

EVALUATION ON COMPREHENSIVE PERFORMANCE OF SULFATE ACTIVATED SLAG SELF-COMPACTING CONCRETE

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Abstract

The current study deals with the production and engineering properties of a self-compacting concrete (SCC) produced with sodium sulfate activated slag binder. A mixture of 5% calcium hydroxide and sodium sulfate varied at different values of 1, 3, 5, 7, and 10% by mass was used as the primary activator of the binder. The fresh properties of the concretes were identified by adapting slump flow, L-Box, air entrained volume, and unit weight. On the other hand, the properties of the hardened concretes were assessed by using the tests on dried density, flexural strength, compressive strength, drying shrinkage, ultrasonic pulse velocity (UPV) and water absorption. Experimental results illustrated that by properly adjusting dosage of superplasticizer (SP) in order to avoid an issue related to lost set, SCC productions were only successfully achieved with sulfate amount limited at 5%. The increment of the sulfate in the range of 1–5% seemed to denser the SCC structure due to the increased unit weight, reduced air entrains, improved strengths, decreased drying shrinkage, increased UPV, and decreased water absorption. Further increase of the sulfate amount in the range of 5–10% induced the hardened concretes with reductions on mechanical strengths, UPV, and water absorption, possible due to the reduced flowability. In this study, 5% of sulfate was considered as the optimum value to produce the concrete with the best quality except the drying shrinkage still decreasing with the sulfate increment.

Keywords: sulfate activated slag; self-compacting concrete; mechanical strengths; drying shrinkage; ultrasonic pulse velocity; water absorption

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1. Introduction

For many past decades, cement has played a dominant role in resolving an issue related to rapid increase in population but cement manufacture must be on duty for approximately 5–7% of global greenhouse gas emissions [1]. Previous literature review indicated that each ton of clinker production released 0.87 tons of carbon dioxide [2]. Developing alternative binder with reduced energy has been urgent for establishing sustainable development [3]. Among the potential candidates, sulfate activated slag has been under special regard from worldwide researchers. When compared with other existing friendly binders such as supplementary cementitious materials and alkali activated materials, hydration of the sulfate activated slag occurs under a lower alkaline environment, which implies a safer working condition. For producing sulfate activated slag, neutral sodium sulfate coinciding with a minor amount of lime/cement is commonly used as the main activator [4]. Generally, the sulfate dosage concentration equivalent to values varied in range of 1–3% sodium oxide by mass has been considered as the suitable amount to manufacture the binder with satisfactory performance [5]. Nevertheless, the existing issues related to the prolonged setting and low early mechanical properties

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of the sulfate activated slag have been reported [5]. For enhancing the performance of the cementitious material at early ages, addition of limestone powder as partial replacement of slag was previously suggested for accelerating the hydration of the main precursor [6]. In addition, the sodium hydroxide based strong alkali was possibly used to enhance the hydration kinetic and early strengths of the sulfate activated slag [7]. Based on the study [7], the superior mechanical properties of the alkali modified sulfate activated slag were attributed to a well-packed microstructure of the hydrates.

Self-compacting concrete (SCC) has been well-known to own excellent flowing, passing, and filling capacity under its' self-weight without bleeding and/or segregation. As such, the SCC has been specializing in its applicability for construction sites where high-quality assurance independent of craftsmanship is seriously required [8–10]. Nevertheless, by being comprised of a high cement dosage, the SCC productions has been criticized for contributing to environmental challenges. To settle the problem, instead of plain cement, using supplementary cementitious material (SCM) with pozzolanic property (such as slag, fly ash, and silica fume, etc.) and/or inert filler (such as limestone and alternative impure limestone powder) for reducing cement content has been preferable [11, 12]. Especially, neglecting the cement role in SCC proportion by utilizing no-cement binders such as alkali activated materials [13–15] and low energy super-sulfated cement [16] has been considered as an innovative way to alleviate the environmental impact induced by SCC manufacture. As previously mentioned, sulfate activated slag with sodium sulfate also contains no-cement content and thus its applicability as an alternative binder of the friendly environmental SCC should be considerably evaluated.

According to the above review points, developing no-cement binder based SCC applicable for specific construction sectors is always encouraged. Besides the SCCs using alkali activated materials and super-sulfated cement, there is lack of study focusing on production and properties of sulfate activated slag based SCC. Such research gap urges the current study to explore the sulfate activated slag based SCC trial and the changed performance of SCC on a large range of sulfate dosage. Obviously, the significance of the current study lays on proposing a guideline for manufacturing the practical sulfate activated slag SCC. Based on the experimental conduction on the macro-behavior of the concrete the study also contributed to visualizing the applicability of such the potential binder for a so-called promising field of greener SCC. In order to get the research achievement, the sodium sulfate is used as the partial replacement of slag at various levels in the SCC proportions. The experimental tests on both fresh and hardened properties of the resultant SCCs are conducted. By assessing the impacts of sulfate content on the comprehensive performance of the SCCs, the SCC production possibly applied for the construction sectors is suggested.

2. Experimental program

2.1. Materials

Industrial by-products of ground granulated blast furnace slag were used as the crucial precursor of the binder. To trigger the hydration of slag, commercial calcium hydroxide accompanied with sodium sulfate based alkaline-sulfate activators were used. For producing self-compacting concrete (SCC), low calcium Class F fly ash (FFA), crushed sand, and crushed stone were used as the fine, medium, and coarse aggregates, respectively. The chemical and mineral compositions of the powders including slag, fly ash, sodium sulfate, and calcium hydroxide were detected by applying X-Ray fluorescence (XRF) and X-Ray diffraction (XRD) as illustrated in Table 1 and Fig. 1, respectively.

Accordingly, the slag was comprised of amorphous phases rich in oxides of calcium, silica, and aluminum, which was normally assigned to the latent hydraulic material. On the other hand, fly ash mostly contained mullite and quartz crystals being stable in low- to mid-alkali environment such

Table 1. Physicochemical properties of materials

Properties	Slag	Fly ash	Sodium sulfate	Calcium hydroxide
Specific gravity	2.9	2.2	2.7	2.2
SiO ₂ , %	38.01	58.77	-	-
Al ₂ O ₃ , %	13.13	26.11	-	-
Fe ₂ O ₃ , %	0.55	5.61	-	-
CaO, %	36.80	2.07	-	-
MgO, %	5.77	1.66	-	-
SO ₃ , %	1.36	0.21	-	-
Na ₂ O, %	0.13	0.27	-	-
K ₂ O, %	0.78	1.48	-	-
TiO ₂ , %	0.45	0.66	-	-
L.O.I, %	3.01	3.11	-	-
Na ₂ SO ₄ , %	-	-	> 95%	-
Ca(OH) ₂ , %	-	-	-	> 95%

Note: L. O. I = Loss on ignition.

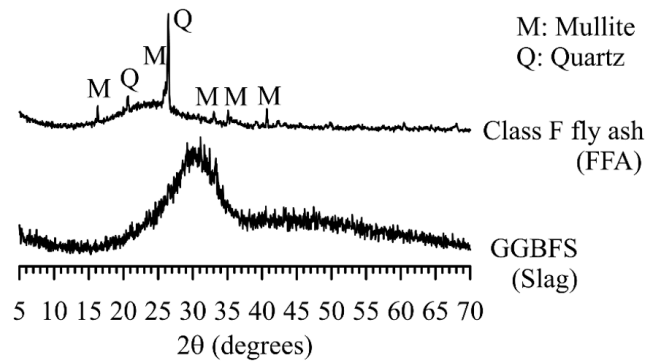
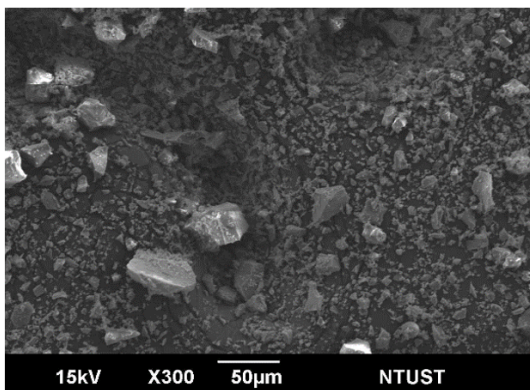
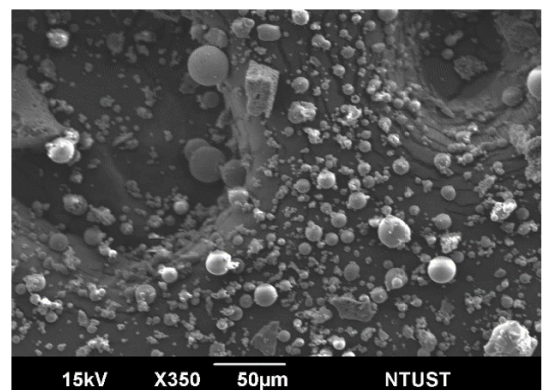


Figure 1. XRD patterns of materials



(a) slag



(b) class F fly ash

Figure 2. SEM images with of materials

as cement pore solution and hydrated lime solution. Therefore, it was considered to serve as the filler in SCC proportions. SEM images as shown in Fig. 2 indicated the fact that the FFA particles

were almost spherical in comparison with the angular shapes of the slag. The specific gravity, water absorption, and fineness modulus of the crushed sand were 2.85, 3.8%, and 3.2, respectively. In addition, the specific gravity, water absorption, and maximum size of the crushed stone were 2.75, 2.6% and 10mm, respectively. To control the workability of the fresh SCCs, Type G super plasticizer (SP) was used. In this study, all raw materials are available in Vietnam.

2.2. Mixture proportions

Previous studies [7] have indicated that approximately 5% sodium sulfate is typically used in sulfate-activated slag binders. In this study, the binder composition of the sulfate activated slag based SCC was designed by partially replacing slag with sodium sulfate at different values of 1, 3, 5, 7, and 10% by mass. For generating sufficient alkali to trigger the slag hydration, calcium hydroxide was used as partial replacement of the total amount of binder, i.e., mixture of slag, sodium sulfate, and calcium hydroxide, at 5% by mass. The composition and volume of aggregates including FFA, sand, and stone were identified based on the particle packing theory for minimizing void volume as previously suggested [17]. According to preliminary experiment, simultaneously setting both ratio of FFA to FFA-sand mixture at 14 mass.% (i.e., $\text{FFA}/(\text{FFA}+\text{sand}) = 0.14$) and ratio of the FFA-sand mixture with optimized proportion to total amount of FFA, sand, and stone at 54 mass.% (i.e., $(\text{FFA}+\text{sand})/(\text{FFA}+\text{sand}+\text{stone}) = 0.52$) led to the most expected result, and thus they were applied in this study. For all SCC mixtures, the water to binder ratio by mass was fixed at a value of 0.4 (i.e., $\text{water}/(\text{slag}+\text{sodium sulfate}+\text{calcium hydroxide}) = 0.4$). Different from the typical SCCs using cement, the SP dosage in this study was properly adjusted based on the crucial requirements of avoiding an issue related to lost set followed by satisfactory flowing and passing ability. Preliminary experimental results illustrated that the SP dosage reaching the so-called threshold value of 0.9% by mass of the total binder induced the sulfate activated slag binder to become a lost set. Therefore, this value of SP was applied as the limited value in all SCC proportions. The mixture proportions of the SSCs are shown in Table 2.

Table 2. Mixture proportions of the SCCs, kg/m³

Mixes	Na ₂ SO ₄	Slag	Ca(OH) ₂	Water	Fly ash	Sand	Stone	SP
N1	5.5	521	27.7	222	113	711	691	4.2
N3	16.6	510	27.7	222	113	711	691	6.1
N5	27.7	498	27.7	222	113	711	691	6.5
N7	38.7	487	27.7	222	113	711	691	7.0
N10	55.3	470	27.6	222	113	711	691	7.4

2.3. Test methods

The flowing and passing ability of the SCCs was estimated based on National Vietnam Standard TCVN 12209:2018 [18]. Accordingly, the SCC flowing ability was assessed based on the value of slump flow diameter (d) by adapting the Abrams cone slump flow test. On the other hand, the SCC passing ability was estimated by conducting the 2-bar L-box test. By the test, the concrete depths at two ends of horizontal part, denoted as H_{\min} , and vertical part, denoted as H_{\max} , of the L-box were recorded and used for computing passing ability ratio, denoted as $PL = H_{\min}/H_{\max}$. The SCC was considered to have satisfactory passing ability as the PL value reached the level of 0.8. Besides flowing and passing ability, the fresh properties of the SCCs were additionally estimated based on the unit weight and air entrained volume in accordance with TCVN 3108:1993 [19] and TCVN 3111:1993 [20].

Immediately after the testing on the fresh properties of the SCCs, the $150 \times 150 \times 150 \text{ mm}^3$ cubes of SCCs were cast, cured in molds at ambient temperature for 24 hours, removed from the molds, cured in air at $27 \pm 2 \text{ }^\circ\text{C}$ and $50 \pm 4\%$, and used for the tests of dried density, compressive strength, ultrasonic pulse velocity (UPV), and water absorption accordant to TCVN 3115:1993 [21], TCVN 3118:2022 [22], TCVN 13537:2022 [23], and TCVN 3113:2022 [24], respectively. In addition, the SCC prisms with the dimensions of $150 \times 150 \times 600 \text{ mm}^3$ and $75 \times 75 \times 285 \text{ mm}^3$ were also cast for the flexural strength and drying shrinkage tests in accordance to TCVN 3119:2022 [25] and TCVN 8824:2011 [26], respectively. After being cured in mold for 24 hours, all the concrete prisms were removed from the molds and cured in air at $27 \pm 2 \text{ }^\circ\text{C}$ and $50 \pm 4\%$ of RH until testing ages of 1, 3, 5, 7, 21, and 28 days. In this study, the compressive strength tests were run at 3, 7, and 28 days of curing. The tests of UPV and drying shrinkage were monitored at various ages up to 28 days and the flexural strength and water absorption was conducted at 28 days. In each test, three specimens were used, and when the minor standard deviation was within the acceptable range, only the mean values were reported in the experimental results.

3. Results and discussions

3.1. Fresh properties

The fresh properties of the SCCs were conducted and the results were summarized in Table 3.

Table 3. Fresh properties of the SCCs

Properties	N1	N3	N5	N7	N10
SP, %	0.51	0.74	0.79	0.8	0.85
d , cm	60	60	60	43	38
$PL = H_{\min}/H_{\max}$	0.8	0.8	0.8	0.2	0.1
Unit weight, kg/m^3	2384	2429	2475	2464	2452
Entrained air volume, %	2.9	2.7	2.3	2.6	2.5

According to the table, by controlling the SP dosages at the values less than 0.9% of total amount of binder to assure a good set of the specimens as aforementioned, the fresh SCC mixtures with satisfactory slump flow diameter (d) values reaching 60 cm, which is assigned to the typical SCC, were only achieved with the addition of sodium sulfate amount less than 5%. Further increase in the sulfate amount beyond 5% led to the serious increase in the demand of SP and thus induced the issue of lost sets. The passing ability ratio (PL) of the satisfactory flowability SCC was computed to be 0.8 which was normally assigned to the SCCs with sufficient passing ability. Table 3 illustrates that the sulfate increment in the range of 1–5% increased the unit weight and decreased the air entrained volume of the fresh SCC, which was possibly due to more condensed concrete structure. But, further increased amount of sulfate reduced the unit weight and increased the air entrained volume of the fresh concrete due to the decreased self-compaction of the fresh concrete. Consequently, the increase in sulfate addition induced a negative impact on the rheological property of the fresh SCCs using sulfate activated slag. Such a result was attributed to the initial hydration of sulfate that led to the increased water demand for maintaining the workability of the fresh concrete. As previously suggested [27], the addition of the sodium sulfate substituting slag possibly accelerated the dissolution rate of the cementitious ingredients and enhanced the ion concentration of the mixture, which in turn thus resulted in the fresh binder characterized by a more favorable aqueous state. In a previous study [28], the decreases in both workability and setting time of the fresh sulfate activated slag due to the increment of sodium sulfate were also obtained.

3.2. Dried density

The dried density of the hardened SCCs is illustrated in Fig. 3. According to the figure, the dried density result was obviously in good consistence with the obtained unit weight result as previously discussed. Indeed, as can be seen from Fig. 3, the dried density of the hardened SCC increased with the increased amount of sulfate in the range of 1–5%, which was attributable to the enhanced structure of the modified SCCs. However, as the sulfate dosage increased at value in the range of 7–10%, the dried concrete dried density continuously decreased. As previously discussed, the sulfate increment in the range of 1–5% seemed to induce the SCC structures to become more condensed, which was related to the increased dried density values. But excessive addition of sulfate coinciding with limited SP amount remarkably reduced the concrete workability and thus reduced the dried density of the hardened concrete.

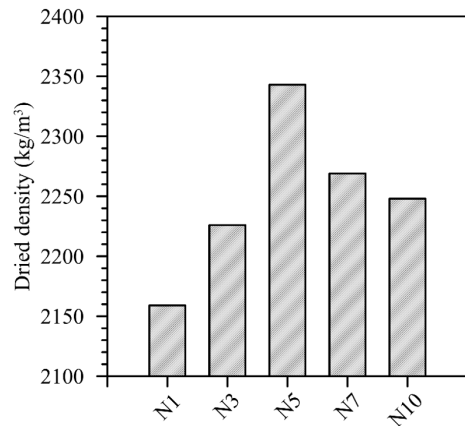


Figure 3. Dried density of the SCCs

3.3. Mechanical strengths

The compressive and flexural strengths of the hardened SCCs are illustrated in Fig. 4 and Fig. 5, respectively. As expected, the compressive strengths of the concretes increased as the curing ages increased due to the enhancement of the binder hydration. Irrespective of the ages of curing, the compressive strengths of the hardened concretes increased with the increment of the sulfate amount in the range of 1–5% but continuously decreased with further increase of sulfate in the higher range of 7–10%.

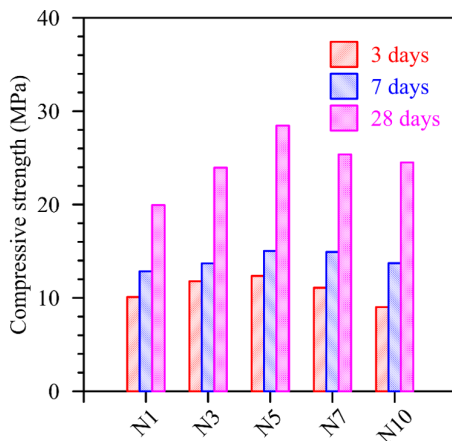


Figure 4. Compressive strengths of the SCCs

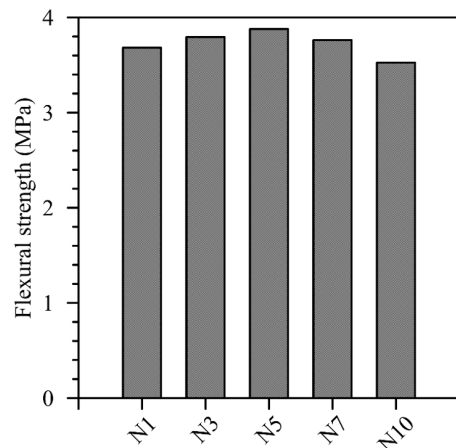


Figure 5. Flexural strengths of the SCCs

As such, 5% of sulfate activator was considered as the optimum amount for producing the SCC with the highest compressive strengths of 12.4, 15.0, and 28.5 MPa at 3, 7, and 28 days of curing, respectively. Obviously, this SCC qualified technical requirements in a widespread construction applicability, particularly emphasized on the structural concretes. The 28-day flexural strengths of the hardened concretes as illustrated in Fig. 4 was apparently in consistence with the compressive strength

result. A comparison illustrated that effects of sulfate addition on the changes of the flexural strengths and compressive strengths were similar. Consequently, the value of 5% of sulfate activator was also the optimum amount to manufacture the hardened SCC (N5 mix) with the maximum flexural strength of 3.9 MPa at 28 days. In this study, the changed mechanical strengths of the sulfate activated slag SCCs on the sulfate dosage was possibly explained based on the degree of structural condensation of the concrete sample. In fact, as afore-mentioned in Section 3.2, addition of the sulfate activator at the so-called optimum amount, i.e., at 5%, induced the structure of the concrete to become enhanced due to the increased dried density. Additionally, the improved concrete structure due to the addition of the proper amount of the sulfate activator was also clarified by the other evidence as subsequently illustrated. According to previous findings [7], the so-called optimum sodium sulfate dosage of 3–5% produced a compressive strength improvement of approximately 23–35%, even though sodium hydroxide was employed as the primary alkali activator, which differs from the approach adopted in the present study.

3.4. Ultrasonic wave velocity (UPV)

UPV measurement has been considered as one of the indicators reflecting the quality of the concrete. Normally, the higher UPV values are associated with the concretes with the higher quality and thus the higher mechanical properties, particularly emphasized on compressive strength. In this study, the UPV of the hardened concretes was conducted and illustrated in Fig. 6. As shown in the figure, the UPV values were enhanced with ages due to the enhanced hydration of the binder. On all days, the UPV increased as the sulfate activator varied in the range of 1–5% but decreased as the sulfate amount reached higher values in the range of 7–10%. Such results obviously confirmed the previous discussion on the effect of sulfate addition on the improved structure and strength of the concrete. Additionally, the UPV result also indicated 5% of the sulfate activator was the optimum amount to produce the hardened SCC (N5 mix) with the highest 28-day UPV value of 3984 m/s and thus the highest quality. Consequently, coinciding with the dried density result, the obtained result of UPV provided additional evidence supporting the mechanical strength of the hardened SCCs modified with sulfate.

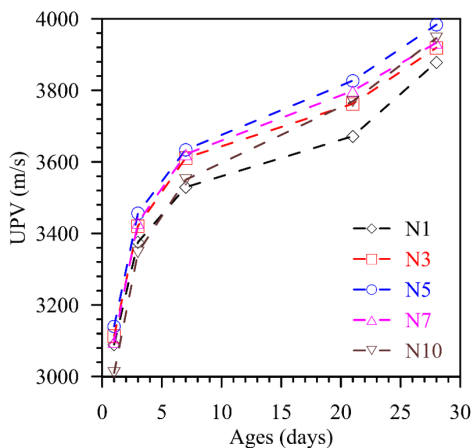


Figure 6. UPV values of the SCCs

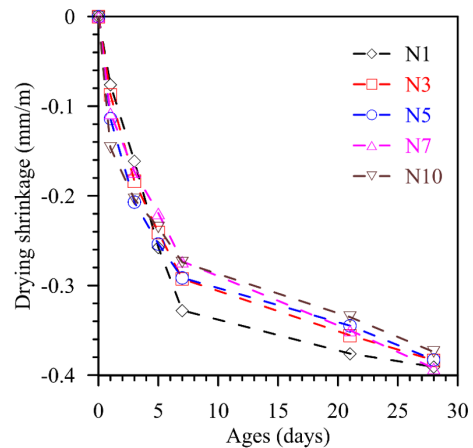


Figure 7. Drying shrinkage of the SCCs

3.5. Drying shrinkage

Drying shrinkage has been normally applied for estimating the volume stability of the material using cementitious binder. The lower drying shrinkage is associated with the specimen with less volume

changed and thus less vulnerable to crack issues induced by the shrinkage strain. In this study, the drying shrinkage of the hardened concretes is shown in Fig. 7. Accordingly, the drying shrinkage values varied in the ranges of 0.08–0.15 mm/m, 0.17–0.22 mm/m, 0.22–0.26 mm/m, 0.32–0.38 mm/m, and 0.37–0.4 mm/m at 1, 3, 5, 21, and 28 days. Generally, the increase in sulfate addition reduced the drying shrinkage of the hardened SCC, particularly as 10% of sulfate was used. A comparison showed that at 28 days, the drying shrinkage of the hardened concrete reduced at 4.3% as the sulfate amount increased from 1% to 10%. Such a result was possibly attributed to generation of expansive hydrates, particularly emphasized on ettringite crystals. The obtained experimental result obviously paid a promising way for improving resistance of the concretes to crack problems by Sproperly adjusting the sulfate dosage.

3.6. Water absorption

The water absorption serves as a durability performance indicating condensation level of microstructure and thus resistance of the concrete to chemical penetrating attack. The higher condensed structure of the concrete has been related to the lower water absorption of the sample. In this research, the 28-day water absorption of the hardened concretes is illustrated in Fig. 8. According to the figure, the changed amount of sulfate in the range of 1–10% induced the water absorption of the hardened concrete varied in a corresponding range of 4.8%–6.9%. The increased amount of sulfate in the range of 1–5% induced the hardened SCC with significant improvement on the water absorption. But continuous increment of the sulfate amount in the range of 7–10% increased the water absorption of the hardened concrete. 5% of the sulfate activator was the optimum amount due to the lowest value of the water absorption. Apparently, such results effectively supported the improvement on the mechanical strengths of the hardened concretes due to proper adjustment on the sulfate amount. In this study, as compared with the SCC containing the lowest amount of sulfate, i.e., 1%, the SCC with optimum amount of sulfate, i.e., 5%, had the water absorption remarkably reduced up to 30.7%.

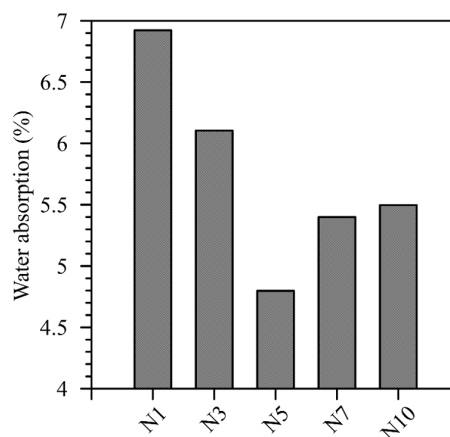


Figure 8. Water absorption of the SCCs

4. Conclusions

The influence of sodium sulfate activator, varied in the range of 1–10% by mass, on the performance of sulfate-activated slag-based self-compacting concrete (SCC) was investigated. The experimental findings indicated that satisfactory fresh properties were achieved when the sulfate content was controlled below a threshold of 5% to prevent setting loss. The mechanical strengths and durability, assessed by ultrasonic pulse velocity (UPV) and water absorption, improved within the range of 1–5% sulfate activator but declined as the dosage increased beyond 5%. Accordingly, 5% sulfate activator was identified as the optimum dosage. At this level, the sulfate-activated slag SCC achieved 28-day compressive and flexural strengths of 28.5 MPa and 3.9 MPa, respectively, together with a UPV of 3984 m/s and a minimum water absorption of 4.8%. With respect to dimensional stability, increasing the sulfate activator content up to 10% effectively reduced drying shrinkage, with the lowest 28-day value of 0.374 mm/m observed at 10%. Nevertheless, given the limited scope of the experimental

data, further investigations are required to optimize the activator dosage with respect to shrinkage behavior. Moreover, advanced microstructural analyses are essential to provide mechanistic insights that support the macro-scale performance trends reported in this study.

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