DOKUZ EYLÜL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

MECHANICAL AND SHRINKAGE PROPERTIES OF SLURRY INFILTRATED FIBER CONCRETE (SIFCON) MODIFIED WITH SUPERABSORBENT POLYMERS

by
Bayram TUTKUN

June, 2022
İZMİR

MECHANICAL AND SHRINKAGE PROPERTIES OF SLURRY INFILTRATED FIBER CONCRETE (SIFCON) MODIFIED WITH SUPERABSORBENT POLYMERS

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M.Sc THESIS EXAMINATION RESULT FORM

We have read the thesis entitled **"MECHANICAL AND SHRINKAGE PROPERTIES OF SLURRY INFILTRATED FIBER CONCRETE (SIFCON) MODIFIED WITH SUPERABSORBENT POLYMERS"** completed by **BAYRAM TUTKUN** under supervision of **PROF.DR. HALİT YAZICI** and we certify that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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"For everything in the world; for civilization, for life, for success; science is the truest guide."

Mustafa Kemal ATATÜRK

MECHANICAL AND SHRINKAGE PROPERTIES OF SLURRY INFILTRATED FIBER CONCRETE (SIFCON) MODIFIED WITH SUPERABSORBENT POLYMERS

ABSTRACT

The superior performance of high-performance concretes is directly related to their very dense and impermeable structure. This structure can be damaged due to many factors and cause performance losses in terms of both mechanical and durability. Shrinkage is one of the most important durability problems threatening high-performance concrete. Autogenous shrinkage can be reduced by a technique called internal curing. In this study, superabsorbent polymers (SAP) were selected as internal curing agents; the effect of SAP modification on the mechanical and shrinkage properties of slurry infiltrated fiber concrete (SIFCON) was investigated.

In the scope of the study, two SAP with different particle sizes and two additional internal curing water ratios were selected for each SAP. In addition, to investigate the effectiveness of SAP in field conditions, water and relative humidity curing (50% RH, 20 °C) have been applied to all mixtures. The autogenous and drying shrinkage measurements were made from the beginning of setting time by non-contact laser method for up to 7 days. Also, ring test has been applied to investigate the cracking sensitivity of the mixtures under drying conditions. For both types of SAP, reductions in shrinkage were obtained as the additional internal curing water increased. However, the larger SAP was found to be more effective against shrinkage. In this pioneering study of SAP modification in SIFCON, it has been demonstrated that SAP can reduce the shrinkage problem of SIFCON without significant loss of mechanical properties.

Keywords: Superabsorbent polymers, SIFCON, shrinkage, internal curing

SÜPER EMİCİ POLİMERLER İLE MODİFİYE EDİLMİŞ ÇİMENTO BULAMACI EMDİRİLMİŞ LİFLİ BETONUN (SIFCON) MEKANİK VE BÜZÜLME ÖZELLİKLERİ

ÖZ

Yüksek performanslı betonların üstün performansı, çok yoğun ve geçirimsiz yapılarıyla doğrudan ilişkilidir. Bu yapı birçok faktörden dolayı zarar görebilir ve hem mekanik hem de dayanıklılık açısından performans kayıplarına neden olabilir. Büzülme, yüksek performanslı betonu tehdit eden en önemli dayanıklılık problemlerinden biridir. Otojen büzülme, içsel kürleme adı verilen bir teknikle azaltılabilir. Bu çalışmada, içsel kürleme ajanı olarak süper emici polimerler (SAP) seçilmiş ve SAP modifikasyonunun çimento bulamacı emdirilmiş lifli betonun (SIFCON) mekanik ve büzülme özellikleri üzerindeki etkisi araştırılmıştır.

Çalışma kapsamında her bir SAP için farklı parçacık boyutuna sahip iki SAP ve iki ilave içsel kür suyu oranı seçilmiştir. Ayrıca SAP'ın saha koşullarında etkinliğini araştırmak için tüm karışımlara su ve nem kürü (%50 BN, 20 °C) uygulanmıştır. Otojen ve kuruma çekmesi ölçümleri, priz süresinin başlangıcından 7 güne kadar temassız lazer yöntemiyle yapılmıştır. Ayrıca kurutma koşulları altında karışımların çatlak hassasiyetini araştırmak için halka testi uygulanmıştır. Her iki SAP türü için, ilave içsel kürleme suyu arttıkça büzülmede azalmalar elde edilmiştir. Ancak, daha büyük SAP'ın büzülmeye karşı daha etkili olduğu bulunmuştur. SIFCON'da SAP modifikasyonuna dair bu öncü çalışmada, SAP'ın mekanik özelliklerde önemli bir kaybı olmadan SIFCON'un büzülme problemini azaltabileceği gösterilmiştir.

Anahtar kelimeler: Süper emici polimerler, SIFCON, büzülme, içsel kürleme

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CHAPTER ONE
INTRODUCTION

A few decades ago, it was not possible to build sky towers with conventional concrete systems. With the development in the chemical admixture field, today it is possible to produce high-strength, long-lasting concretes which are called ultra-high-performance concretes (UHPC). UHPC is a competitor to conventional reinforced concretes, even steel, and other composites. Fibers with different origins and sizes are used in UHPC designs (Wang, Shi, Wu, Xiao, Huang, & Fang, 2015). A typical UHPC contains a 2% fiber ratio (by volume) (Xue, Briseghella, Huang, Nuti, Tabatabai, & Chen, 2020) and the fiber ratio could be increased up to 6% (Renuka & Rajasekhar, 2021). Yet, it is known that fibers have a negative effect on workability. The concept of laying down the fibers into the molds and pouring the cement-based slurry onto it has gave the possibility to use fibers up to 30% by volume (Yalçınkaya, Beglarigale, & Yazıcı, 2014). Thus, the very high ratios of fibers have led the slurry infiltrated fiber concrete (SIFCON) to achieve superior tensile and flexural performance.

To achieve desired performance in UHPC, some precautions such as lowering the water/cement ratio and increasing the binder dosages must be taken. However, at the same time, these precautions are mostly the reason for a deleterious shrinkage problem which could shorten the service life of the UHPC. Since SIFCON has a very dense and connected fiber network, it could be considered dimensionally stable. Yet, the regions between fibers could crack due to shrinkage strains since fibers restrain the matrix (Gilani, 2007). Thus, even though there are no remarkable length changes, SIFCON could suffer shrinkage cracks. Furthermore, it is known that the cracks formed due to shrinkage cause a uniform void network by connecting all the voids in the concrete and increasing the permeability (Hearn, 1999).

One of the most advantageous properties of UHPC is certainly high impermeability. Due to the very low water/cement ratios and high binder dosages, UHPC reaches a minimum amount of voids. Even though minimizing the voids is the key parameter for reaching high-strength values, it is also preventing adequate water curing especially for thick specimens/concrete structures. Furthermore, since there is a limited amount of water in the UHPC designs, thus water evaporates -or internal relative humidity drops- during the hardening state. Thus, self-desiccation phenomena occur for most of the UHPC mixtures. To ensure adequate curing (Bentz & Weiss, 2011), and prevent self-desiccation (Jensen & Hansen, 2001) internal curing technique is one of the best solutions for cementitious composites.

1.1 Aim and Scope

Literature data have shown that many UHPC types can be produced to provide the desired strength properties (e.g., compressive strength, flexural strength, tensile strength, ductility, toughness, etc.). Furthermore, literature data has also shown that UHPC has superior durability properties due to its very dense and impermeable structure. One of the most important durability problems in UHPC could be named shrinkage. Over the past decade, the internal curing technique has been used successfully for UHPC to mitigate or prevent the shrinkage and cracks formed due to shrinkage.

Even though SIFCON has very limited application fields, it promises very efficient applications such as slabs, repair and reinforcement applications, structures subject to impact loads, and structures subject to the explosion (Tuyan, Yazıcı, & Yazici, 2012). Thus, the durability properties of SIFCON should be examined in detail to support and verify these kinds of applications. Moreover, the shrinkage properties and the effect of internal curing in SIFCON has still a gap in the literature. To ensure all these gaps and problems, this study aims to investigate the mechanical and shrinkage properties of SIFCON which has been modified by superabsorbent polymers for internal curing.

CHAPTER TWO
LITERATURE REVIEW

Internal curing in UHPC has a very complex mechanism behind it. To understand this mechanism both the main structure of UHPC and the internal curing mechanism should be known. This chapter has contained detailed knowledge of UHPC. Literature data about internal curing needing, methods, and application parameters has also been presented. Consequently, two main sections -UHPC and internal curing- have merged at the end of the section to shed a light on the topic of the study.

2.1 Design Parameters and Raw Materials for Ultra-High-Performance Concretes

Traditional concrete could be roughly defined as a combination of cement, water, sand, and gravel. For a long time, traditional concrete has been used with steel rebar reinforcement. This combination has known as reinforced concrete. The need for high-strength and long-lasting concrete has led researchers to develop the properties of traditional concrete. Properties of traditional concrete can be developed by lowering the w/c ratio, decreasing the maximum aggregate size, using pozzolanic additives, and often using fibers (Shi, Wu, Xiao, Wang, Huang, & Fang, 2015). In the mid-1980s researchers were achieved to improve the strength of concrete up to 100 MPa by using a w/c ratio between 0.3 and 0.4. Then, in the early 2000s, thanks to the chemical admixture developments, the w/c ratio was able to be decreased down to 0.15 and 300 MPa strength was obtained (Figure 2.1). Furthermore, with specific applications like pre-setting pressurization, heat treating, and using steel aggregates, even 810 MPa compressive strength was obtained (Richard & Cheyrezy, 1995). This advanced concrete can be defined as high-performance concrete (HPC) or ultra-high-performance concrete (UHPC). Even though the differences between HPC and UHPC are not defined clearly, some researchers claimed that UHPC has to be more workable and less porous (Marvila, De Azevedo, De Matos, Monteiro, & Vieira, 2021). ACI Committee 239 (*ACI Committee 239, 2018*) has defined UHPC as "*concrete that has a minimum specified compressive strength of 22,000 psi (150 MPa) with specified* *durability tensile ductility and toughness requirements; fibers are generally included to achieve specified requirements*".

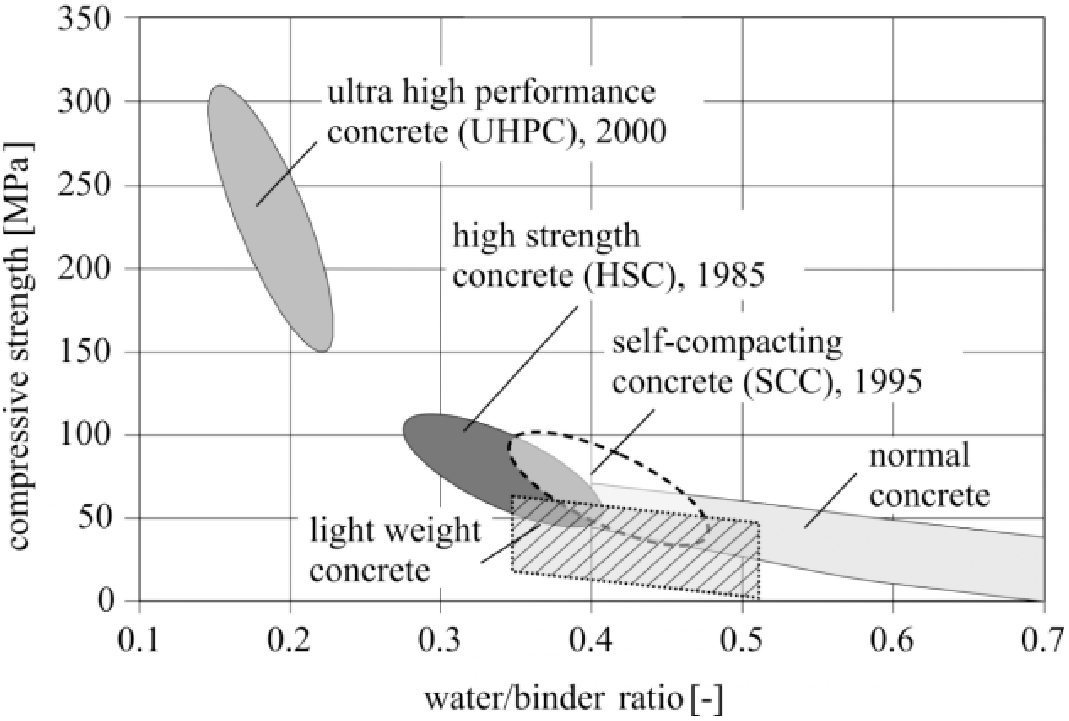

Figure 2.1 Water/binder ratio and compressive strength relationship for various types of concretes (Müller, 2007)

The key parameter in the design of UHPC could be named maximum homogeneity and minimum porosity (Gu, Ye, & Sun, 2015). To obtain desired properties, a phenomenon called "packing density" must be taken into account. Packing density could be defined and calculated as the ratio of water to solids (including all binders and fine materials) (Geisenhansluke & Schmidt, 2004). Decreasing the maximum aggregate size, optimizing the aggregate gradation, using filler materials, and using pozzolanic additives are one of the most used ways to increase the packing density. A typical illustration of packing density in conventional concrete and UHPC has presented in Figure 2.2 (Du, Meng, Khayat, Bao, Guo, Lyu, Abu-obeidah, Nassif, & Wang, 2021).

For such specific and delicate concrete, the raw materials also play an important role. To achieve higher strength values, it is known that high binder dosages must be used. Also, pozzolanic materials should be used to increase the packing density and impermeability. Moreover, binders must be dimensionally stable, generate lower hydration heat, and preferably eco-friendly. Since the matrix strength has very high as compared to the normal concretes, aggregate strength has to be exceeded the matrix strength to make use of the higher matrix strength.

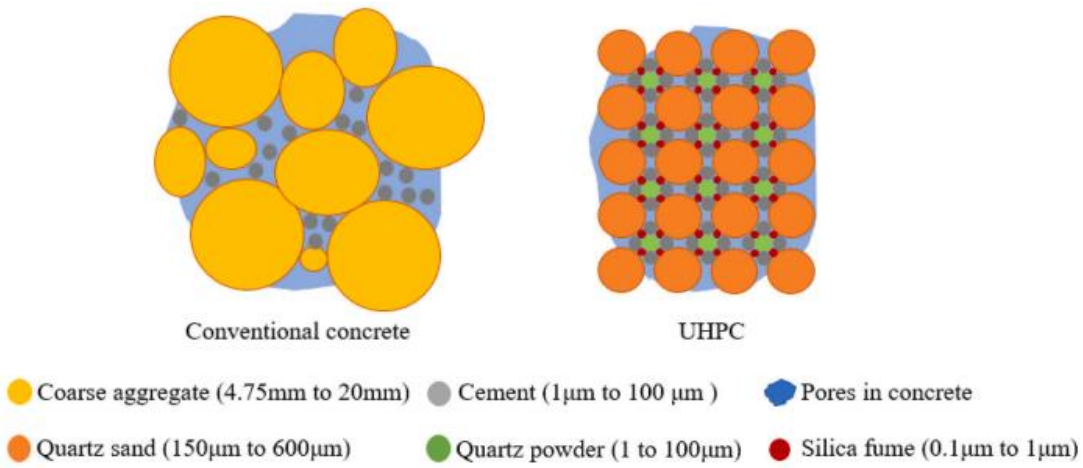

Figure 2.2 Schematic view of packing density in conventional concrete and UHPC (Du et al., 2021)

In UHPC designs, cement dosage must be limited around 800-1000 kg/m^3 since higher dosages could cause hydration heat and dimensional stability problems (Shi et al., 2015). It is also recommended to use cement with low C_3A content since it reduces water content, ettringite formation, and hydration heat (Talebinejad, Bassam, Iranmanesh, & Shekarchizadeh, 2004). It is very common to use pozzolanic materials in UHPC since pozzolanic materials are cost-effective and eco-friendly. Silica fume (SF) has an exclusive role in UHPC designs. Since SF has a very fine particle size distribution, it is vital for an UHPC for improving the packing density (Figure 2.3). Besides the filler effect of SF, it is also known that SF has a positive effect on viscosity (Benaicha, Roguiez, Jalbaud, Burtschell, & Alaoui, 2015), hydration heat (Langan, Weng, & Ward, 2002), fiber-matrix bond (Chan & Chu, 2004), corrosion resistance and permeability (Khan & Siddique, 2011). Some researchers were tried to find alternatives for SF since it is not cost-effective. Rougeau and Borys (2004) have investigated the usability of metakaolin, pulverized fly ash (FA), limestone micro filler, siliceous micro filler, and micronized phonolith as a replacement for SF. Authors claimed that even though all the used materials were suitable for UHPC mixtures, the best performance has obtained when SF was used. Besides the SF, other pozzolanic materials such as FA, granulated blast furnace slag (GGBFS), metakaolin (MK), and limestone powder could be listed as the most used pozzolanic materials in UHPC mixtures (S. Park, Wu, Liu, & Pyo, 2021).

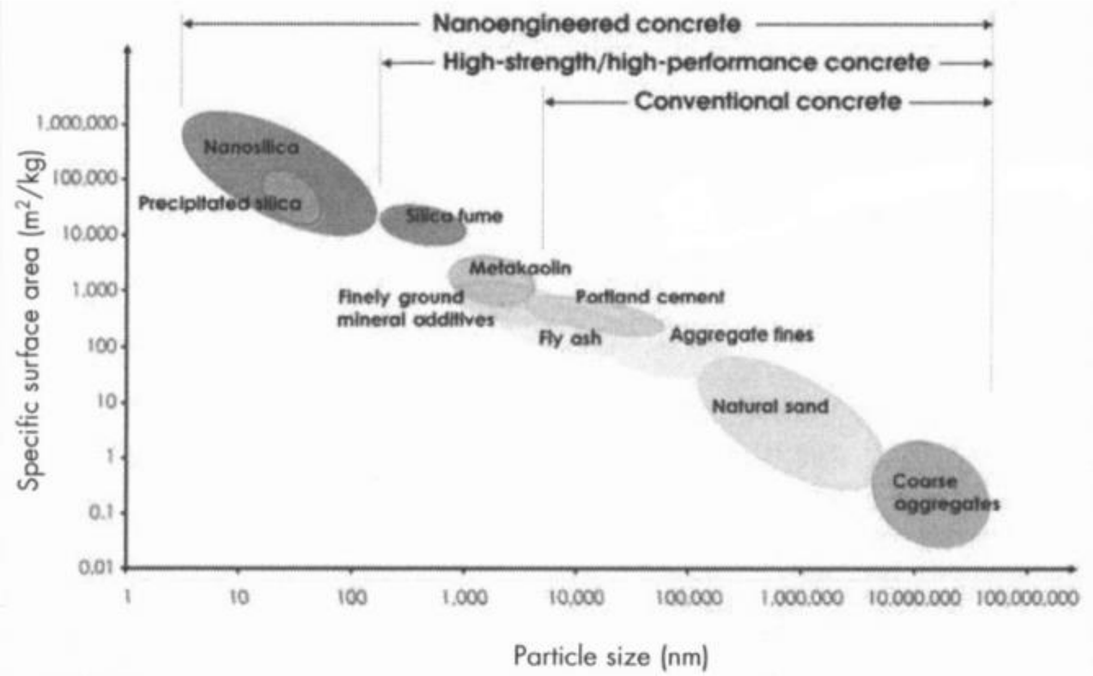

Figure 2.3 Particle-size and specific-surface-area scale related to concrete materials (Sobolev & Gutiérrez, 2005)

The effect of aggregate origin on the UHPC properties can be divided into two main sections: i) quality of aggregate, and ii) aggregate-matrix interfacial transition zone (ITZ). UHPC aggregates must be dimensionally stable, not reactive (e.g. alkali-silica reaction, alkali-aggregate reaction), and high-strength. Research has been made on the suitability of different aggregates for UHPC such as recycled aggregate (Zhang, Ji, Zeng, Yang, Lin, & Liang, 2018), bauxite (Y. Liu & Wei, 2021), iron ore tailings (Zhao, Fan, & Sun, 2014), waste glass (Jiao, Zhang, Guo, Zhang, Ning, & Liu, 2020), etc. Moreover, it could be said that quartz is one of the most used aggregate in UHPC designs. Since these kinds of aggregates mostly have higher strength than the matrix, the ITZ will be the decisive phase when it comes the strength. As aforementioned, ITZ could be improved by increasing the packing density by decreasing aggregate size and using filler materials.

It is obvious that, without the superplasticizers, researchers could not be able to produce UHPC. Yet, superplasticizers must be selected and used at the optimum amount to not increase the cost and avoid side effects. Superplasticizers have an effect on workability, workability life (e.g., slump loss), setting time, strength, and hydration temperature (Hirschi & Wombacher, 2008). As expected, using superplasticizer causes an increase in workability. Yet, it could also cause bleeding or segregation when used over the optimum amount. UHPC should have constant workability at a specific time interval. At this point, the main structure of the superplasticizer plays an important role. Even though the slump loss is related to mainly the hydration process of cement, it has been pointed out that carboxylic acid-based superplasticizer showed less slump loss than lignosulfonate, sulfonated melamine formaldehyde, sulfonated naphthalene formaldehyde-based superplasticizers (Chandra & Björnström, 2002). The setting time is directly related to the chain structure of the superplasticizer. The hydrolysis process causes a decrease in the number of attached side-chains of the superplasticizer. It has been reported that acrylic copolymer-based superplasticizer was less retarded in the setting time than carboxylate terpolymer and polyoxyethylene copolymer due to the chain structures (Felekoğlu & Sarikahya, 2008).

Besides the advanced compressive strength properties of UHPC, flexural strength is also one of the promising features. To enhance the flexural strength and performance different fibers could be used. Using fibers completely changes the behavior of the concrete. Using fiber in brittle materials to enhance flexural performance goes back to ancient times such as reinforcing clay bricks with organic fibers (Katzer, 2006). In the early 1900s, asbestos fibers have used as reinforcement in the production of roofing plates and pipes (Brandt, 2008). Later, the study of Romualdi and Mandel, (1964) have took the attention to using fibers in conventional concrete.

Many different types of fibers could be used as reinforcement in concrete such as steel, glass, wood, asbestos, cotton, bamboo, rockwool), polypropylene, kevlar, nylon, and polyester (Wafa, 1990). Fiber type should be selected considering the needing and environmental conditions. For example, in cases in which the cost is foreground, recycled or organic fibers could be preferred. Moreover, synthetic fibers are drawn attention over the past few decades with their low-cost and high-performance properties. On the other hand, even though corrosion is still a problem, the deformation capacity of steel fibers is making steel fibers indispensable.

Fiber length, geometry, aspect ratio, and dosage have a remarkable influence on concrete properties. As shown in Figure 2.4, while microfibers are effective to control the opening and propagation of microcracks, longer fibers are effective to control the larger cracks and contribution to toughness (Betterman, Ouyang, & Shah, 1995). It is known that the cracking starts at the micro-level in the concrete. If there are the same amount (%, by volume) of micro and micro fibers in the concrete, it is expected that the microfibers have more intensity. Thus, microfibers are more effective to propagate micro cracks. Yet, since the embedded length of micro fibers is shorter, pull-out energy also lower than longer fibers. So, it could be said that longer fibers have contributed to the toughness due to their longer embedment lengths.

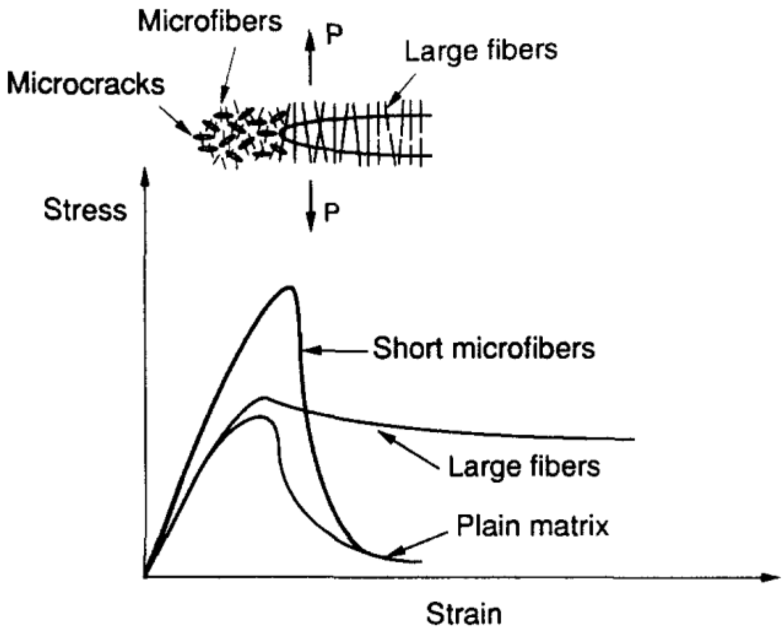

Figure 2.4 Illustration of different sizes of fibers on crack bridging (Betterman et al., 1995)

2.2 Applications of Ultra-High-Performance Concretes

Thanks to the many studies on UHPC over the past few decades, detailed information about the behavior of UHPC is available today. This situation paved the way for the spread of UHPC applications. In UHPC applications, three main ways can be mentioned: 1) building the entire structural member with UHPC, 2) supporting the structural member by UHPC, and 3) urban furniture and decorative arts (Bajaber & Hakeem, 2021). Building the entire structural member with UHPC could be considered expensive. Yet, for the needing of long span and of thick structural members, buildings or members completely UHPC such as a roof for a large silo, cooling towers of nuclear power plants and deck units have been made, too (Hajar, Resplendino, Lecointre, Petitjean, & Simon, 2004). As seen in Figure 2.5, the world's first reactive powder concrete bridge has been manufactured with UHPC in Canada (Sherbrooke Overpass) (Blais & Couture, 1999). Since that, UHPC has been used in many other bridges (Zhou, Lu, Song, & Lee, 2018). In addition to these, many other studies have focused on the use of UHPC for many applications such as architectural applications (Fenzy, Perez, & Bernardi, 2016), public art (Maspoli, 2019), and urban furniture (Bajaber & Hakeem, 2021).

Figure 2.5 Sherbrooke Overpass, the world's first reactive powder concrete bridge (Blais & Couture, 1999)

Supporting the weak structural member by UHPC (strengthening) has very wide application fields (Zhu, Zhang, Hussein, & Chen, 2020). SIFCON occupies a wide place among the application areas of UHPC. Thanks to the very high deformation capacity of SIFCON, it is possible to obtain efficient results with thin sections, especially in reinforcement applications, without disturbing the layout of the existing structure (Abdollahi, Bakhshi, Mirzaee, Shekarchi, & Motavalli, 2012; Al-Rousan & Shannag, 2018; Balaji & Thirugnanam, 2018).

2.3 Slurry Infiltrated Fiber Concrete (SIFCON)

Since the fibers occupy a rigid place in the concrete, they reduce the workability. Depending on the fiber properties, the fiber limit can be specified as 3% by volume for proper mixing of conventional or special fiber concretes (Bentur & Mindess, 2006). SIFCON is a special type of concrete with a very high fiber content (up to 30%) that exceeds these limits with a different process (Yalçınkaya et al., 2014). In SIFCON, the fibers are laid randomly or oriented in the mold, then a very fluid slurry is poured over these fibers. SIFCON design parameters and raw materials are quite in line with the previously UHPC, as aforementioned. Only the coarsest aggregate size is expected to be small enough to pass through the fibers (1-2 mm). A typical SIFCON design and fresh properties with different mineral admixtures has presented in Table 2.1 (Yazıcı, Aydın, Yiğiter, Yardımcı, & Alptuna, 2010). Although short-cut steel fibers are generally used as fibers in SIFCON, polypropylene fiber (Ipek & Aksu, 2019), aramid fiber (Nanni, 1992), waste steel fibers (Sengul, 2018), waste rubber fibers (Canbaz & Çelikten, 2020), Waste agricultural fibers (Awogboro, Olafusi, Olutoge, & Oni, 2015) were used alone or in hybrid form with various fibers. The main important factors in the mechanical properties of SIFCON are matrix strength, fiber volume, fiber type and fiber orientation. Matrix strength and modulus of elasticity naturally directly affect the properties of the mixture. Since fiber type or geometry affects the peeling properties, its mechanical properties are a determining parameter (Tuyan et al., 2012). Although it is difficult to make structures made directly from SIFCON due to the production steps, SIFCON produced as layers has many applications.

Table 2.1 Typical SIFCON design and fresh properties with different mineral admixtures (Yazıcı et al., 2010).

Component	Control	FA50	GGBS50	I-GGBS50
Fly ash (%)	0	50	0	0
Slag (%)	0	0	50	50
Cement (kg/m^3)	800	400	400	400
Fly ash (kg/m^3)	0	400	0	0
Slag (kg/m^3)	0	0	400	400
Silica Fume (kg/m^3)	80	80	80	80
Water (kg/m^3)	339	339	339	231
0-1 mm limestone (kg/m^3)	397	330	388	489
0 – 125 µm limestone (kg/m^3)	397	330	388	342
0–75 µm quartz (kg/m^3)	0	0	0	147
Superplasticizer (kg/m^3)	7.3	6.3	5.2	19.40
Water/cement	0.42	0.85	0.85	0.85
Water/binder[a]	0.39	0.39	0.39	0.26
Minislump flow (mm)	355	375	380	370
V-funnel (s)	6.0	11.1	6.1	8.0

[a]Binder=cement+SF+FA or GGBS.

2.3.1 Mechanical Properties of SIFCON

2.3.1.1 Compressive Behavior of SIFCON

The typical stress-strain relationship of parallel and perpendicular fiber-oriented SIFCON is shown in Figure 2.5 (Homrich & Naaman, 1987). Generally, when the compressive strength is reached, the strain is above 1.5%. In addition, SIFCON can retain more than 60% of its compressive strength, although it deforms more than 10%. In the same strength class, the toughness of SIFCON material is 50 times higher than normal concrete. Naaman and Homrich, (1989) investigated the effects of fiber type, fiber orientation, matrix strength, and die size on the compressive strength of SIFCON in their study using cylindrical samples with a diameter of 75 mm and a height of 150 mm. In that study, hooked and deformed fibers with a rough surface were used. It is stated that in the case of orientation parallel to the load, deformed fibers perform better, in the case of orientation perpendicular to the load, hooked and deformed fibers give higher strength, but the drop after maximum stress is higher. And hooked fibers generally shows higher compressive strength than the deformed type. When hooked fibers are compared, it is seen that the use of 30 mm long fiber increases the compressive strength compared to the use of 50 mm fiber (Lankard, 1984). It is stated that if the sample size is less than three times the fiber length, the core samples can show 15-30% higher compressive strength compared to the molded samples. In the case of fibers oriented perpendicular to the loading axis, the compressive strength can be considerably higher than in the case of parallel orientation. However, the drop after maximum stress is less when the fibers are parallel to the load.

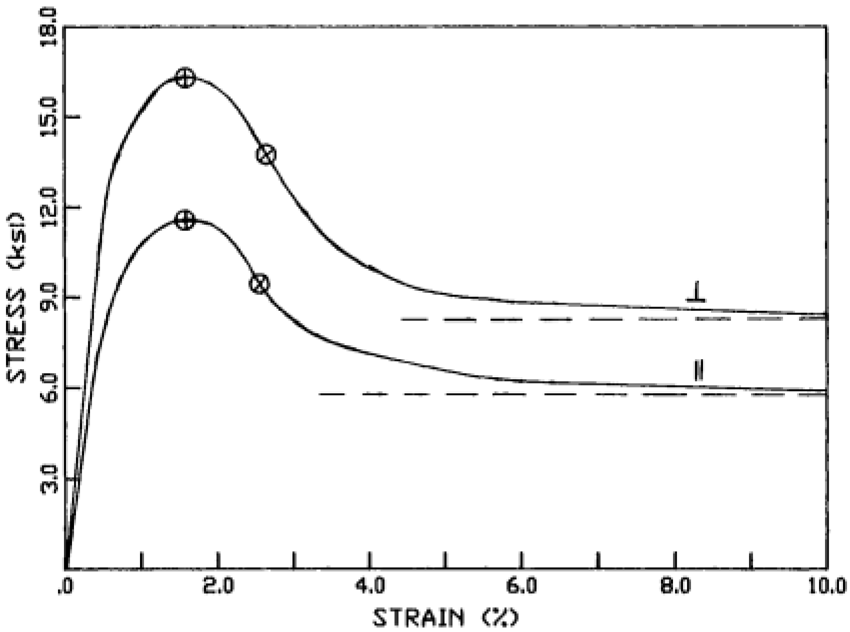

Figure 2.6 Typical stress-strain relationship of parallel and perpendicular fiber-oriented SIFCON (Homrich & Naaman, 1987)

2.3.1.1 Tensile Behavior of SIFCON

Due to the very dense fiber structure, the tensile behavior of SIFCON depends on the bonding of the fibers and the crack structure. Naaman and Homrich, (1989) performed direct tensile tests on 76x38 mm SIFCON samples with high and low fiber content. 80-90% of the fibers are oriented parallel to the loading axis. All specimens showed multiple crack development at low and medium stresses. Cracks that occur up to the maximum load are quite small. However, after the maximum load is reached, the breakage of the sample occurs with the opening of a single crack and the stripping of the fibers. It has been observed that many other minor cracks close after the main crack is formed and the load is reduced. In fact, the elongation measured after this stage indicates the opening of this critical crack. Therefore, the unit deformation calculated after this point is misleading. The researchers transformed the crack opening that occurred after this stage into unit deformation by limiting the unit deformation to 2%. Figure 2.7 shows the load-elongation curve and the transformed stress-strain curve in tension.

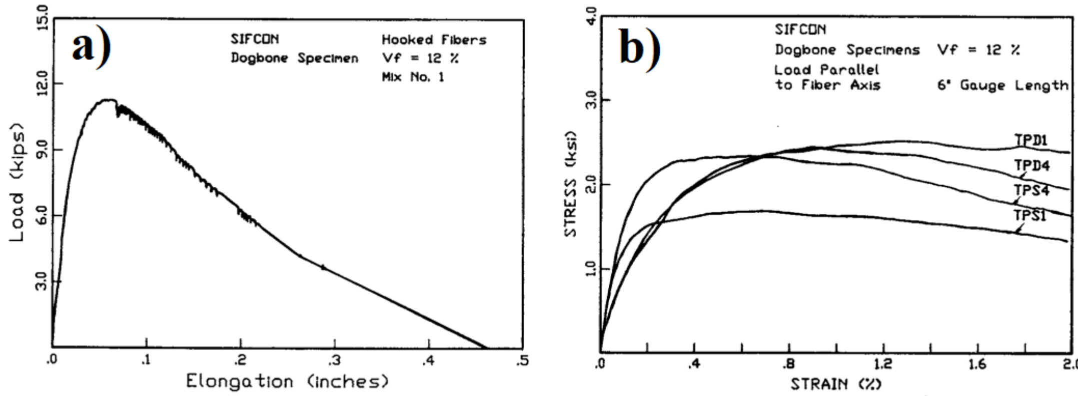

Figure 2.7 Typical load-elongation curve (a) and the transformed stress-strain curve (b) of SIFCON in tension (Naaman & Homrich, 1989)

2.3.1.1 Flexural Behavior of SIFCON

Although flexural behavior in SIFCON is similar to tensile behavior, flexural behavior is of greater importance for the design of structures or structural member strengthening. Under bending, the first crack occurs randomly in the tensile region. However, it does not always start from the fiber which has located bottom of the specimen. In the case of continuation of the loading, cracks similar to the old cracks occur. Crack width does not increase with loading in SIFCON containing dense fiber, but the number of cracks increases. The first crack formation is used to estimate the flexural strength (Naaman, Reinhardt, Fritz, & Alwan, 1993). In the study by Yazıcı et al., (2010), it is seen in Figure 2.8 that the flexural strengths of the reference mixture without fiber and the reference mixture with random fiber distribution are quite similar. However, a slight increase has been observed in the flexural strength of the reference mixture containing oriented fiber. As expected, there is a significant difference in toughness between fiber-free mixtures and fibrous mixtures. The authors stated that when fibers are oriented perpendicular to the loading, both flexural strength and fracture energy have improved. Moreover, the effect of fiber orientation has more impact on fracture energy than flexural strength. In the same study, it has noted that a high amount (50%) of Class C fly ash replacement considerably improved the flexural properties. This indicates that producing eco-friendly SIFCON is both possible and logical.

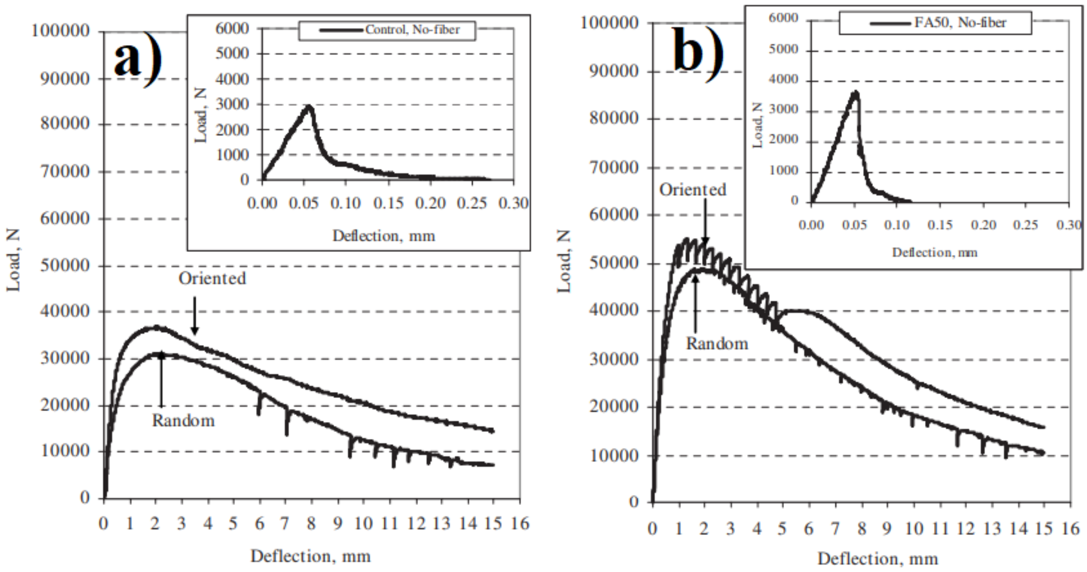

Figure 2.8 Flexural behavior of oriented and randomly distributed fiber-containing SIFCON with and without fiber (control (a) and 50% fly ash containing (b) mixtures) (Yazıcı et al., 2010)

2.3.2 SIFCON Applications

Due to the very high fiber volume, SIFCON can offer superior properties unlike other types of concrete. As stated before, it is difficult to make a structure entirely with SIFCON due to the manufacturing process. For this reason, SIFCON is mostly produced in desired dimensions, assembly on-site, or for reinforcement purposes. It has been stated that when used in the tensile zone as a thick layer, SIFCON has 90% more energy absorption capacity than conventional reinforced concrete and 55% more energy absorption capacity than fiber reinforced concrete (Balaji & Thirugnanam, 2018). Moreover, by using SIFCON at beam-column joints as a strengthening material, earthquake resistance could be improved (Misir & Kahraman, 2013). Sabar and Kadhum, (2022) used SIFCON jackets to strengthen beams damaged by fire. Authors indicated that SIFCON jacketing has improved the energy absorption capacity up to 54% in fire-damaged beams when compared to the undamaged and non-strengthened beams.

It can be said that the closest competitor of SIFCON in the field of reinforcement is glass fiber reinforced plastic (GFRP). In the study by Abdollahi et al., (2012), various SIFCON jacket thicknesses and GFRP layers were compared. According to the results of the study, the authors have suggested that the reinforcement method (SIFCON jacket or GFRP) to be selected should be selected according to the quality of the concrete to be strengthened. The authors stated that the SIFCON layer thickness is the most important parameter affecting the performance after reinforcement. It can also be said that similar results have been obtained for SIFCON and GFRP reinforcement in most cases.

2.3.3 Durability Properties of SIFCON

Apart from the mechanical properties, there are very limited studies on the durability properties of SIFCON. Armagan and Canbaz, (2016) showed that the flexural strength of SIFCON samples exposed to freeze-thaw increased until the 50th cycle, but a sharp decrease was observed after the 50th cycle. It has stated that this situation was related to the formation of cracks in the 50th cycle and the fact that these cracks affected the fiber-matrix adhesion and caused sharp decreases in flexural strength. In studies on the performance and permeability of SIFCON in harmful chemical environments; SIFCON has lost about 5% compressive strength under the influence of sulfate (Deepesh and Kanase, 2016), and the sulfate and acid resistance of SIFCON increased with the substitution of steel slag as fine aggregate (Shelorkar and Jadhao, 2019), and the water absorption capacity decreased with the use of pozzolanic materials (Kani and Raj, 2016). It is clear that SIFCON is faced with the danger of corrosion due to the use of steel fibers, and the absence of any cover thickness. In a study by Alrubaie et al., (2019), SIFCON samples were exposed to wetting-drying cycles and a chloride-containing environment directly. The mixtures containing more than 6% fiber showed higher corrosion potential than the plain sample. Although the corrosion potential of the reinforcement bar was measured in this study, it is seen that SIFCON increases this corrosion potential due to the corrosion of the fibers. In a previous TÜBİTAK project (106M283), the effect of curing type on corrosion has examined. And the corrosion potentials of the near-surface fibers and the central fibers of the SIFCON samples were measured after the wetting-drying cycles. Autoclave cured samples have higher corrosion potential than steam cured and standard cured samples (Figure 2.9). And the fibers on the surface has higher corrosion potential than the central fibers. All samples have found to be more than 90% likely in the active corrosion class (negative half-cell potential more than -270mV) after 350 wetting-drying cycles.

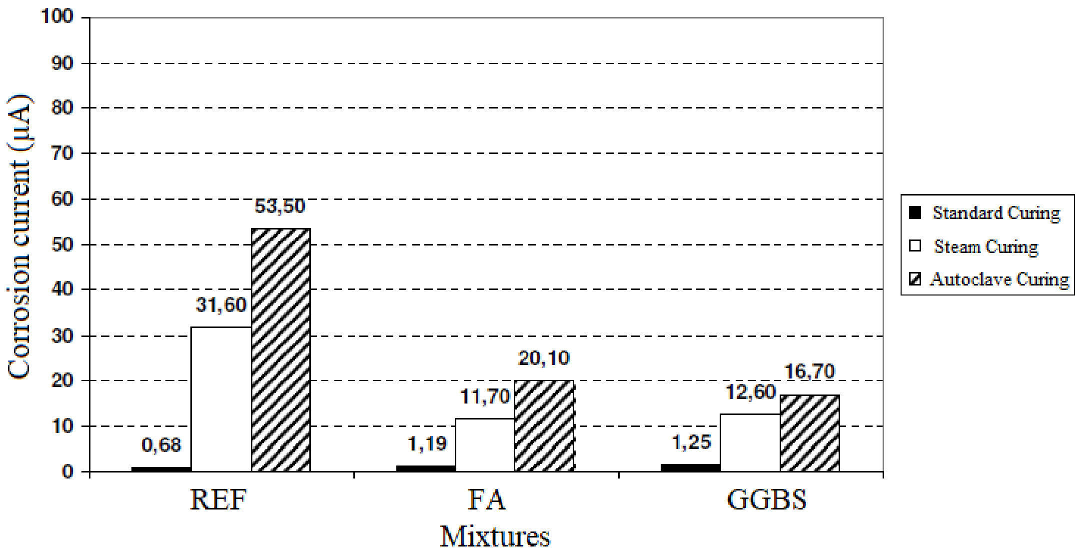

Figure 2.9 Corrosion currents of the fibers at the center of the specimens after 350 wetting-drying cycles

2.3.1.1 Shrinkage Properties of SIFCON

It has stated that the SIFCON matrix (fibreless SIFCON) showed very high drying shrinkage, while the shrinkage values of SIFCON were low. This situation can be interpreted as the very dense fiber structure preventing shrinkage by forming a continuous network with each other (Gilani, 2007). In this study, it is seen in Figure 2.10 that the number of microcracks increases with the increase in fiber content in the images after crack mapping on the drying shrinkage samples. Although the shrinkage values of SIFCON remain at low values, the very high shrinkage of the SIFCON matrix causes cracks locally due to the restriction effect of the fibers. There is no study on the effect of these cracks on durability. According to the studies carried out on other types of concrete in cracked conditions, it has been stated that drying shrinkage is all over the sample, causing it to form a uniform void network by combining all the voids in the sample and increasing the water permeability. On the other hand, in the cracks formed due to loading, the permeability increased locally only in the loading region, and the other regions were not affected (Hearn, 1999). Therefore, cracks due to drying shrinkage in SIFCON tend to cause durability problems. At the same time, considering that steel fibers are generally used in SIFCON, negative effects due to drying shrinkage can be seen in long-term performance. The very high paste volume and low w/c ratio of the SIFCON matrix will have a negative effect on both autogenous and drying shrinkage.

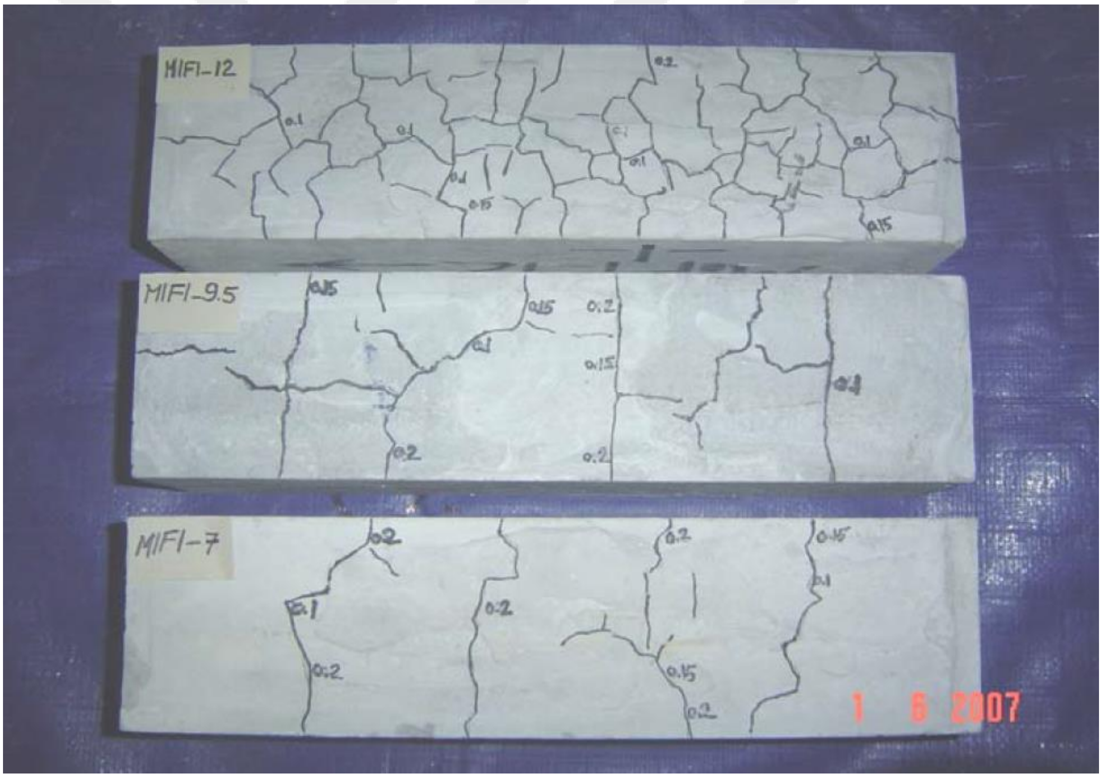

Figure 2.10 Crack formations due to drying shrinkage in SIFCON (fiber ratios are 12%, 9.5%, and 7%, from top to bottom) (Gilani, 2007)

In a previous TUBITAK project, crack sensitivities of various SIFCON matrices have determined by ring testing. Crack times vary depending on the type of mineral additive but range from 51 to 186 hours. When compared to the results of the ring test in other UHPC studies in the literature (Shen, Wen, Kang, Shi, & Xu, 2020; Yeon, 2021), it can be said that the SIFCON matrix cracks very quickly.

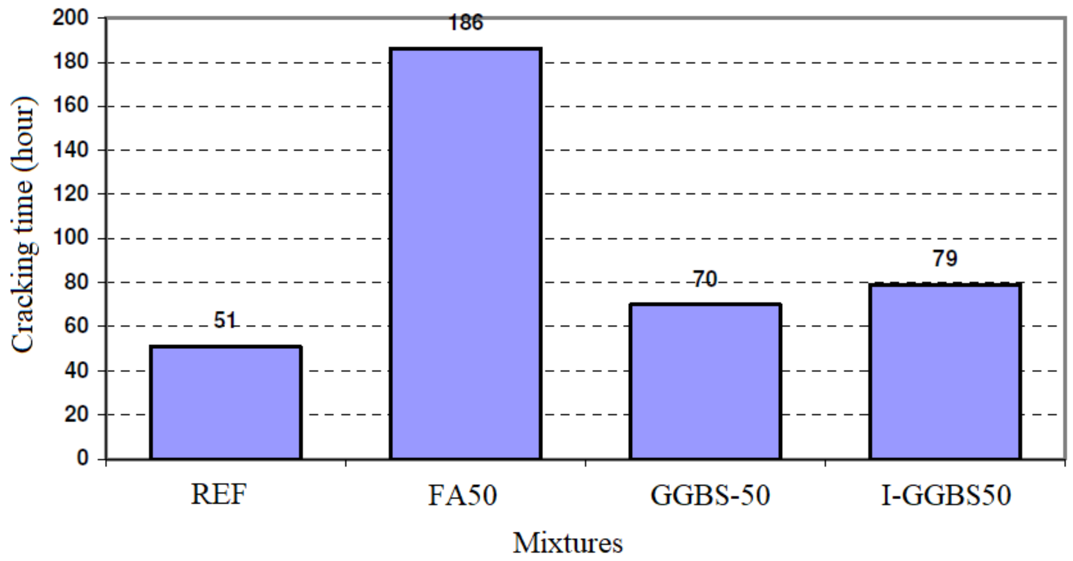

Figure 2.11 First cracking time of SIFCON mixtures according to ASTM C1581 standard ring test (FA: Class C fly ash, GGBS: ground granulated blast furnace slag, I: improved mixture, 50: the cement replacement ratios)

Apart from the above studies, there is a lack of studies in the literature focused on the shrinkage properties of SIFCON. However, it can be seen from the above studies that SIFCON may have problems in terms of shrinkage. For these reasons, shrinkage and shrinkage prevention methods should be investigated in SIFCON.

2.4 Internal Curing

The purpose of internal curing can be explained as preventing the cement reactions from being interrupted by water loss. Unlike conventional concretes, cement-based composites cannot cured adequately by providing external water. As seen in Figure 2.12 due to the dense internal structure of cement-based composites, externally applied water cannot reach the entire section. With the widespread use of ultra-high-performance cement-based composites, the problem of shrinkage caused by water loss and therefore crack formation has gained importance. Lightweight aggregates and agents with high water absorption capacity such as SAP can be used for internal curing.

When the cement reactions start, the water trapped inside the agents' releases and provides the necessary water for the reactions.

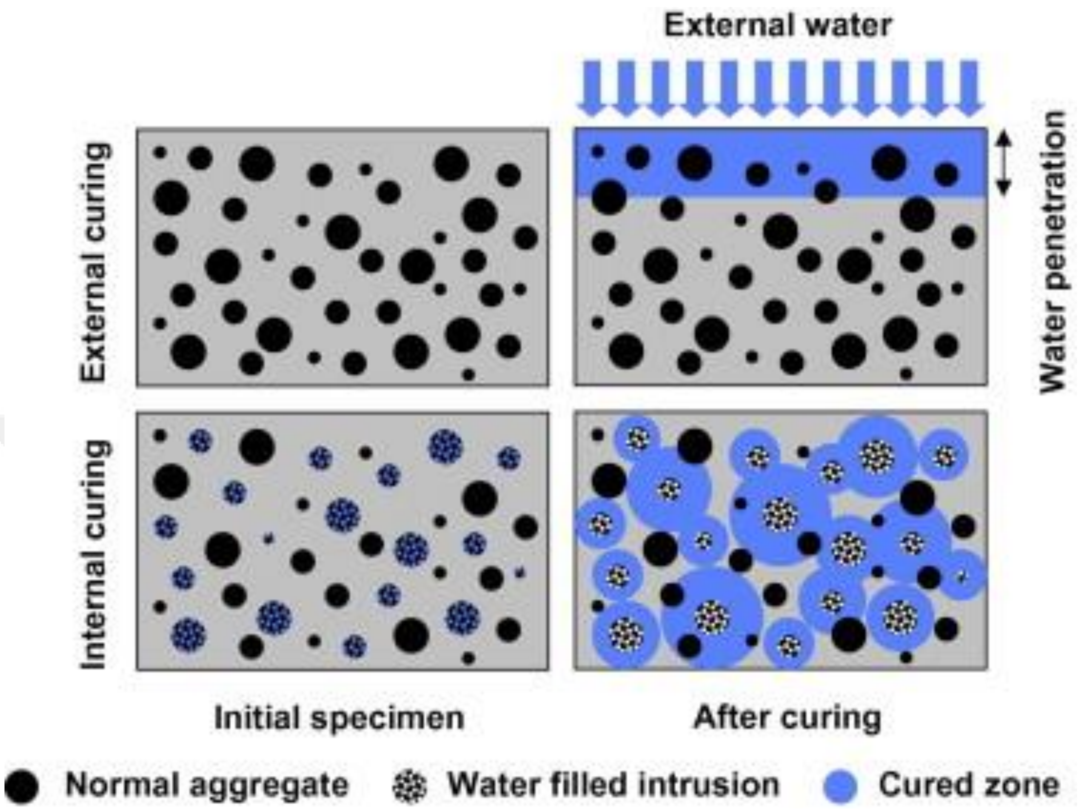

Figure 2.12 Differences between external curing and internal curing (Bentz & Weiss, 2011)

The purpose of internal curing is to prevent shrinkage due to water loss and thus crack formation in cement-based composites. For this purpose, successful studies have been carried out using many internal curing agents in the literature. Materials with water-absorbing and holding until the desired time capacity such as lightweight aggregates, SAP, wood fibers and powders, microcapsules and emulsified water can be used as internal curing agents (Jensen & Lura, 2006). Over the years, many approaches have emerged with developments in how the internal curing agents are added to the mixture, how much water is needed, and the behavior mechanics of the agents in the mixture. SAP is a material with a water absorption capacity of up to 300-400 times its weight. SAP has been used effectively in many fields until today. These areas include hygiene products (diapers, etc.), agricultural use (holding the water needed by plant roots over time), water purification systems, and the pharmaceutical industry (Mignon, Snoeck, Dubruel, Vlierberghe, & De Belie, 2017). In addition to these areas, it has started to be used for internal curing thanks to its high water absorption capacity and its slow release of water into the mixture after the concrete has been set. As an internal curing agent, SAP differs from lightweight aggregates in its very high water holding capacity. The amount of internal curing water increases as the w/c ratio decreases. Therefore, SAP can provide internal curing water in high-performance concrete mixes with only 1-2 kg/m^3 dosage and a much higher amount of lightweight aggregate. For this reason, the negative effects of using a high amount of lightweight aggregate in high-performance concrete on strength and permeability can be prevented. After Jensen and Hansen, (2002) published a detailed publication on the subject as a pioneer, studies on the use of SAP as an internal curing agent gained momentum.

2.4.1 Effect of Internal Curing on Mechanical Properties

While creating extra space in the mixtures using SAP, it also increases the hydration with internal water curing. Whichever of these two effects is dominant, there will be a development in that direction. In general, it is believed that the use of SAP slightly reduces the mechanical properties, but this reduction can be neglected due to its benefits. (Hasholt, Jespersen, Jensen, Tange Hasholt, Seneka Jespersen, & Mejlhede Jensen, 2010) stated that SAP slightly reduces the compressive strength when used in mixtures with an s/c ratio of 0.5, but the decrease in compressive strength is negligible in mixtures with an s/c ratio of 0.35 (Figure 2.13). The reason for this situation was explained by the authors as the increase in the degree of hydration at low w/c ratios, and the fact that SAP had no effect on the degree of hydration at high w/c ratios. Moreover, the authors modeled void volume structure using Bolomey's formula and Power's model based on gel space ratio and claimed that at low w/c ratios, less compressive strength loss is experienced than should be due to voids formed by SAP.

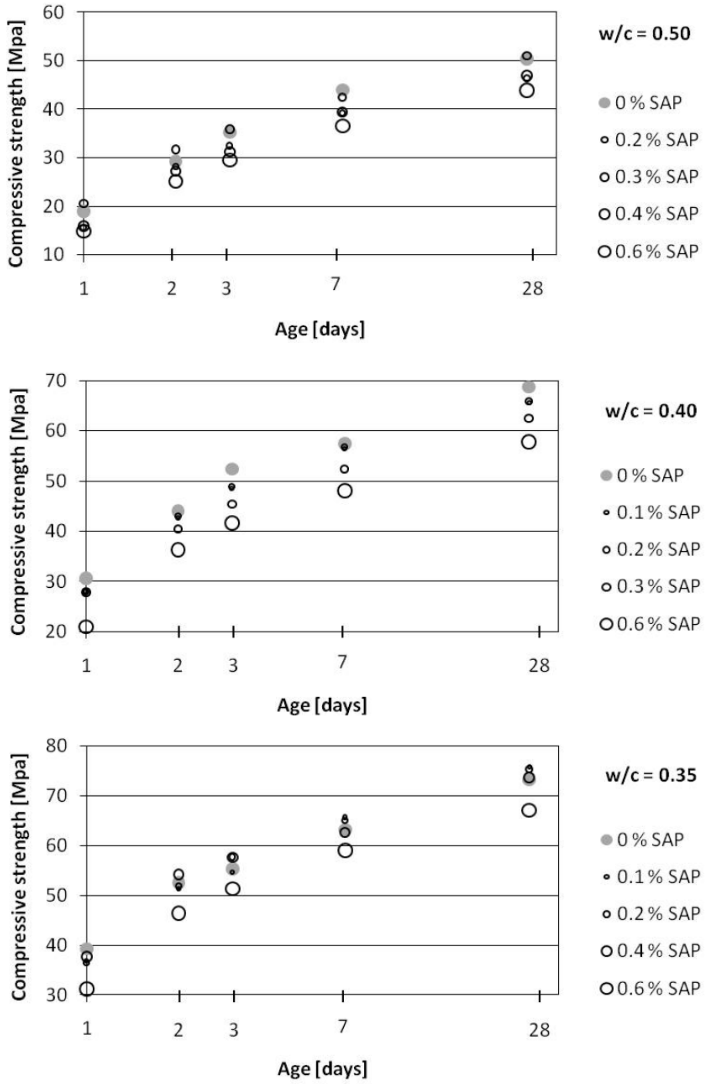

Figure 2.13 Compressive strengths of SAP-modified mixtures (Hasholt et al., 2010)

Justs et al., (2014) stated that the reference mixture hydrated more in the first 24 hours, following the hydration heat of the mixtures containing SAP, which they made for 3 days, but the hydration degrees of the mixtures containing SAP has increased in advancing ages. The degree of hydration has increased with the increase of SAP dosage. The degree of hydration appears to be an important parameter in mixtures containing SAP. Esteves et al., (2007) followed the compressive strength of mixtures containing SAP and reference mixtures at various relative humidity (95%, 50%, and 30%). According to this study, the mixtures using SAP are less sensitive to the decrease in relative humidity, and the difference between them and the reference mixtures approached zero as the relative humidity decreased. The authors also stated that when measuring compressive strength, mixtures containing SAP may yield lower results due to the presence of internal humidity.

2.4.2 Effect of Internal Curing on Shrinkage Properties

Many studies show that SAP reduces autogenous shrinkage when used in appropriate dosages and properly added to the mixture. In a round-robin test with 13 participants, the effect of two types of SAP on autogenous shrinkage has investigated using several autogenous shrinkage measurement methods. According to the results of the study, the most effective results were obtained with the larger particle size SAP (Figure 2.14). Shen, Shi, Tang, Ji, & Jiang et al., (2016) observed a decrease of up to 56% in autogenous shrinkage on the 28th day compared to the control mixture, with the increase in the amount of SAP in their autogenous shrinkage measurements up to 28 days. Justs et al., (2015) stated that SAP caused negligible changes in the setting start and end times. In this study, the increase in the amount of SAP from 0.206% to 0.313% by weight of the cement dosage and a slight increase in the w/c ratio caused a serious decrease in autogenous shrinkage. These results also show the importance of SAP dosage.

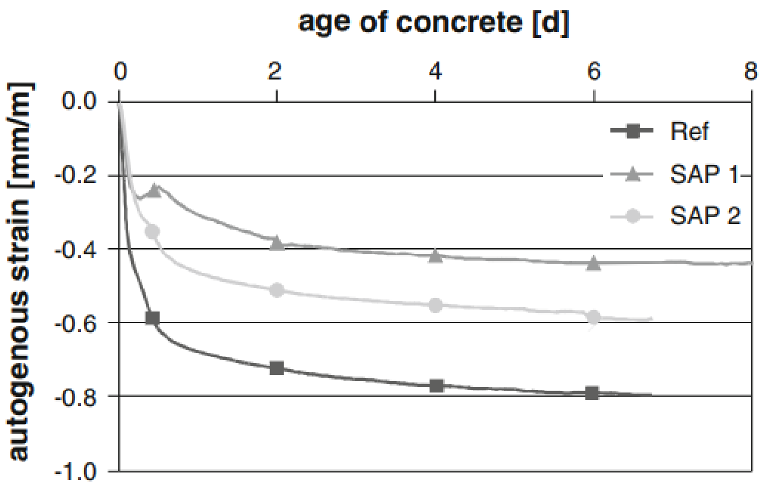

Figure 2. 14 Average autogenous shrinkage results of RILEM round-robin test (Mechtcherine et al., 2014)

In the study by Schröfl et al., (2012), two types of SAP and various w/c ratios has used. The authors stated that autogenous shrinkage decreased, and error expansion was observed in mixtures containing SAP. Furthermore, the authors also stated that the expansion state was caused by the addition of a very high ratio of SAP or the method of adding SAP to the mixture. In this study, SAP was added to the mixture in a pre-activated form. Pre-activation means that SAP is added to the mix after the required internal curing water has been absorbed. It has been revealed in further studies by the same authors that this situation is not suitable for mixing (Mechtcherine, Secrieru, & Schröfl, 2015). This is because SAP tends to leave the water it holds due to external forces, therefore, during mixing, pre-activated SAP gives some of the water to the mixture, therefore increasing the w/c ratio. At the same time, Schröfl et al., (2012) stated that the main factor in the water absorption capacity of SAP is the $Ca^{+}2$ ion. SAP could have different absorption capacities according to the pH of the solution. In this study, the authors also revealed that the SAP type has a great effect on internal curing. It has been observed that SAP, which starts to give out the absorbed water after the start of the set, is effective in preventing autogenous shrinkage.

Drying shrinkage occurs as a result of the evaporation of water in very small voids in the concrete and the convergence of these voids. Shrinkage reducing admixture (SRA) can be used to prevent this situation by reducing the surface tension of the water in these spaces (Folliard & Berke, 1997). Significant reductions in drying shrinkage have been achieved with the use of SRA in high-performance concretes (Folliard & Berke, 1997; Quangphu, Linhua, Jiaping, Qian, & Tienquan, 2008). However, internal curing methods mostly reduce the autogenous shrinkage in high-performance concretes (Zhutovsky, Kovler, & Brameshuber, 2010). Studies has showing that SAP is also a solution to prevent drying shrinkage and cracking. In a study, authors used SAP as a coating material, and it is reported that using SAP is an effective method of preventing cracks in the coating (Beushausen & Gillmer, 2014). Lefever et al., (2020) studied the crack behavior of mixtures using nanosilica and SAP, while mixtures without SAP cracked in 2 weeks in the ring test, mixtures with SAP did not crack for 4 weeks. In a similar study, Shen, et al., (2016) stated that as the SAP dosage increases, the time to first crack increased in the ring test. Yang et al., (2019) observed a reduction in drying shrinkage in the mixtures in which they used SAP 0.2%, 0.4%, and 0.6% by weight of cement. The maximum reduction in drying shrinkage was 0.4%. Mönnig, (2006), compared SAP with the lightweight aggregates. The authors stated that both lightweight aggregate and SAP reduced drying shrinkage from the first week (Figure 2.15). On the other hand, there have been authors reporting negative effects on drying shrinkage, Mechtcherine et al., (2008) observed higher drying shrinkage in the mixtures they used SAP. Authors stated that this may be due to the presence of more free water in the environment and therefore the increase in the amount of water to be removed by drying.

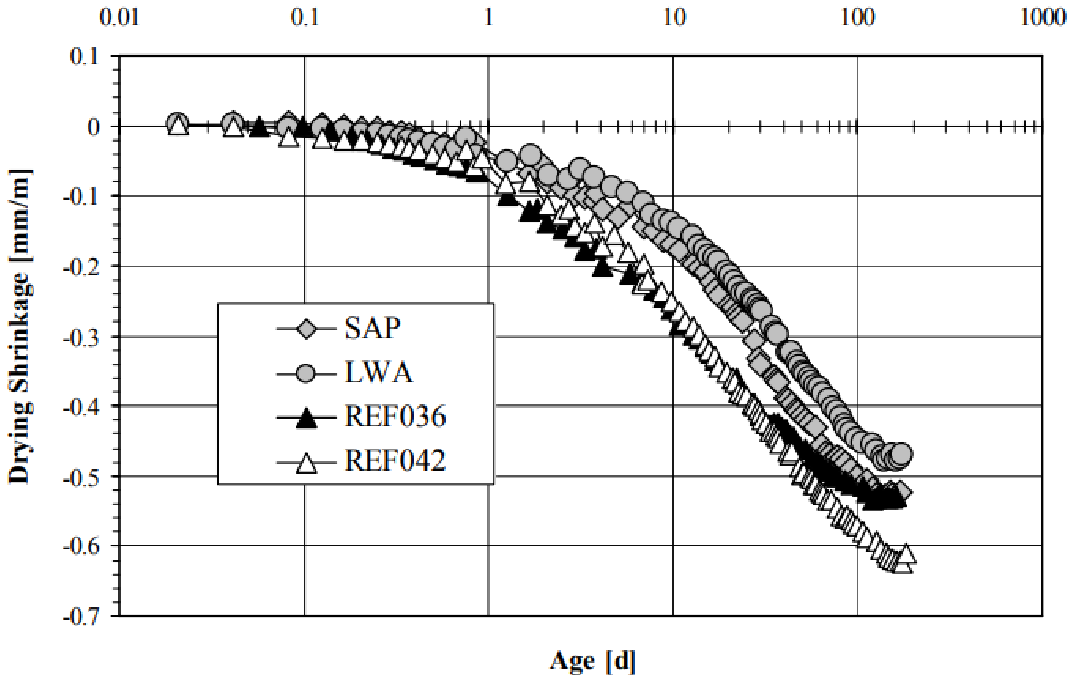

Figure 2.15 Comparison of drying shrinkage values of LWA and SAP-containing mixtures (Mönnig, 2006)

It is known that the use of SAP reduces deformations and delays crack formation in the ring test. Shen, et al., (2016) used ring test to evaluate the restrained shrinkage behavior of SAP-containing mixtures. While the control mixture cracked on the 10th day, with the increase in the SAP dosage, the cracking occurred on the 19th day. In the same study, while the control mixture reached stress of 5 MPa just before cracking, this value decreased to 3 MPa with increasing SAP dosage. Similarly, Lefever et al., (2020) found that while the control mixture cracked on the 10th day, the mixtures containing SAP did not show any cracks until the 30th day, which is limited to the test period. In another study, while the control mixture and a mixture containing a low amount of SAP cracked, other mixtures containing SAP did not crack during the test period which has limited to 28 days (Figure 2.16). At the same time, similar to other studies, the stresses had much lower in all mixtures using SAP (Lyu, Shen, Mo, Chen, He, Li, & Qin, 2020).

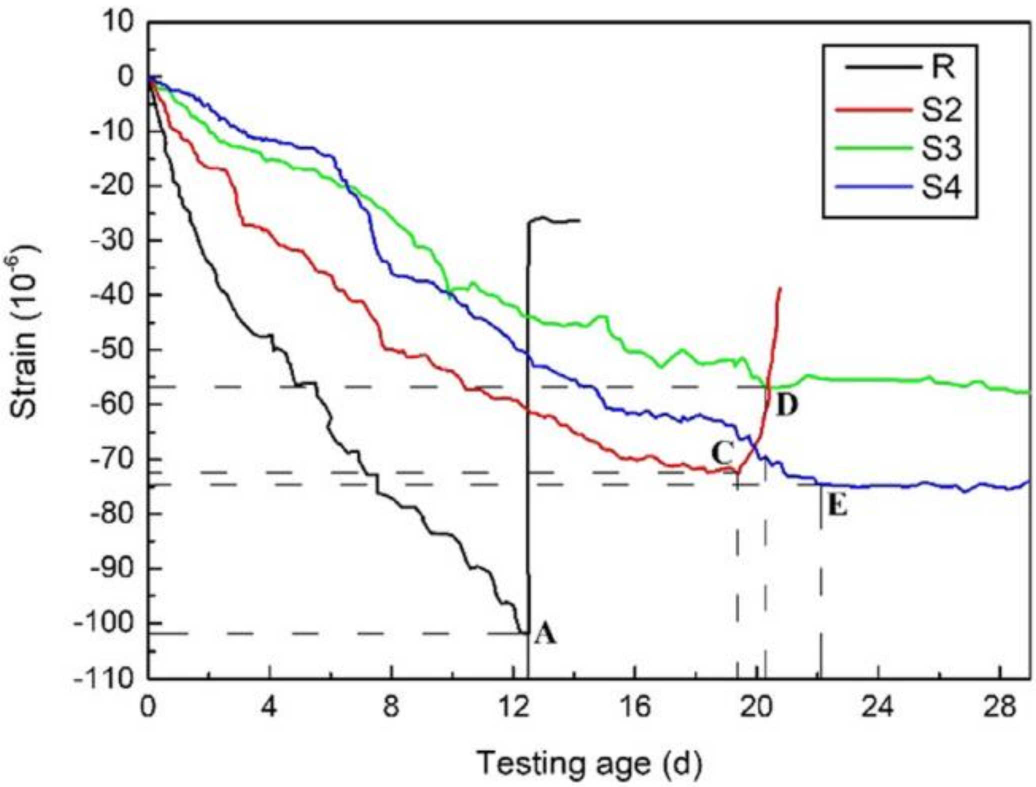

Figure 2.16 Restrained shrinkage behavior of SAP under drying conditions (R: reference, S2: 0.7185 kg/m3, S3: 0.8754 kg/m3, S4: 1.032 kg/m3 SAP containing mixtures) (Lyu et al., 2020)

CHAPTER THREE
MATERIALS AND METHODS

3.1 Materials

3.1.1 Cementitious Materials

CEM I 42.5 R type Portland cement with a specific surface area of 395 m^2/kg was supplied from Soma Cement. Silica fume (MasterRoc® MS 610) with 20,000 m^2/kg nitrogen absorption fineness and 92.25% SiO_2 content was supplied from BASF. Material properties of cement and silica fume are presented in Table 3.1.

Table 3.1 Chemical compositions of cement and silica fume

Chemical Composition (%)	Cement	Silica Fume
SiO_2	19.79	92.25
Al_2O_3	4.78	0.88
Fe_2O_3	3.39	1.98
CaO	63.71	0.51
MgO	1.78	0.96
Na_2O	0.32	0.45
K_2O	0.78	0.12
SO_3	2.84	0.33
Cl^-	0.0098	-
Free CaO	-	-
Loss on Ignition	2.09	-

3.1.2 Superabsorbent Polymers

Two SAP based on acrylamide and acrylic acid were supplied from SNF Floerger. The finer SAP (D50: 215 µm) was named SAP A and the coarser SAP (D50: 725 µm) was named SAP B. Master Sizer Hydro 3000 model particle size analyzer was employed for determining the particle size distributions of the used SAP. Particle size distributions of used SAP are given in Figure 3.1.

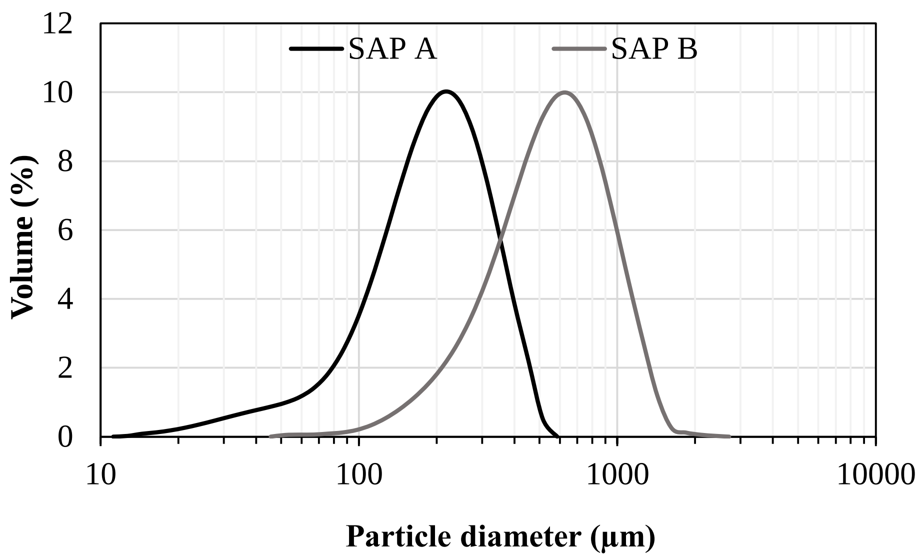

Figure 3.1 Particle size distributions of SAP A and SAP B

3.1.3 Aggregates

Three different sizes (0.4–1, 0–0.4, and 0–0.075 mm) of quartz aggregate were used in the experimental studies. The specific gravity and water absorption of the quartz aggregate were 2.61 and 0.10%, respectively. Aggregate gradation is presented in Figure 3.2. A finer aggregate gradation was preferred and the maximum aggregate size was limited to 1 mm to ensure adequate filling into the dense fibers.

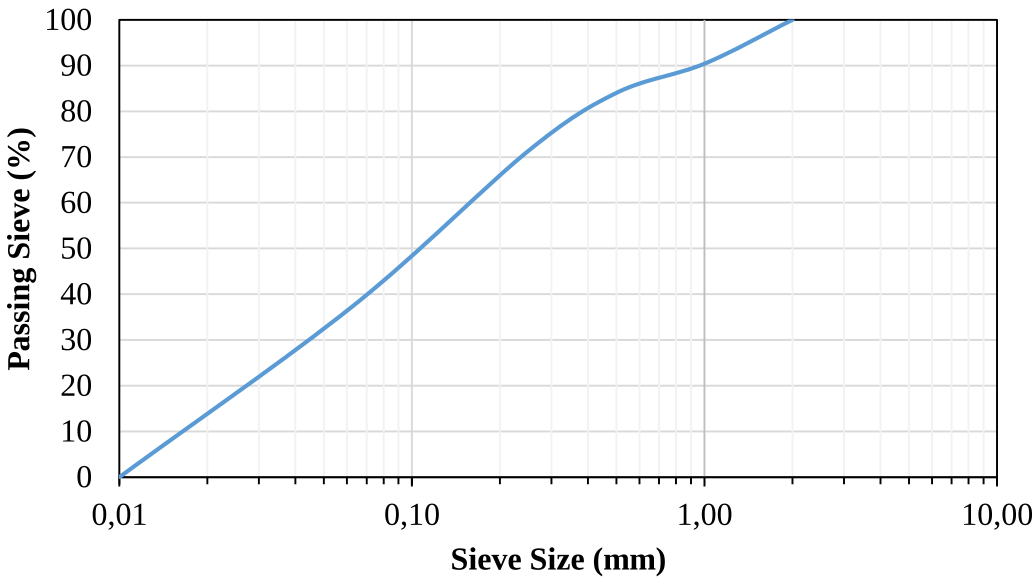

Figure 3.2 Gradation of used quartz aggregate

3.1.4 Superplasticizer

Polycarboxylic ether-based high-range water reducer (MasterGlenium® ACE 450) supplied from BASF was used in the SIFCON matrix to reach the aimed fresh state properties without any bleeding and segregation. Properties of the superplasticizer are given in Table 3.2.

Table 3.2 Properties of the used superplasticizer

Properties	**Explanations**
Form	Liquid
Structure of materials	Polycarboxylic ether-based
Color	Amber
pH value	5 – 7 (20 °C)
Density	1.069 – 1.109 kg/lt (20 °C)
Solubility in water	Miscible

3.1.5 Fibers

Hooked-end steel fibers with 35 mm in length and 0.55 mm in diameter were used in the experimental studies. The aspect ratio and tensile strength of steel fibers were 45 and 1200 MPa, respectively. Images of the used steel fibers are presented in Figure 3.3.

Figure 3.3 Steel fibers (Personal archive, 2022)

3.2 Methods

3.2.1 Mixture Designs and Mixing Procedure

SIFCON matrix with a water/cement ratio of 0.28, water/binder ratio of 0.25, and water/powder ratio of 0.18 was designed. The fiber ratio was 10% by volume and the fibers were randomly placed.

There are two approaches in the literature for the amount of water required for internal curing. Bentz & Snyder (1999) approach and Jensen & Hansen (2001) approach are given in Equation 3.1 and Equation 3.2, respectively.

$$M_{LWA} = \frac{C_f * CS * \alpha_{max}}{S * \phi_{LWA}} \quad (3.1)$$

where:

M_{LWA} = mass of (dry) LWA needed per unit volume of concrete (kg/m3);

C_f = cement factor (dosage) for concrete mixture (kg/m3);

CS = chemical shrinkage of cement (g of water/g of = chemical shrinkage of cement (g of water/g of cement);

α_{max} = maximum expected degree of hydration of cement;

S = degree of saturation of aggregate (0 to 1);

ϕ_{LWA} = absorption of lightweight aggregate (kg water/kg dry LWA).

$$(w/c)_e = \begin{cases} 0.18(w/c); & for\ w/c < 0.36 \\ 0.42 - (w/c); & for\ 0.36 \le w/c \le 0.42 \\ 0; & for\ w/c > 0.42 \end{cases} \tag{3.2}$$

where:

$(w/c)_e$ = additional water needed for internal curing (g of water per g of cement)

These approaches recommend the same ratio of internal cure water up to 0.36 w/c ratio. Bentz & Snyder (1999) approach suggests a constant additional internal curing water for w/c ratios higher than 0.36. In this case, according to Bentz & Snyder (1999) approach, it can be said that all concrete types and classes require internal curing. On the other hand, Jensen & Hansen (2001) approach suggests additional internal curing water that increases up to 0.36 w/c ratio and decreases up to 0.42 w/c ratio to zero. The comparison of the two approaches is presented in Figure 3.4 (Castro, Spragg, & Weiss, 2012).

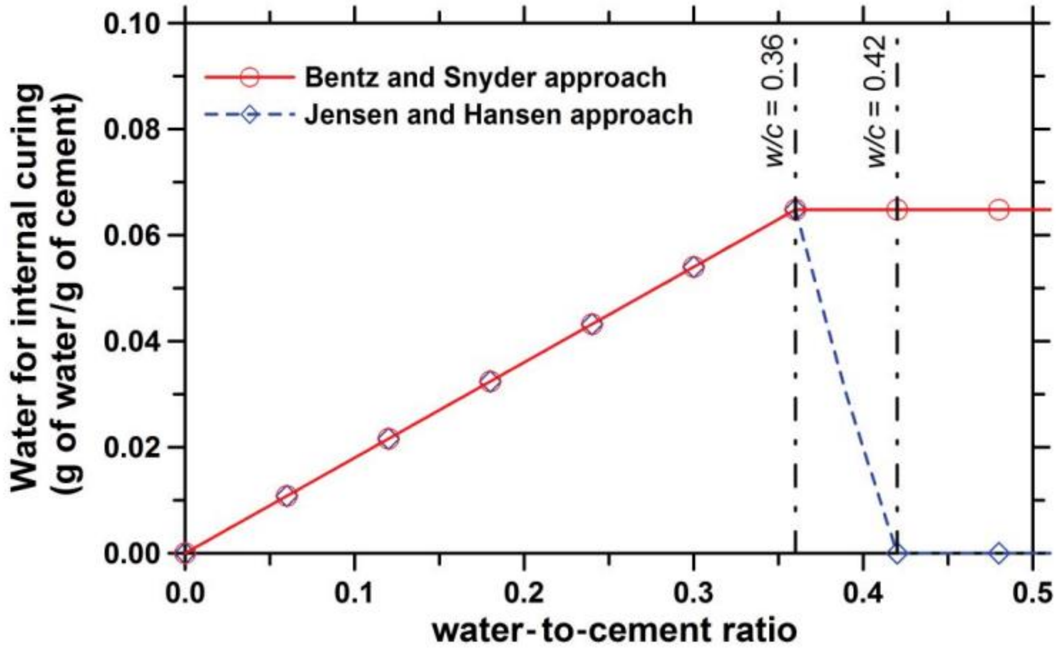

Figure 3.4 Comparison of the two different approaches for calculating additional internal curing water (Castro et al., 2012)

Since the w/c ratio of the mixtures used in this study is constant at 0.28, the same additional water is obtained with both approaches. This amount was found to be 36 kg/m^3. Since all conditions such as full hydration of cement, water access to all cement, etc. were not ideal, as expected, a higher additional internal curing water (60 kg/m^3) have also been selected. Thus, for both SAP, two additional internal curing water (30 kg/m^3 and 60 kg/m^3) have used. In addition, water has added to the top of the mixture, and SAP has weighted to absorb the desired amount of additional internal curing water. The absorbency of SAP has been determined by the "slump flow method", which will be explained in detail in later chapters. According to the water absorption rates found by the slump flow method, 0.11% and 0.19% (by weight of cement) SAP were used for the mixtures of SAP A 36 and SAP A 60, respectively. These ratios were 0.14% and 0.23% for SAP B 36 and SAP B 60 mixtures, respectively. In previous studies, additional internal curing water has changed by keeping the used SAP ratio constant (J. Liu, Farzadnia, Khayat, & Shi, 2021; Mechtcherine et al., 2014). However, in this study, it was thought that equalization of the additional internal curing water would allow a more objective comparison between the two SAP.

Mix designs of the SIFCON and SIFCON slurries are given in Table 3.3 and Table 3.4, respectively. Two different designs are given since some tests (e.g. shrinkage tests) will be performed only on the non-fibrous SIFCON matrix (which will be called SIFCON slurry throughout the study), and some tests (e.g. compressive strength, bending strength) will be performed on fibrous and non-fibrous SIFCON. In the designs given in Table 3.4, the fiber was removed and the remaining volume was distributed proportionally to the other components. Aggregate and cement volumes have stayed constant for SAP mixtures. Thus, the mixture designs for 1 m^3 has changed. However, the additional SAP volumes were minor. For mixtures containing SAP, the first part of the mixture codes indicates the SAP type, and the second part indicates the additional internal curing water amount (e.g. SAP A 60: the mixture containing SAP A and 60 kg/m^3 additional internal curing water).

Table 3.3 Mixture designs of the SIFCON composites

	REF	SAP A 36	SAP B 36	SAP A 60	SAP B 60
Cement	800	800	800	800	800
Silica Fume	80	80	80	80	80
Water	220	256	256	280	280
SAP	0	0.90	1.09	1.50	1.82
Quartz (0-0.075 mm)	328	328	328	328	328
Quartz (0-0.4 mm)	419	419	419	419	419
Quartz (0.4-1 mm)	140	140	140	140	140
Steel Fiber	785	785	785	785	785
Superplasticizer	17.0	17.0	17.0	17.0	17.0

*units are kg/m^3

Table 3.4 Mixture designs of the SIFCON slurries

	REF	SAP A 36	SAP B 36	SAP A 60	SAP B 60
Cement	880	880	880	880	880
Silica Fume	88	88	88	88	88
Water	242	268	268	295	295
SAP	0	0.99	1.20	1.65	2.00
Quartz (0-0.075 mm)	361	361	361	361	361
Quartz (0-0.4 mm)	461	461	461	461	461
Quartz (0.4-1 mm)	154	154	154	154	154
Superplasticizer	18.7	18.7	18.7	18.7	18.7

*units are kg/m^3

A Hobart mixer with 2 liters capacity was used to prepare mixtures. SAP has added to the mixtures in dry form and additional internal curing water has weighed with mixture water. All dry ingredients of the mixture were premixed for 1 min. Then, 2/3 of the water and superplasticizer with 1/3 of the water were added to the mixture respectively. Mixture with water and superplasticizer have mixed at normal speed for 9 min and mixing has continued another 9 min at high speed. Since the SIFCON matrix has a very high powder content and relatively high superplasticizer amount, long mixing periods were preferred to ensure the superplasticizer dispersed homogenously.

After the mixing finished, the SIFCON matrix has poured into the molds which has already been filled with 10% (by volume) steel fibers. The slurry was placed gradually in 10 steps to ensure that slurry has filled adequately between fibers. No vibration was applied to the samples and molds were kept in an environment with 95% relative humidity for 24 hours. After the specimens were removed from the mold, half of them were placed in a standard water cure (20 °C) and the other half placed in a climate cabinet (20 °C and 50% relative humidity). At least 3 samples were taken for each experiment and mean values with standard deviation bars are presented in the graphs.

3.2.2 Teabag Method

The teabag method has used to determine the absorption kinetics of SAP in deionized water and cement pore solution. Illustration of the teabag method are presented in Figure 3.5. A cement slurry has been prepared with a w/c ratio equal to 5 and continuously stirred for 24 h. Then, the cement slurry has filtrated with a filter paper which has a 2.5 μm mesh size to separate cement particles from pore solution as recommended by Snoeck et al., (2018). For each solution and SAP, 3 specimens were prepared. Tests have been conducted in an environment that has a 20 °C constant temperature. Teabags containing 0.2 g SAP has put into 200 ml solution and taken out at 1, 5, 10, 30, 60, 180 min. For each interval, the surface water has gently wiped with a towel, and specimens were weighed. To prevent evaporation, beakers were immediately covered with a parafilm after weighing.

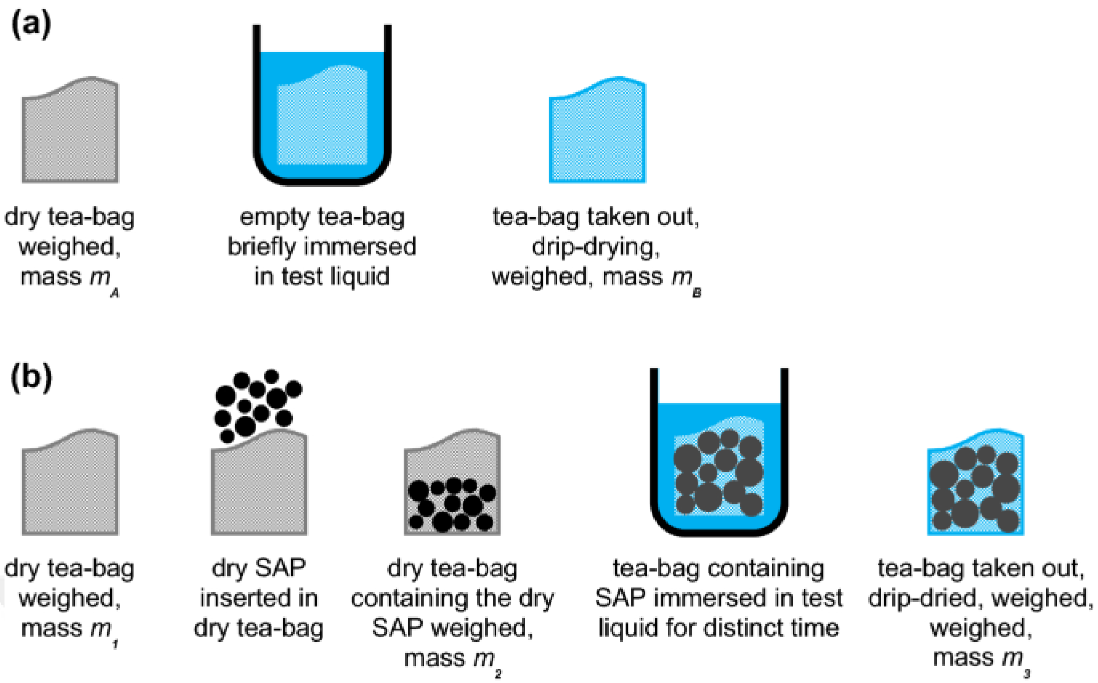

Figure 3.5 Illustration of teabag method (Assessment of wet dead load (a) and assessment of SAP sorption (b)) (Snoeck et al., 2018)

The teabag method is a reference method to determine the absorption capacity of SAP. The teabag method could be used to understand the characteristics of SAP, in terms of absorption and desorption kinetics. Most realistic results can be obtained in the teabag method by using concrete pore solution as a test liquid. Yet teabag method results may not always accurately reflect the water absorption capacity of SAP in cementitious environments. Since there are no external forces or external pressure, the teabag method reflects the "free sorption" of SAP (Snoeck et al., 2018). Also, many researchers stated that interstitial water between the SAP particles in the teabag method caused overestimated water absorption capacity values (Esteves, 2011; Zhao, Jensen, & Hasholt, 2020; Zhong, Wang, Wang, Liu, Li, & Zhou, 2020). It is also known that finer SAP particles decreased the amount of interstitial water (Omidian, Hashemi, Sammes, & Meldrum, 1999). Thus, SAP with different particle sizes will cause different experimental errors. For all the above reasons, the absorption capacity of SAP should be determined with more accurate test methods such as the slump flow test.

3.2.3 Slump Flow Method

Slump flow test are based on adding extra water into the SAP-containing mixtures to obtain the same slump flow value the reference mixture (Schröfl et al., 2012). Slump values at 30 min are taken into account, and water was added to the SAP mixtures to equal the slump values with reference mixtures. Slump flow test was conducted on standard mortar mixtures (w/c ratio 0.5 and quartz aggregate used with standard gradation) since SIFCON's fresh properties are very delicate and contain very fine particles which could lead to misinterpretation. The flow values were determined after 15 drops, according to the TS EN 1015-3 standard.

3.2.4 Free Flow Test

Free flow test was conducted with a cone with the internal dimensions of 100 mm diameter at the base, 70 mm diameter at the top, and a height of 60 mm. The test was conducted according to EFNARC guidelines. Details of the cone are presented in Figure 3.5-a. The measurements have taken 29 to 31 min after the start of the mixing.

3.2.5 Mini V-Funnel Test

Mini V-Funnel test was conducted with a funnel with the internal dimensions of 30 mm width at the base, 270 mm width at the top and a height of 300 mm. Test was conducted according to EFNARC guidelines. Details of the mini v-funnel are presented in Figure 3.5-b. The measurements have taken 29 to 31 min after the start of the mixing.

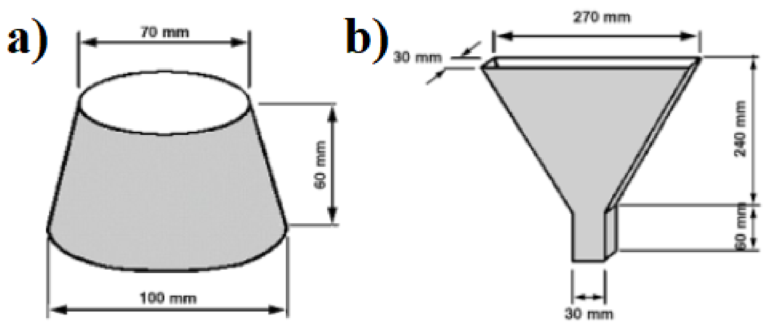

Figure 3.6 Details of the slump cone (a) and mini v-funnel (b) (Yalçınkaya, 2015)

3.2.6 Setting Times

The vicat test was performed to find the start time of the shrinkage measurements. The starting moment of the setting was considered the first moment when the sample was able to support itself, and the lid on the laser-facing side of the mold was removed at this time. The start and end of the setting time of the specimens were determined with an automatic vicat device. In autogenous measurements, the vicinity of the vicat chamber was filled with parafilm oil, which does not mix with the concrete, in such a way as to pass the sample by approximately 5 mm, and the ambient temperature was set to 20 °C. In the drying shrinkage measurements, the environment was kept at 50% relative humidity and 20 °C. If the needle penetration is 4 mm from the base of the mold, it is noted as the initial setting time, and when the needle penetration is 2.5 mm from the top, it is noted as the final setting time according to TS EN 480-2 standard.

3.2.6 Compressive Strength Test

The compressive strength of the mixtures has determined with 71 × 71 × 71 mm cubes. Specimens were kept in molds for the first 24 h in a room which has 20 °C temperature and 90% relative humidity. Then the specimens were demolded, and half of the specimens were cured in the lime saturated water at 20 °C and the other half of the specimens were cured climate cabinet which has in 20 °C temperature and 50% relative humidity. At the age of 28 days, all of the specimens were taken from the curing environments and dried in the oven at 40 °C for 3 days. This 3-day process was applied so that the relative humidity of the specimens due to their internal humidity does not make a difference in the compressive strength. The specimens were dried rather than saturated so that the SAP would not retain too much water and cause false results. It has also been stated by Esteves et al., (2007) that internal humidity can lead to different incorrect results in mixtures using SAP. After the curing periods were finished, specimens were loaded at a 2.40 kN/sec loading rate.

3.2.7 Flexural Strength Test

The flexural strength of the mixtures has determined with 25 × 50 × 305 mm plates. This specimen type has been used in previous SIFCON studies (Beglarigale et al., 2016; Yazıcı et al., 2006). Specimens were kept in molds for the first 24 h in a room which has 20 °C temperature and 90% relative humidity. Then the specimens were demolded, and half of the specimens were cured in the lime saturated water at 20 °C and the other half of the specimens were cured climate cabinet which has in 20 °C temperature and 50% relative humidity. At the age of 28 days, all of the specimens were taken from the curing environments and dried in the oven at 40 °C for 3 days. This 3-day process was the same as the compressive strength testing process. The samples were subjected to a three-point bending test (Figure 3.6). SIFCON slurry specimens and SIFCON specimens have loaded at 0.05 mm/min and 0.5 mm/min loading rates, respectively. The support span has been selected as 270 mm and the maximum displacement has been limited to 10 mm in SIFCON specimens. Toughness was calculated by the area under the load-displacement curve.

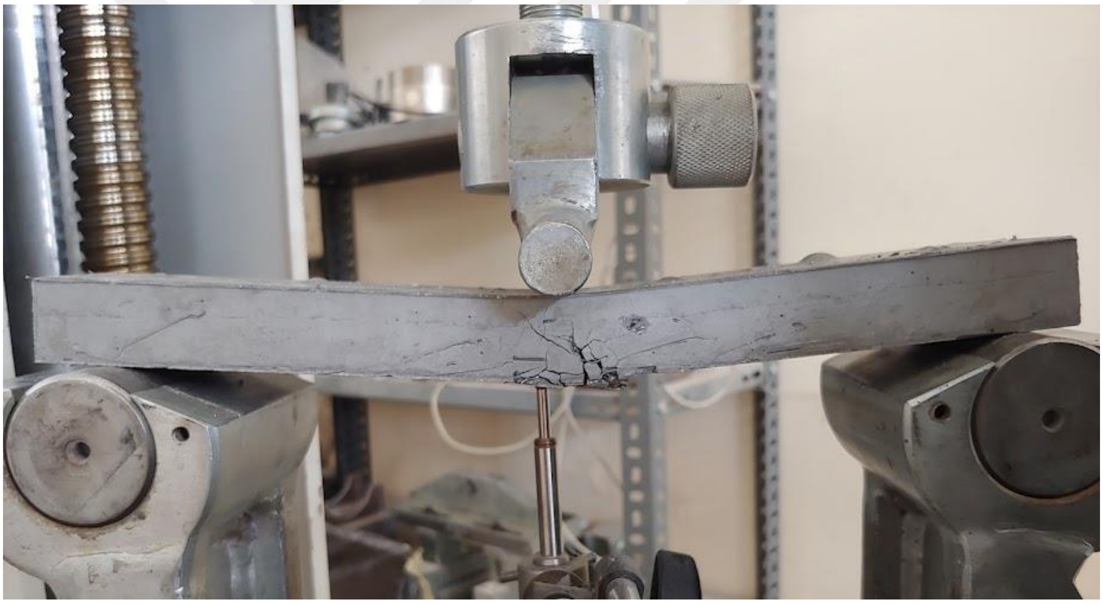

Figure 3.7 Three-point bending test setup (Personal archive, 2022)

3.2.8 Restrained Ring Test

Restrained ring test were conducted in accordance with ASTM C 1581 standard. The mold dimensions have compatible with the dimensions given in the relevant standard. The casted ring test specimens were kept at 20 °C and 95% relative humidity (sealed conditions) for 24h. Then outer rings were demolded, and specimens were subjected to 20 °C and 50% relative humidity until the age of cracking. Details of the ring test system are presented in Figure 3.6.

Figure 3.8 Restrained ring test system

3.2.8 Early Age Shrinkage Measurements

Shrinkage measurements were made with a non-contact laser system up to 7 days from the start of the setting time. Specimens with dimensions 25 x 25 x 280 mm have used for the shrinkage measurements. For autogenous shrinkage measurements, the shrinkage system was kept at 20 °C and 95% relative humidity. In addition to the ambient system conditions, specimens were also covered with parafilm to prevent any water evaporation. For the drying shrinkage measurements, the shrinkage system was kept at 20 °C and 50% relative humidity. The illustration of the non-contact laser system is presented in Figure 3.6.

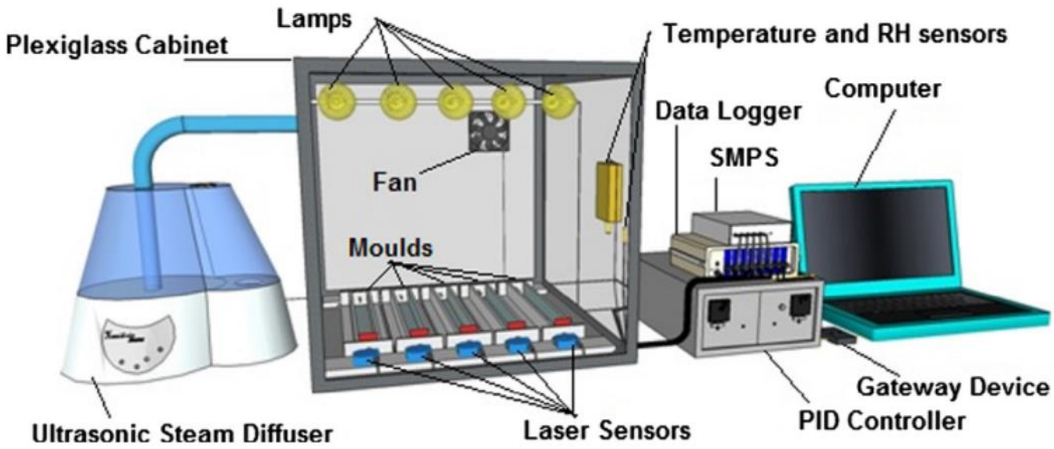

Figure 3.9 Shrinkage measurement system (Yalçınkaya & Yazıcı, 2017)

CHAPTER FOUR
RESULTS AND DISCUSSION

4.1 Teabag Method

Absorption results for SAP A and SAP B in deionized water are presented in Figure 4.1 and Figure 4.2, respectively. Absorption results for SAP A and SAP B in cement pore solution are presented in Figure 4.1 and Figure 4.2. The absorption capacity of SAP is dependent on the ionic composition of the solution. For example, while an SAP has absorbed deionized water 350 times of its mass, this rate has 50 times for concrete pore solution (Schröfl et al., 2012). As can be seen in the figures, while both SAPs absorb 250-300 times their own weight in deionized water, this rate is 30-40 times in cement pore water. The absorption kinetics of both SAP have dependent on the test solution. While both SAP continued to absorb in deionized water, desorption after a quick absorption has been observed in cement pore solution. Absorption kinetics in deionized water were agreed with the "interstitial water" theory (Omidian et al., 1999). Therefore, it could be inferenced that, the coarser SAP has more interstitial water than the finer SAP. On the other hand, absorption kinetics in cement pore solution was different than in deionized water. SAP A has reached its peak absorption capacity at 5 min, after that, desorption continued until the end of the test. SAP B was more constant than SAP A in the cement pore solution. Quick desorption kinetics in cement pore solution has shown in previous studies in SAP which has the same origin (acrylamide and acrylic acid) and similar crosslinking density (Schröfl et al., 2012). The authors stated that this type of quick desorption kinetics is not allowed to give an exact dosage of additional water. Since desorption of SAP A was stable in cement pore solution after 30 min, it could be classified as proper SAP for internal curing.

Accurate determination of the absorption capacity of SAP is vital since the w/c ratio could be affected. If a lower absorption capacity than true absorption capacity is used, the w/c ratio will decrease. Similarly, if a higher absorption capacity than the true absorption capacity is used, the w/c ratio will increase. In addition, SAP characteristics are also important for adequate internal curing. Mechtcherine et al., (2021), have emphasized that absorption and desorption properties of SAP should be examined prior to use since some SAP could not be served as an internal curing agent.

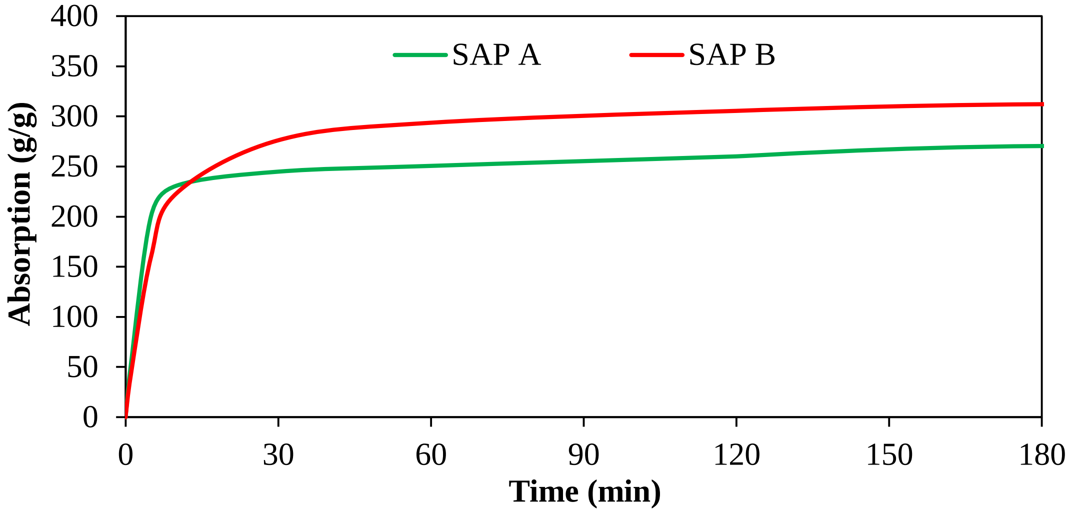

Figure 4.1 Time-dependent absorptions of SAP A and SAP B in deionized water up to 180 min.

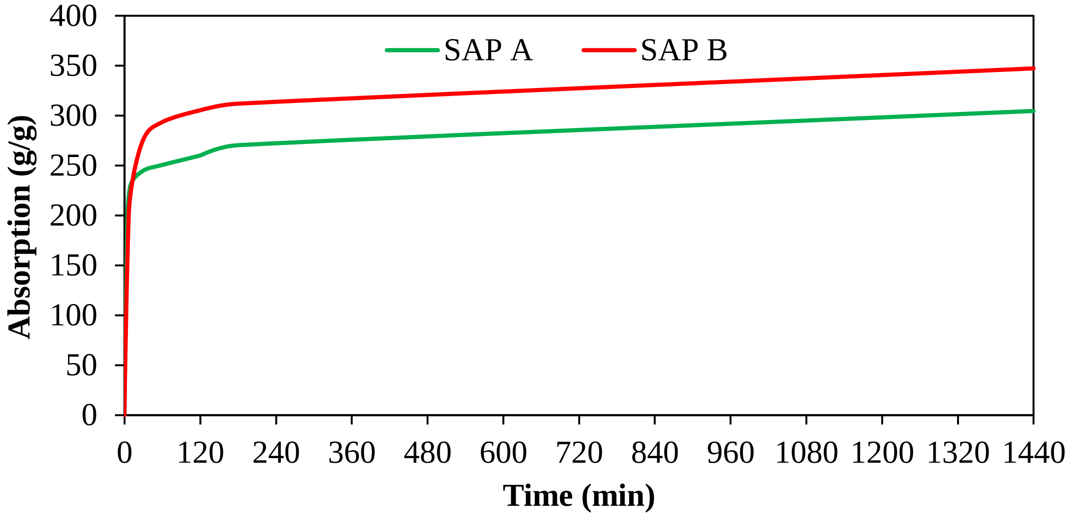

Figure 4.2 Time-dependent absorptions of SAP A and SAP B in deionized water up to 1 day.

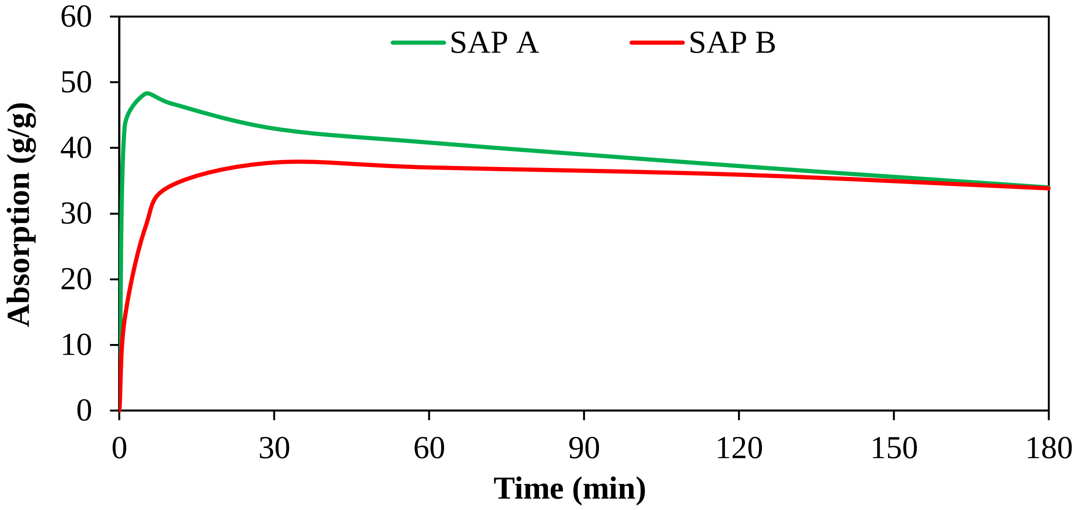

Figure 4.3 Time-dependent absorptions of SAP A and SAP B in cement pore solution up to 180 min

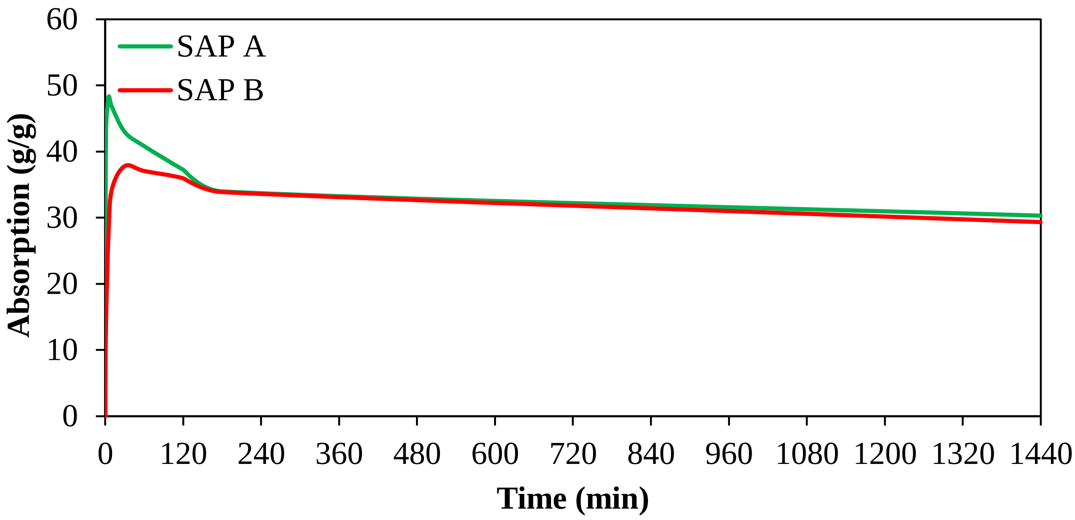

Figure 4.4 Time-dependent absorptions of SAP A and SAP B in cement pore solution up to 1 day

4.2 Slump Flow Method

According to the slump flow test, the absorption capacities of SAP A and SAP B were found to be 40 g/g and 33 g/g, respectively. The filler effect of SAP has not been taken into account in the slump flow test (Zhao et al., 2020). This could cause misleading results between finer and coarser SAP. On the other hand, it is advantageous that there will not be any compaction differences between mixtures prepared according to slump flow test results. Also, similar results have obtained at 30 min with slump flow test and teabag. It could be indicating that the neglected filler effect errors in the slump flow test and interstitial water errors in the teabag method were the same.

4.3 Free Flow and Mini V-Funnel Test

The mini v-funnel passing time and free flow diameters are presented in Figure 4.5. It can be said that all SAP-containing mixtures have similar v-funnel passing time except SAP A 36 mixture. It is known that the addition of SAP could increase (J. Liu, Khayat, & Shi, 2020; Venkateswarlu, Deo, & Murmu, 2021) or decrease (Azarijafari, Kazemian, Rahimi, & Yahia, 2016; Toledo Filho, Silva, Lopes, Mechtcherine, & Dudziak, 2012) the v-funnel times depending on the SAP size and origin. The free flow diameters of SAP-containing mixtures were slightly decreased compared to the reference mixture, but a similarity was observed among the mixtures containing SAP. In addition, the unstable water absorption behavior of SAP A (Figure 3.4) may have led to misleading results in the v-funnel test. Although the fresh state tests were carried out exactly 30 minutes after the cement and water meet, according to the teabag result of SAP A, there is very rapid water absorption and desorption before the 30th minute and slight desorption after the 30th minute. This behavior could differ in the mixture and instability may have been observed at the time of the v-funnel test. However, the fact that this behavior was not seen in the SAP A 60 mixture undermines all these theories. In addition, it has been stated that there is a non-linear relationship between the amount of SAP and viscosity (Geng, He, Chen, & Niu, 2022). For these reasons, the out-of-trend behavior in the SAP A 36 mixture should be examined with detailed rheological tests. However, it can be said that all mixtures have sufficient workability for a SIFCON mixture.

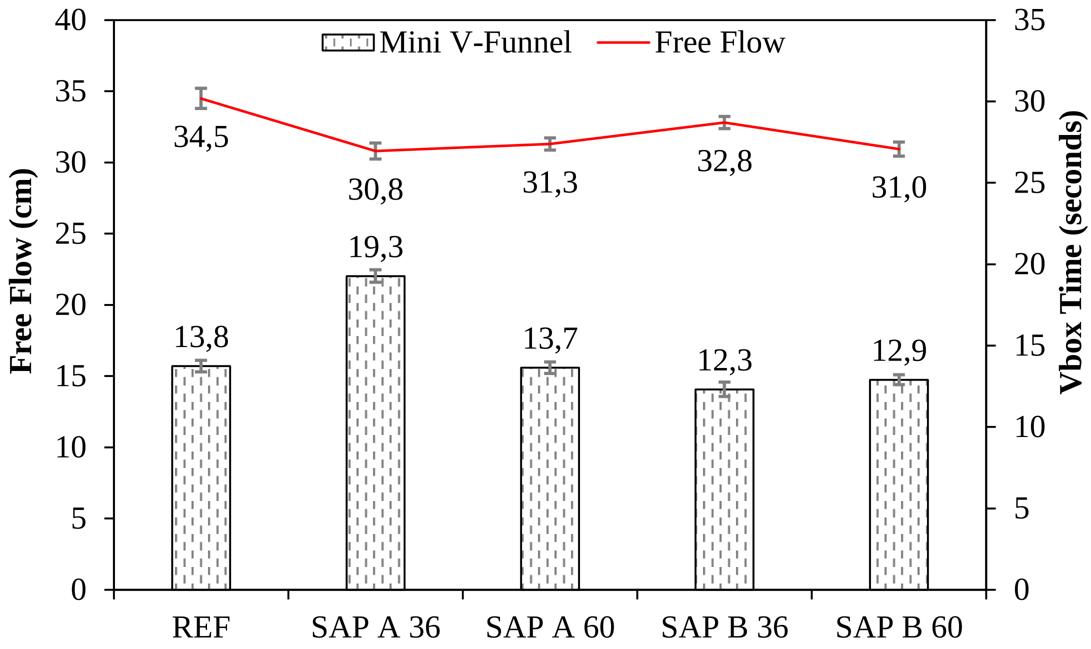

Figure 4.5 Mini v-funnel and free flow values of the mixtures

4.4 Setting Times

Initial and final setting times in autogenous conditions are presented in Figure 4.6 and the drying conditions are presented in Figure 4.7. While setting times were not affected by SAP modification under autogenous ambient conditions, SAP modification increased setting times significantly under drying conditions.

In terms of autogenous conditions, the change of setting times is directly related to the ion concentration. As the w/c ratio decreases, the amount of water per cement will increase, so concrete with a lower w/c concentration with a higher concentration of ions will hydrate faster and therefore set faster (Ge, Wang, Sandberg, & Ruiz, 2009). In theory, if the water absorption ratio is correct, it is assumed that the addition of SAP does not change the effective w/c ratio in the fresh state. Considering that the w/c ratio and the setting time are directly related, it can be said that the setting times in autogenous conditions indicate that the water absorption rates found by the slump flow method are correct. Similarly, Castro, Varga, & Weiss (2012), stated that if the water absorption value of the lightweight aggregate is used correctly, the hydration heats of the reference and the lightweight aggregate mixtures will be similar. Although there are many factors affecting setting times, it can be said that setting times can be used as an auxiliary test method to control whether the water absorption value of SAP was correct or not.

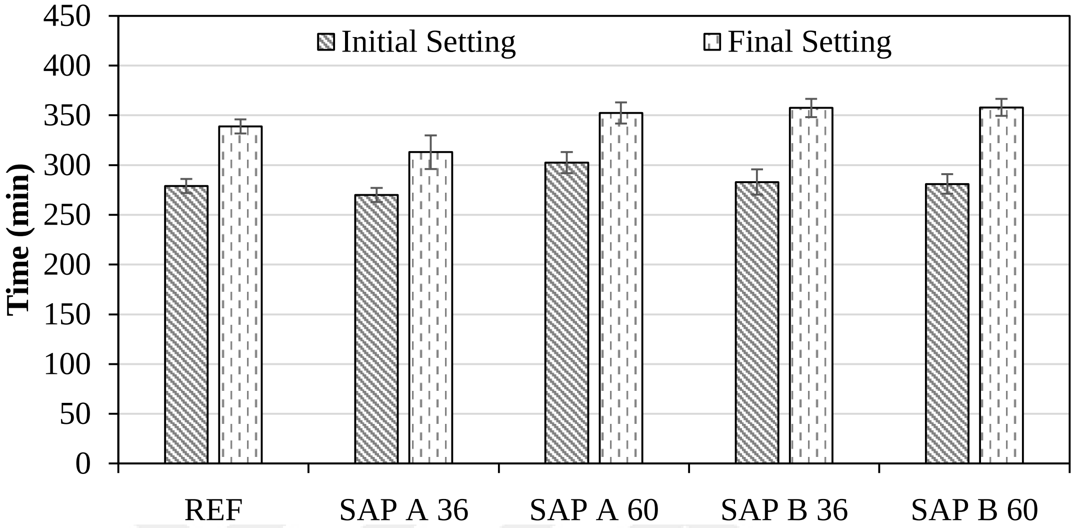

Figure 4.6 Initial and final setting times of the mixtures under autogenous conditions

If the relative humidity of the environment is low, the relative humidity in the concrete will try to decrease until it equalizes with the ambient conditions. Ahmadi, (2000) stated that with the decrease in the relative humidity of the environment, there was a significant decrease in the setting times. The decrease in the relative humidity in the concrete occurs with the evaporation of the water that has not yet reacted. In this case, the w/c ratio will decrease due to the loss of water. As mentioned before, it is known that with the decrease of the w/c ratio, the setting times are shortened. The decrease in the setting time of the reference mixture under drying conditions can be explained by the decrease in the w/c ratio due to water loss. On the other hand, it is seen that the mixtures containing SAP have prolonged setting times in the drying conditions (Figure 4.7). In fact, it is seen that the setting times of some mixtures in the drying conditions are longer than the setting times in the autogenous conditions. This may be because SAP gives water to the environment as the environment dries since SAP works with osmotic pressure. Thus, specimens in the drying conditions have a higher water content than the autogenous conditions. It has been reported that SAP can keep the internal relative humidity of concrete high even under drying conditions (Shen, Wang, Chen, Wang, & Jiang, 2015). The setting times of the mixtures containing SAP are not shortened in drying conditions, this can be explained by the fact that their high internal relative humidity even under drying conditions.

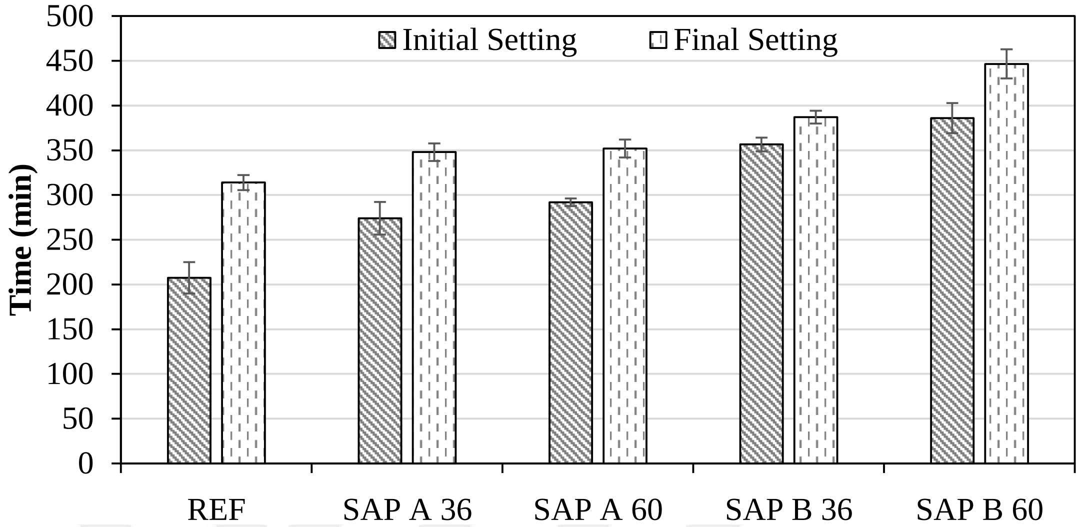

Figure 4.7 Initial and final setting times of the mixtures under drying conditions

4.5 Compressive Strength Test

The compressive strengths of SIFCON slurry mixtures are presented in Figure 4.8, and the compressive strengths of SIFCON mixtures are presented in Figure 4.9. For both SAP, a decrease in compressive strength was observed with the increase in the SAP ratio. The reference mixture was more affected and lost more strength due to the harsh curing conditions (50% RH curing) than the mixtures containing SAP. In SIFCON slurry mixtures, the strength losses of REF, SAP A 36, SAP B 60, SAP B 36 and SAP B 60 mixtures at 50% RH cure are 4%, 1.1%, 2.3%, -2.9% and 1.4%, respectively. The same values are 3.1%, 2.9%, 2.2%, 1.9% and 1.4% for SIFCON mixtures, respectively. Esteves et al., (2007) stated that SAP-containing mixtures are not affected by external relative humidity drops due to SAP's ability to keep internal relative humidity high, therefore, SAP-free mixtures experience more severe strength loss in low relative humidity environments. Mixtures containing SAP kept their internal humidity high and prevented water loss. Therefore, strength losses due to the water loss in 50% RH curing are mitigated. Moreover, SAP contributed to strength with additional hydration. However, due to the voids created by SAP, mixtures containing SAP have lost their strength. As a result, it can be said that the negative effect of the voids created by SAP outweighs the positive effects such as reduction in water loss and additional hydration. However, since the primary use of SAP is to reduce shrinkage, the strength losses are at a compensable level. However, regardless of shrinkage, it can be said that SAP has the ability to reduce the curing needing in field conditions.

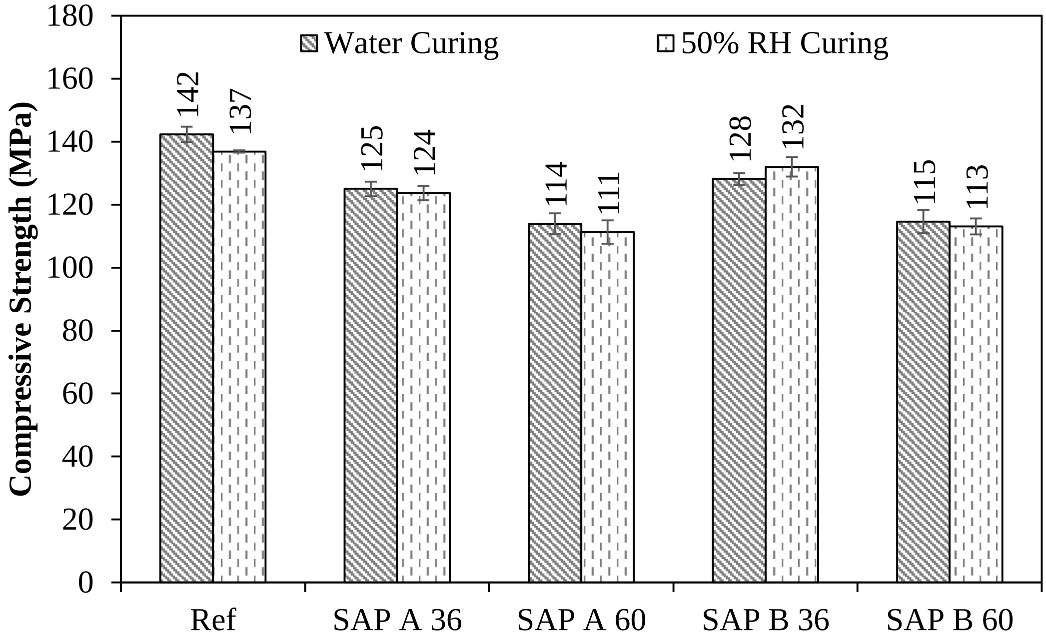

Figure 4.8 Compressive strengths of SIFCON slurry mixtures

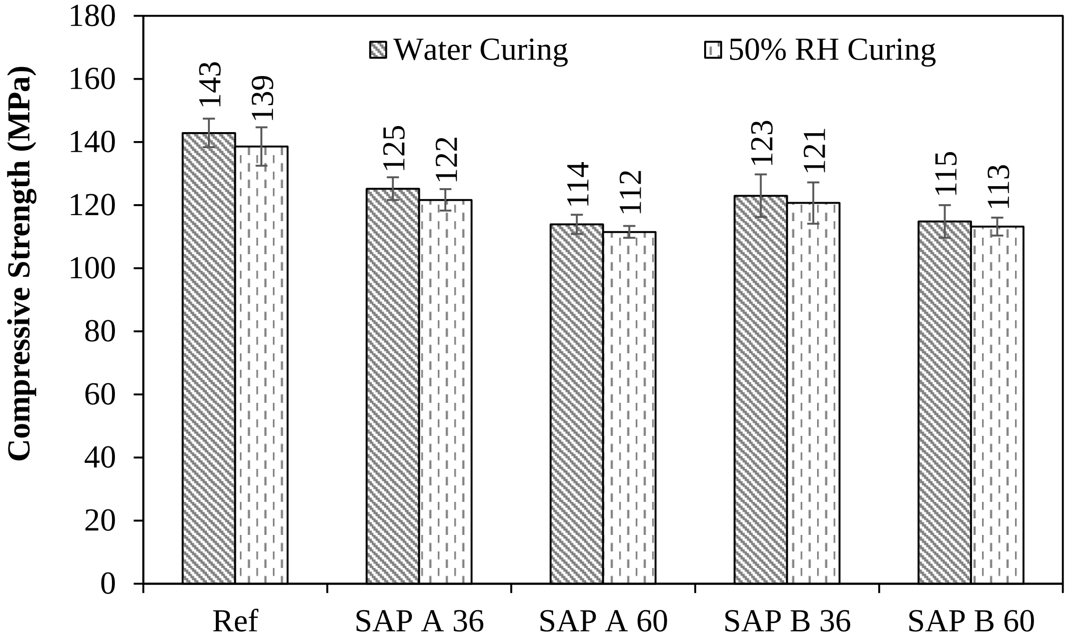

Figure 4.9 Compressive strengths of SIFCON mixtures

The effect of fiber usage on compressive strength in water curing and 50% RH curing are presented in Figure 4.10 and Figure 4.11, respectively. It can be said that the use of fiber has almost no effect on the compressive strength in both curing conditions. There is a lack of information in the literature about the relationship between fiber orientation and compressive strength. Yazıcı et al. (2010) obtained a higher compressive strength by using randomly distributed fibers than the oriented and fibreless specimens. The authors have explained the losses in compressive strength in case of parallel or perpendicular fiber orientation by anisotropy. On the other hand, it has been reported that there is no change in compressive strength in the use of random fiber compared to the fibreless samples (Sengul, 2018). It can be said that fiber orientation and compressive strength relationship depend on many parameters such as matrix strength, fiber geometry, and even specimen dimensions (Riedel & Leutbecher, 2021).

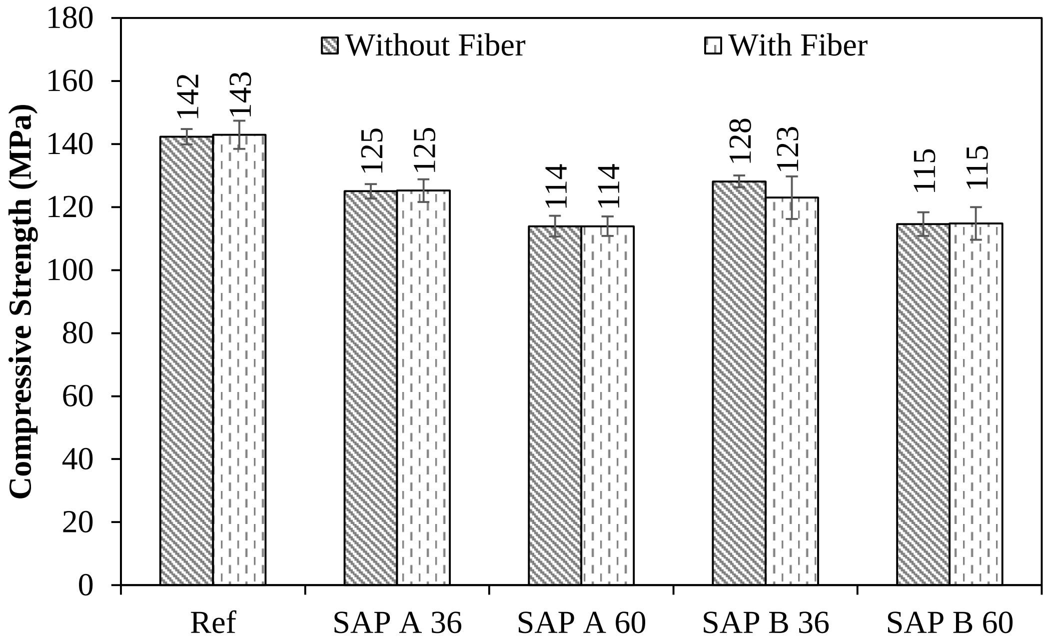

Figure 4.10 Compressive strengths of water-cured specimens

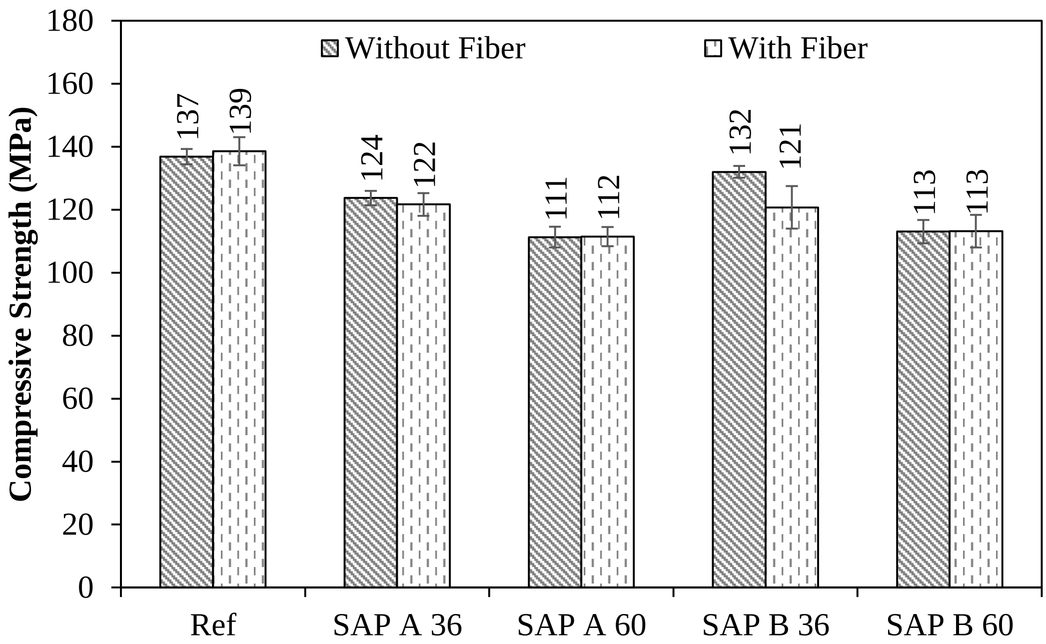

Figure 4.11 Compressive strengths of 50% RH cured specimens

4.6 Flexural Strength Test

Flexural strength of water and 50% RH cured SIFCON slurry specimens are presented in Figure 4.12. Contrary to compressive strength, flexural strength increased with increasing SAP ratios. Due to the very high compressive strength of the specimens, the effect of additional hydration provided by SAP may have been much lower than the effect of additional voids created by SAP. On the other hand, it is possible that an increase was observed due to the additional hydration provided by SAP since the flexural strengths are not very high.

Similarly, the lower strength of the water-cured specimens can be explained by hydration. While SAP acts only as a void in the water curing, it also has an auxiliary effect on hydration in the 50% RH curing. However, the reason why the strength of the reference specimen is lower in water curing may be different. This may indicate that the additional drying time of 3 days was not enough to remove all the water in the specimens.

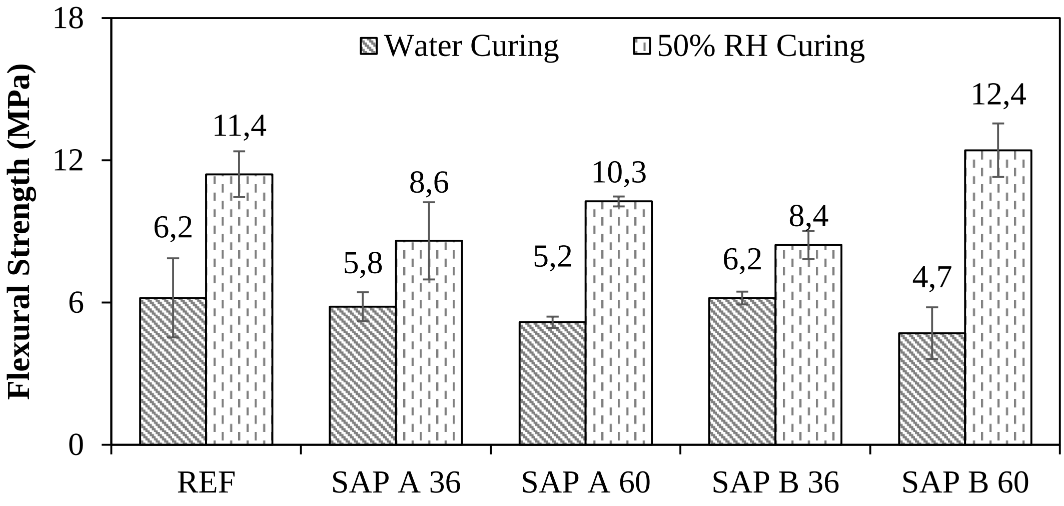

Figure 4.12 Flexural strengths of SIFCON slurry specimens

The toughness values of water and 50% RH cured plain specimens are presented in Figure 4.13. The toughness values have quite parallel with the flexural strength values.

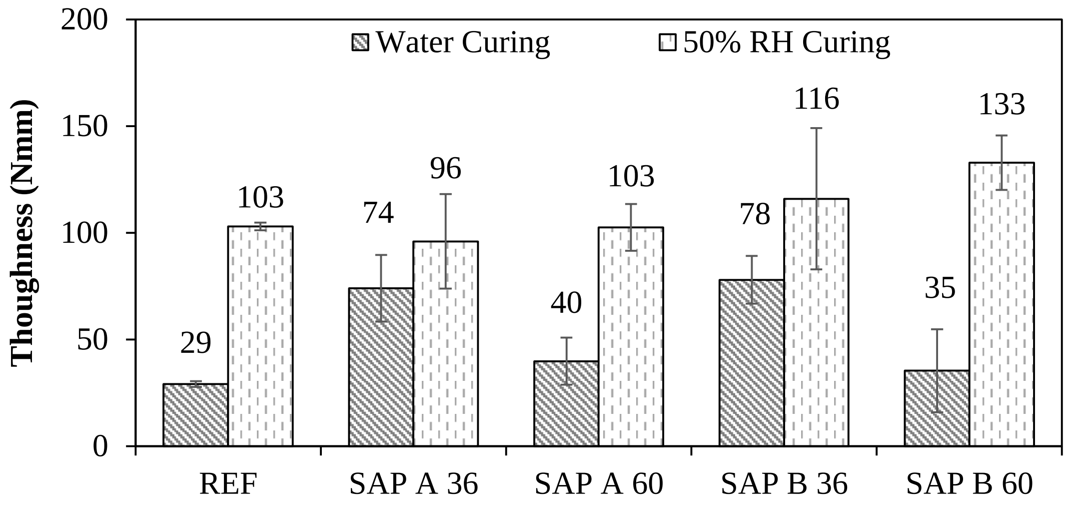

Figure 4.13 Toughness values of SIFCON slurry specimens

Load-displacement curves of the water-cured SIFCON specimens are presented in Figure 4.14. Contrary to the compressive strength and SIFCON slurry flexural strength values, it can be said that the mixtures containing SAP B performed quite poorly. It is possible that since the SAP B is coarser and expanded due to swelling in the mixture, some particles may have not penetrated between the fibers. In this case, the extra voids may have caused a loss in flexural strength.

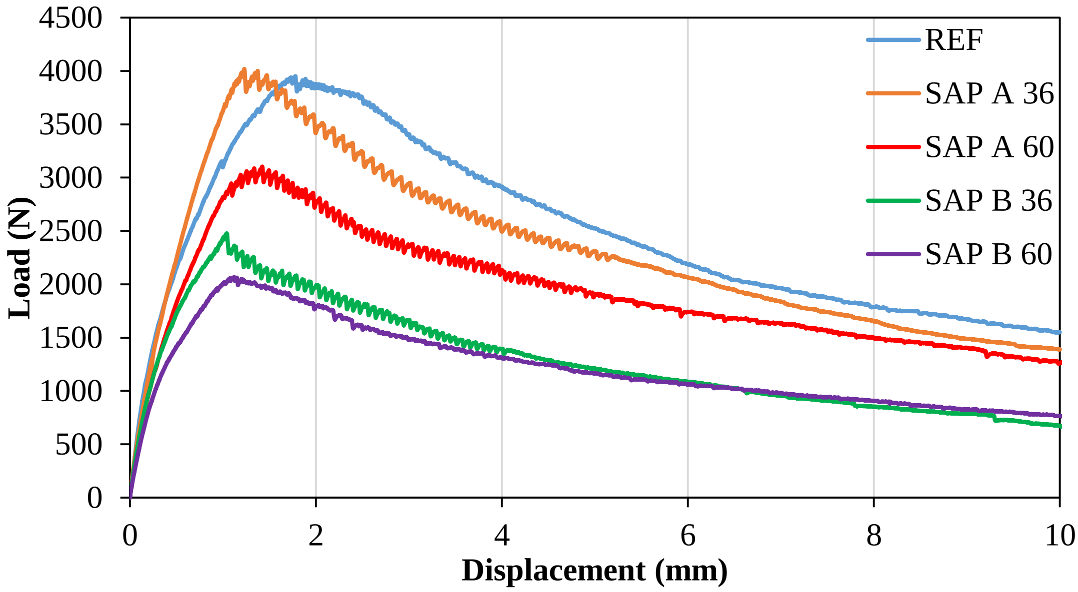

Figure 4.14 Load-displacement curves of water-cured SIFCON specimens

The flexural strength values which have been obtained with the value at the peak point of the stress-strain curves are presented in Figure 4.15, and the toughness values which have been calculated with the area under these curves are presented in Figure 4.16. The flexural strength and toughness values are decreased with the increase in the SAP ratio. The SAP A 36 mixture was slightly exceeded the reference mixture. As mentioned before, SAP B mixtures were performed quite poorly as they could not pass through the fibers. The fact that the better performance of the SAP A 36 mixture can be explained by the increased fiber-matrix adherence due to hydration. However, considering that these mixtures were in the water, it is theoretically expected that the entire specimen will be saturated with water and provide the necessary water for hydration. Thus, there will be no positive effect of internal curing. It is possible that the entire specimen is not saturated with water due to the low permeability of the specimens. On the other hand, it is also possible that the small-sized SAP A filled the fiber-matrix interface as a filler.

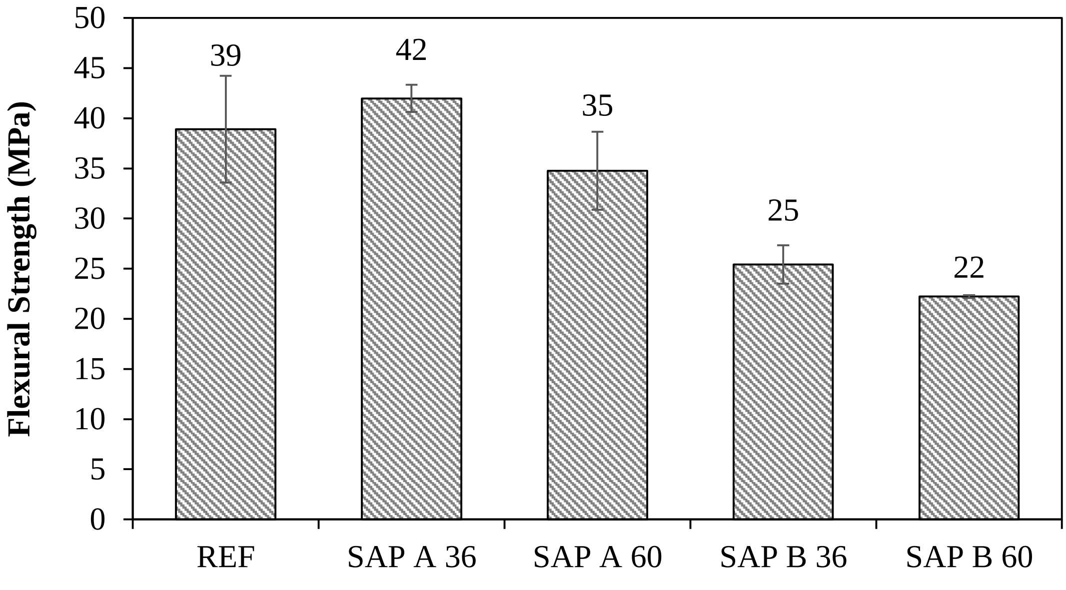

Figure 4.15 Flexural strengths of water-cured SIFCON specimens

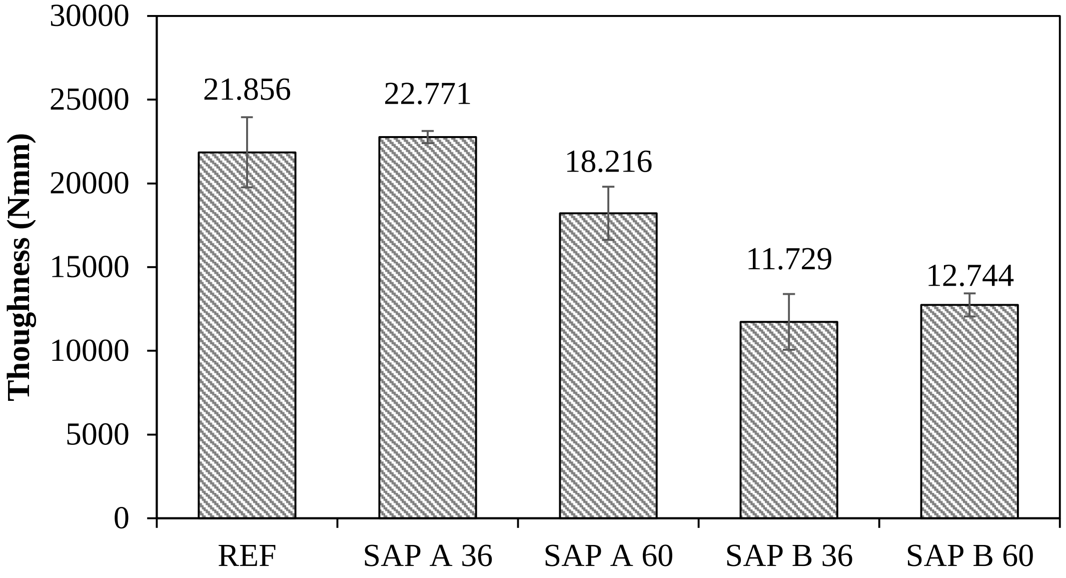

Figure 4.16 Toughness values of water-cured SIFCON specimens

Load-displacement curves of the 50% RH cured SIFCON specimens are presented in Figure 4.17. The performances of the 50% RH-cured specimens are quite similar to the water-cured specimens. The strength was slightly decreased in the 50% RH cured specimens.

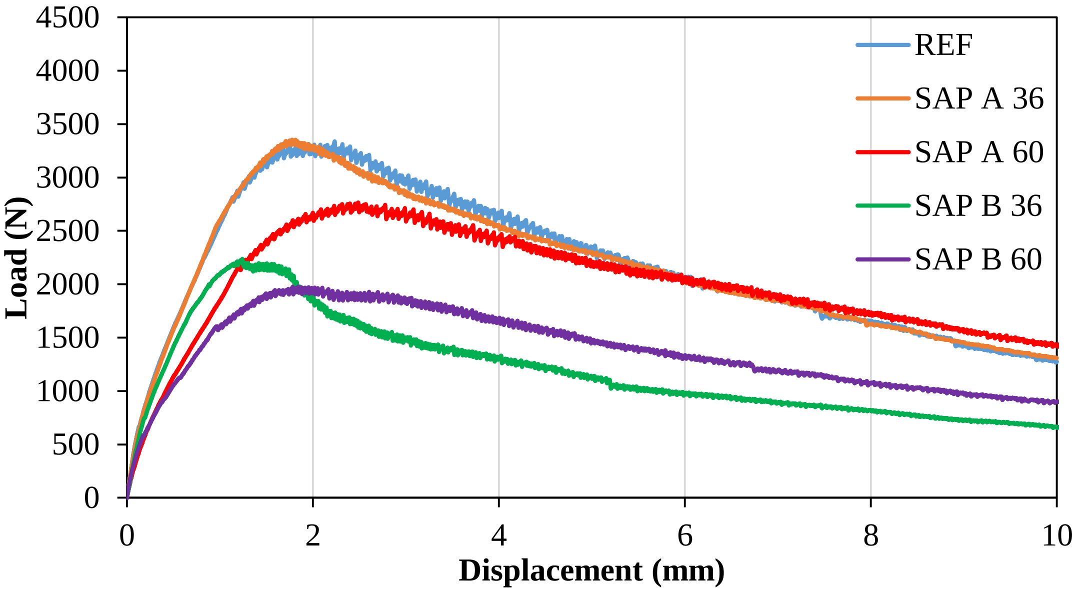

Figure 4.17 Load-displacement curves of 50% RH cured SIFCON specimens

The flexural strengths of the 50% RH cured specimens are presented in Figure 4.18, and the toughness values are presented in Figure 4.19. Again, behaviors were quite similar to the water-cured specimens.

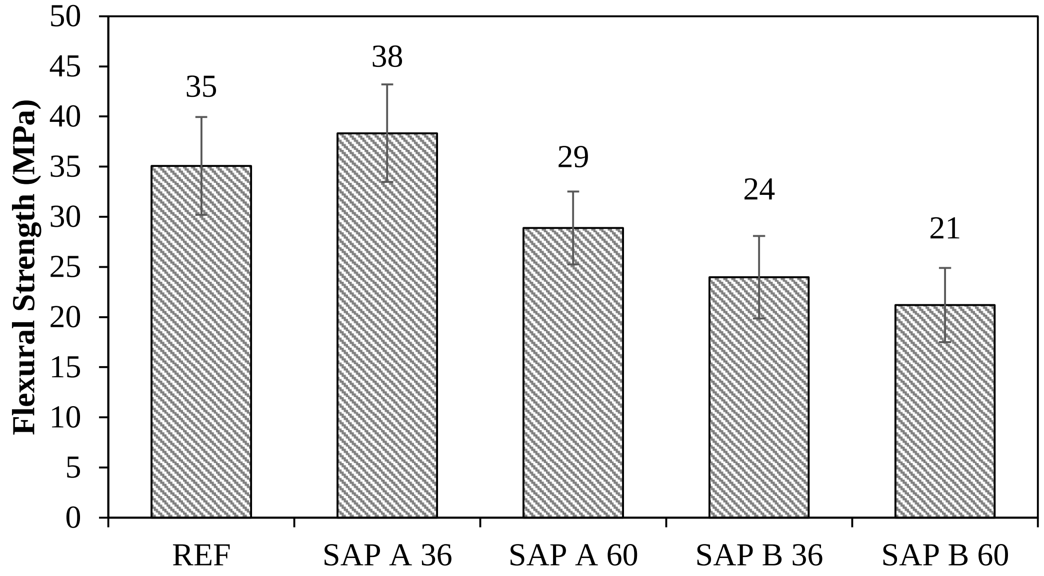

Figure 4.18 Flexural strengths of 50% RH cured SIFCON specimens

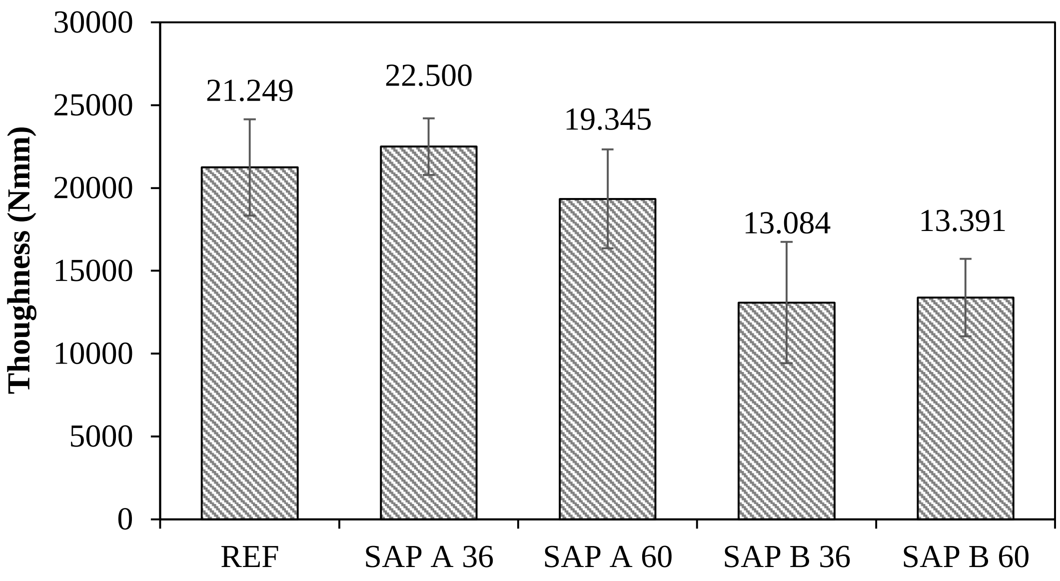

Figure 4.19 Toughness values of 50% RH cured SIFCON specimens

The comparison of flexural strength and toughness values are presented in Figure 4.20 and Figure 4.21, respectively. Since the use of SAP is expected to reduce shrinkage as well as reduce the need for curing in field conditions, SAP-containing mixtures are expected to be less affected by 50% RH curing than reference mixtures. All the SAP-containing mixtures have less affected by the 50% RH curing. REF, SAP A 36, SAP A 60, SAP B 36 and SAP B 60 mixtures have lost 10.9%, 9.5%, 20.3%, 6.0% and 4.8% strength in 50% RH curing compared to water curing, respectively. These values were 2.9%, 1.2%, -5.8%, -10.4%, -4.8% for toughness.

It is seen that the mixtures containing SAP A are affected by the 50% RH curing as much as the reference mixture, and even a serious loss of strength is experienced with the increase in SAP A dosage. Mixtures containing SAP B were less affected by 50% RH curing. Even though SAP B was more effective in 50% RH curing, the flexural strength values have not promising. Some of the parameters affecting the compressive strength and flexural strength are different. The only major difference between compressive strength and flexural strength is the fibers' role. Fibers play a vital role in flexural strength. Therefore, it indicates that the changes in the fiber-matrix interface are very important. SAP may have affected the interfacial transition zone between fiber and matrix in two ways; i) higher hydration degree due to the additional available water and ii) less shrinkage due to the shrinkage-reducing effect of SAP.

In the case of a higher hydration degree, the fiber bond must be improved. It is well known that the degree of hydration increases with the use of SAP (Esteves, Lukošiute, & Česniene, 2014; Justs et al., 2014). It is also known that the bond between fiber and matrix could increase with the increased hydration degree (Feng, Yang, & Qian, 2021; J. Liu, Farzadnia, & Shi, 2019). Moreover, Liu et al. (2019) have examined the pull-out behavior of SAP-modified mixtures. The authors have stated that additional hydration and reduced micro-cracking due to the SAP increased the bond between fiber and matrix.

In case of less shrinkage, the fiber bond must be worsened. It is known that reductions in shrinkage affect the fiber bond negatively due to the decreased clamping pressures (Stang, 1996). Yet, previous studies have only focused on the effect of SRA on fiber bond (S. H. Park, Ryu, Koh, & Kim, 2014; Stang, 1996). SRA is a new generation additive that simply reduces the surface tension of the pore water and prevents slower/less separation of the water from the capillary pores in the event of evaporation and the capillary pores coming closer to each other due to this separation and thus, forming the shrinkage. Since SRA is directly related to the surface tension and capillary pressure, the theory of increased bond with increased shrinkage may not only be related to shrinkage. The fiber bond and shrinkage relationship may have been affected by capillary pressure changes due to the usage of SRA. Any study has not yet been conducted to examine the relationship between fiber bond and internal curing agents in this respect.

Generally, specimens under bending reach their peak load shortly after the first crack. This is usually related to the strength of the matrix rather than the fibers. When peak load is reached, most fibers have not been activated yet. On the other hand, the more the fibers resist peeling, the longer the load on the sample will be able to remain without falling. This leads to an increased toughness. For these reasons, it is healthier to evaluate the fiber-matrix adhesion with the toughness data below this curve instead of the flexural strength, which is the peak point of the load-displacement curve.

Samples in the water cure can be assumed to be in autogenous conditions, while samples in the 50% RH cure can be assumed to be in drying shrinkage conditions. As it is known and will be seen in the following sections, shrinkage in drying conditions is much higher than shrinkage in autogenous conditions. Therefore, shrinkage reductions with the use of SAP are also much greater under drying conditions. In water-cured samples, the effect of fiber compression will make little difference between the mixtures. At the same time, considering that the samples in the water cure are completely saturated with water, it is doubtful whether additional hydration will occur with the use of SAP. In this case, it is difficult to distinguish between the above two theories and to say that they were valid in water-cured samples.

It is thought that both theories will be more effective in the 50% RH curing than water curing. In other words, with the use of SAP, shrinkage will decrease (the fibers will be less compressed) and hydration degree will increase in 50% RH curing. It could be seen from the toughness results that higher results are obtained with SAP containing 50% RH cured specimens than the water cured specimens (Figure 4.21). Thus, since the development is in a positive direction, namely, the toughness increases, it could be said that the effect of hydration was more dominant than the compaction effect. In addition, the voids formed by SAP under loading may have contributed to the toughness by extending the crack path or increasing the number of multiple cracks. Additional experiments such as pull-out, degree of hydration and SEM analyses are needed to separate all parameters from each other.

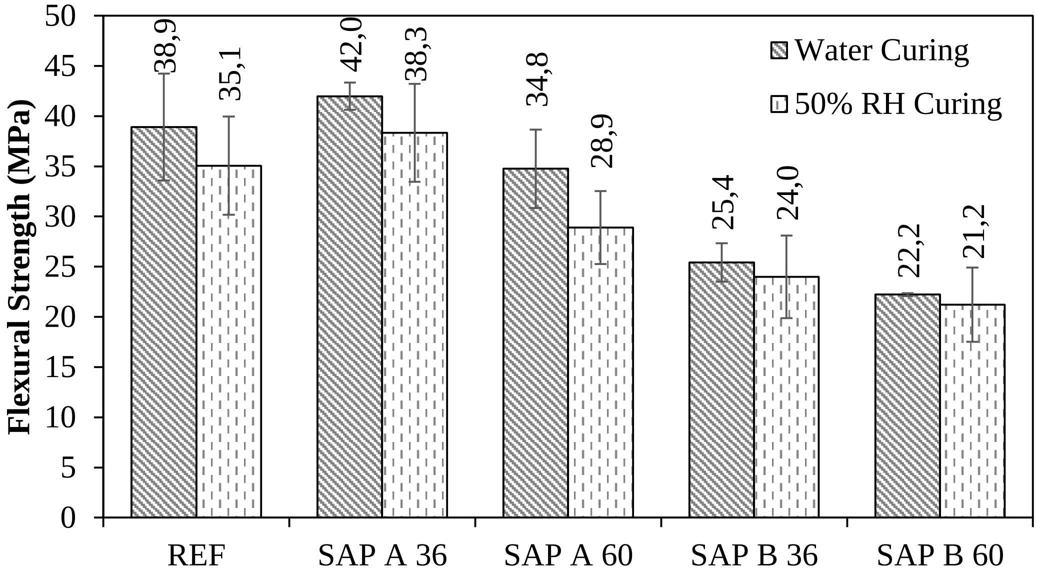

Figure 4.20 Flexural strengths of water and 50% RH cured SIFCON specimens

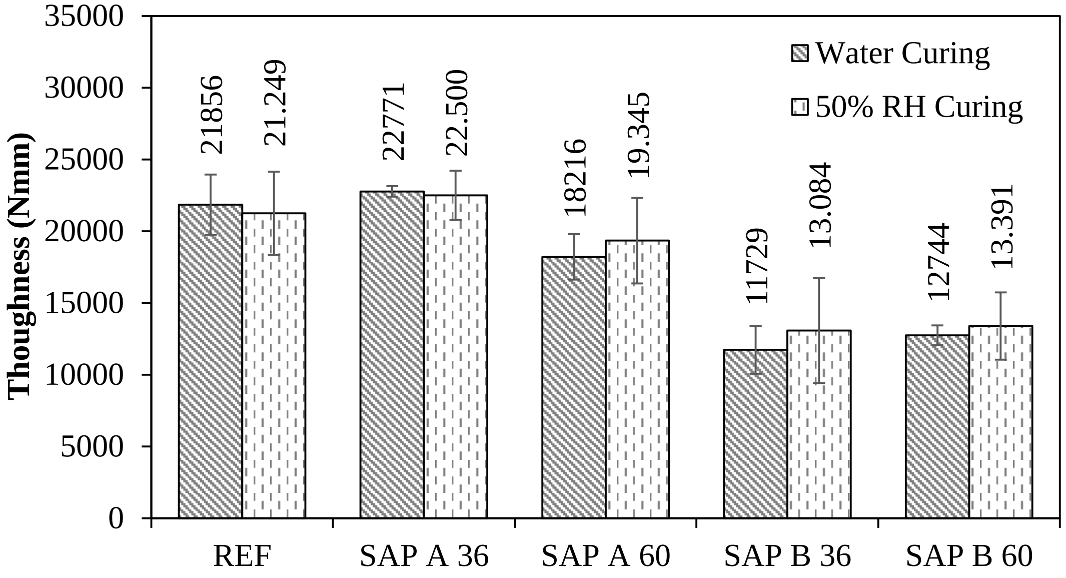

Figure 4.21 Toughness values of water and 50% RH cured SIFCON specimens

4.7 Restrained Ring Test

Steel strain values after the initiation of the drying of the SIFCON slurry mixtures are presented in Figure 4.22. It can be seen that higher SAP ratios were more effective in delaying the cracking time, as expected. The first crack times of REF, SAP A 36, SAP A 60, SAP B 36 and SAP B 60 mixtures are 19, 20, 30, 24, and 52 hours, respectively. Furthermore, SAP B has shown better performance than SAP A. While all mixtures except SAP B 60 mixture cracked at similar strain values, SAP B 60 mixture cracked at a higher strain value. In many studies in the literature, SAP has been used successfully to increase the first crack time. In these studies, the crack time of the mixtures containing SAP was delayed or not cracked at all (Fan, Shen, Guo, Zhao, Yang, & Wang, 2020; Lefever et al., 2020). The results of this study also show parallelism with the literature in terms of improvement in first cracking time with the usage of SAP. However, considering that the SIFCON slurry reference mixture was cracked in 19 hours and this time can be increased up to 52 hours with the use of SAP, all mixtures have in the high potential cracking class according to ASTM C 1581 standard. Thus, it can be said that SIFCON slurry exhibits a very serious shrinkage and is under the threat of shrinkage cracks.

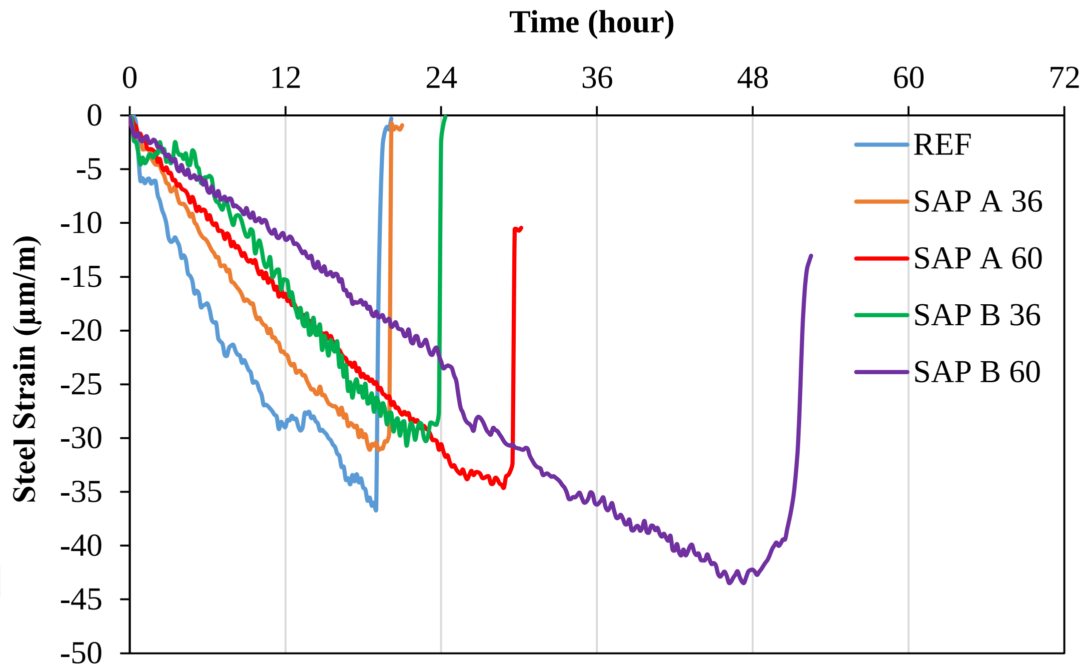

Figure 4.22 Steel strain and cracking time of the SIFCON slurry mixtures

4.7 Early Age Shrinkage

Autogenous shrinkage results of the SIFCON slurry mixtures are presented in Figure 4.23. It is seen that all mixtures showed very high shrinkage in the first few hours. After the first few hours, a little expansion has been seen. This could be explained by the crystallization pressure of the calcium hydroxide or the re-absorption of the bleeding water (Sant, Lothenbach, Juilland, Saout, Weiss, & Scrivener, 2011). As expected, with the increase of the SAP ratio, a decrease in autogenous shrinkage has also observed. While the shrinkage strains of the mixtures with 60 kg/m^3 additional water were almost constant after 1 day, mixtures with 36 kg/m^3 additional water has continued to shrink. It can be seen that the 36 kg/m^3 additional internal curing which has taken into account according to the Equation 1 and Equation 2 was not enough to mitigate the autogenous shrinkage. On the other hand, more sufficient mitigation in autogenous shrinkage has obtained with 60 kg/m^3 additional internal curing water. In these equations, chemical shrinkage constant has calculated with the cement dosage for this study. However, Bentz (2007) stated that the pozzolanic materials should be also taken into account in to the calculation of the chemical shrinkage constant. Yet, the pozzolanic materials could not be added to the cement dosage, they should be tested prior to using and a specific chemical shrinkage constant should be found. Thus, it should be noted that 36 kg/m^3 additional water was not accurate since the reactions was not enough and silica fume was not taken into account.

There is no limit value for shrinkage. The point where shrinkage is dangerous is the point where the shrinkage stresses exceed the tensile strength (Gribniak, Kaklauskas, Kliukas, & Jakubovskis, 2013). For this reason, since the strength development and shrinkage development for each concrete will be different, a critical shrinkage value cannot be mentioned for a specific concrete. However, with the use of SAP, it can be said that autogenous shrinkage is effectively reduced with both SAP types and both additional internal curing water dosages.

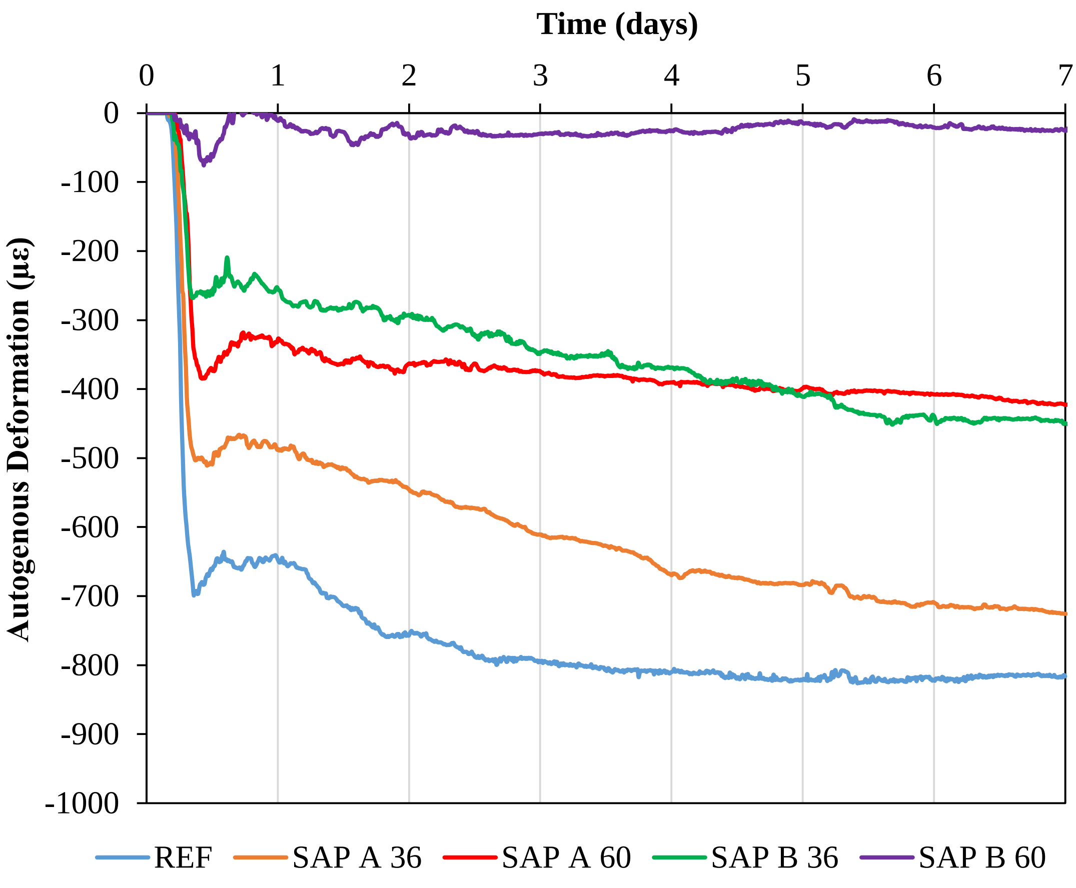

Figure 4.23 Autogenous shrinkage of the SIFCON slurry mixtures

The shrinkage results of the SIFCON slurry mixtures under drying conditions (50% RH and 20 °C) are presented in Figure 4.24. Results are quite agreed with the restrained ring test results. As mentioned earlier, there is not a general agreement on the behavior of the internally cured mixtures under drying conditions. While some researchers have reported an increase in shrinkage under drying conditions (Mechtcherine et al., 2008), contrary results have also been reported (Mönnig, 2006; Yang et al., 2019). In this study, it is clear that SAP decreased the shrinkage under drying conditions at an early age. However, according to these data, it cannot be clearly said that the use of SAP reduces the "drying shrinkage", it can be said that it reduces the shrinkage under "drying conditions". This could be caused by the decrease of plastic shrinkage with the use of SAP in the first few hours. SAP has been used to mitigate plastic shrinkage and plastic shrinkage cracks in many studies (Boshoff et al., 2020; Snoeck, Moerkerke, Mignon, & De Belie, 2020).

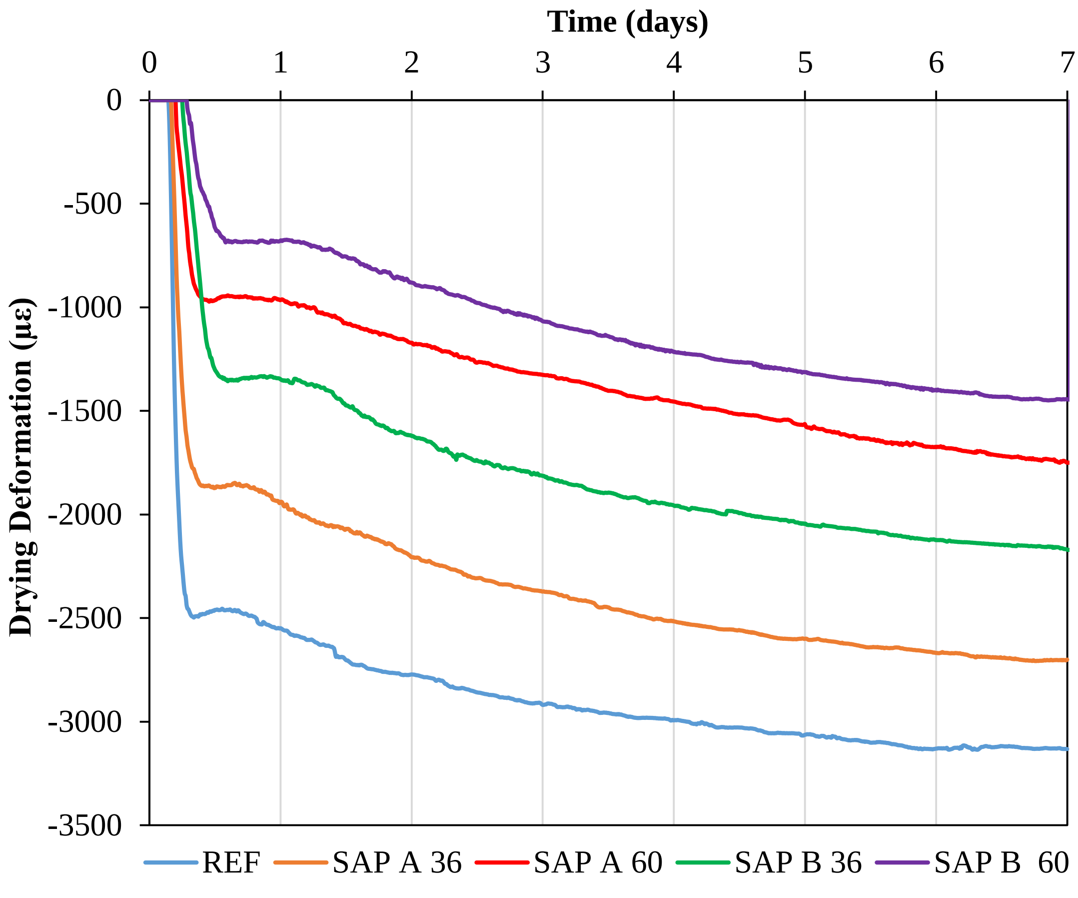

Figure 4.24 Shrinkage under drying conditions of the SIFCON slurry mixtures

Plastic shrinkage is a type of shrinkage that occurs in the plastic state of concrete. When the evaporation rate exceeds the bleeding rate, water evaporates from the capillary spaces and the capillary spaces shrink into each other, thus, plastic shrinkage occurs (Lerch, 1957). It is known that plastic shrinkage begins within the first few hours and continues until the final setting (Kovler & Zhutovsky, 2006). Thus, roughly, the time between the initial to final setting can be named the plastic shrinkage stage. In Figure 4.25, shrinkage values from initial to final setting and final to day 7, under drying conditions are presented.

It can be seen that SAP is very effective in reducing the shrinkage from initial to final setting times. From the final setting to day 7, the REF mixture has showed the least shrinkage. This finding partly supports the theory that an increase in shrinkage in drying shrinkage is due to the internal curing with SAP. This phenomenon can be explained with a couple of reasons; i) postponed capillary pressure breakthrough due to the more available water (Snoeck et al., 2020), and ii) less bleeding due to bleeding water bound by SAP. Also, Schröfl, Erk, Siriwatwechakul, Wyrzykowski, & Snoeck (2022), stated that the absorption and desorption kinetics of SAP are vital for plastic shrinkage mitigation since plastic shrinkage occurs in a short time window. Moreover, the shrinkage between the initial and final setting times in the REF mixture was greater than the shrinkage up to the final and up to the 7th day (Figure 4.25). On the other hand, the shrinkage between the initial and final setting times in the SAP containing mixtures was less than the shrinkage up to the final and up to the 7th day.

However, it should be noted that the shrinkage values presented in Figure 4.24 include plastic, drying, and autogenous shrinkage values together. It is very difficult to determine the types of shrinkage, which one is effective, and how much. However, it can be said that SAP reduces shrinkage since it is very effective from the initial setting until the final setting. Although additional tests such as capillary pressure and evaporation rate are required here to determine the plastic shrinkage, it can be said that SAP also reduced plastic shrinkage and is therefore the total shrinkage and cracking potential.

According to the above theory, it could be seeming like SAP is mostly effective in the first few hours. Thence, it can be concluded that preventing the water loss of SIFCON on the first day with traditional methods can contribute to the shrinkage problem as much as SAP. Yet, according to the ring test results in Figure 4.22, it can be seen that SAP delayed the cracking time in the samples which have kept in the autogenous environment on the first day, then drying conditions until the cracking. This contrary result can be explained by the fact that the stresses caused by the reduced autogenous shrinkage due to SAP on the first day of the ring test are reduced and this effect is effective in drying conditions. As a result, it can be said that SAP is very effective in the first few hours under drying conditions, but it is also effective in reducing shrinkage in the following days, although not as much as in the first few hours. In addition, it is seen that the measurement system has great importance when evaluating such results. If measurements had taken from the end of the setting or day one, it would be seen that the SAP-containing mixtures showed higher shrinkage than the REF mixture.

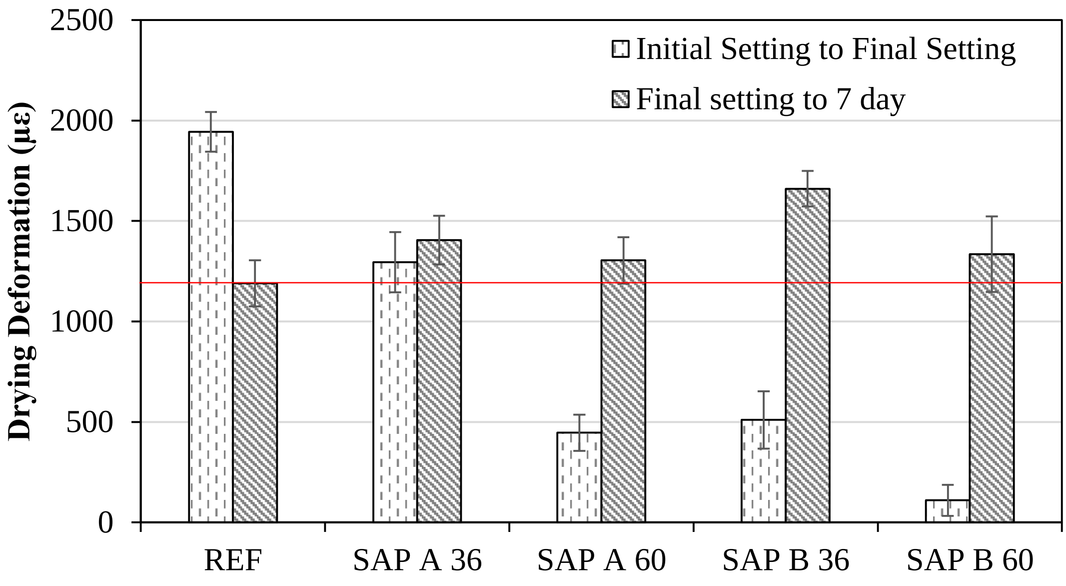

Figure 4.25 Change in drying deformation from 1 to 7 days

The shrinkage values under autogenous conditions can theoretically be subtracted from shrinkage under drying conditions and, thus, "drying shrinkage" values could be obtained (Yalçınkaya, 2015). However, as discussed earlier, plastic shrinkage under drying condition is also considered to be effective. The theoretically obtained drying shrinkage values are presented in Figure 4.26. It can be said that the order of reduction in shrinkage is the same except for the SAP B 60 mixture. Since the SAP B 60 mixture prevents almost all shrinkage in autogenous conditions, the total shrinkage value of SAP B 60 mixture, in theory, actually represents drying shrinkage. This disruption in order can be interpreted as the out-of-trend behavior of the SAP B 60 mixture in autogenous shrinkage.

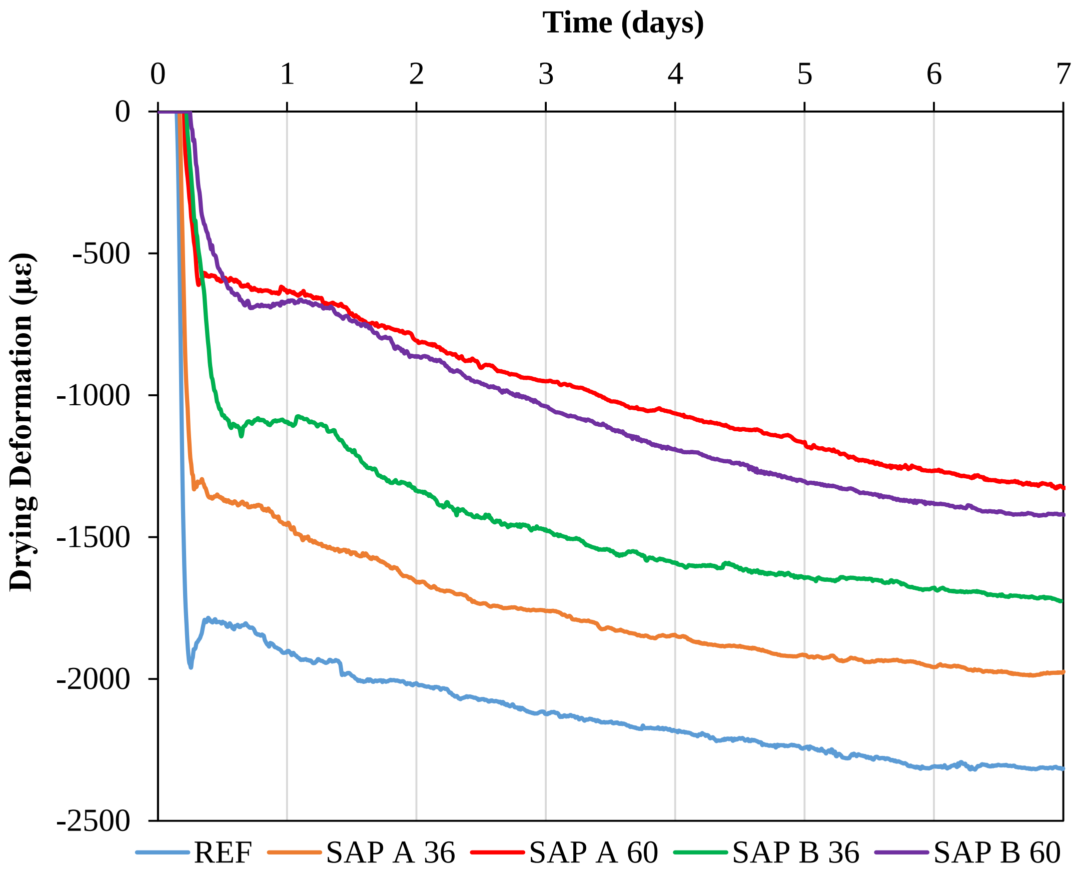

Figure 4.26 Theoretical drying shrinkage values of the SIFCON slurry mixtures

CHAPTER FIVE
CONCLUSIONS AND RECOMMENDATIONS

In this study, the effect of modification of SIFCON matrix with SAP on its mechanical and shrinkage properties was investigated. This study showed that even though SIFCON has a very dense fiber structure, the SIFCON matrix has very high shrinkage values. To prevent shrinkage and shrinkage cracks, internal curing can be applied with superabsorbent polymers with a tolerable loss in mechanical properties. The conclusions and recommendations can be drawn as follows.

5.1 Conclusions

- Fresh state properties were negatively affected only in the SAP A 36 mixture containing 36 kg/m^3 of additional internal curing water. Other mixtures were not significantly affected by the SAP modification.

- While the initial and final setting times were not affected by the SAP modification under autogenous conditions, the setting times were prolonged under drying conditions.

- The SIFCON and SIFCON slurry mixtures' compressive strengths have decreased slightly with the SAP modification. With the increase in the SAP ratio, the decreases in strength were also increased.

- Minor decreases have shown in the relative humidity cured specimens' compressive strengths. However, SAP-containing mixtures have less affected by relative humidity curing. In fact, relative humidity cured SAP B 36 mixture has exceeded the water cured SAP B 36 mixture. This may be indicating that SAP reduces the need for curing in field conditions.

- It has been observed that the use of randomly placed fibers has no effect on the compressive strengths. It can be said that the fibers could not make any contribution due to the very high matrix strength.

- Relative humidity cured SIFCON slurry mixtures performed better than water cured SIFCON slurry mixtures in terms of flexural strength and toughness. This may be due to the fact that the water and relative humidity cured samples could not be brought to a similar internal humidity ratio. However, since the variations of flexural strengths and toughness values according to the curing type are very large, various reasons such as the constant swelling of the SAP in the water cure and the causing a more void structure, the inability of the water penetration in the SIFCON slurry specimens may have caused this.

- SIFCON mixtures containing SAP B performed very poor flexural behavior in both cure types. It is possible that the mixtures containing SAP B were not properly filled between the fibers since SAP B is larger than SAP A and the SAP expands while absorbing the water inside the mixture.

- While water cured specimens outperformed relative humidity cured specimens in flexural strengths, toughness values were quite similar to each other for both curing types. This behavior may have been caused by the increase in the shrinkage in the relative humidity cure and therefore increase in adherence due to fibers compressed by the matrix.

- With the increase in the SAP ratio, the first crack time was delayed in the restrained ring test. The SAP B 60 SIFCON slurry mixture performed quite well in the restrained ring test. Mixtures containing 36 kg/m^3 of additional internal curing water have not significantly contributed to crack sensitivity.

- In autogenous shrinkage, all SAP-containing mixtures reduced the autogenous shrinkage significantly. As expected, the rate of reduction in autogenous shrinkage increased with the increase of the SAP ratio. While the mixtures containing SAP B showed very little shrinkage after the first day, the mixtures containing SAP A continued to shrink surprisingly.

- Under drying conditions, all SAP-containing mixtures were successful in reducing shrinkage. The internal curing agents used in this study also provided a reduction in drying shrinkage, contrary to some studies in the literature. The drying shrinkage results and the restrained ring test results showed quite a parallelism.

5.2 Recommendations

- Even though the teabag method is not a good method to obtain SAP absorption ratios in cementitious environments, it must be done to understand the absorption and desorption kinetics. To understand the untrendy fresh state behavior of the SAP A 36 mixture, detailed rheological experiments should be done.

- The fact that the SAP modification causes a direct decrease in the compressive strength and some contributions to the flexural behavior is thought to be related to hydration. In order to consolidate this theory, it is recommended to perform hydration degree, permeability tests, and internal structure examinations in future studies.

- In this study, two different sizes of SAP from a single origin were used. It is recommended to compare SAP varieties from different origins in further studies.

- It is recommended to examine the effect of SAP modification on SIFCON with different fiber ratios, geometries, and orientations in further studies.

- The effect of SAP in SIFCON should also be examined in terms of long-term shrinkage behavior, internal humidity tracking, and crack mapping.

- In further studies, the pull-out test should also be done to better understand fiber-matrix adherence.

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