exposed to magnesium sulfate attack were 1.21, 1.49, 1.59, 2.32, and 2.42 km/h for 300, 350, 400, 450, and 500 kg/m<sup>3</sup>, respectively. There was a percentage difference of 22.93%, 8.02%, 7.02%, 6.83%, and 3.97 % between specimens subjected to sodium sulfate and magnesium sulfate attack for 300, 350, 400, 450, and 500 kg/m<sup>3</sup>, respectively. These percentage values were similar to the percentages of  $f_c$ . The results of UPV of samples exposed to acid attack and freeze-thaw were given in Figure. 7.



**Figure 7.** The UPV results of specimens subjected to freeze-thaw after acid attack.

While the UPV results of samples subjected to 1% acid attack were 1.61, 1.78, 1.84, 2.46, and 2.66 km/h, the UPV of samples subjected to 1% acid attack were found as 1.31, 1.48, 1.56, 2.08 and 2.46 km/h for 300, 350, 400, 450 and 500 kg/m<sup>3</sup>, respectively. There was a percentage difference of 18.63%, 16.85%, 15.22%, 15.45%, and 7.52 % between specimens subjected to sodium sulfate and magnesium sulfate for 300, 350, 400, 450, and 500 kg/m<sup>3</sup>, respectively. Zhu et al. (Zhu et al. 2021) evaluated the freeze-thaw resistance of concrete using UPV. They found that the rate of loss of UPV due to freeze-thaw increased linearly. The UPV results in this study also increased linearly as the cement content increased.

### 3.4. The relative dynamic modulus of elasticity

This study calculated the relative dynamic modulus of elasticity (RDME) of samples using the UPV at the end of 56 freeze-thaw cycles. The equation used in calculating the RDME of specimens is given below (International 2008).

$$P_c = 100 \times \left( \frac{U_L}{U_f} \right)^2 \quad (1)$$

Where,

$P_c$ =The RDME,

$U_f$ = The UPV results of samples at 0 cycles,

$U_L$ = The UPV results of samples at 56 cycles.

The RDME results of samples are given in Table 3.

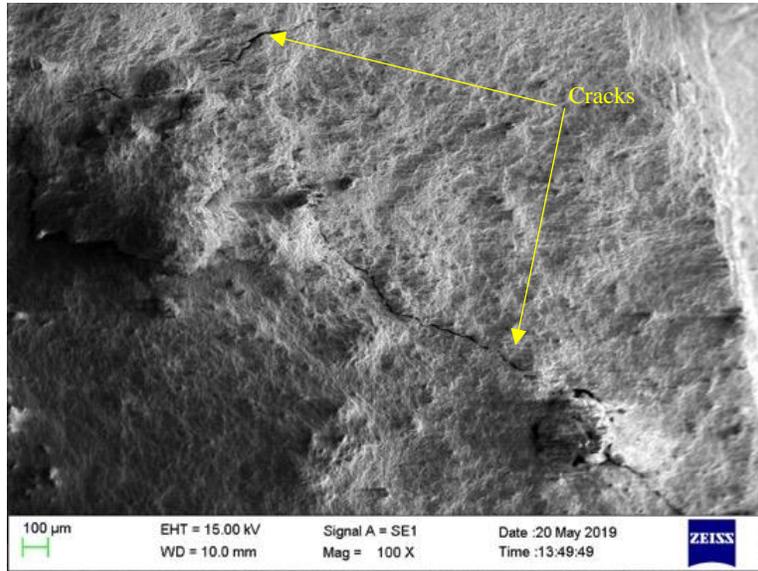
As shown in Table 3, the highest RDME results for sulfate attack were obtained from the samples exposed to sodium sulfate attack. When the samples subjected to acid were examined, the highest RDME results were found from the samples subjected to the 1% acid attack. If all samples exposed to both acid and sulfate attacks were compared, the highest results were obtained with the samples exposed to both 1% acid. Furthermore, it was observed that the RDME results increased as the cement content increased in all samples. Sun et al. (Sun et al. 2002) investigated the effect of sodium chloride in samples subjected to freeze-thaw and externally loaded.

They said that it decreased rapidly in the RDME results of samples if the stress was high. Zhang et al. (Zhang et al. 2019) researched the effects of high temperatures on concrete containing fly ash subjected to freeze-thaw. They found that the RDME in specimens with fly ash was higher than the reference concrete at above 150 °C. Li et al. (Li and Shen 2019) examined the effects of combined dry-wet and freeze-thaw on concrete containing the aeolian sand. They said that the relative dynamic elastic modulus of samples exposed to first the freeze-thaw and then the dry-wet was 2.2 times lower than samples subjected to first the dry-wet and then the freeze-thaw. Xiao et al. (Xiao et al. 2019) studied recycled concrete subjected to combined sulfate attack and freeze-thaw.

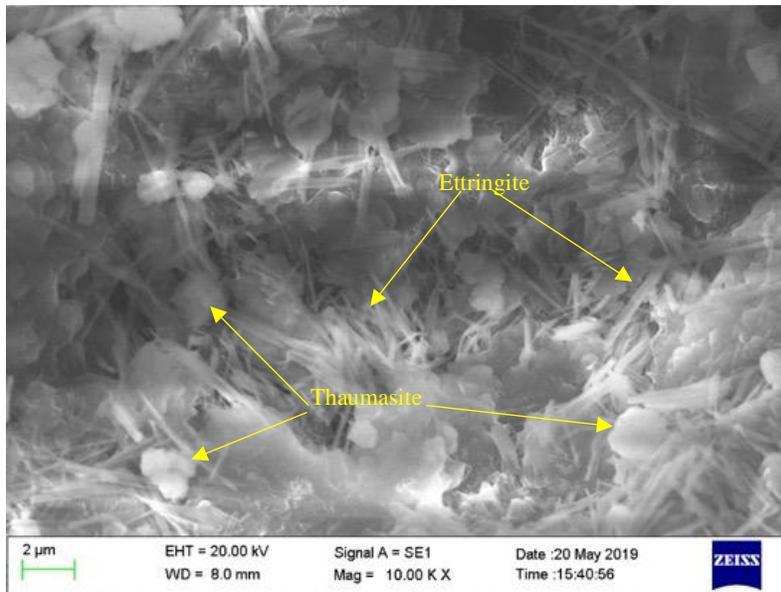
When concretes exposed to the sulfate effect were exposed to freeze-thaw, more cracks would occur. Thus, freeze-thaw damage would also be increased. They mentioned that this damage could cause the RDME to drop rapidly. In this study, the RDME decreased with the decrease in cement content in all samples. Thus, it can be said that more cracks formed and more damage in concrete at low doses. When each durability effect test was evaluated in itself, and the samples containing 300 kg/m<sup>3</sup> and 500 kg/m<sup>3</sup> cement content were compared, the decreases in the RDME of specimens exposed to the sodium sulfate, magnesium sulfate, 1% acid attack and 2% acid attacks were found to be 55.05%, 71.05%, 57.58%, and 67.16%, respectively. As the cement content increases, the compressive strength also increases. Therefore, concrete became more resistant to sulfate attack.

### 3.5. Microstructure

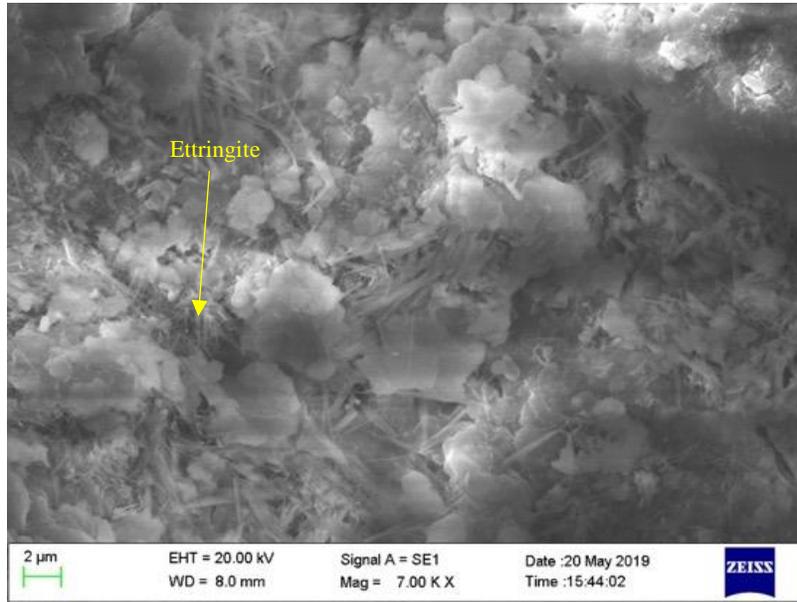
In this study, the microstructure of the samples was investigated to determine the damages that occurred by the combined durability effects. Figures. 8-9 showed the SEM images of samples exposed to freeze-thaw after magnesium and sodium sulfate attack.



a)

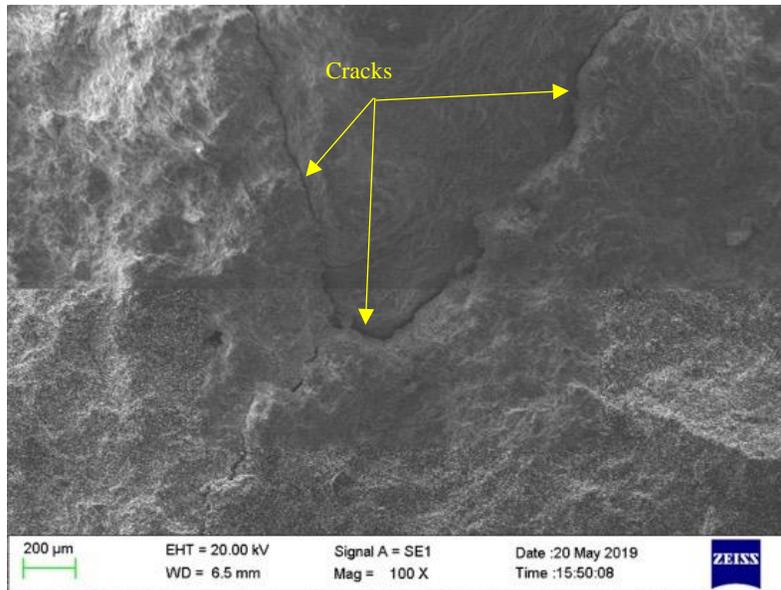


b)

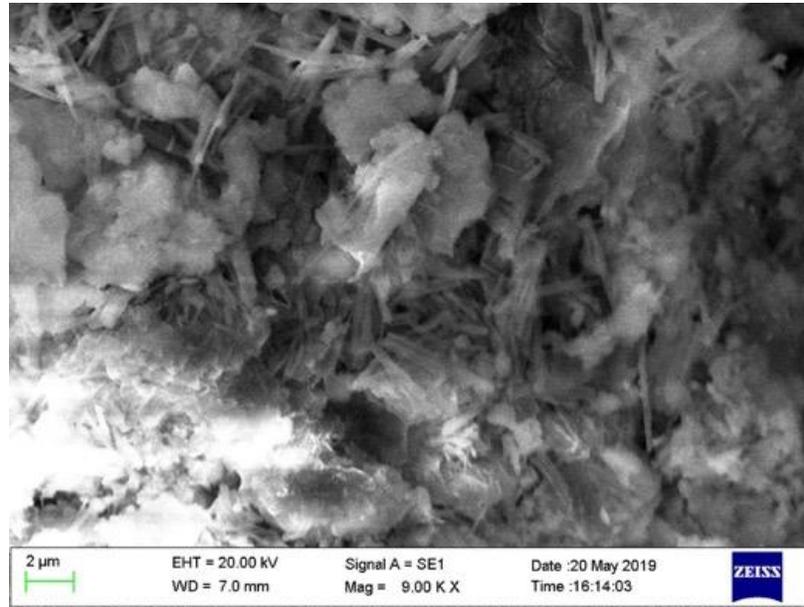


c)

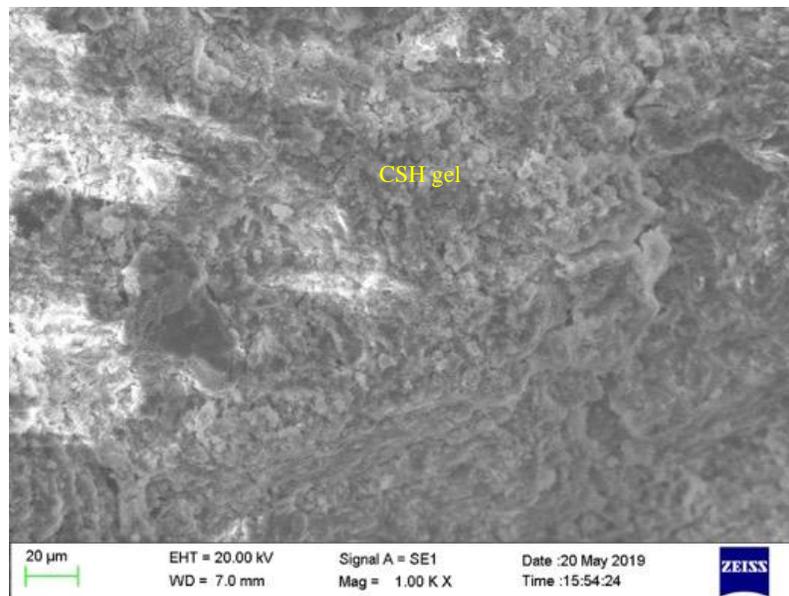
**Figure 8.** The SEM images of specimens subjected to freeze-thaw after magnesium sulfate attack



a)



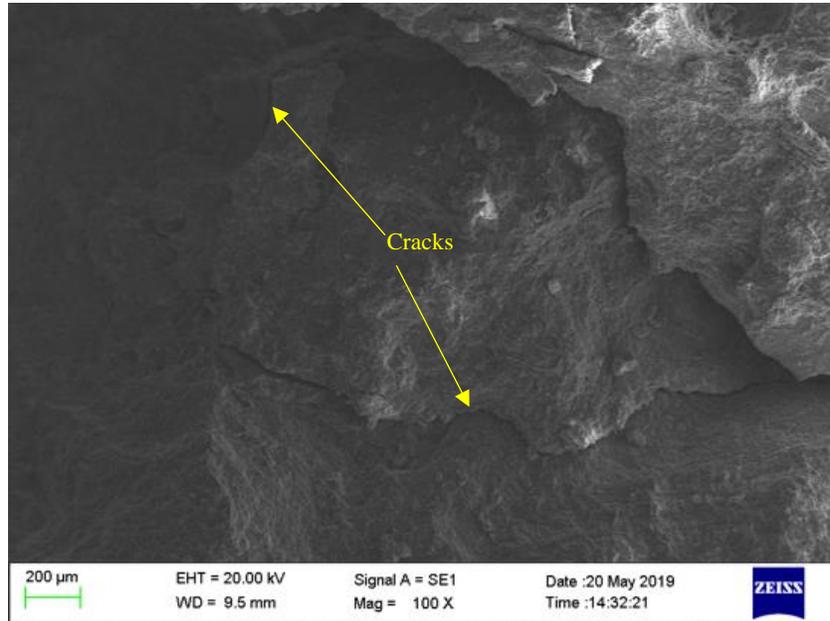
b)



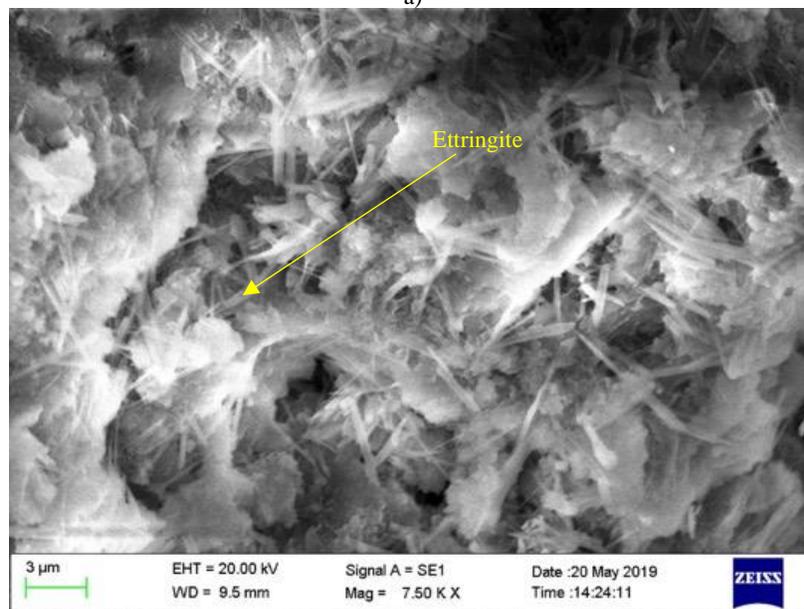
c)

**Figure 9.** The SEM images of specimens subjected to freeze-thaw after sodium sulfate attack

As shown in Figures.8a and 9a, the cracks occurred in concrete due to freeze-thaw. The single crack was not formed. Multiple cracks were formed. The gypsum and ettringite occurred products because of sulfate attack. It can be shown in Fig. 7b and Fig. 8b that the ettringite and thaumasite occurred in the microstructure of the samples. Due to the freezing event occurring at  $-20 \pm 2$  °C, the diffusion of sulfate ions is reduced. The gypsum is not seen in concrete because of this event (Rahman and Bassuoni 2014). The gypsum was not found in both the magnesium and sodium samples. The thaumasite occurs with the reaction between C-S-H and Calcite if there is moisture (Rahman and Bassuoni 2014). The C-S-H gels can see in Figure 8c. Figure 9-10 shows the SEM images of samples exposed to freeze-thaw after 1% and 2% acid attacks.

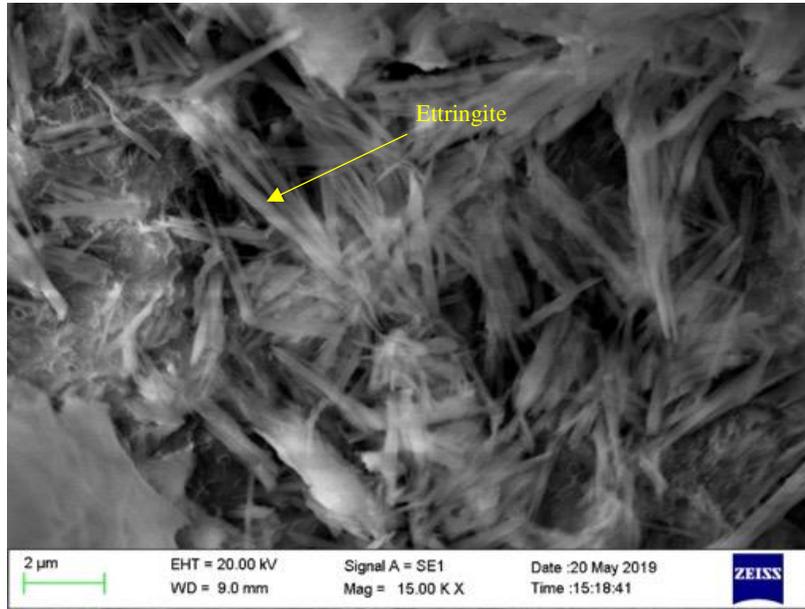


a)



b)

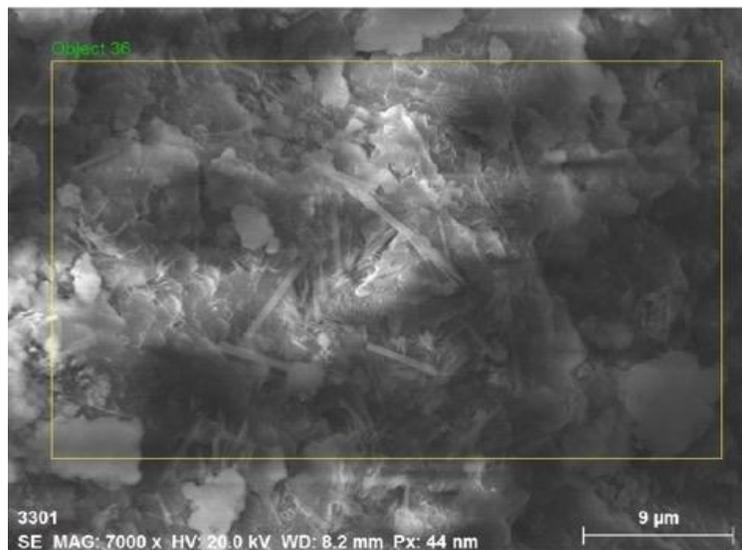
**Figure 10.** The SEM images of specimens subjected to freeze-thaw after 1% acid attack



b)

**Figure 11.** The SEM images of specimens subjected to freeze-thaw after 2% acid attack.

Fig. 9 a and b were shown cracks that occurred from freeze-thaw. Fig. 9 and 10 showed the products formed in concrete exposed to acid attack. Because of the formation of harmful materials such as ettringite and thaumasite, the microstructure was harmed. This causes the concrete to be more permeable. This allows the entry of more harmful substances. As a result, spalls in concrete and a decrease in strength properties were observed (Yu et al. 2018). The EDS analysis of samples exposed to freeze-thaw after magnesium sulfate attack was given in Fig. 11.



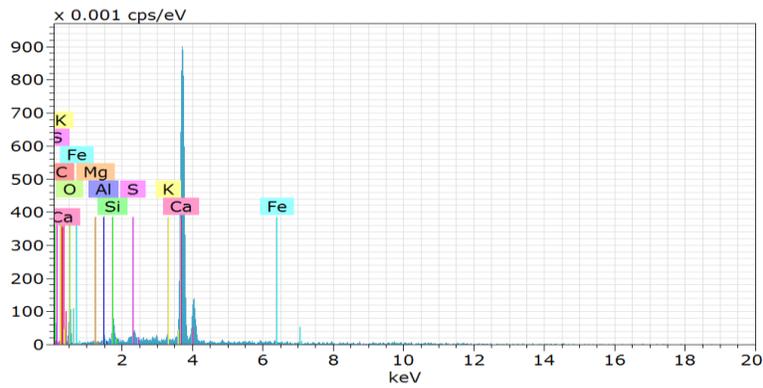


Figure 12. The EDS analysis of sample subjected to freeze-thaw after magnesium sulfate attack.

According to this analysis, Ca, O, Si, Al, S, C, Mg, and K in the sample were found to be 63.33%, 28.29%, 1.26%, 0.49%, 1.24%, 4.92%, 0.30%, and 0.15%, respectively. The EDS analysis of the sample exposed to freeze-thaw after sodium sulfate attack was given in Figure. 13.

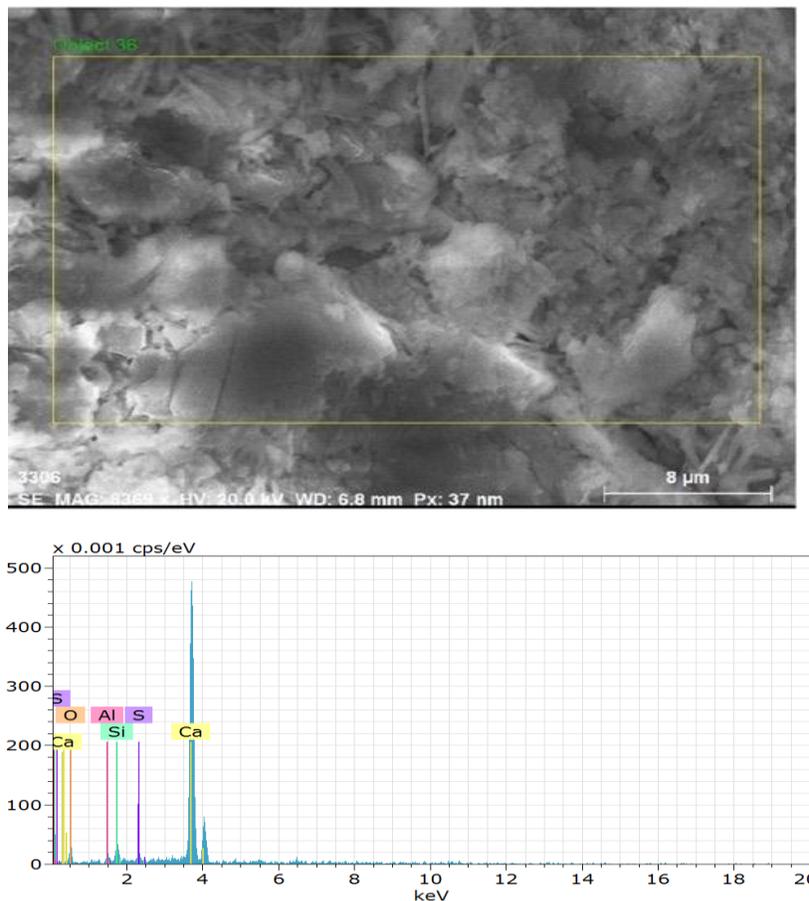
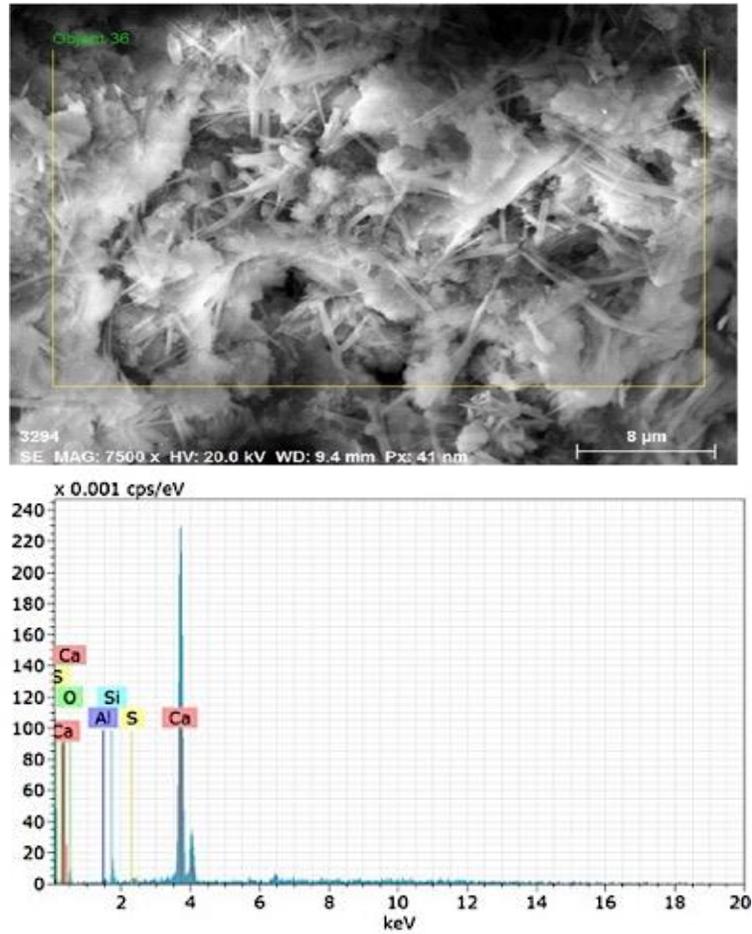


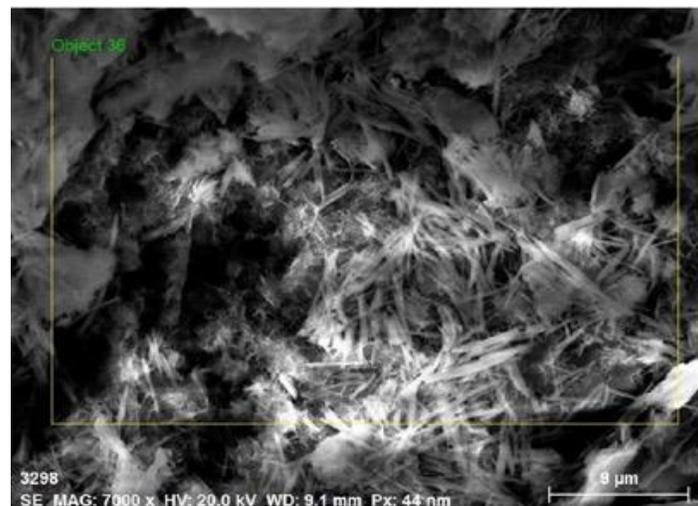
Figure 13. The EDS analysis of sample subjected to freeze-thaw after sodium sulfate attack.

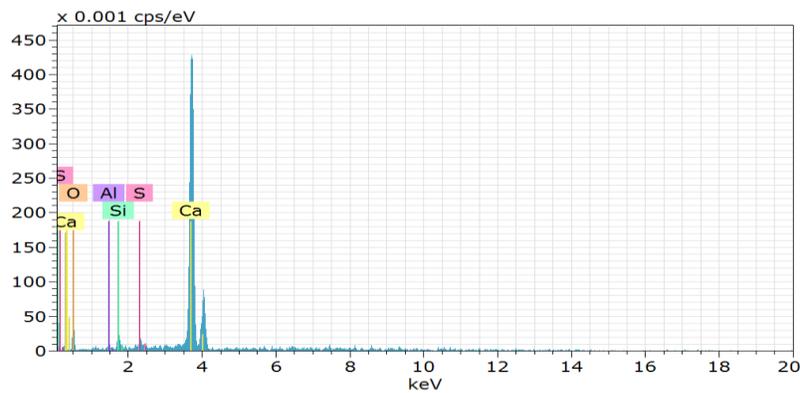
Fig. 12 found that Ca, O, Si, Al, and S in the sample were 65.71%, 30.20%, 1.67%, 1.24%, and 1.18%, respectively. The EDS analysis of the sample subjected to freeze-thaw after the 1% acid attack was shown in Figure. 14.



**Figure 14.** The EDS analysis of specimens subjected to freeze-thaw after 1% acid attack

In this analysis, Ca, O, Si, Al, and S in the sample were 70.5%, 27.23%, 1.44%, 0.68%, and 0.15%, respectively. The EDS analysis of the sample subjected to freeze-thaw after the 1% acid attack was given in Fig. 14.





**Figure 15.** The EDS analysis of specimens subjected to freeze-thaw after 1% acid attack.

Figure. 15 found that Ca, O, Si, Al, and S in the sample were 62.28%, 35.02%, 1.43%, 0.78 %, and 0.49 %, respectively. As the percentage of acid increased, the amount of Ca decreased. In the EDS analysis, the Si and Ca peaks showed that C-S-H occurred. The S and Al were evidence of the presence of ettringite in samples (Huang et al. 2015). The highest ettringite formation occurred in samples exposed to sulfate attack when these analyses were examined. Therefore, the  $f_c$  of this sample was obtained as the lowest in all samples.

#### 4. Conclusions

This study investigated the strength properties of concrete subjected to combined durability effects. The samples were cured at  $20 \pm 2$  °C for 23 months +2 days after standard curing for 28 days. Then, these samples were subjected to freeze-thaw after being exposed to sulfate and acid attacks for four days. The experimental results are shown below:

1. The lowest weight loss was obtained from concrete with  $500 \text{ kg/m}^3$  cement content. When the combined durability effects were examined, the lowest weight loss was obtained from the samples exposed to freeze-thaw after being exposed to the 1% acid attack. This loss was 3.24%. The samples containing  $300 \text{ kg/m}^3$  cement content exposed to both freeze-thaw and magnesium sulfate have the highest weight loss with % 6.13.
2. The highest  $f_c$  was obtained from concrete with  $500 \text{ kg/m}^3$  cement content exposed to freeze-thaw after being exposed to 1% acid attack for four days. The  $f_c$  of this sample was obtained as 40.39 MPa. The lowest  $f_c$  was a sample with  $300 \text{ kg/m}^3$  exposed to both freeze-thaw and magnesium sulfate with 7.25 MPa.
3. Similar to the  $f_c$  results, the highest UPV was obtained from concrete with  $500 \text{ kg/m}^3$  cement content exposed to freeze-thaw after being exposed to 1% acid attack for four days. The UPV of this specimen was found as 2.66 km/h. The UPV of the sample with  $300 \text{ kg/m}^3$  exposed to both freeze-thaw and magnesium sulfate was found as the lowest value with 1.21 km/h.
4. The RDME results found that the samples exposed to sodium sulfate and freeze-thaw were less affected by the increase in cement dosage.
5. This study found that the most damaging durability effect among the combined durability effects was the magnesium sulfate and freeze-thaw effect. Furthermore, this study found that the least damaging to concrete was the 1% acid effect and freeze-thaw. It may be suggested that concrete or reinforced concrete exposed to the combined durability effects should have a high cement content or use pozzolanic additives.

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