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EARLY STRENGTH ENHANCEMENT OF HIGH-VOLUME FLY ASH MORTAR USING ACCELERATING ADMIXTURES

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Abstract. High-volume fly ash mortar is considered a "green" material, a sustainable development solution for the construction industry, in which the fly ash replacement Portland cement content is over 50%. However, this material has the major disadvantage of slow strength development, especially at an early age, which limits its applicability. To overcome the above disadvantage, the individual or combined addition of lime $Ca(OH)_2$, silica fume, and/or accelerating admixtures including sodium thiocyanate (NaSCN), diethanolamine (DEA), and glycerol (Gly), etc. have been interested recently. In this study, to enhance the early strength of mortars with fly ash content to replace 60÷80% of Portland cement, a three-component accelerating admixture of NaSCN, DEA, and Gly (2:1:0.5 by the mass) was used with different ratios of 0.21÷0.49 % by the total powder mass, combined with a reasonable amount of lime, silica fume supplement as presented in previous research. Experimental results showed that the optimal accelerating admixture content is 0.35% by weight of powder, combined with 6% silica fume and the additional amount of lime is 5%; 10%; and 15% corresponding to mortars using fly ash of 60%; 70% and 80%, respectively to improve early strength of mortars that are suitable for structural use (R3 ≥ 13MPa, 3 days old). In addition, the influence of the additives used on the initial setting time of high-volume fly ash mortars was also discussed.

Keywords: mortar, high-volume fly ash, lime, silica fume, accelerating admixture, strength.

1. INTRODUCTION

At present, in Vietnam, more than 25 million tons of coal ash are discharged each year mainly from power plants belonging to Vietnam Electricity, the Vietnam National Coal-Mineral Industries Group, Vietnam Oil and Gas Group, and other enterprises. If the amount of coal ash is not recycled, it is estimated the storage will reach 422 million tons by 2030 [1]. Currently, most of this ash is transported to the landfill outside the environment and if not used, in the long run, will adversely affect the surrounding environment and waste resources. Therefore, the research and manufacture of mortar and concrete with high fly ash content for construction works is essentially necessary. High-volume fly ash mortar meets the necessary criteria for a "green", "environmentally friendly" material, a sustainable development solution for the construction industry in which the replacement of Portland cement by fly ash is over 50% [2].

Because the content of Portland cement used in high-volume fly ash mortar is very little, there will be a lack of lime $Ca(OH)_2$ (which is the hydration product of cement generated) to participate in the pozzolan reaction with fly ash, as well as ensuring alkalinity to protect reinforcement [3]. The major disadvantage of this mortar is slow strength development, especially at an early age, which limits its applicability [4]. To promote early strength development for such mortars, in addition to the traditional approaches of using Portland cement with high C_3A content [5], lowering the water/powder ratio [6], etc., the reasonable addition of lime $Ca(OH)_2$ [7], silica fume [8], and/or the individual or combined application of chemical accelerating admixtures, i.e. sodium thiocyanate (NaSCN) [9], diethanolamine (DEA) [10], glycerol (Gly) [11], etc. have also been interested recently. According to the preliminary research results of the authors, when adding lime and silica fume with a reasonable ratio, the initial time setting, consistency of fresh mortar, and compressive strength of high-volume fly ash mortar were improved. Specifically, to achieve early age strength properties (3 and 7 days old) and improve setting time, the optimal lime addition content was determined to be 5%; 10%; and 15% by total binder mass corresponding to mortars using fly ash of 60%; 70% and 80%, respectively. Meanwhile, the reasonable rate of silica fume selection was 6% in all grades [12]. In addition, previous studies have shown that using a combination of chemicals can produce a higher acceleration effect on cement hydration than the single use of each chemical. Aggoun et al. [13] reported that the combination of $Ca(NO_3)_2$ with triethanolamine or tri-isopropanol amine accelerated the setting time and early strength for all the types of cement used in their research. Riding [14] presented that the 1-day strength of mortars using 30% fly ash replacement was increased by 28% when using a combination of calcium chloride and diethanol-isopropanolamine as additives. However, the combination of additives did not significantly affect the 07-day and 28-day compressive strength. Recently, Kien Hoang et al. [15] studied the effect of three-component admixture to enhance the early and late compressive strength of cement-fly ash mortar (cement/fly ash = 70/30) at both low and normal temperatures by increasing the degree of hydration of the cement. The three-component additive is made from a combination of NaSCN, DEA, and Gly in small dosages by the weight of the binder. Research results showed that the combination of these three-component additives contributes to enhanced acceleration performance compared to the ingredients when used individually. From the above literature review, it can be preliminary stated that the reasonable addition of lime and silica fume content, combined with the use of chemicals as accelerators for cement in mortars using high fly ash content is promising in providing high technical efficiency, and suitable for structural use (early strength gain, early/faster de-moulding, etc.). It also contributes to expanding the scope of application of this sustainable building material in practice.

In this study, in order to improve the early strength of mortars with fly ash content to replace 60÷80% of Portland cement, a three-component accelerating admixture of NaSCN: DEA: Gly (2:1:0,5 by mass [15]) was used with different ratios from 0.21 to 0.49% according to the powder weight, combined with a reasonable amount of lime, silica fume supplement (preliminary research results of the authors [12]). This experimental assessment is based on two important criteria, which are the initial setting time and compressive strength of the high-volume fly ash mortar compared with the control sample without using additional accelerating additives. The mortar with high fly ash content (replacing up to 80% of fly ash) that meets the minimum compressive strength at an early age (R3 ≥ 13MPa, 3 days old) and (R28 ≥ 40MPa, 28 days old) will make an important contribution in sustainable development, reducing CO_2 emissions by replacing Portland cement with fly ash, saving natural resources, and limiting environmental pollution on the impact of thermal power coal ash and construction industry using Portland cement. Moreover, with such required strength, it is suitable for the actual construction of civil works, or prefabricated components that need to be demoulded early and is equivalent to commercial products with the same compressive strength [16-19].

2. MATERIALS AND RESEARCH METHODS

2.1. Materials

The cement used in this research is Nghi Son PC40 with a density of 3.11 g/cm^3, in accordance with the technical requirements for Portland cement (TCVN 2682:2020). Type F Fly ash of Duyen Hai 1 thermal power plant (oxide content $SiO_2 + Al_2O_3 + Fe_2O_3$ = 86% > 70%) with a density of 2.28 g/cm^3, suitable as an admixture for concrete and cement according to TCVN 10302:2014.

The sand used is a mixture of 50% natural sand (Dong Nai River sand) and 50% crushed sand originating from Tan Cang quarry (Dong Nai province), with a density and fineness modulus of 2.65 g/cm^3 and 2.73, respectively, in accordance with the requirements for concrete sand (TCVN 7570:2006). Local tap water taken from the South Institute, Vietnam Institute for Building Materials is used as mixing water, following TCVN 4506:2012. Sika's polycarboxylate-based superplasticizer (Viscocrete 8650) is added to reduce high levels of mixing water and ensure the workability of the mortar, in class F according to TCVN 8826:2011.

In this study, in order to improve the compressive strength at an early age as well as the setting time for high-volume fly ash mortar, lime $Ca(OH)_2$, silica fume, and three-component accelerating admixture of NaSCN: DEA: Gly were added in reasonable amounts. The silica fume (Microsilica 940U) provided by Elkem Silicon Materials, is a highly active mineral additive, containing 93.8% SiO_2 and the density is 2.24 g/cm^3. Meanwhile, the lime used is hydrated lime, the chemical formula is $Ca(OH)_2$, containing 62.2% (CaO+MgO) and the density is 2.21 g/cm^3. In addition, accelerating admixture including sodium thiocyanate (NaSCN, density of 1.74 g/cm^3), diethanolamine (DEA, density of 1.09 g/cm^3), and glycerol (Gly, density of 1.26 g/cm^3) was applied in solution with the typical dosage recommended by [15] being 0.2%; 0.1% and 0.05% of powder mass, respectively (Figure 1). Specifically, it could be observed from the left image of Figure 1 that the particle size of fly ash is much larger than silica fume particles (middle image), and most fly ash particles possess a spherical smooth surface giving the characteristic properties of fly ash. Furthermore, the particle size of silica fume is much finer than that of fly ash, and the particle shape is quite diverse.

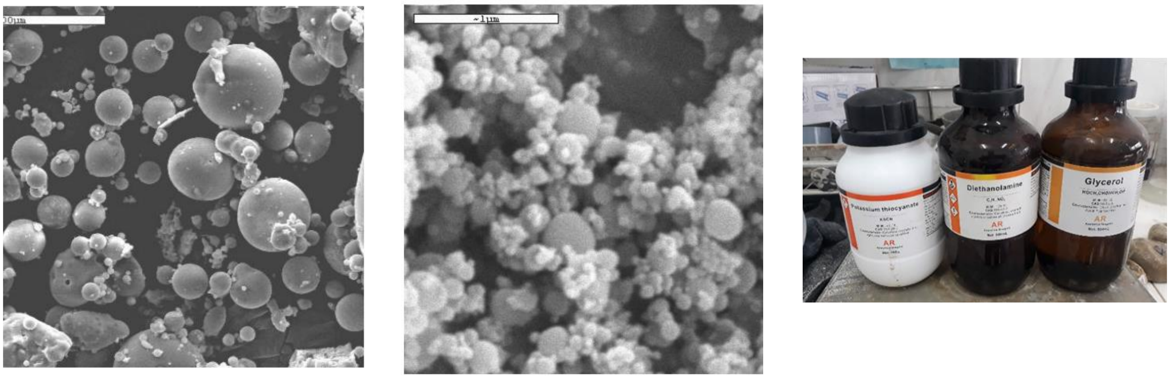

Figure 1. SEM image of fly ash (300x magnification) – left [12]; silica fume – middle; Three-component accelerating admixture – right (digital image by authors).

Table 1. Designed mix proportion of high-volume fly ash mortars using lime, silica fume and accelerating admixtures.

TT	**Designation**	**The ratio of material components**							**Binder**
		Cement	**Fly ash**	**Lime**	**Silica fume**	**NaSCN**	**DEA**	**Gly**	
		(kg)	(kg)	(%)	(%)	(%)	(%)	(%)	(kg)
1	T60H0	180	270			0	0	0	450
2	T60H0.21	180	270			0.12	0.06	0.03	450
3	T60H0.28	180	270	5	6	0.16	0.08	0.04	450
4	T60H0.35	180	270			0.20	0.10	0.05	450
5	T60H0.42	180	270			0.24	0.12	0.06	450
6	T60H0.49	180	270			0.28	0.14	0.07	450
7	T70H0	135	315			0	0	0	450
8	T70H0.21	135	315			0.12	0.06	0.03	450
9	T70H0.28	135	315	10	6	0.16	0.08	0.04	450
10	T70H0.35	135	315			0.20	0.10	0.05	450
11	T70H0.42	135	315			0.24	0.12	0.06	450
12	T70H0.49	135	315			0.28	0.14	0.07	450
13	T80H0	90	360			0	0	0	450
14	T80H0.21	90	360			0.12	0.06	0.03	450
15	T80H0.28	90	360	15	6	0.16	0.08	0.04	450
16	T80H0.35	90	360			0.20	0.10	0.05	450
17	T80H0.42	90	360			0.24	0.12	0.06	450
18	T80H0.49	90	360			0.28	0.14	0.07	450

In this study, the determination of the high-volume fly ash mortar compositions is according to Bolomey-Skramtaev's method, in which a theoretical calculation method combined with experimental tests, based on the "absolute volume" theory. i.e. the total absolute (completely solid) volume of materials in 1m3 of mortar is equal to 1000 litres. Based on the preliminary results obtained by the authors [12], the optimal ratio of silica fume content is 6% for all researched mixes, while the optimal dosages of lime are 5%, 10%, and 15% for the mixes with different replacement ratios of fly ash 60; 70; and 80%, respectively. The content of the three-component accelerating admixture has a relative ratio of 2:1:0.5 to each other, and the dosages used increase gradually from 0; 0.21; 0.28; 0.35; 0.42, and 0.49% of powder mass, as detailed in Table 1. Moreover, the mix proportion of mortars used in the study is designed with the following fixed parameters: water/powder ratio of 0.3 (powder = cement + fly ash + lime + silica fume); superplasticizer content using 1% of the total powder mass. Samples are symbolized in two parts: the first letter T - the sample using fly ash, H - the sample using a three-component accelerating admixture; the numbers 60; 70; and 80 followed by T is the percentage of replacing cement with fly ash, while the numbers 0.21; 0.28; 0.35; 0.42; 0.49 after H is the percentage of accelerating admixture used. For example, in sample T60H0.21, the content of lime, silica fume, and accelerating admixture was 5%, 6%, and 0.21%, respectively with fly ash replacing cement at 60%.

2.2. Research methods

In this study, the evaluation through two important criteria of the mixture both in fresh and hardened states, that is the initial setting time of fresh mortar (TCVN 3121-9:2003) and compressive strength of hardened mortars (TCVN 3121-11:2003) from 3 to 90 days compared with the control sample without using accelerating additives. From that, the optimal ratio of accelerating admixtures used is determined to increase the early strength of high-volume fly ash mortars at the replacement rates of cement by fly ash of 60, 70, and 80% by mass, together with the reasonable addition of lime and silica fume. Because the content of the three-component accelerating admixture has almost no influence on the flowability of the mortar, its effect is not presented in this paper.

3. EXPERIMENTAL RESULTS AND DISCUSSION

Besides the application of optimal dosages of lime and silica fume, the addition of three-component accelerating admixtures with concentrations from 0 to 0.49% by weight of powder affects the initial setting time and significantly changes the compressive strength of the mortars as shown in Figure 2 to 5.

3.1. Effect of three-component accelerating admixtures on the initial setting time of mortar

According to Figure 2, the initial setting time of mortar without accelerating admixture is 335; 350, and 365 minutes in mixes using 60; 70, and 80% fly ash replacing cement. When gradually increasing the content of accelerating admixtures from 0 to 0.28%, the setting time of the fresh mortar does not change significantly (<1.5%), especially for mortar mixture using 60% fly ash even to 0.35% additive content. Therefore, the addition of these chemicals needs to be high enough to activate the cement-fly ash binder, producing an increase in compressive strength. If the additive content continues to increase, the process of shortening the setting time of the mortar is stronger. For example, the time to the initial set is accelerated by 5-10 minutes (an average 2.4% time reduction) at a dosage of 0.35%. The setting time of mortar is

315; 325 and 340 minutes for mortar with a fly ash ratio of 60; 70 and 80% corresponding to the amount of accelerating admixture used is 0.49% (reduced by about 6.0, 7.1, and 6.8% respectively compared to the control mortar sample without using accelerator) as shown in Figure 2. This result is consistent with the previous study [15], because the heat development of the hydration process of the mortar sample when using the three-component complex admixture, has the highest efficiency in promoting the hydration rate of the C_3S and (C_3A + C_4AF) phases and is, therefore, more effective in shortening the setting time of high-volume fly ash mortars. In addition, it can be remarked that, within the scope of this study, the effect of three-component accelerators is more significant when the fly ash content replaces cement higher than 60%. This can be explained by the synergistic effect of the three-component accelerating admixture, where Glycerol is a polyol, each molecule has three hydroxyl groups (-OH) that contribute to the addition of $Ca(OH)_2$, especially affects the crystallization of ettringite and calcium hydroalumino-monosulfate, accelerates the consumption of sulfate ions [20] and also the pozzolanic reaction of fly ash proceeds further and completely.

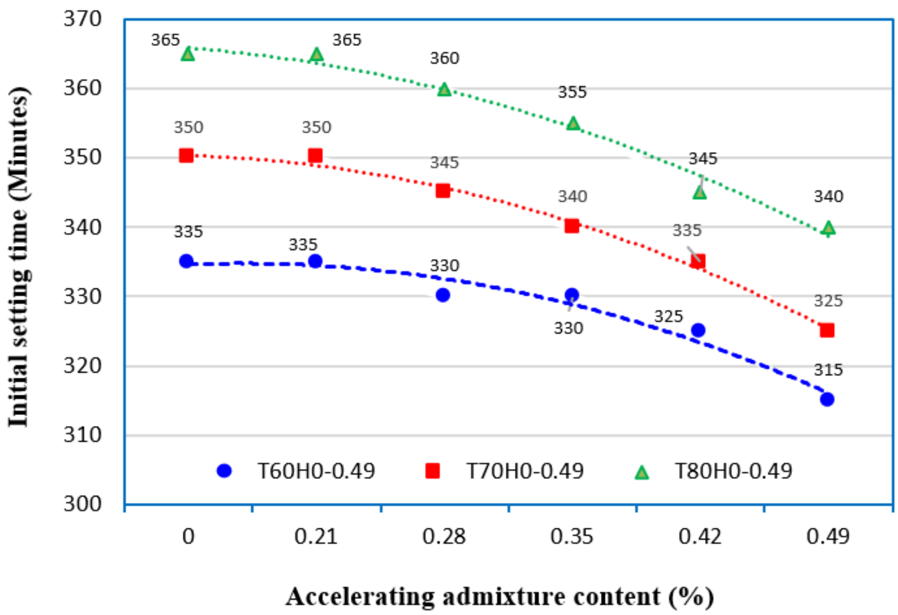

Figure 2. Effect of accelerating admixture on the initial setting time of mortar.

3.2. Effect of three-component accelerating admixtures on the compressive strength development of hardened mortar

The effect of accelerating admixture content on the compressive strength of mortar when replacing 60; 70 and 80% fly ash over time are shown in Figures 3, 4, and 5, respectively. Compressive strength at 3 days old of mortar without accelerating admixtures with the ratio of using fly ash to replace cement 60, 70, and 80% (T60H0, T70H0, and T80H0) are 28.1; 23.3 and 13.2 MPa, respectively, while those at 28 days age are 62.3; 58 and 42.3 MPa, respectively. The results showed that most of the compressive strength at all additive addition dosages (0.21 to 0.49%) in the study resulted in higher results than the control sample without using this additive at all different ages. However, the rate of increase in strength of mortar samples using accelerating admixtures at early ages (3 and 7 days) and late ages (after 28 days) is different. Typically, at 3 days old, the compressive strength of mortar samples of T60H0.21÷0.49 increased by 1.35; 4.20; 10.95; 12.02, and 13.80%, respectively compared with sample T60H0 (28.1 MPa) corresponding to the amount of accelerating additive of 0.21;

0.28; 0.35; 0.42 and 0.49%. Meanwhile, the improvement ranges of compressive strength of mortar samples T70H0.21÷0.49 and T80H0.21÷0.49 are 2.3-14.7% and 0-15.2%, respectively. Similarly, compressive strength at 7 days of the age of mortar using accelerators increased by 4-12.73%, 4.8-18.5%, and 2.5-26.2% with samples T60H0.21-0.49, T70H0.21-0.49, and T80H0.21-0.49, respectively. This can be explained by the synergistic effect of the ternary admixture contributing to the acceleration of hydration of the silicate phases, the intermediate phase of Portland cement, and the increased amount of AFm (especially calcium hemicarboaluminate hydrate) in hydration products. The increased formation of hemicarboaluminate consumes more calcium hydroxide and unreacted binders, and also at the same time produces more ettringite. This results in a less porous microstructure and thus higher mortar strength [15]. On the other hand, the 56-day age compressive strength of mortar with the ratio of using fly ash to replace cement of 60, 70, and 80% increased insignificantly, by the maximum rate of 3.89%; 8.1%, and 7.6%, respectively and almost insignificant changes (<6%) at 90 days. Similar trends were observed in previous studies of early strength enhancement for high-volume fly ash mortars using chemicals [13-15].

In addition, also on the above results, it could be recommended that the necessary content of a three-component accelerating admixture is 0.35% to obtain the highest compressive strength in most ages and mixtures with different ratios of fly ash to replace cement (Figure 3÷5). When the dosage used of three-component admixtures was low (up to 0.28%), the early strength at 3 and 7 days of mortar samples increased insignificantly, by a maximum rate of 6.6% and 13.2%, respectively. The compressive strength of mortar supplemented with 0.35% three-component accelerating admixture (T60H0.35, T70H0.35, and T80H0.35) is 31.2; 25.8 and 14.7 MPa, respectively at 3-day age (an increase of 10.8-11.4% compared to the control sample) and 47; 33.5 and 24 MPa, respectively at 7-day age (an increase of 11.73-21.8% compared to the control sample). With the further addition of the accelerator additive to the mixture, the compressive strength of the mortar over time is improved but not significantly (<5%).

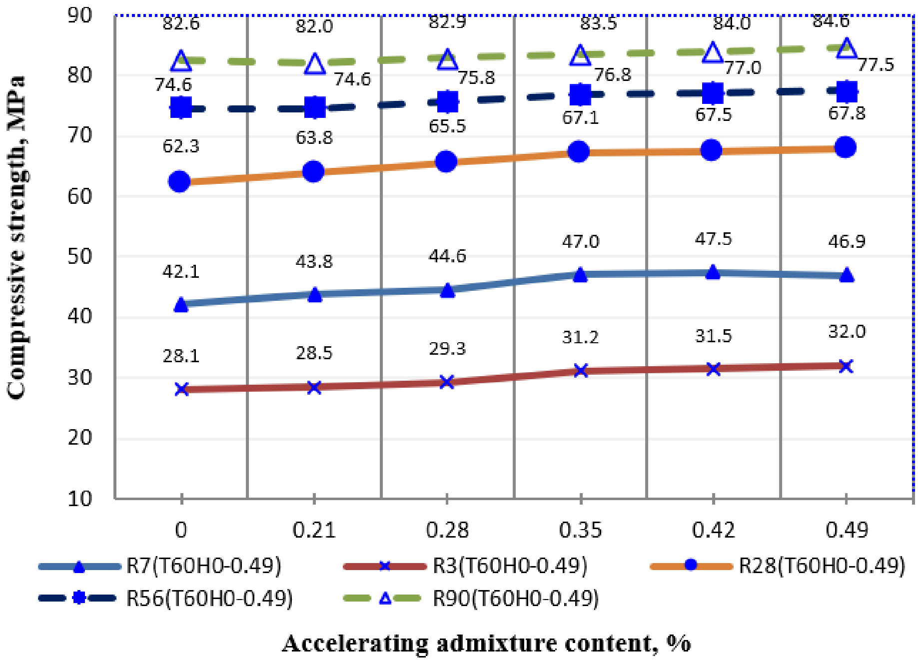

Figure 3. Effect of accelerating admixture on compressive strength of mortar when replacing 60% of fly ash.

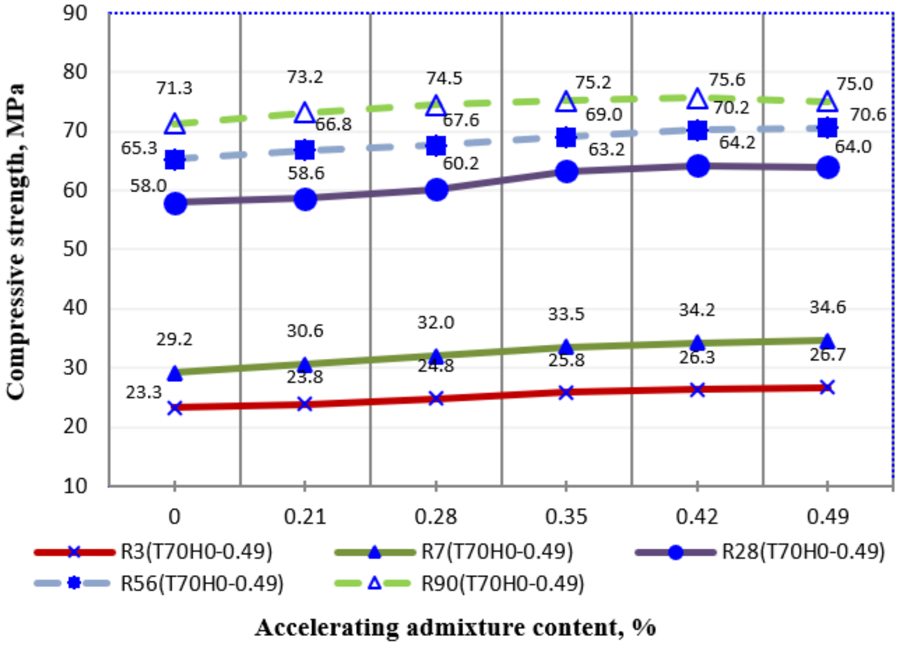

Figure 4. Effect of accelerating admixture on compressive strength of mortar when replacing 70% of fly ash.

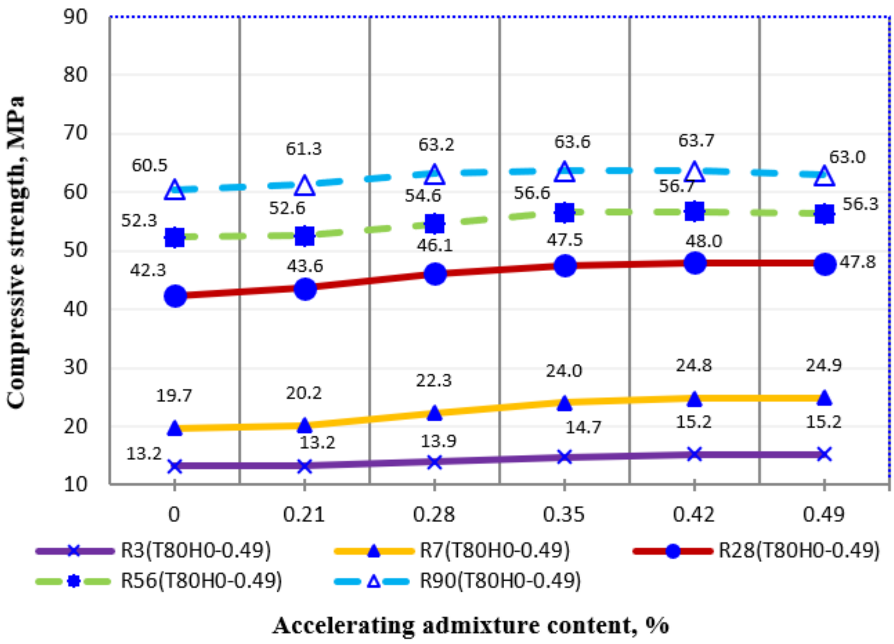

Figure 5. Effect of accelerating admixture on compressive strength of mortar when replacing 80% of fly ash.

4. CONCLUSION

From the above-obtained results, it is shown that when adding lime, and silica fume at a reasonable ratio, and simultaneously using the three-component accelerating admixture including NaSCN: DEA: Gly (2:1:0.5 by mass) with different ratios from 0.21÷0.49% of powder weight, the properties of initial setting time and compressive strength of mortars containing high-volume fly ash content was improved. The three-component accelerating admixture can be considered as a potential chloride-free, early setting and strength development accelerator for cement binders with high fly ash content.

However, the effect of setting time reduction of the mortar mixture when using accelerating admixtures will be significant when the fly ash content to replace cement is more than 60% and the accelerating additive dosage used is greater than 0.35%. In addition, the required ternary additive content is 0.35% for the compressive strength to reach the highest value on most days of age and in all mixtures with the use of fly ash to replace cement in this study. This is due to the synergistic effect of the three-component accelerating admixture which accelerates cement hydration and increases the amount of AFM (especially calcium hemicarboaluminate hydrate) in the hydration products. High-volume fly ash mortar (replacing up to 80% fly ash) meets minimum compressive strength at an early age ($R3 \geq 13MPa$, 3 days old) and ($R28 \geq 40MPa$, 28 days old). It can be found that the potential application range of this mortar is completely the same as that of normal commercial mortar for load-bearing concrete structures such as columns, floor beams, etc., or suitable for transporting mortar/concrete that requires a longer time and further distances than conventional mortar/concrete.

Although further research and development are still needed, the initial results of this study will make an important contribution to sustainable development, reducing CO_2 emissions by replacing Portland cement with fly ash. At the same time, it will make a great contribution in terms of technology for manufacturing materials using domestic raw materials, diversifying sources of raw materials to replace Portland cement by using waste in large quantities, and is one of the steps in the design and manufacture of materials to serve the needs of life and to solve the real problems of today's society.

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