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Effects of gypsum and phosphoric acid on the properties of sodium silicate-based alkali-activated slag pastes

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Abstract

In this paper, the effects of gypsum and phosphoric acid on the properties of sodium silicate-based alkali-activated slag paste were examined. Alkali-activated slag is recognized as a good performing binder. However, it suffers from fast setting and drying shrinkage. Phosphoric acid has been used as a retarder and gypsum has been used as a drying-shrinkage inhibitor in previous studies. However, it is not known what will happen if one adds both of them to alkali-activated slag. In this study, the effects of gypsum and/or phosphoric acid on the compressive strength development, setting time and drying shrinkage of alkali-activated slag were examined. Experimental results indicated that adding gypsum shortened the setting time, and at the same age, the compressive strength reached a higher value when the amount of used gypsum is higher. Drying-shrinkage decreased when the amount of used gypsum increased; however, when 0.82 M phosphoric acid was added in the activator with gypsum the results were somewhat different. With added phosphoric acid, adding more gypsum shortened the setting time as well. The compressive strength of specimens with added gypsum was lower than that of control specimens under 28 days. Drying-shrinkage increased with phosphoric acid as the amount of used gypsum increased.

Keywords: Alkali-activated slag; Gypsum; Phosphoric acid; Setting time; Drying shrinkage

1. Introduction

Alkali-activated slag (AAS) is a new type of binder used in concrete manufacturing. This binder, based on 100% slag plus activator, has been used in Eastern Europe, Scandinavia and China and was discussed in two extensive reviews [1,2]. This slag produced from industrial by-products, requires less production energy than ordinary portland cement (OPC) and is associated with low CO_2 emissions, which is recognized as the main cause of the so-called 'green house effect'. Alkali-activated slag concrete (AASC) has recently received much attention from the academic field. Many valuable research results on this material have been reported. AASC has been found to have some superior properties as compared to ordinary portland cement concrete (OPCC), namely, low hydration heat, high early strength and excellent durability in an aggressive environment [3–6]; however, many alkali-activated slag properties remain to be explored. For example, in a recent published paper, Collins and Sanjayan [7] reported that microcracks in AAS concrete increased from lack of curing moisture. A survey of the published literature showed that this new binder system has some serious problems such as rapid setting [8] and high drying shrinkage [9]. These problems must be resolved before it can be used in commercial practice.

It has been reported that the strength development of AASC depends on the type of activator and concentration [10,11]. Sodium silicate-based activator (sodium silicate and sodium hydroxide) was found to have the best strength development performance. However, the sodium silicate-based activator also produces rapid set. Phosphate [8] and malic acid [12] were used as retarders in previous studies. In engineering practice, a binder should have a reasonable setting time. This means that concrete or mortar made using a binder should have a setting time long enough to permit transport time for the concrete or mortar. However, it is not economical if the setting time is too long. The setting time (initial and

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final) [8] has been used to study the retarding effect of phosphate.

It has been reported that alkali-activated slag concrete has higher drying shrinkage than ordinary portland cement concrete does. Kutti et al. [13] measured the drying shrinkage of $NaOH + Na_2CO_3$-activated AASC over 12 months. The drying shrinkage of AASC was 1.82 times to that of OPCC when exposed to 59% relative humidity (RH). Malolepszy and Deja [14] and Hakkinen [15] reported that alkali-activated slag mortar exhibited 1.6 times the drying shrinkage of OPC mortars. Collins and Sanjayan [16] also found that AASC had 57% higher drying shrinkage than that of OPCC at 50% RH. The pore size distribution and the calcium silicate hydrate gel characteristics have a critical influence on the drying shrinkage [17,18]. Bakharev et al. [19] used gypsum to reduce the drying shrinkage of AASC.

In this paper, we studied the effects of gypsum and phosphoric acid on some properties of the sodium silicate-based alkali-activated slag paste (AASP), namely, the compressive strength development, setting time and drying shrinkage. We especially focused on the combined effects of gypsum and phosphoric acid.

2. Experimental scheme

The chemical composition and physical properties of the slag used are tabulated in Table 1. Blast furnace slag is a granulated product ground to a fineness of about 383 m^2/kg, with a particle size range of 5–75 μm. This material is neutral with a basicity coefficient of $K_b = (CaO + MgO)/(SiO_2 + Al_2O_3)$ equal to 0.93. The hydration modulus (HM) is defined as

$$HM = \frac{CaO + MgO + Al_2O_3}{SiO_2}. \tag{1}$$

The hydration modulus of slag should exceed 1.4 to ensure sound hydration properties. From Table 1, the hydration modulus of slag is 1.75.

Table 1
The chemical and physical properties of slag

The main chemical composition of slag (wt %)	SiO_2 (%)	33.87
	Al_2O_3 (%)	14.42
	Fe_2O_3 (%)	0.69
	CaO (%)	39.54
	MgO (%)	5.35
	SO_3 (%)	2.47
	Basicity coefficient $K_b = (CaO + MgO)/(SiO_2 + Al_2O_3)$	0.93
Physical properties	Specific gravity	2.90
	Ignition loss (%)	0.28
	Fineness (m^2/kg)	383

Sodium silicate has a chemical composition of $\%SiO_2 = 37.0$ and $\%Na_2O = 17.7$, and a mole ratio of 2.16. A sodium silicate-based activator was used. This means that the activator contains sodium silicate and sodium hydroxide. Because the activator was not our major concern, we fixed the activator composition with $Na_2O = 122$ g/l and $SiO_2 = 123$ g/l.

Phosphoric acid was chosen as the retarder. In a previous study [7], sodium phosphate was used as the retarder. The reason we adopted phosphoric acid instead of sodium phosphate is described as follows. Once sodium phosphate is used, it will affect the Na_2O concentration. In order to keep Na_2O concentration in the activator-liquid constant, we used phosphoric acid as a retarder. Various concentrations of phosphoric acid were adopted to determine the retarding effects of phosphoric acid. In this phase, the liquid/slag ratios of 0.44, 0.54 and 0.64 respectively, were used to examine how liquid/slag ratio affects the appropriate amount of retarder. The liquid/slag ratio is the weight ratio of the liquid phase including sodium silicate, sodium hydroxide and/or phosphoric acid over the solid phase, i.e., the slag.

In the followings, the liquid/slag ratio was kept at 0.54. Several dosages of gypsum were added into the binder system as a drying-shrinkage inhibitor. The gypsum used was plaster of Paris ($CaSO_4 \cdot 0.5H_2O$). To determine the setting time, 0%, 2%, 3%, 4% and 6% gypsums (by slag mass) were used. For comparison, an ordinary portland cement paste (OPCP) with the same *w*/*c* ratio (0.54) was made. All specimens were placed into standard curing conditions after demolding.

ASSP and OPCP properties including compressive strength development (liquid/slag = 0.54), setting time and drying shrinkage (liquid/slag = 0.54) were examined. The compressive strength tests were conducted following ASTM C109. The initial and final setting times were measured using the Vicat test following ASTM C191. The drying shrinkage was measured following ASTM C596. The focus was on the effects using phosphoric acid and gypsum together. For the drying shrinkage and compressive strength, the amounts of gypsum used were 0%, 2% and 4% by slag mass.

3. Results and discussions

3.1. Phosphoric acid retarding effect

The retarding effects of phosphoric acid on the setting times are shown in Figs. 1 and 2. It was observed that the retarding effect was not significant before some critical amount of phosphoric acid was used. As the critical amount of phosphoric acid was achieved, the setting time increased fast and immediately exceeded the reasonable region. This means that the retarding

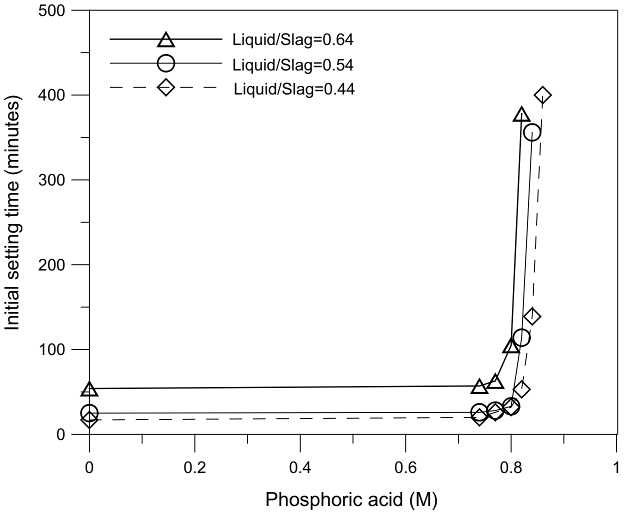

Fig. 1. Retarding effects of phosphoric acid on the initial setting time.

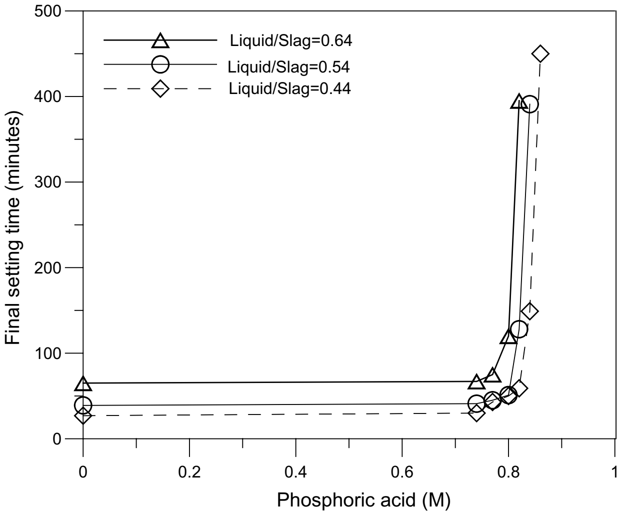

Fig. 2. Retarding effects of phosphoric acid on the final setting time.

effect was very sensitive to the amount of phosphoric acid. Excess phosphoric acid might retard the setting too much. Insufficient phosphoric acid, on the other hand, cannot retard the setting time to make the binder satisfy engineering requirements. For example, a minimum 45 min initial setting time for concrete transportation is needed in Taiwan. The appropriate amount of retarder depends on the type of retarder, the activator and its composition. Results from this research can only be used under the same conditions, i.e., phosphoric acid is used as the retarder with sodium silicate-based activator with $\%SiO_2 = 37.0$ and $\%Na_2O = 17.7$. Because only 0.82 M phosphoric acid has the best setting time performance for liquid/slag ratio of 0.54, this retarder concentration was used for other experiments. It can also be observed from these two figures that the difference between initial and final setting times was within 20 min. This result is very similar to that reported in Ref. [8]. Such a small difference might make the work of concrete finish impossible. To seek an appropriate retarder such that the rapid setting problem can be resolved and the difference between initial setting and final setting can allow concrete finish is a challenge in the future.

In addition, it can be found that a higher phosphoric acid concentration is required when the liquid/slag ratio is lower for reaching the same retarding effect. These observations relate to the mechanism of retarding. According to [8], the formation of $Ca_3(PO_4)_2$ was one of the reason for retrading effect in alkali-activated red mud slag paste. It has been pointed out [20] that the rapid setting of sodium silicate-based alkali-activated slag paste was due to a quick formation of CSH gel within several minutes. In [20], it was also mentioned that the slag hydration process must be considered with regard to the oxygen forms present in slag, double fixed in the $\equiv$Si—O—Si$\equiv$ bond, single fixed in $\equiv$Si—O—Me bonds, and “free” in =Me—O—Me— bond and the conditions of hydrate formation. The first destruction step under high concentration of OH^- can be described as the breaking of Me–O (Me$\equiv$Ca, Mg), Si—O—Si, Al–O–Al and Al–O—Si of the slag grain. Since the Me–O bonds are much weaker than Al–O and Si–O bonds, more Ca and Mg enter into water than Al and Si, and Si/Al-enriched layer forms quickly on the surface of slag [21,22]. The phase structure of the slag provides channels for OH^- ions to enter the bulk to complete the hydration process. Besides, the dispersed medium shows a high concentration of $[SiO_4]^{4-}$ from water glass, and the first step may correspond to the hydration and hardening of the water glass or reaction between $[SiO_4]^{4-}$ and Ca^{2+} dissolved from the surface of slag grain. Adding phosphoric acid will result in low soluble $Ca_3(PO_4)_2$, which correspondingly reduces the concentration of calcium ions. Consequently, the formation of CSH, which is known as the products for the alkali-activated slag system in the beginning, slows down. Such a retarding becomes noticeable only when most calcium ions were consumed by phosphoric acid. Following this logic, it can be said that when the liquid/slag ratio is smaller the concentration of calcium ions (remember they come from slag) is higher. Therefore, to reach the same retarding effect a higher phosphoric acid concentration is required.

3.2. Effects of gypsum

Gypsum was used to inhibit the drying shrinkage of ASSC as mentioned in the previous section. As shown in Table 2, adding gypsum reduced the setting times, both

Table 2
Setting times of AASP adding gypsum (liquid/slag = 0.54, SiO_2 = 123 g/l, Na_2O = 122 g/l)

	Initial setting time (min)	Final setting time (min)
Gypsum 0% (by slag mass)	25	39
Gypsum 2% (by slag mass)	21	32
Gypsum 3% (by slag mass)	19	27
Gypsum 4% (by slag mass)	17	25
Gypsum 6% (by slag mass)	10	19
Gypsum 0% (by slag mass) + 0.82 M phosphoric acid	114	128
Gypsum 2% (by slag mass) + 0.82 M phosphoric acid	98	111
Gypsum 3% (by slag mass) + 0.82 M phosphoric acid	87	98
Gypsum 4% (by slag mass) + 0.82 M phosphoric acid	78	89
Gypsum 6% (by slag mass) + 0.82 M phosphoric acid	61	74

initial and final. A higher gypsum dosage resulted in a less setting time. The reduction in setting time is explained as follows. First, heat was generated when the gypsum was added into liquid. According to thermodynamics, particles would own a higher free energy under a higher temperature such that the chemical reaction rate increased. Furthermore, additional calcium ions entered the liquid when the gypsum was used. As explained above, a higher concentration of gypsum resulted in a higher generation rate of CSH gel. Consequently, a faster setting was observed when the gypsum was added.

The main purpose of adding gypsum is to reduce the problem of ASSC drying shrinkage. It was observed that adding gypsum reduced the drying shrinkage as shown in Fig. 3. In this figure, OPCP represents ordinary portland cement paste with $w/c = 0.54$. When gypsum was not used to inhibit the drying shrinkage, the drying shrinkage of AASP was 3.2 times to that of OPCP when aged 7 days. This ratio reduced to 2.3 times at 14 days, 1.8 times at 21 days and 1.93 times at 28 days. This figure shows that the drying shrinkage for AASP had an increasing trend at 28 days while the drying shrinkage of OPCP already reached a stable value. It was reported that the drying shrinkage of AAS mortar and concrete was 1.6–2.1 times greater than that of OPC mortar and concrete. Our results indicated that the relative drying-shrinkage ratio between AASP and OPCP might be higher than the comparison between AAS and OPC mortars, or a comparison between AAS concrete and OPC concrete at an early age. This result comes from the volumetric stability provided by the aggregates. When a mortar or concrete is considered, aggregates inhibit the drying shrinkage. Paste is freer to shrink than mortar or concrete. It was also observed in this figure that the drying-shrinkage reduced by adding gypsum increased as the amount of gypsum was increased. At 7 days, no difference between the different AASP groups with and without gypsum could be found. However, at 21 days the drying shrinkage of AASP with gypsum reached a stable value and at 28 days the difference between the AASP groups with and without gypsum was apparent. An approximate 22% reduction in drying shrinkage at 28 days using 4% gypsum was found. A 16% reduction in drying shrinkage was found when using 2% gypsum. In Refs. [13,17,18,23], they have mentioned that the formation of moisture-sensitive silica gel contributes to the higher shrinkage of AASC. In Ref. [9], they reported that the higher shrinkage of AASC was also due to the pore size distribution. Pore sizes can be classified into four levels according to the International Union of Pure and Applied Chemistry (IUPAC) system [24]. The capillary pores comprise both of mesopores and macropores and constitute water filled space between original cement grains, whereas the micropores constitute part of calcium silicate hydrate gel component [9]. Shrinkage under practical conditions depends on loss of water from the mesopores and also the size of the macropores, which determine how easily water may be lost from the mesopores [18]. When pore water is lost, pore contraction occurs, i.e., overall volumetric contraction. This pore contraction is more apparent for mesopores than macropores because the water surface

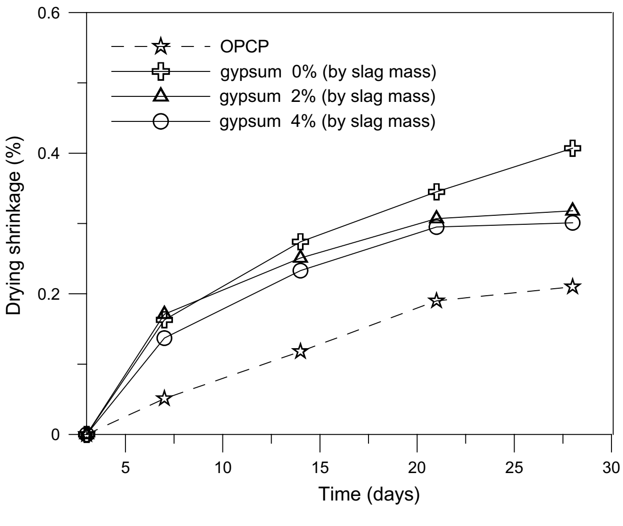

Fig. 3. Effects of gypsums on the drying shrinkage.

tension force is more significant in mesopores. Water is not easy to lost from micropores due to capillary force. As explained above, the pore size distribution becomes very important as studying the drying shrinkage. In the case of alkali-activated slag, the proportion of pores in the micropore size range tends to be higher than OPC [25,26]. The number of pores within the capillary range, however, is lower than OPC [26]. Shi [27] found OPC to have a continuous pore size distribution within 5- to 1200-nm pore sizes whereas sodium silicate-activated slag contained pores either less than 10 nm or greater than 200 nm. Collins and Sanjayan [9] showed that AASP had a much higher proportion of pore sizes within the mesopore limits than OPCP. The proportion of pores within the mesopore classification ranges from 74% to 82% for AASP compared with 24.7–36.4% for OPCP [9]. They [9] then concluded that the higher total volume of mesopores in AASP could explain the higher magnitude shrinkage of AASC. The formation of more CSH gel using the gypsum in the AASP makes the volume for micropores increase. Consequently, the drying shrinkage is reduced.

The compressive strength development is illustrated in Fig. 4. It can be found that AASP had a much higher compressive strength than OPCP at all ages. AASP, no matter gypsum was used or not, reached 80% of the 28 days compressive strength within 1 day. The AASP (without gypsum) had a compressive strength of 71.7 MPa at 28 days while OPCP reached 29.3 MPa only at the same age. Adding gypsum to AASP further increased the gap between the AASP and OPCP compressive strengths. With more added gypsum, a higher compressive strength was obtained for AASP. At 28 days, AASP with 4% added gypsum had a compressive strength 2.7 times to that of OPCP.

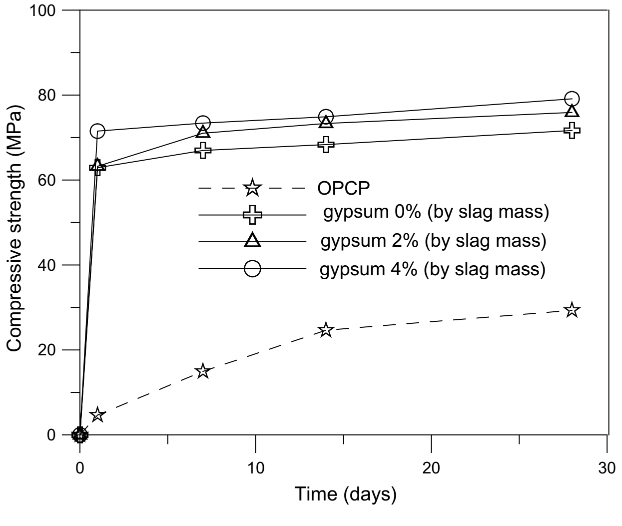

Fig. 4. Effects of gypsums on the compressive strength.

3.3. *Effects of a combined use of gypsum and phosphoric acid*

To our best knowledge, the combined gypsum and phosphoric acid effects have not been explored in the literature. It is then interesting to explore if using gypsum and phosphoric acid together can concurrently reduce the drying shrinkage and increase the setting time. In this series, 0.82 M phosphoric acid was used as a retarder because it had the best setting time performance. Gypsums 0%, 2% and 4% were added by slag mass.

The initial and final setting times for AASP using gypsum and phosphoric acid are illustrated in Table 2. It was observed that adding gypsum and phosphoric acid together blocked the retarding effect of the phosphoric acid. As the amount of gypsum increased, the setting time decreased. Within the range we studied, although the retarding effect in setting time reduced, yet the setting time is still longer than the control specimen (i.e., AASP without the phosphoric acid and gypsum). The reason is explained as followings. When the gypsum was added with the phosphoric acid, the concentration of calcium ions increased. Consequently, the existing phosphoric acid concentration is not enough to maintain the same retarding effect. Remembering that the retarding mechanism of phosphate acid is the formation of calcium phosphate as explained earlier. When more calcium ions entered into the solution due to the gypsum, in order to reach the same retarding effect a higher concentration of phosphate acid then was required. This effect became more apparent when the amount of gypsum was increased. That is there remain extra calcium ions that can be consumed by the hydration reaction of CSH gel; thus, the setting time reduces.

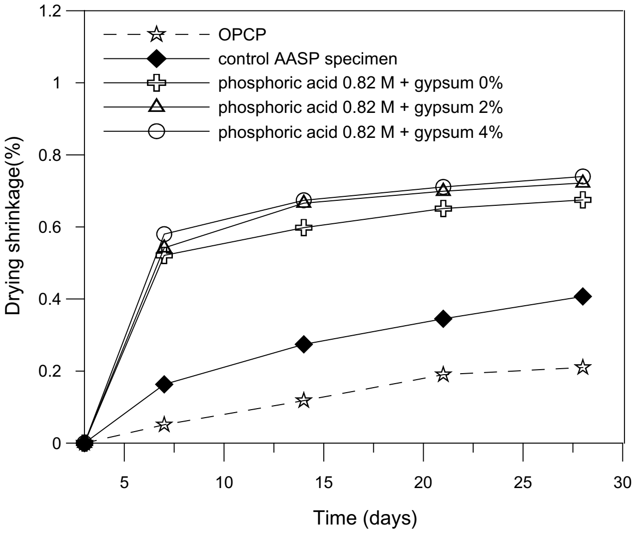

Fig. 5. Effects of a simultaneous use of gypsum and phosphoric acid on the drying shrinkage.

The next set of experiments involved a combination of gypsum and phosphoric acid to determine if gypsum inhibited drying shrinkage. In these tests, only 0%, 2% and 4% gypsums (by slag mass) were used. The results are shown in Fig. 5. It was found that adding phosphoric acid alone to AASP increased drying shrinkage in comparison with blank AASP (without gypsum and phosphoric acid). AASP drying shrinkage was more apparent than OPCP. The reasons why AAS binder produces higher drying shrinkage than OPC binder does have been explained in Ref. [9]. Following previous mentioned reasons, adding phosphoric acid to AASP increased the drying shrinkage, implying a higher total volume of mesopores were formed. The formation of $Ca_3(PO_4)_2$ may condense the macropores such that additional mesopores were observed. It can be observed that when the gypsum and phosphoric acid were used together, the gypsum can no longer inhibit drying shrinkage of AASP. The reason also comes from the formation of $Ca_3(PO_4)_2$ as explained earlier.

In Fig. 6, the compressive strength development using both of gypsum and phosphoric acid in AASP is illustrated. It was found that at 3 days, the blank AASP group had the highest compressive strength. All other AASP groups had close compressive strengths with respective to the OPCP group. Adding phosphoric acid, whether or not gypsum was used, retarded the chemical reaction in alkali-activated slag. It was not surprising that compressive strengths for AASP groups with added phosphoric acid were very close. The AASP group (0.82 M phosphoric acid) with a higher addition of gypsum showed a lower compressive strength. The product yielded by the gypsum and phosphoric acid chemical reaction, $Ca_3(PO_4)_2$, was a weaker phase compared to the alkali-activated slag product (CSH gel at early age and $CaO \cdot Al_2O_3 \cdot MgO \cdot SiO_2$ at later stage of hydration). The ratio between volumes of the stronger phase ($CaO \cdot Al_2O_3 \cdot MgO \cdot SiO_2$, CSH gel) and weaker phase ($Ca_3(PO_4)_2$) decreased as the amount of gypsum increased. It was then expected that an AASP group with 0.82 M phosphoric acid additions would have a stronger compressive strength when less gypsum was added, which was actually observed and confirmed in this study. As indicated in the figure using phosphoric acid (with or without gypsum), an increasing trend was exhibited in the long-term compressive strength when the blank AASP reached its final strength.

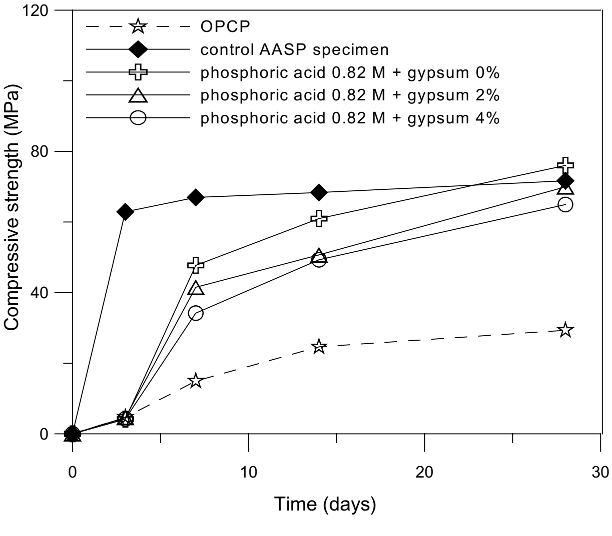

Fig. 6. Effects of a simultaneous use of gypsum and phosphoric acid on the compressive strength.

4. Conclusions

In this paper, effects of gypsum and phosphoric acid on setting time, compressive strength development and drying shrinkage of AASP were examined. The combined use of gypsum and phosphoric acid was the main focus. It was concluded that using phosphoric acid alone increased the setting time, reduced the compressive strength at an early age and increased the drying shrinkage. On the other hand, adding gypsum alone reduced the setting time, increased the compressive strength and decreased the drying shrinkage. The combined use of phosphoric acid and gypsum blocked the retarding effect of phosphoric acid, the compressive strength development was similar to that using phosphoric acid alone and the drying shrinkage reducing effect of gypsum disappeared. With the idea of using phosphoric acid as a retarder gypsum to reduce drying shrinkage is not attainable. A compatible system to reduce drying shrinkage and retard setting time for AAS binder is necessary.

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