PROPERTIES OF SULFATE-ACTIVATED BINDER INCORPORATING VARIOUS FLY ASH-GROUND GRANULATED BLAST-FURNACE SLAG MIXTURES

CÁC TÍNH CHẤT KỸ THUẬT CỦA CHẤT KẾT DÍNH SULFATE HOẠT HÓA ĐƯỢC CHẾ TẠO TỪ CÁC HÀM LƯỢNG KHÁC NHAU CỦA TRO BAY VÀ XỈ LÒ CAO NGHIỀN MỊN

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Abstract - Finding new eco-binders as an alternative to conventional Portland cement in construction activities has recently attracted many researchers worldwide. The feasibility of activating blended fly ash (FA)-ground granulated blast-furnace slag (GGBFS) by commercial Na_2SO_4 to produce sulfate-activated binder (SAB) was introduced in this study. The SAB samples were prepared with different FA/GGBFS weight ratios of 5/95, 10/90, 15/85, 20/80, and 30/70. Results show that the SAB mixtures exhibited better flowability with more FA inclusion. Before 7 days, the mechanical strength of the SAB samples declined with increasing FA content. However, the samples with 20 and 30% FA exhibited higher strength values at 28 days. Water absorption of the SAB samples was in the ranges of 12.29 – 14.11%. Besides, the inclusion of more FA was beneficial in reducing the drying shrinkage of the SAB samples. The FA/GGBFS ratio of 30/70 was recommended for producing SAB for green and sustainable construction.

Key words - Sulfate-activated binder; fly ash; ground granulated blast-furnace slag; sodium sulfate; setting time; compressive strength

Tóm tắt - Việc nghiên cứu chất kết dính mới thân thiện hơn với môi trường để thay thế cho xi măng Poóclăng truyền thống trong các hoạt động xây dựng đã thu hút nhiều nhà nghiên cứu trên thế giới. Tiềm năng của việc hoạt hóa hỗn hợp tro bay (FA)-xỉ lò cao nghiền mịn (GGBFS) bằng Na_2SO_4 công nghiệp để sản xuất chất kết dính sulfate hoạt hóa (SAB) được giới thiệu trong nghiên cứu này. Các mẫu SAB được chuẩn bị với các tỷ lệ FA/GGBFS (theo khối lượng) là 5/95, 10/90, 15/85, 20/80 và 30/70. Kết quả cho thấy hỗn hợp SAB thể hiện khả năng chảy tốt hơn với hàm lượng FA cao hơn. Trước 7 ngày, cường độ của các mẫu SAB giảm khi hàm lượng FA tăng. Tuy nhiên, cường độ của các mẫu SAB chứa 20 và 30% FA có xu hướng tăng lên ở 28 ngày tuổi. Độ hút nước của các mẫu SAB ở 28 ngày dao động từ 12,29% đến 14,11%. Bên cạnh đó, sử dụng nhiều FA mang lại hiệu quả tích cực trong việc giảm độ co khô của các mẫu SAB. Tỷ lệ FA/GGBFS = 30/70 được khuyến nghị để sản xuất SAB cho mục đích xây dựng bền vững.

Từ khóa - Chất kết dính sulfate hoạt hóa; tro bay; xỉ lò cao nghiền mịn; natri sulfate; thời gian đông kết; cường độ chịu nén

1. Introduction

Cement is known as the most common binder used in the construction industry. However, it was reported that the manufacturing of cement produces a large amount of CO_2, and the annual value of carbon emission accounted for 5-8% of global, and this large amount of carbon emission gives a big challenge to the sustainable development of the world [1, 2]. Thus, it is needed to find a solution to reduce the use of cement, a new material namely alkali-activated material (AAM) has been investigated as a new cementitious material for cement replacement [3, 4]. It was reported that AAM reduces CO_2 emission by up to 75% because of the difference in preparation, material design as well as the calculation method of CO_2 emission [5–7].

Adesina and Kaze indicated that AAMs produced similar performance in comparison with normal Portland cement concrete [8]. Nevertheless, one limitation of AAMs is the application of large-scale and commercial applications using conventional activators. In general, the conventional activators of AAMs commonly include sodium hydroxide (SH) or sodium silicate (SS) [9–11]. These conventional activators do not exist in nature and are also quite expensive with high-energy consumption as well as CO_2 emission due to processing [8]. It was also reported that the CO_2 footprint of AAMs is strongly dependent on the type, content, and

concentration of the activator [12]. As a result, the usage of these activators would increase the overall cost as well as the environmental impact [13, 14]. In addition, these conventional activators (SH and SS) bring a negative effect on fresh and some hardened characteristics of AAMs [15, 16]. For instance, the use of a SS activator has been found to produce lower workability and higher shrinkage, thus AAMs using that activator is only suitable for specific application [15, 16]. The investigation of new activators with low environmental impact is vital to mitigate the CO_2 emission of AAMs.

Over the years, the development of AAM has indicated that alternative cheaper, and more sustainable activators can be used for conventional activators [17–19]. Some alternative activators for AAM are lime, sodium carbonate, and sodium sulfate (Na_2SO_4, NS). The use of NS as an activator could considerably reduce the CO_2 emissions of AAM because NS can be easily attained from natural minerals (i.e., thenardite) without a complicated synthesis [20, 21]. Previous studies indicated that NS could activate slag to form calcium-silicate-hydrate (C-S-H) gel and ettringite [22, 23], but the strength evolution of NS-activated slag is slow due to low initial pH. Some previous studies used limestone and silica fume to enhance the compressive strength of NS-activated slag [20, 24]. Besides, it was reported that the CO_2 emission of fly ash (FA) is much

lower than that of slag, thus FA was partially added to the binder of NS-activated fine slag (ground granulated blast-furnace slag (GGBFS)) to reduce CO_2 emission [25]. A previous study found that 20% FA addition in NS-activated GGBFS reduced compressive strength at 3 days, but the 28 day-compressive strength was improved [1].

Based on the above literature, it is accepted that there are limited studies on NS-activated GGBFS with FA addition. Thus, this study is aimed to examine the properties of NS-activated GGBFS and FA blends. Both fresh properties (workability and setting time) and hardened properties (compressive and flexural strengths, water absorption, and drying shrinkage) of the sulfate-activated binder (SAB) samples were extensively investigated.

## 2. Materials and experimental programs

2.1. Materials

The SAB samples were prepared using GGBFS, FA, and NS while grade-40 Portland cement was used to prepare the control mixture for comparison. The specific gravities and chemical compositions of cement, GGBFS, and FA are shown in Table 1.

Table 1. *Characteristics of cement, GGBFS, and FA*

Cementitious materials	Cement	GGBFS	FA
Specific gravity	2.99	2.85	2.22
Chemical composition (wt.%)			
SiO_2	23.5	36.6	59.2
Al_2O_3	6.0	13.7	26.7
Fe_2O_3	3.7	0.3	6.1
MgO	2.0	7.7	0.9
CaO	59.9	39.2	1.1
SO_3	0.4	0.2	0.1
Others	1.1	2.3	3.9

The scanning electron microscope (SEM) images and X-ray diffraction (XRD) patterns of these materials are presented in Figures 1 and 2, respectively. On the other hand, it is noted that NS used was an industrial chemical that was commercially available in the market in form of white powder with a specific gravity of 2.66.

(a) Cement   *(b) GGBFS*

(c) FA
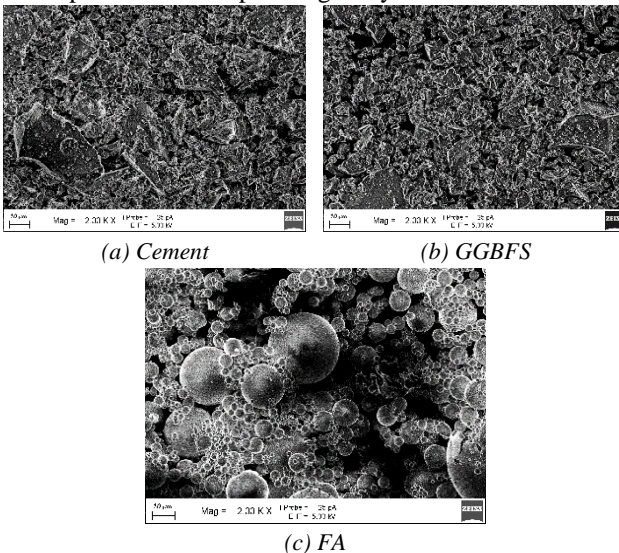
Figure 1. *SEM images of cement, GGBFS, and FA particles*
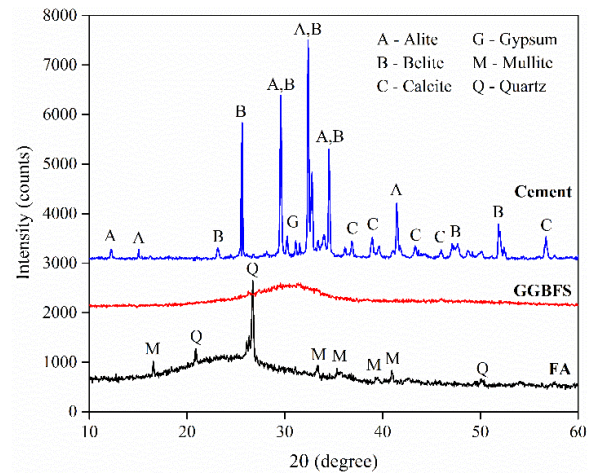
Figure 2. *XRD patterns of cement, GGBFS, and FA*

2.2. Mix design and proportions

In this study, based on the preliminary trials in the laboratory, five SAB mixtures were prepared with various proportions of GGBFS and FA (see Table 2) to investigate the properties of the binder.

Table 2. *Mix proportions of SAB samples*

Mix ID.	Material proportions (unit: kg)				
	Cement	GGBFS	FA	NS	Water
C100	1575.1	0	0	0	445.8
F05S95	0	1403.6	73.9	88.7	443.3
F10S90	0	1322.4	146.9	88.2	440.8
F15S85	0	1242.1	219.2	87.7	438.4
F20S80	0	1162.7	290.7	87.2	436.0
F30S70	0	1006.4	431.3	86.3	431.3

In detail, five SAB mixtures were prepared with FA/GGBFS weight ratios of 5/95 (F05S95 mix), 10/90 (F10S90 mix), 15/85 (F15S85 mix), 20/80 (F20S80 mix), and 30/70 (F30S70 mix). Besides, the only Portland cement sample (C100 mix) was also prepared for comparison. Based on the preliminary trials, a water/powder (including cement, GGBFS, FA, and NS) ratio of 0.28 was fixed for all mixtures.

2.3. Samples preparation and test methods

Table 3. *Experimental details*

Items	Sample size (mm)	No. of sample	Sample's age (day)	Standard reference
Flowability	-	-	After mixing	ASTM C230
Setting time	-	-	After mixing	ASTM C191
Water absorption	$50 \times 50 \times 50$	3	7, 28	ASTM C1403
Flexural strength	$40 \times 40 \times 160$	3	3, 7, 28	ASTM C348
Compressive strength	$40 \times 40 \times 40$	3	3, 7, 28	ASTM C349
Drying shrinkage	$25 \times 25 \times 285$	3	1, 3, 7, 14, 28	ASTM C596

To prepare the SAB samples, all of the raw materials were firstly prepared based on Table 2. NS was then well dissolved in water to create an activator solution and the

mixing procedures were started. The GGBFS and FA were mixed in a laboratory mixer for 1 min. Activator solution was then gradually added to the mixer and mixing continued for 5 min to obtain a homogeneous mixture (the same for the C100 mix). Right after mixing, the fresh SAB mixtures were tested for flowability and setting time. Then, the SAB samples were prepared for testing hardened properties including water absorption, flexural strength, compressive strength, and drying shrinkage. Experimental details for all experiments are summarized in Table 3.

3. Results and discussion

3.1. Flowability and setting time

The result of the flowability of fresh SAB mixtures is presented in Table 4. It can be seen that all of the SAB mixtures registered flow diameter values within the range of 230 ± 10 mm. Test results showed that the flowability of the fresh SAB mixtures slightly increased with increasing FA content in the mixture. This could be attributed to the spherical shape of the FA particles (see Figure 1c), which reduces frictional forces, enhances lubrication between particles in the mixture, and thus increases the mixture's flowability [1].

Table 4. *Properties of fresh SAB mixtures*

Mix ID	FA content (%)	Flow diameter (mm)	Initial setting (min)	Final setting (min)
C100	0	230	188	247
F05S95	5	220	157	217
F10S90	10	220	174	297
F15S85	15	230	220	370
F20S80	20	230	263	437
F30S70	30	240	387	569

Regarding the setting time, it can be seen that the initial setting times (IS) of all mixtures were more than 2 hours, with the values ranging from 157 to 387 minutes. In which, the IS of the reference mixture (C100 mix) was approximately 188 minutes. The IS of the SAB mixtures containing 5% and 10% FA were 31 and 14 minutes shorter than that of the C100 mixture, respectively. Both the IS and final setting times (FS) of the SAB mixtures containing 15, 20, and 30% FA were found to be increased considerably. For instance, the IS and FS of the F15S85 (15% FA) were 220 and 370 minutes, respectively while the respective IS and FS of the SAB mixtures with 20% and 30% were 263 and 437 minutes and 387 and 569 minutes. As a result, the inclusion of FA leads to an increment in setting time, which could be attributed to the presence of higher CaSO4 in the mixture with higher FA content as stated in the previous studies [26, 27].

3.2. Water absorption

The water absorptions of different SAB mixtures at 7 and 28 days are shown in Figure 3. For all mixtures, the water absorption of the samples at 28 days was reduced in comparison to that of the samples at 7 days. This reduction is attributed to further cement hydration as well as the pozzolanic reaction of FA. Besides, it can be seen that the water absorptions of SAB mixtures were larger than those of the reference mixture. In detail, the water absorption value of the SAB mixture at 7 days ranged from 14.62 to 18.09%; While at 28 days, these water absorption values varied from 12.29 to 14.11%. Whereas, the C100 mixture registered water absorption values at 7 and 28 days of 10.37 and 9.24%, respectively.
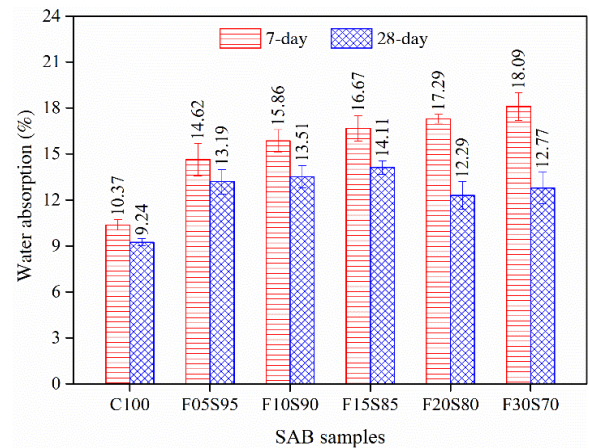

Figure 3. *Water absorption of SAB samples*

At 7 days, the gradual increase in water absorption could be attributed to two following reasons: (1) The FA particle has a porous structure (see Figure 1c), resulting in a higher water absorption rate in FA particles as compared to that of cement particles and (2) both FA and GGBFS are supplementary cementitious materials, which normally have a slower pozzolanic reaction in comparison with cement hydration reaction [28]. As a result, the amount of C-S-H formed from the pozzolanic reaction maybe not be as much as the cement hydration products. Thus, the micro-structure of the SAB samples was less dense as compared to the reference mixture, and the higher amount of FA leads to a higher water absorption rate. However, at 28 days, the water absorption increased when the FA content raised from 0 to 15% (F15S85 mix), then the water absorption decreased when FA content increased to 20% (F20S80 mix) and 30% (F30S70 mix). The lower water absorption levels of the F20S80 and F30S70 samples at 28 days as compared to the other mixtures could be attributable to a higher reaction rate of FA and GGBFS, resulting in a denser microstructure and consequently reducing water absorption of these samples.

3.3. Flexural strength

Figure 4 shows the flexural strength of SAB samples for up to 28 days. For all mixtures, the flexural strength grew up with curing time from 3 to 28 days primarily due to continuous chemical reactions in the samples. Besides, the flexural strength was generally decreased with the increment of FA content for the cases of 3 and 7 days. At 28 days, the flexural strength of the SAB samples fluctuated around 5 MPa and the difference in flexural strength value between the SAB samples with different FA content was insignificant; while the flexural strength of the reference samples at the same age was 8.3 MPa. The decrease in the flexural strength due to the increment of FA content at 3 and 7 days was due to the porous microstructure of FA particles (see Figure 1c) as well as the slow reaction of FA and GGBFS particles, as reported in the previous studies [1,29,30]. At 28 days, when the FA content varies from 5 to 30%, the flexural strength was changed insignificantly. This is attributed to the further hydration as well as the

pozzolanic reaction of both FA and GGBFS in a sulfate environment, generating more hydration products (i.e., C-S-H gel), which improve the microstructure and maintain the flexural strength of the SAB samples [1].
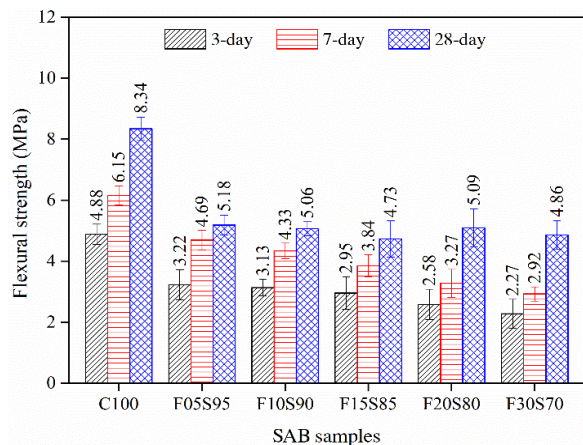

Figure 4. *Flexural strength of SAB samples*

3.4. Compressive strength

The compressive strength development of the SAB samples until 28 days is shown in Figure 5. For all samples, the compressive strength increased with curing time from 3 to 28 days due to cement hydration (in C100 samples) and pozzolanic reaction (in SAB samples). It can be observed that the compressive strength of the SAB samples was lower than that of the reference mixture for all curing ages. This is because FA and GGBFS are pozzolanic materials, which normally have a slower chemical reaction in comparison with cement hydration. This finding agreed well with the result found in the previous studies [29–31]. In addition, it can be seen that at 3 and 7 days, the compressive strength decreased when FA content was increased from 5 to 30%. The increment of FA content was equivalent to the reduction of GGBFS amount, which reduced the amount of CaO. As a result, this reduction of CaO content leads to a decrease in compressive strength, as reported in the previous study [29].
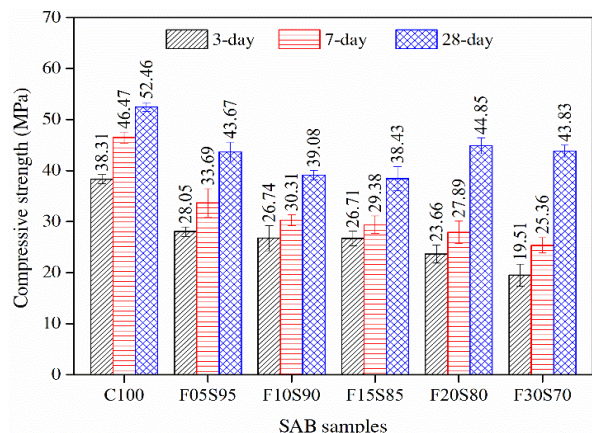

Figure 5. *Compressive strength of SAB samples*

On the other hand, at 28 days, the compressive strength of the SAB samples decreased when the FA content raised from 5 to 15%. In contrast, when the FA content was increased from 20 and 30%, the compressive strengths were higher than those of the samples containing 5 to 15% FA content. Specifically, the compressive strength values of the

samples with 5, 10, and 15% FA were 43.67, 39.08, and 38.43 MPa, respectively, while the compressive strength values of the samples with 20 and 30% FA were 44.85 and 43.83 MPa, respectively. Similar to the flexural strength development pattern (presented in Section 3.3), the highest compressive strength gained for the mixtures with 20% FA (F20S80 mix) can be explained as follows: The replacement of GGBFS with FA increases the actual concentration of the activator solution, and the high concentration solution promotes the activation of GGBFS to produce hydration products sooner [1]. This can compensate for the reduction in mechanical strength caused by GGBFS replacement. In addition, at a later age (at least 28 days), the pozzolanic reaction of FA could also contribute to the increment of the mechanical strength of the SAB [1].
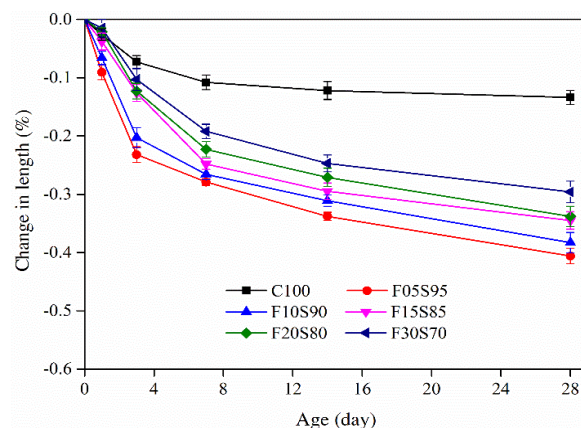
3.5. Drying shrinkage

Figure 6. *Drying shrinkage of SAB samples*

The drying shrinkage of SAB samples is presented in Figure 6. Within 28 days of curing, the drying shrinkages of all mixtures were in the range of 0 to -0.5%. The change in length of the reference mixture was lowest (-0.134%), followed by the SAB samples containing 30%, 20%, 15%, 10%, and 5% FA content with the respective change in length values of -0.296%, -0.338%, -0.345%, -0.383%, and -0.406%. The drying shrinkages of the SAB samples were higher than that of the reference sample (C100). As above mentioned, this is primarily due to the slow reaction of FA and GGBFS in SAB samples, which leads to an increment of drying shrinkage of SAB samples. Besides, it can be seen that for the SAB samples, an increase in the amount of FA from 5 to 30% results in a lower change in length. This is consistent with the result found in the previous studies [1, 32]. As already discussed in the previous section (Section 3.3), the replacement of GGBFS with FA increases the concentration of the activator solution, promoting the activation of GGBFS to produce more hydration products, resulting in lower drying shrinkage.

4. Conclusions

This study evaluates the possibility of using FA and GGBFS to prepare a new SAB. From the experimental results, some main remarks can be drawn as follows:

(i) All of the SAB mixtures had flow diameter values in the ranges of 230 ± 10 mm. Also, the SAB mixtures including more FA content exhibited better flowability.

Similarly, the prolonged setting time of the SAB mixtures was recorded for samples containing more FA.

(ii) Both the flexural and compressive strengths of the SAB samples at 3 and 7 days declined with increasing FA content. At 28 days, the flexural and compressive strength of the SAB samples were in the respective ranges of 4.73 – 5.18 MPa and 38.43 – 44.85 MPa. It is found that the SAB's strength reduced when the FA content increased from 5% to 15%, while the sample's strength improved when the FA content increased from 15% to 30%.

(iii) Water absorption of the SAB samples had a similar variation pattern as the strength development and the water absorption levels of all SAB samples at 28 days were in the ranges of 12.29 – 14.11%. The SAB sample incorporating 20% registered the lowest water absorption level (12.29%).

(iv) The drying shrinkage of SAB samples was higher than that of the reference sample. This study found that the inclusion of more FA was beneficial in reducing the drying shrinkage of the SAB samples.

Although the F20S80 samples gave better results, based on the experimental results, with the high FA consumption and acceptable properties, the FA/GGBFS ratio of 30/70 (the F30S70 mixture) was suggested for producing SAB for green and sustainable construction. However, the long-term performance of the SAB samples should be considered in future studies to support the applicability of this new SAB in real practice.

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