



Investigation of Different Superplasticizers Effect on Workability and Strength Parameters in Ultra High Performance Concretes

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Article Info

Research article

Received: 11.03.2022

Revision: 09.05.2022

Accepted: 21.06.2022

Keywords

UHPC

Superplasticizer

Compressive Strength

Workability

UPV

Abstract

The use of ultra-high performance concretes (UHPC) in the modern construction industry is increasingly widespread. UHPCs are a type of concrete that provides advantages in solving many engineering problems. UHPCs have superior properties compared to conventional concretes in terms of workability, self-settling, as well as high strength and durability. However, although UHPCs have many advantages, achieving the desired workability is one of the biggest challenges of the production procedure, since they contain high amounts of powder materials. Therefore, the aim of this study is to determine the most suitable superplasticizer (SP) additive in terms of workability and strength by using different SP additives in UHPC mixtures. In this study, workability and strength parameters were tested on UHPC mixtures using 8 different SP additives. The SPs used were named A, B, C, D, E, F, G, H. First of all, the spreading diameters of the obtained mixtures were measured. For each mixture, compressive strength, unit weight, ultrasound velocity, Schmidt hammer rebound and Leeb hardness measurements were performed on 70x140 mm sized cylindrical samples taken on days 2, 7 and 28. Since SPs have a working principle at the interfaces of particles in the internal structure of concrete, different behaviors were observed on workability, even if a little. All the results obtained have been compared with the literature and it has been proven that they meet the UHPC specifications. As a result of the study, the best compressive strength value (127.83 MPa) was achieved with the G superplasticizer, and the flow diameter value was determined as 230 mm.

1. INTRODUCTION

High strength and modulus of elasticity, continuity and long life, low creep, low permeability and long-term cost reduction are most important of the features of ultra-high performance concretes (UHPCs). The most important usage areas of UHPCs can be modern building facades, shell structural elements and bridge elements. In the last two decades, with significant developments in concrete technology, significant efforts have been made for UHPCs that can be used in modern bridge engineering [1, 2]. According to EN 206: 2013 standard, the compressive strength of the UHPC should be above 100 MPa [3]. Also, ASTM C1856 [4] has specified the compressive strength of UHPC at least 120 MPa and the amount of slump flow between 20-25 mm. In order to produce such a concrete, fine aggregate (< 5 mm), micro silica fume, high performance cements and other powder additives must be included in the mixture [3, 5]. To significantly reduce the water need, select SP additives must be used. Also, the flexural, tensile and shear strength of the UHPC to be produced can be increased by adding an amount of fibers that do not adversely affect the compressive strength. Considering the number of articles published on UHPCs related since 2015, a significant increase is observed. This shows how much application potential UHPCs have in the construction industry [3]. In addition, the durability of UHPCs against chemical and physical factors has been examined by many researchers and it has been confirmed that they have superior performance compared to conventional concretes [6–9].

Many factors affect the compressive strength of UHPCs, such as the type of material used, its quantity, property and the water/cement ratio [10]. Pourbaba et al. [11] examined age effect on UHPCs containing different proportions of steel fiber. The results revealed that the compressive strength increases with the increase of steel fiber. In addition, the 18-day compressive strength of all mixtures reached 90% of the finally compressive strength. 10% silica fume may not affect the processability of UHPCs much. In addition, the dosage and type of SP is also important. The SP dosage depends on the properties of the cement (C3A and alkali sulphate content). In low water / cement based materials, slump loss is observed over time and increases with higher rates. The use of silica fume up to 20% increases the compressive strength but does not exceed 15% [12]. The flexural strength of UHPCs has been reported around 30 MPa on average [13]. This value can be managed with different fiber types and ratios.

Due to its many advantages, the non-destructive method has attracted great attention from engineers for field applications. The continuous measurability of microstructural change in concrete and the strong relationship between ultrasonic pulse velocity (UPV) and cement hydration is a known theory for the UPV test method [14]. One of the other non-destructive test methods, the Schmidt rebound hammer test was first developed by a Swiss engineer in the late 1940s [15]. Estimated compressive strength is obtained based on the values obtained as a result of the Schmidt hammer test result. Also, the Leeb hardness test, provides information about the hardness and estimated strength of construction materials such as stone and concrete [16].

Micro-sized fillers are used to fill the gaps between aggregate particles and cement in UHPCs with very low water / cement ratios and high strength. As such, silica fume is widely used, having an average particle size of about 1/10 of the cement. Therefore, these powder materials, which are necessary to increase the strength, cause low workability of concrete. Consequently, in order to avoid this problem in UHPC production, a select SP additive material should definitely be used in the mixture. SPs are generally based on polycarboxylate chemistry and work mainly by creating a steric barrier between particles. However, polycarboxylates based on methacrylic acid ester (MPEG) have been confirmed to be effective dispersants for UHPC [17]. The developments in the field of chemistry and the advancement of polymer technology have led to the discovery of highly effective plasticizers since the mid-1980s. These plasticizers, which have high water reducing ability, also increase the workability of fresh concrete. This effect, provided by the new generation of plasticizers, has led scientists to investigate to eliminate the compression process required during the placement of fresh concrete. The new generation superplasticizers, which have long polymer chains, accumulate on the surface of fine particles (as adsorbed) and provide dispersal of cement particles by means of electrical impulse and steric effect. While traditional superplasticizers are based on sulfone naphthalene formaldehyde or sulfonated melamine formaldehyde, new generation superplasticizers are copolymers with a carboxylic group in the main chain and a polyethylene glycol group in which the polyethylene glycol group is attached as a side chain [18].

Chryso Premia based SPs have been used in many studies and their positive effects on the workability and strength of concrete have been reported. The amount of Chryso Premia SP varies according to factors such as water/cement ratio and powder material content. For example, Babatunde et al. [19] used 1-3.4% in the UHPC mixture, Boshoff et al. [20] 0.4% in the cement composite mixture, and Kruger et al. [21] 0.7% by weight of binder in the 3D concrete mixture. Stearic acid is an additive that reduces the surface contact of fine particles and improves the stability of magneto-rheological liquids thanks to its long carbon chains. Chryso Optima 100 is often used in the cement industry as a surfactant polymer additive material that reduces the proportion of particles to volume [22]. Voit et al. [23] have used 1-2.5 wt% a BASF ACE type SP, while Rios et al. [24] have used 5.5%. While Kalkan et al. [25] examined the effect of shrinkage-reducing admixtures on the mechanical properties of self-compacting concrete, they used 0.7-0.9% BASF Master Glenium 51 as SP. SPs such as Sika [26, 27] and Iksa Polycar [28] preferred by other researchers are also available as water reducing additives.

Due to the use of too many fine materials and the low water/binder ratio in UHPC mix designs, problems are experienced in the consistency of fresh concrete and during pouring, and accordingly, factors with high permeability and adversely affecting the strength and durability parameters emerge. By using new generation superplasticizers, UHPC productions with improved workability are provided in the fresh concrete phase. Since the SPs used in UHPC mixture designs have a great effect on the consistency of fresh concrete, a study was conducted with the SP supplied from different suppliers and the best SP recommended

to be used in UHPC mixtures was determined. In this study, 8 different SP: A, B, C, D, E, F, G and H additives it is aimed to examine the effects on UHPC properties. For this, UHPC with 8 different SP added mixtures was produced and workability, compressive strength, UPV, Schmidt and Leeb hardness tests were carried out on the obtained 70 x 140 mm cylinder specimens. In terms of competition in the product market for chemical additives, and accordingly, in order to avoid conflicts and ethical values, the brand and company names of the SPs are not shared, and therefore their names are indicated with letters.

2. MATERIALS AND METHODS

2.1. Materials

CEM I / 52.5 R Portland cement has been used as a binder in the mixtures. White Portland Cement is a product, that has been used for more than 100 years around the world, preferred for obtaining aesthetical appearances and high strength levels. Particle size range of the cement used is given in Figure 1. Silica sand with a grain size range of 90-850 µm and quartz powder with a grain size range of 2-850 µm were used as filling material. The SiO₂ content of the quartz sand used is 96% (minimum), its specific gravity and specific surface area are 2.2 g/cm³ and 15m²/g (minimum), respectively. Microwhite silica fume (SF) was added to the mixtures as pozzolanic material. Microwhite silica fume is a dry silica fume powder. This silica fume is often used to improve the properties and performance of high performance concrete and specialty mortar formulations. This SF, which is also reactive, is often used to improve the properties and performance of high performance concrete and special mortar formulations. Stainless micro steel fiber (MSF) with a length of 12.5 mm and a diameter of 0.175 mm was preferred as the fiber. The tensile strength and modulus of elasticity of the fiber used are 2800 MPa and 210 GPa, respectively. For the suitable selection of SP, which is the main purpose of this study, 8 different types of plasticizers were used in the mixtures. The properties of plasticizers named from A to H are given in Table 1. Company information of SPs used due to ethical rules is not provided.

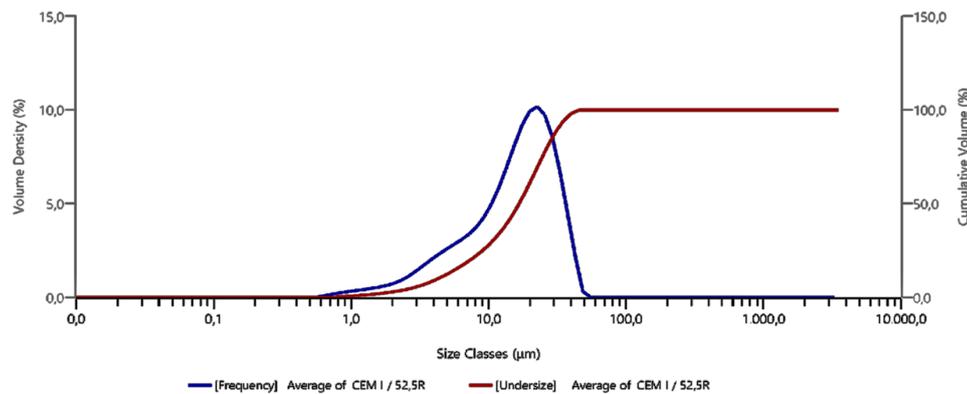


Figure 1. The particle size range and volume density of the cement used.

Table 1. Technical characteristics of 8 different superplasticizers.

SP type	Based on	Color	Density (g/cm ³)	PH	Chloride Content	Alkali Content
A	polycarboxylate	Brown	1.08	4	≤%1	-
B	Polycarboxylate	Dark yellow	1.075	4	≤%1	-
C	Polycarboxylate ether	Yellow	1.072-1.112	5-7	≤%1	≤%3.0
D	Polycarboxylate ether	Brown	1.082-1.142	6-7	≤%1	≤%3.0
E	Polycarboxylate	Light brown	1.09	3-7	≤%1	-
F	Polycarboxylate	Light brown	1.06-1.1	3-7	≤%1	-
G	Polycarboxylate	Light brown	1.07	5	-	≤%10
H	Polycarboxylate	Light yellow	1.08	4.4	-	-

2.2. Mix Design

In the production of UHPCs in the experiments, firstly, aggregate ratios were determined by using the modified Andreassan model, which is one of the particle packaging model types to ensure maximum occupancy. Amounts of 90-850 μm silica sand and 2-850 μm quartz powder were determined in granulometry in accordance with the curve shown by the model. The amount of cement in the mixture was determined as 800 kg/m^3 by examining the literature studies conducted for UHPC production. According to the cement amount, SF ratio was determined as 25 wt%. The amount of SD used in UHPC production has been calculated according to the literature and company recommendations. All dry mixes used in concrete were mixed for 2 minutes. The SP ratio in all mixtures was chosen as 4% of the total binder. By adding 70% of the water and SP to the mixture, the mixing process continued for about 5 minutes. Then, the remaining water is added to the mixture and the mortar was mixed until it reaches the consistency. The water/binder ratio was determined as 0.20 in all mixtures. The average water temperature used in the mixtures was 7.5 $^{\circ}\text{C}$. Then, 4% of the total weight MSF was added and the mixture was continued for the last 2 minutes and then poured into cardboard cylinder molds. The amounts of materials used in the UHPC mix are given in Table 2.

Table 2. Mixture content (kg/m^3) for 1 m^3 volume.

Cement	Silica sand	Quartz	Silica fume	Superplasticizer	Water	Micro Steel fiber
800	800	200	200	40	200	93

2.3. Test Methods

After the mixtures were prepared, Slump test has been performed according to ASTM-C 1611 / C 1611M - 05 standard [29] to control their workability. 2-7-28-day 70 X 140 mm cylindrical samples produced for the determination of compressive strength were tested according to TS EN 12390-3 standard [30]. Ultrasonic pulse velocity (UPV), Schmidt and Leeb hardness tests were performed as non-destructive methods before subjecting them to compressive strength on the same specimens. Both sides of the samples were tested with a Schmidt hammer according to the ASTM C805 standard [31] and average values were obtained after 12 tests. UPV tests were performed according to ASTM C597 [32]. The ASTM A956 standard [33] was used to determine the Leeb hardness of the produced UHPC samples. In addition, unit weights were calculated by measuring the dry weight and dimensions of all 28-day samples. Schematic images representing test methods are shown in Figure 2.

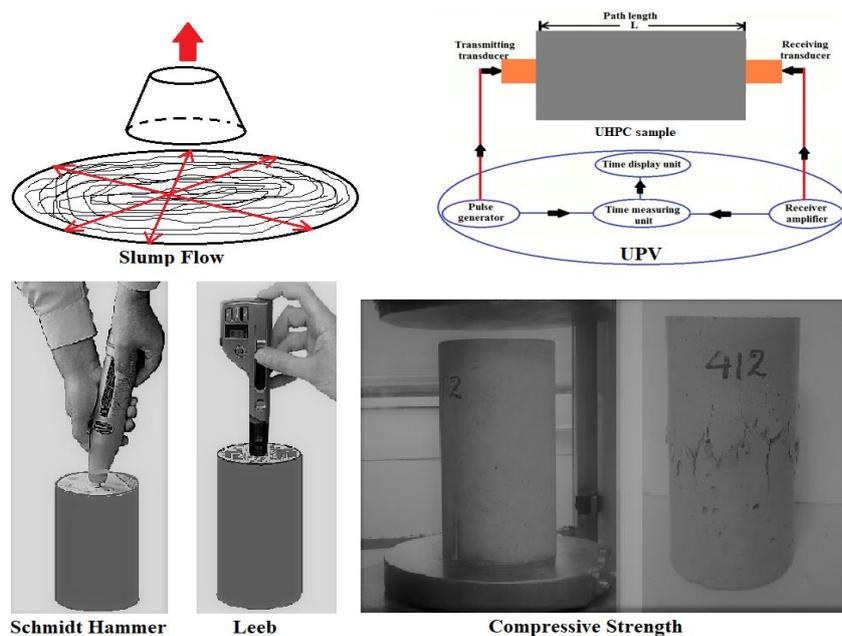


Figure 2. Schematic images of test methods.

3. RESULTS AND DISCUSSION

3.1. SP Effects on the Fresh State Test Results

Although different SP additives were used in the same ratio (4 wt%) in all 8 mixtures, the Slump flow diameters varied between 222-260 mm. The comparison of slump flow diameters is given in Figure 3. Considering the results, it is observed that A-D, F-G and B-C-E values are equal to each other. The mixtures with the lowest workability were B-C-E added mixtures. Slump flow diameters of A and D added mixtures were higher than these mixtures by 8%. The slump flow value of F and G added mixtures 3.6%, and the value of the H added mixture 17% were higher than the B-C-E added mixtures. It is thought that the changes between the flow diameters are due to the PH value and chloride content. Also, considering all the results, G presented the maximum compressive strength of the mixture containing SP. This may be due to the fact that the alkali content is higher than the others.

The high specific surface area of the materials used in concretes such as UHPC containing high dosages of powder materials causes the water used to be inadequate. This leads to workability problems. Therefore, in order to avoid this problem, generally 12-25% water-reducing super plasticizer additives are used. SP additives wrap around the cement particles, charge the particles with a negative electric charge, allowing the cement particles to repel each other and prevent agglomeration. The resulting Slump flow diameters have been confirmed to be a suitable workability level for UHPCs. This proves the purity of the aggregates used. In the [34] study, it was stated that the low workability was due to impurities such as clay in aggregates. Optimizing the workability level is important as it affects the strength and rheological properties. Excessive workability prevents sufficient C-S-H gel production and leads to a decrease in compressive strength [35]. Therefore, it is beneficial to examine not only the workability behavior but also the mechanical properties for the selection of a SP as a water reducing admixture in concrete production. Previous research implies that the slump flow loss involves chemical and physical processes. This is mainly attributed to the w/binder ratio, type and dosage of SP, as well as SO₃ content, alkali content, C-S-H formation, load characteristic, C₃S/C₂S ratio [34].

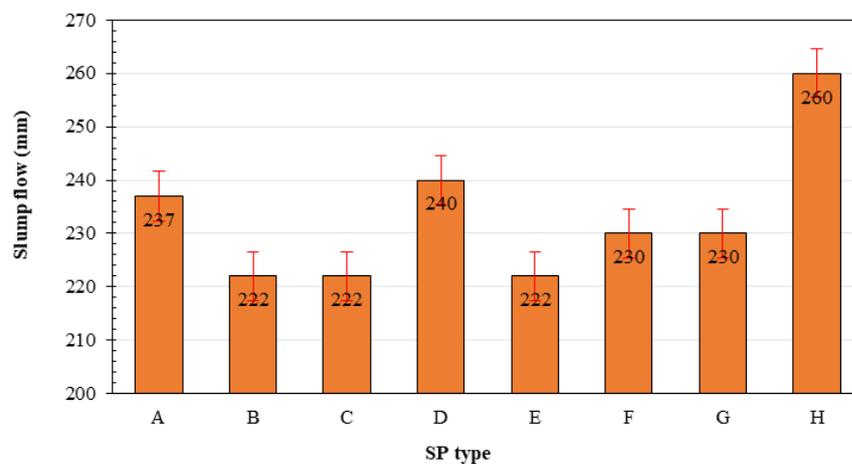


Figure 3. Comparison of slump flow diameters.

3.2. SP Effects on the Hardened Properties

The unit weights of the samples obtained from 8 different mixtures are shown in Figure 4 and the effects of different SP additives are compared. No serious differences have been observed between results. The unit weights of UHPCs produced with A, B, C and E additives have been obtained equal. However, unit weight of D added composite was slightly lower with a difference of 0.4% compared to this group. Compared to the unit weights of these mixtures, the maximum unit weight value was obtained for the F added mixture with a difference of 0.9%. Also, the minimum unit weight value was obtained for the G added mixture with a difference of 2.2%. The unit weight of H added mixture was 0.9% less than the unit weight of these four mixtures. Since the binder, filler and water / cement ratios used in UHPC mixtures are the same, it is normal for unit weight values to be similar to each other. However, the difference observed

in samples such as the G added mixture may be due to air bubbles or test errors during mixing. When the slump flow and unit weight results were compared, no significant relationship was found between them.

The unit weight values of 2.22-2.29 g / cm³ obtained in this study have been confirmed by the literature, and the reason why UHPCs are denser than conventional concretes is that large aggregates are not used, prefer fine aggregates using special packing unit weight, and the addition of fibers. The reason why UHPCs are heavy in mass is the use of heavy aggregates such as quartz and silica sand. Meeting the requirement for optimum classification of quartz sand for uniformity of the UHPC matrix and optimum packing unit weight is one of the challenges in UHPC manufacturing [36].

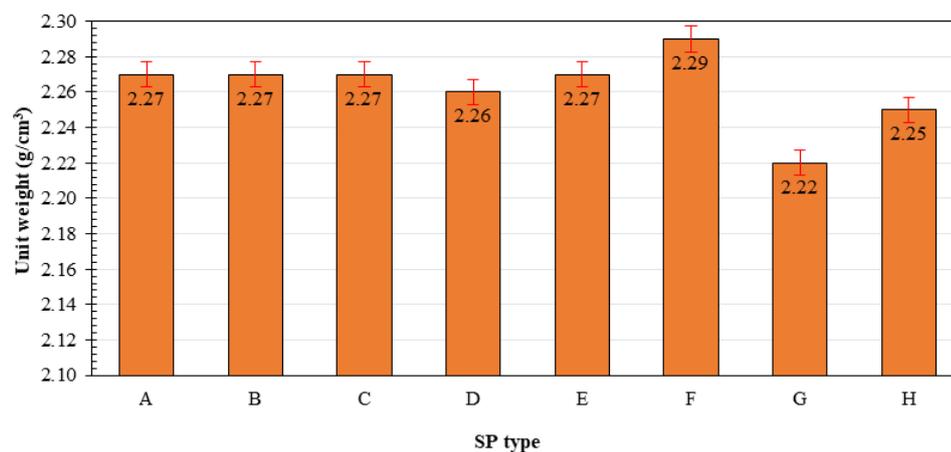


Figure 4. Unit weights of the samples obtained from 8 different mixtures.

Considering the compressive strength results (Figure 5), it is understood that SP types do not have a significant effect. In fact, the 28-day compressive strengths of C-D-F added mixtures are almost equal, and the strengths of the A-E added mixtures are also equal. However, according to the early strength results, the minimum compressive strength was obtained as 75.39 MPa for the H added mixture and the maximum compressive strength as 85.58 MPa for the E added mixture. Also, according to the 28-day compressive strength results, the minimum compressive strength was obtained as 115.26 MPa for the H added mixture and the maximum compressive strength as 127.83 MPa for the G added mixture. Maximum strength difference due to SP effects was approximately 10% for 28-day while maximum strength difference was approximately 13% for 2-day. This situation, in which the SP effect is more pronounced at an early age, has been confirmed by the literature [34]. When the compressive strengths were examined according to the age of the samples, the average strength of the 7-day and 28-day samples increased by 26% and 46%, respectively. When the strength results are compared with the unit weight results, it is observed that there is a parallel relationship between them.

Compressive strengths of 120 and above obtained for 28-day UHPCs prove that the trapped air content in the concrete is low. The chemical additives used may swell in the mixture and cause the formation of air gaps, which directly leads to a decrease in the compressive strength [37, 38]. The 0.2 water / binder ratio in this study was obtained as a result of many preliminary experiments. If the water / binder ratio falls below this level, more SP will be needed. This will cause the particles to separate from each other, the formation of air gaps and thus a decrease in the compressive strength [37]. The change in the strength of UHPCs is not limited to chemical additives, there are many other factors such as the curing temperature. Steam curing of UHPC at high temperatures is also a factor affecting strength. Microstructure develops due to pozzolanic reactions between C-H resulting from hydration of cement and complementary materials such as silica fume and nanosilica. Exposure to high temperature affects the reaction of the pores in the composite tissue and causes an increase in compressive strength compared to samples cured under ambient conditions [39]. Therefore, if the samples in this study were cured with steam at high temperatures, their compressive strength would probably have increased much more. Well-chosen steel fiber dosage can increase ductility and reduce autogenous shrinkage of UHPC. However, the increase in the amount of steel fiber may reduce the workability of the concrete and lead to a decrease in its strength.

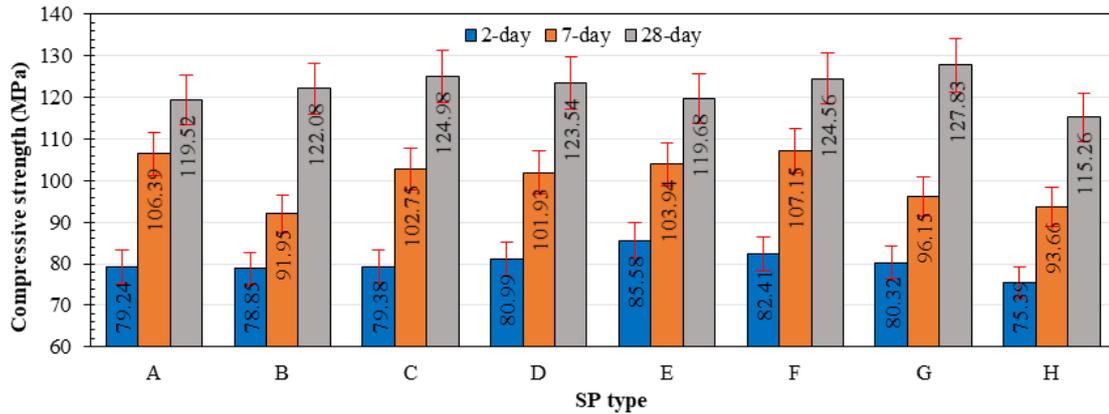


Figure 5. 2, 7 and 28-days compressive strength results.

Conventional test methods that examine the properties of concrete cannot provide information about its microstructural and physicochemical properties. The UPV test method has been suggested by many researchers to examine the microstructural changes and strength of concrete. According to the results of many studies, it has been confirmed that there is a strong relationship between UPV values and cement hydration [40].

The 2, 7, and 28 days UPV values are shown by comparison in Figure 6. When the UPV results were examined, a significant difference was observed between the 28-day UPV values, while the effects of the SP types at early age were insignificant. The 2, 7 and 28 days UPV values were measured as 4.16-4.25, 4.27-4.43, and 4.39-4.55 km/s, respectively. UPV values of UHPCs produced with all additives increased according to the sample age. This situation is also valid in conventional concretes and while it is more evident in the first days, the rise rate decreases after 28 days [41]. Although different additives do not have a significant effect on the UPV values, the small amount of change observed is due to the hydration rate and the porosity ratio in the microstructure. The increase in the number and ratio of pores causes a decrease in the ultrasonic pulse velocity. Also, the increase and concentration of hydration products in older ages create a more intense structure and exhibit higher UPV results. This situation is more effective in the first ages due to the high hydration rate. When UHPC values containing C additive are examined, there is a 5% difference between 2 and 7, while a difference between 7 and 28 is 0.7%. For example, there is a 5% difference between 2 and 7 and a 3% difference between 7 and 28 in D added UHPC values. This difference between C and D is related to the working principle of SPs. It can be concluded that the hydration rate in D is faster in older ages compared to C. A similar situation can be observed for other contributions. The UPV levels obtained were compared with the literature, and confirmed by the results of UHPCs studied in many studies [14, 42, 43]. Fodil et al [43] measured 4-5 km/s UPV values for concretes with compressive strength above 80 MPa.

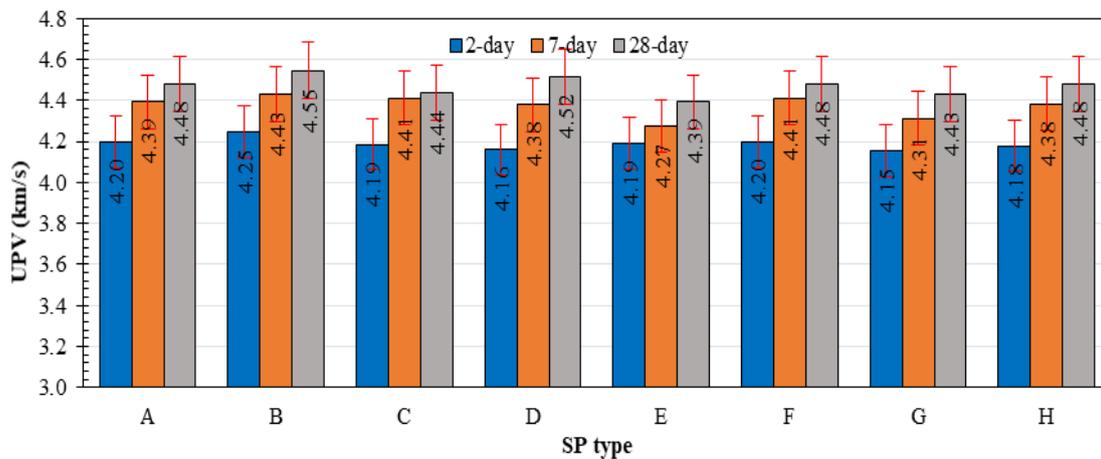


Figure 6. 2, 7 and 28-days ultrasonic pulse velocity test results.

The measured Schmidt hammer results for UHPCs are compared in Figure. 7. Comparing the Schmidt hammer test results with the compressive strength results, it is observed that they are more similar to the UPV test results in terms of sample age effect. Comparing the Schmidt hammer test results with the compressive strength results, it is observed that they are more parallel to each other than the UPV test results in terms of age effect. In other words, while a significant difference was observed between 2 and 7 days results, the difference was considerably reduced between 7 and 28 days. According to the SP type, the 2, 7 and 28 days Schmidt hammer test results were obtained as 18-20, 31-39 and 42-47 R, respectively. There was an 84% difference between the average results of 2 and 7 days and a 27% difference between 7 and 28 days. Considering the Schmidt numbers of the 7-day samples according to the SP type, a 10% difference was observed between the minimum value (D) and the maximum value (A, B, C and E). This difference was calculated as 25% and 12% for 7 and 28-day samples, respectively.

Schmidt values are mostly affected by the aggregate type in the concrete. For example, it is known that the Schmidt numbers of concrete containing coarse limestone aggregate are 7 points lower than concrete containing coarse aggregate, resulting in a 7 MPa difference in the compressive strength of concrete. Schmidt numbers of lightweight concretes also vary compared to normal concretes. There is difference in the rebound numbers of two concrete samples containing the same aggregate from different sources [15]. However, according to the results reported by Xu and Li [44], it was found that the Schmidt hammer numbers at different positions of a single piece of concrete were close to each other. When the rebound results obtained are compared with the literature, it is concluded that the values are suitable for high strength concrete. In concretes with low strength, microstructural aggregate roughness and gaps caused by the weak bond between aggregate-cement cause a decrease in Schmidt values [15]. In this study, in samples with low Schmidt numbers, the SP used probably caused an increase in the porosity ratio in the concrete by reducing the contact surface between particles. Therefore, the use of these SPs in high dosages in UHPC mixtures may lead to deterioration of other properties along with the compressive strength.

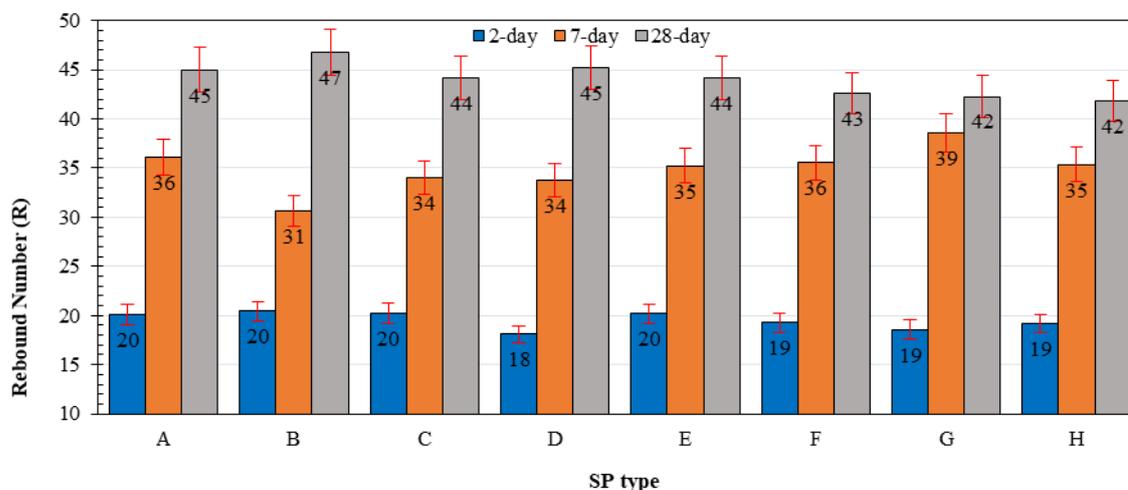


Figure 7. 2, 7 and 28-days Schmidt hammer test results.

The Leeb hardness test, which is a non-destructive method, provides information about the hardness and estimated strength of materials [16, 45]. In the present study, the hardness of cylindrical samples produced from UHPC mixtures was measured at different ages and compared in Figure. 8. The measurement was taken from a total of 6 points, namely the back front surfaces of the sample, and the average hardness value was calculated. When the hardness values obtained are compared with the other non-destructive test results, it can be concluded that there is a parallel relationship between them. An average of 16% difference between 2 and 7 day Leeb hardness results and 9% difference between 7 and 28 day results were observed. This situation is directly related to the cement hydration rate as in other test results. According to the 28-day Leeb hardness results, the maximum and minimum values were measured for A and G added UHPCs, respectively. 8% Difference has been observed between these values.

There are a few Leeb hardness studies on normal concretes, although limited. Song et al. [46] investigated the Leeb hardness of sodium silicate based concrete and normal C30 concrete and concluded that the average hardness value of normal concrete was 362 HL and that of sodium silicate based concrete was 405

HL. The hardness values of the 28-day GRC specimens produced in this study were measured between 509 and 548 HL. Therefore, the results prove to be compatible when compared to normal concrete. The 2 and 7 day Leeb hardness results were measured as 396-438 and 456-510, respectively. Gomez-Heras et al. [47] stated that the finer the grain size, the higher the Leeb hardness. This situation is directly related to the filling of fine-grained minerals into micro and macro pores [48].

4. CONCLUSION

In this study, using 8 different superplasticizer additives, their effects on UHPCs with the same mixture content were investigated. Workability, compressive strength determination and non-destructive test methods were carried out on the cylindrical samples obtained from the mixtures. The results obtained can be summarized as follows;

A 17% difference was observed between the slump flow diameters. This may be due to the difference in behavior of SPs on C-S-H. All the slump flow results obtained for all 8 mixtures have been compared with the literature and confirmed that they are at an appropriate level.

No significant difference was observed between the unit weight of UHPCs. Minor differences may be related to the working principle of SPs. This proves that the ratio of the 8 different SPs used are suitable and compatible with powder materials.

The fact that 28-day compressive strengths are generally above 120 MPa proves that all used SPs are suitable for UHPCs. Some of the SPs have shown their effects on UHPC characteristics at an early age, some in later ages. However, in older ages, the strength and other properties of the samples have reached a similar level. This is all about the hydration rate of the cement and the proportion of C-S-H products produced over time. Therefore, in the selection of SP, it is important to consider factors such as the intended use of the UHPC and the expected durability time.

A parallelism was observed between nondestructive test results considering the sample age and SP type. While the effect of SP type was more pronounced in early ages, it decreased in later ages. UPV, Schmidt hammer and Leeb hardness tests provide information about the internal structure of concrete, porosity, capillary voids and C-S-H formation. Since the type and ratio of SP also has a significant effect on the formation of C-S-H, the slight differences that occur between nondestructive test results may be related to the working principle of different SPs.

As a result, all test results show that 8 different SPs can be used in UHPCs. However, if it is necessary to make a selection according to the compressive strength, which is important among the UHPC properties, it is recommended to produce the mixture containing G additive. The compressive strength of the sample obtained from this mixture was slightly higher than the others as 127.83 MPa. The slump flow diameter for this mixture was also measured as 230 mm, which is consistent with the literature.

ACKNOWLEDGMENTS

In this study, we would like to thank Fibrobeton company and R&D personnel who contributed to material supply, test production and some tests.

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