

DEVELOPMENT AND EVALUATION OF SHORT-TERM STRENGTH AND DURABILITY CHARACTERISTICS OF AN ECO-FRIENDLY SULFATE-ACTIVATED BINDER

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Abstract

The potential use of locally sourced ground granulated blast-furnace slag (GGBFS) and commercial sodium sulfate (Na_2SO_4) in the development of an eco-friendly sulfate-activated binder (SAB) was investigated in this study. The influence of Na_2SO_4 contents (1.5, 3.0, 4.5, 6.0, and 7.5% by mass of GGBFS) as a crucial sulfate activator on mechanical strength and durability performance of the SAB samples was evaluated through the tests of compressive strength, porosity, chloride ion penetration, and ultrasonic pulse velocity. The correlations among these properties were also established and discussed. The experimental results show that the Na_2SO_4 content had significant influences on both the strength and durability of the SAB samples. The porosity and chloride ion penetration were reduced as increasing the Na_2SO_4 content. Meanwhile, increasing the Na_2SO_4 content increased the compressive strength and ultrasonic pulse velocity of the SAB samples. Furthermore, the test results point out that using 7.5% Na_2SO_4 provided a significant improvement in the mechanical strength and durability performance of the newly developed SAB.

Keywords: sulfate activated binder; sodium sulfate; ground granulated blast-furnace slag; compressive strength; durability.

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1. Introduction

The rapid development of construction around the world requires a large quantity of concrete. In recent years, about 4.4 billion tons of concrete have been produced annually and the demand for concrete is estimated to be continuously rising over 5.5 billion tons by 2050 [1]. Particularly, in Vietnam, more than 140 million m^3 of concrete was consumed in 2019 [2]. This leads to high demand for Portland cement (PC), a very common material that has been used for the construction industry in most of the countries so far. In 2020, Vietnam was ranked in the top 3 leading cement producers

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globally by countries, 96 million metric tons of cement was produced in the country whilst the first ranking China produced over half of the world's cement with 2200 million metric tons of cement [3]. Nevertheless, cement production causes unexpected problems for the environment, which has received much attention in recent years. In detail, producing PC emits a high amount of carbon dioxide into the environment [4, 5] as the progress of making 1 ton of PC discharges about 1 ton of carbon dioxide [6]. In addition, the production process of PC consumes a large amount of energy (about 1800 MJ for every ton of clinker [7] and about 3500 MJ for every ton of PC produced [8]). Therefore, the use of more eco-friendly materials has been considered as an alternative to the PC with consideration of sustainability and conservation of the environment. For this purpose, pozzolanic materials, i.e. fly ash and ground granulated blast-furnace slag (GGBFS), could be considered as either a partial or full replacement for PC to reduce energy consumption and CO₂ emission [9, 10].

GGBFS, a primary by-product obtained from the iron-making process, is widely used for partial replacement of PC in the development of various construction materials. The effectiveness of utilizing GGBFS has been proved through many studies and practical applications [10–12]. In Vietnam, the amount of GGBFS is increasing every year. Five million tons of GGBFS were produced in 2018 and the GGBFS quantity is predicted to reach 10 million tons in 2025 [13], indicating a promising application of GGBFS in the eco-cement industry. In blend with PC, GGBFS can react with calcium hydroxide, which is generated from the hydration of PC in water to form some hydration products. However, the amount of GGBFS for replacing PC in eco-cement is limited due to the slow development of early strength [10]. Besides, researchers have pointed out that GGBFS can be activated by using chemical activators, hence much attention has been given to alkali-activated slag (AAS) systems in recent studies [10–12, 14].

Although AAS has shown promising performance for replacing PC based on specific results from previous researches, it is still limited in commercial application due to the use of unnatural availability and cost- and energy-consuming activators such as sodium silicate and sodium hydroxide [11]. From this point of view, therefore, a cost-effective sodium sulfate (Na₂SO₄), which is naturally found as thenardite and mirabilite was considered as an environmentally friendly activator [11]. Hence, Na₂SO₄ is a potential activator for the development of sodium sulfate-activated binders (SAB) as a potential alternative for PC used in construction activities. In terms of SAB, the presence of Na₂SO₄ increases the SO₄²⁻ concentration in the mixture, leading to an increase in the formation of ettringite (AFt) [15]. As a result, the improvement and densification of the microstructure, as well as an enhancement of mechanical strength of the resulting material, are obtained [12]. On the other hand, the formation of AFt is considered to be beneficial for the encapsulation of hazardous wastes such as heavy metals [16] and potentially problematic radionuclides [12]. Tan et al. [17] suggested that SAB might be a promising material for application in nuclear power plants that Vietnam has an interest to build in near future.

It can be seen from the literature that the combination of Na₂SO₄ and GGBFS in the preparation of SAB has been interested in recent studies. Rashad et al. [10] reported that the compressive strength of SAB was dependent on both the GGBFS fineness and the activator content. Experimental results show that increasing GGBFS fineness from 2500 to 5000 cm²/g resulted in an increment of 28-day compressive strength by 51.7%. Besides, for a given GGBFS fineness, when the concentration of Na₂SO₄ increased from 1% to 3% (Na₂O equivalent), the compressive strength of the SAB was found to be slightly increased [10]. In addition, Mobasher et al. [11] reported that the GGBFS samples activated by 3.9% Na₂SO₄ (by weight of GGBFS) could gain a compressive strength value of 17 MPa after 30 days. So far, most of the previous studies have investigated the influence of Na₂SO₄ dosage

and GGBFS fineness on compressive strength, workability, and setting time of the SAB samples [12]. However, evaluation of durability performance of the SAB samples (i.e. porosity, chloride ion penetration (CIP), and ultrasonic pulse velocity (UPV)) was limited in these studies.

Therefore, to fulfill the gaps in the literature regarding durability performance of the eco-friendly SAB, the primary objective of the present study was to investigate the changes in compressive strength, porosity, CIP, and UPV of the SAB samples prepared with different activator contents. To create the SAB samples, GGBFS was used as a primary precursor while Na_2SO_4 was used as an activator with different contents of 1.5, 3.0, 4.5, 6.0, and 7.5% (by weight of GGBFS). Then, the short-term mechanical strength and durability of the SAB samples were investigated through the tests of compressive strength, porosity, CIP, and UPV. Furthermore, the correlations among these properties were established and discussed. A controlled cement paste sample was also prepared for comparison purposes.

2. Materials and experimental methods

2.1. Materials

Raw materials used for the preparation of SAB samples in this study included type-PCB40 cement supplied by Insee Vietnam (Fig. 1(a)), a commercial GGBFS supplied by Hoa Phat Group (Fig. 1(b)), and an industrial chemical type of Na_2SO_4 powder (99% purity) supplied by VMC Group (Fig. 1(c)). The Na_2SO_4 powder with a density of 2.66 g/cm^3 was used as a sulfate activator. The physical properties and chemical compositions (determined by X-ray fluorescence analysis) of both cement and GGBFS are shown in Tables 1 and 2, respectively.

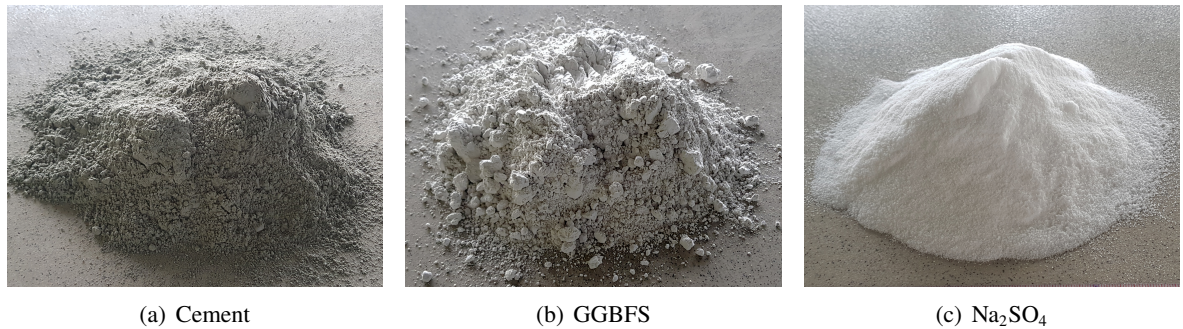


Figure 1. Raw materials used in this study

Table 1. Physical properties of cement and GGBFS

Materials	Specific gravities	Strength activity index at 28 days (%)	Blaine fineness (cm^2/g)	Compressive strength at 28 days (MPa)	Initial setting time (min)	Final setting time (min)
Cement	2.99	100	4020	56	160	190
GGBFS	2.85	96	4500	-	-	-

Table 2. Chemical compositions of cement and GGBFS

Materials	Chemical compositions (% by mass)						
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	SO ₃	Others
Cement	23.5	6.0	3.7	2.0	59.9	0.4	1.1
GGBFS	36.6	13.7	0.3	7.7	39.2	0.2	2.3

2.2. Mixture proportions

As mentioned in the scope of the study, to investigate the effect of the Na₂SO₄ content on the mechanical strength and durability performance of the SAB samples, six mixtures were designed. The material proportions for each mixture are shown in Table 3. The C100 mix used only cement and was considered a control mixture (for comparison purposes). Meanwhile, the other five mixtures used different Na₂SO₄ contents of 1.5, 3.0, 4.5, 6.0, and 7.5% (by weight of GGBFS) to activate the GGBFS and create the SAB samples, denoted as mixes N1.5, N3.0, N4.5, N6.0, and N7.5, respectively. Based on the preliminary trials in the laboratory, a water-to-powder (*w/p*) ratio of 0.3 was fixed for all mixtures.

Table 3. Mixture proportions of SAB samples

Mix IDs	<i>w/p</i>	Cement (kg/m ³)	GGBFS (kg/m ³)	Na ₂ SO ₄ (kg/m ³)	Water (kg/m ³)
C100	0.3	1575	0	0	473
N1.5		0	1524	23	457
N3.0		0	1511	45	453
N4.5		0	1498	67	449
N6.0		0	1486	89	446
N7.5		0	1473	111	442

2.3. Sample preparation

The procedures for the preparation of the SAB samples used in this study can be briefly described as follows: The raw materials were firstly prepared based on their mixture proportions as given in Table 3. Prior to being used, the Na₂SO₄ powder was well dissolved in water. The GGBFS was then mixed with the solution of water and Na₂SO₄ for 5 minutes to obtain a homogeneous mixture. The mixing time was kept the same for the C100 mixture. Right after that, the fresh mixture was poured into the molds with different dimensions to prepare the samples for each test program, which will be mentioned in the test method section. After shaping, all of the samples were placed in the laboratory for 24 hours, then de-molded and cured in water until the designated testing ages.

2.4. Test methods

In the present study, the mechanical strength and durability of the SAB samples were evaluated through the tests of compressive strength, porosity, CIP, and UPV. The compressive strength test (see Fig. 2(a)) was conducted at 1, 3, 7, and 28 days of curing age following the test procedures as described in point 9.2 of the TCVN 6016:2011 [18]. It is noted that the preparation procedures of the

SAB samples for the compressive strength test did not follow TCVN 6016:2011 (see Section 2.3). The porosity of the SAB samples was determined at the age of 28 days using the cubic samples of $50 \times 50 \times 50$ mm with the test procedures as step-by-step described by Nguyen *et al.* [19]. The cylindrical SAB samples of 100 mm in diameter and 50 mm in thickness, which were prepared by cutting the $\varnothing 100 \times 200$ mm sample at 28 days age, were used for the CIP test (see Fig. 2(b)) in accordance with the guidelines of ASTM C1202 [20]. The test of UPV was performed at the age of 28 days using cylindrical samples of $\varnothing 100 \times 200$ mm. In this study, a digital ultrasonic concrete tester from MATEST S.p.A. (see Fig. 2(c)) was used and the test procedures as described by ASTM C597 [21] were followed for determining the UPV values of the SAB samples.

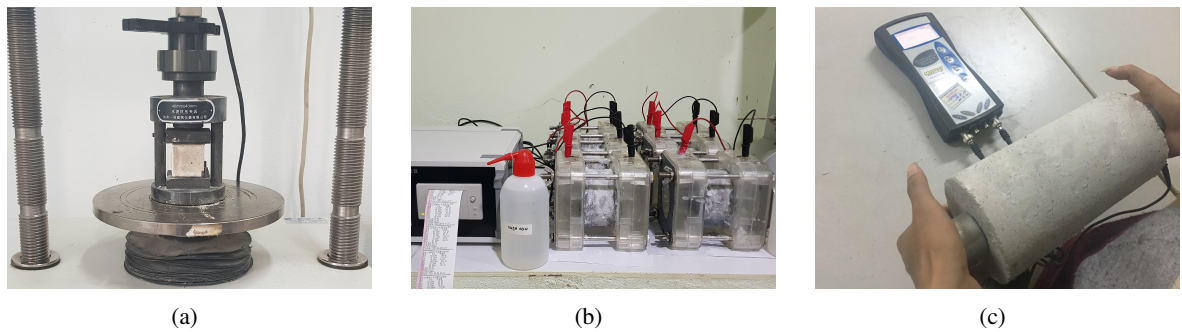


Figure 2. Apparatus used for the tests of (a) compressive strength, (b) CIP, and (c) UPV of the SAB samples

3. Results and discussion

3.1. Compressive strength

The development in compressive strength of the SAB and the control samples up to 28 days is presented in Fig. 3. It is clear to observe that compressive strength increased along with curing time corresponding to the increase of Na_2SO_4 content. Among the SAB samples, the highest strength value of 35.15 MPa was obtained at the N7.5 sample at 28 days. It attained 67% compared to the strength value of the control sample (52.46 MPa). The N1.5 sample exhibited the lowest compressive strength value of 18.86 MPa, which equals 35.95% of the compressive strength value of the C100 sample. Therefore, it can be concluded that higher Na_2SO_4 content provided a sufficient environment for the GGBFS activation, leading to a growth in the mechanical strength of the SAB samples. The enhancement of compressive strength could be explained by the increase in the formation of Aft due to the rise of sulfate concentration, resulting in the enrichment and compaction of the microstructure, and thus subsequently increasing the SAB's strength [12].

The development in compressive strength of the SAB samples can be evaluated through the slope of the curves. It was observed from Fig. 3 that the rate of strength development of the SAB samples

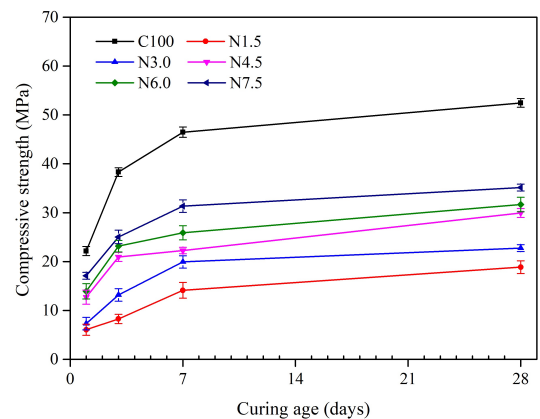


Figure 3. Compressive strength of SAB samples

was lower than that of the control sample in the first two periods of 1 – 3 days and 3 – 7 days. In the third period (7 – 28 days), the rate of strength development of the SAB samples was comparable to that of the control samples (about 0.3 MPa/day as calculated from the strength data). Although the SAB samples gained a lower strength than the control samples, they are still appropriate for utilization as a common construction material [22] whilst its compressive strength could achieve more than 20 MPa at 28 days in case of using Na_2SO_4 content in ranges of 3.0 – 7.5%.

3.2. Porosity

Porosity is one of the vital parameters that affect the strength and durability of cement-based or other binder-based materials [23]. Previous studies have reported that a lower porosity resulted in higher strength for paste and concrete [23, 24]. Generally, the durability of a resulting material is in good association with its porosity as porosity allows the movement and retention of water and other substances inside the resulting material.

In this study, the porosity values of the SAB and the control samples at 28 days are measured as shown in Fig. 4. In which, the C100 sample registered the lowest porosity value of 18.81% among the investigated samples. Whereas, the SAB samples with different Na_2SO_4 contents achieved the porosity values in the ranges of 20.31 – 23.92%. The obtained data also indicate that increasing the Na_2SO_4 content resulted in decreasing the porosity of the SAB samples. As aforementioned, the presence of higher Na_2SO_4 contents promotes the formation of Aft and other hydration products, leading to the denser microstructure [12], and thus reducing the porosity of the SAB samples. The results from Figs. 3 and 4 also indicate that the samples with a higher compressive strength had a lower porosity value. To confirm this trend, the correlation between porosity and compressive strength of the SAB samples was made and discussed further in the next section.

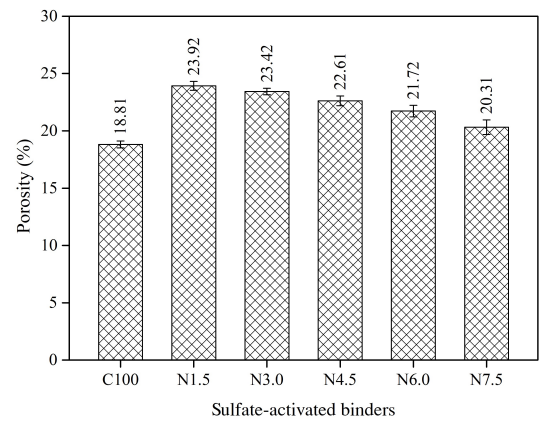


Figure 4. The porosity of SAB samples

3.3. Chloride ion penetration

CIP is a decisive parameter to evaluate the durability of the materials exposing to a severe environment such as a marine environment. Fig. 5 represents the CIP of the SAB and the control samples after 28 days of curing. First of all, the CIP of the SAB samples was inversely proportional to the content of Na_2SO_4 used to activate the GGBFS. Since the Na_2SO_4 content increased 1.5%, the CIP values of the SAB samples declined about 6 – 15%. For instance, the SAB sample with 7.5% Na_2SO_4 exhibited a decline of CIP value by approximately 35% compared to the sample activated with 1.5% Na_2SO_4 . Remarkably, at the same w/p ratio, the N7.5 sample showed a comparable ability to resist chloride ions to the C100 sample. Except for the N1.5 sample, all the other SAB samples can be classified as low chloride penetration [19]. This result is in good agreement with the results of porosity and compressive strength of the SAB samples as mentioned in previous sections. The SAB samples owning high compressive strength possessed low porosity [25, 26]. Consequently, the low porosity led to low penetration of chloride ions. Notably, the reduction of CIP with the increase of Na_2SO_4 content was much steeper than the reduction of porosity, as exhibited in Fig. 4.

In addition to porosity, the CIP strongly depends on the chloride binding capacity of the paste. Chloride ions can be bound by physical and chemical binding [27, 28]. Previous studies indicated that the main hydration products of SAB samples were mainly calcium-silicate-hydrates (C-S-H), AFt, monosulfate (AFm), and trialuminum hydrates (TAH). While C-S-H accounts for not only the strength of SAB but also the physically bound chloride, the AFt, AFm, and TAH are mainly responsible for chemically bound chloride. Furthermore, the high content of MgO in GGBFS is also a factor that greatly impacts the penetration of chloride ions. In addition, paste hydrated from slag cement with high content of MgO normally had a high content of the layered double hydroxide phase (LDH) [29, 30], which can bind chloride chemically [28, 31]. Therefore, the reduction of CIP as shown in Fig. 5 was attributable to not only the refinement of pore structure in the SAB due to more GGBFS being activated but also the chloride binding of activated products.

3.4. Ultrasonic pulse velocity

UPV measurement is a non-destructive method, which is commonly used to assess the homogeneity, cracks, or defects that existed in concrete. The mechanical properties of concrete can be evaluated via UPV results. In this study, the UPV values of SAB and control samples at the age of 28 days are shown in Fig. 6. The increase of Na_2SO_4 content resulted in higher UPV. In detail, since the Na_2SO_4 increased from 1.5% to 3.0%, the UPV rose approximately 9%. After that, the rising rate of UPV for each 1.5% increase of Na_2SO_4 content happened to be significantly lower (about 1.8 to 4.3%). It is found that the SAB sample activated with 7.5% Na_2SO_4 (N7.5) expressed the greatest UPV amongst the SAB samples and was approximately 19.6% higher than that of the N1.5 sample. Besides, the control sample showed much greater UPV than the SAB samples. It should be reminded that the UPV is proportional to the density of the samples [32]. The increase of Na_2SO_4 content possibly improved the activation of GGBFS, forming more hydration products. Therefore, the pores in the samples using high Na_2SO_4 content were filled, resulting in a denser structure and higher UPV.

3.5. Correlations among the properties of SAB samples

To evaluate the evolution of the compressive strength versus porosity of the SAB samples, the relationship between these two parameters is presented in Fig. 7. It could be observed that compressive

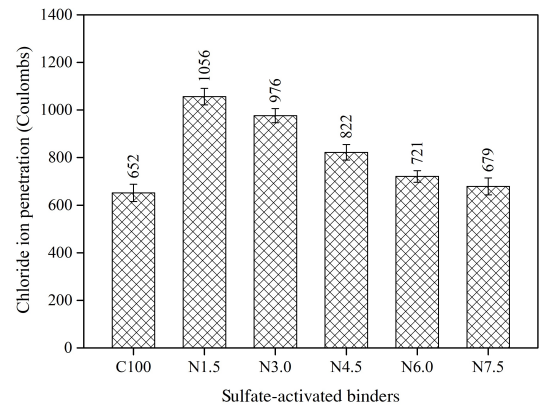


Figure 5. Chloride ion penetration of SAB samples

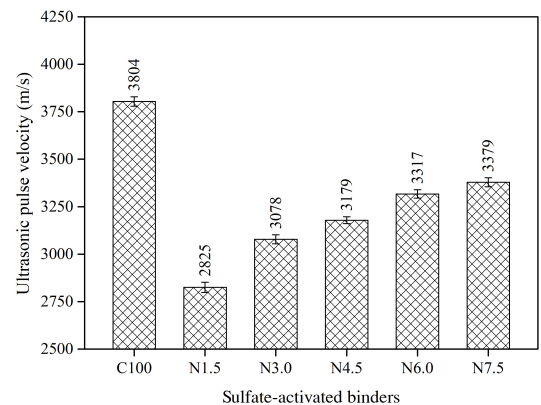


Figure 6. Ultrasonic pulse velocity of SAB samples

strength evolved inversely proportional to the porosity via a linear equation of $y = 158.8 - 5.8x$ with a good correlation coefficient R^2 of 0.91. Thus, the higher the compressive strength, the lower the porosity of the samples. This finding is in good agreement with the results of previous studies [32, 33].

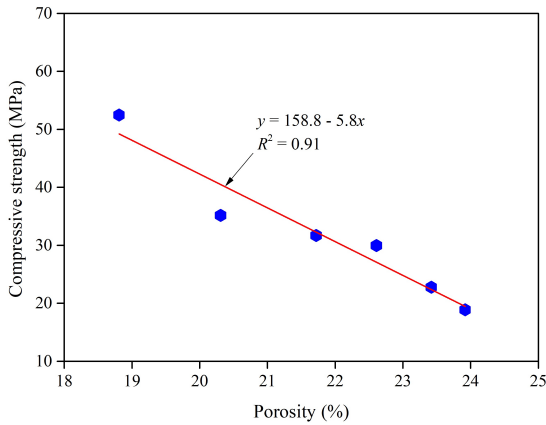


Figure 7. Relationship between compressive strength and porosity of SAB samples

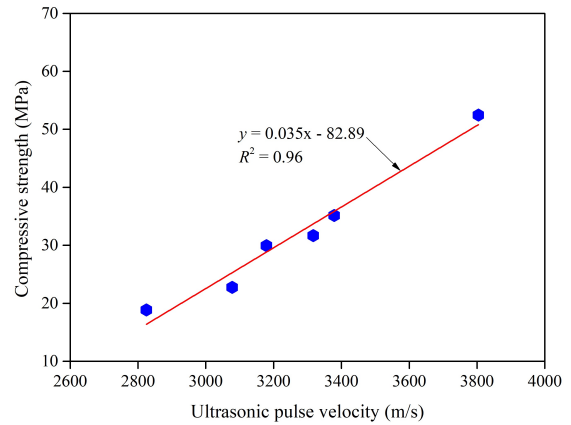


Figure 8. Relationship between UPV and compressive strength of SAB samples

The relationship between compressive strength and UPV of the SAB samples is presented in Fig. 8. It could be observed that the compressive strength of the SAB samples increased proportionally to UPV via a linear equation of $y = 0.035x - 82.89$ ($R^2 = 0.96$). In other words, the higher UPV values were associated with the higher compressive strength of the SAB. The relationships between compressive strength and UPV as well as between compressive strength and porosity expressed a coherent trend for cement-based materials [32, 33].

In addition, the relationship between the UPV and the porosity of the SAB samples is displayed in Fig. 9. An inversely linear relation between these two parameters with a high correlation coefficient ($R^2 = 0.91$) was observed. As above mentioned, the UPV is proportional to the density of the sample. Thus, higher porosity in the SAB samples led to a less dense structure [12] and consequently resulted in lower compressive strength of samples.

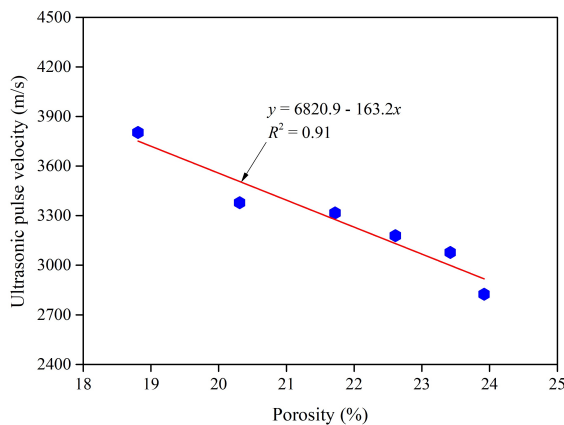


Figure 9. Relationship between UPV and porosity of SAB samples

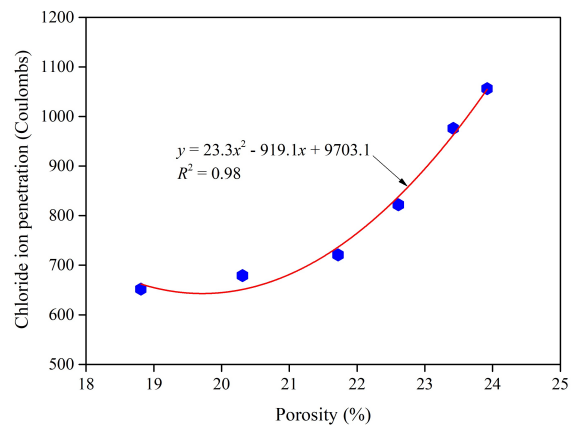


Figure 10. Relationship between CIP and porosity of SAB samples

Fig. 10 displays the relationship between the CIP and the porosity of the SAB samples. Since the

porosity was inferior, the CIP insignificantly varied. However, the CIP increased abruptly afterward. It is found that the relationship between these two parameters was nonlinear progress. As the previous discussion, the transport of ions in samples strongly depends upon not only the porosity but also the chloride binding capacity, which is primarily determined by the hydration products of the binder materials [28, 34, 35].

4. Conclusions

This study investigates the possibility of using a commercial Na_2SO_4 and locally sourced GGBFS in the development of an eco-friendly SAB. The mechanical strength and durability performance of the binders were evaluated using compressive strength, porosity, CIP, and UPV tests. Based on the experimental results, the following conclusions can be drawn:

- The compressive strength values of the SAB samples at 28 days were recorded in the ranges of 18.9 – 35.2 MPa, whereas, that of the control sample at the same age was 52.5 MPa. The increasing trend in compressive strength of the SAB samples was observed as increasing the Na_2SO_4 content. Although the SAB samples exhibited a lower strength than the cement paste, they are still suitable for use in construction as a common construction material.
- An increase in the Na_2SO_4 content led to the reduction in porosity and CIP and an increasing in UPV of the SAB samples. Under the technical aspect, 7.5% of Na_2SO_4 was suggested as a suitable content for the preparation of the good quality SAB samples with the values of compressive strength, porosity, CIP, and UPV at 28 days were 35.2 MPa, 20.3%, 679 coulombs, and 3379 m/s, respectively.
- The good correlations among the properties of the SAB samples increased the confidence in the application of the newly developed SAB samples in real practice. However, further investigations in terms of long-term durability, cost-efficiency, and microstructural characteristics of this binder type should be performed to widespread its application in the construction industry.

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