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Effect of Lightweight Aggregates Incorporation on the Mechanical Properties and Shrinkage Compensation of a Cement-Ground Granulated Blast Furnace Slag-Phosphogypsum Ternary System

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ABSTRACT

Shrinkage-induced cracking is a common issue in concrete structures, where the formation of cracks not only affects the aesthetic appearance of concrete but also potentially reduces its durability and strength. In this study, the effect of ceramsite sand addition on the properties of a ternary system of cement-ground granulated blast furnace slag (GGBFS)-phosphogypsum (PG) is investigated. In particular, the fluidity, rheology, hydration heat, compressive strength, autogenous shrinkage, and drying shrinkage of the considered mortar specimens are analyzed. The results indicate that an increase in PG content leads to a decrease in fluidity, higher viscosity, lower exothermic peak, and lower compressive strength. However, the shrinkage of the mortar specimens is effectively compensated. The incorporation of internal curing water from ceramsite sand improves fluidity, decreases both yield stress and viscosity, enhances the degree of hydration, and induces mortar expansion. However, the inferior mechanical properties of the ceramsite sand generally produce a decrease in the compressive strength.

KEYWORDS

Phosphogypsum; ceramsite sand; internal curing; mechanical property; shrinkage

1 Introduction

The durability of concrete structures is seriously affected by the presence of cracking, a common issue in modern construction engineering. This issue is often a consequence of shrinkage which causes shallow cracks in concrete at an early stage [1–4], not only facilitating the invasion of harmful ions but also accelerating the deterioration of concrete [5–8]. Therefore, it is particularly important to maintain the volume stability of concrete.

Materials such as expansive agents or hydration heat inhibitors are usually used to offset shrinkage or reduce heat release of concrete to improve the volume stability of concrete. Although these methods can be effective to a certain extent, there are still shortcomings. To solve the problem of cracks caused by serious shrinkage of high-strength concrete, Xi et al. [9] studied the effect of expansive agents on the performance of concrete. The results showed that the compressive strength can be improved by expansive agents, but cracks occurred due to excessive and delayed expansion in the later stage. Liang et al. [10] found that hydration heat



inhibitors led to a decline in the early hydration degree of concrete, which significantly reduced the early compressive strength of concrete, and with the increase of hydration heat inhibitors content, the strength of concrete decreased. Zhang [11] and Li et al. [12] respectively discussed the preparation of MgO expansive agents and butyl acrylate copolymer emulsion and found that the preparation of expansive agents and hydration heat inhibitors was complicated and easy to cause environmental pollution.

PG is a solid waste discharged in wet-process of phosphoric acid production, which contains 90% calcium sulfate, and a small amount of impurities such as phosphoric acid residuals, fluorides and heavy metals. The common disposal method of PG is stacked in landfill fields, where PG is harmful to the surrounding environment and human health once the impurities and heavy metals are leaked into the soil. It is estimated that the annual production of PG will reach 250 million tons within the next decade, but only 15% of PG can be utilized [13]. Hence, many researchers are exploring effective utilization ways for PG to serve as an alternative cementitious material in concrete. PG was tried to prepare high-content PG concrete, and although its mechanical properties were reduced, the permeability resistance of concrete was qualified [14–17]. This approach not only reduces the reserves of PG, but also reduces the production and consumption of cement, promoting environmental sustainability and conserving natural resources. Dong et al. [16] found that PG could improve the working performance and hydration process of concrete, and the sulfate in PG would generate the expansion product (ettringite) in concrete. Djayaprabha et al. [18] found that when PG content reached an optimal amount, both the compressive strength and bending strength of mortar were increased, which was also attributed to the formation of ettringite. Costa et al. [19] and Zeng et al. [20] indicated that the addition of PG would significantly reduce water content on the matrix and directly affect the hydration degree. The reason is that the water consumption of ettringite formation is larger [21], resulting in incomplete hydration of cement, increased residual PG, and reduced amount of hydration products that provide strength so that the mechanical properties of concrete decreased and the volume stability became worse.

Lightweight aggregates with water absorption ability can be incorporated into concrete to play the role of reservoir. When the water in the hydration process is insufficient, lightweight aggregates could release water to form an internal curing environment to increase the internal relative humidity of concrete, so that the hydration of cementitious materials could continue and autogenous shrinkage could be compensated. Afifi et al. [22] found that the introduction of internal curing water by perlites could enhance the hydration degree, and reduce the shrinkage and the possibility of cracking. Similarly, Hammam et al. [23] found that the internal humidity of concrete could be improved by pre-wetted perlites and recycled aggregates, and the working performance, durability, and cracking resistance of concrete were all promoted. Zhao et al. [24] indicated that the expansion effect of CaO expansive agent could be improved by the addition of pre-wetted ceramsite sand to reduce the autogenous shrinkage of concrete.

In this study, a cement-GGBFS-PG ternary system was studied, with ceramsite sand partially replacing the sand as an internal curing material. The utilization of internal curing materials could maintain internal relative humidity, providing the necessary water for the reaction of PG, thereby further compensating for shrinkage, and enhancing volume stability.

2 Materials and Experiment Methods

2.1 Raw Materials

The cement used in this study is P. II 52.5 cement with a density of 3.16 g/cm³, a specific surface area of 340 m²/kg, a 28-day bending strength of 10.1 MPa, and a 28-day compressive strength of 59.8 MPa. The PG used in this study primarily consists of dihydrate gypsum (CaSO₄·2H₂O) with D₅₀ = 31.58 μm. The S95 grade GGBFS is used in this study with a density of 2910 kg/m³, a specific surface area of 411 m²/kg, and a fluidity ratio of 101%, the 7-day and 28-day activity index are 77% and 98%, respectively. The chemical compositions of cement, GGBFS, and PG are shown in Table 1. Ceramsite

sand has a 24-h water absorption rate of 27%, with an apparent density of 1123 kg/m³, the microtopography of ceramsite sand is shown in Fig. 1. The sand used is medium sand with a fineness modulus of 2.8 and an apparent density of 2530 kg/m³. The solid content of the polycarboxylate water-reducing agent is 45%. The mixing water is tapping water.

Table 1: The chemical composition of raw materials

Materials	CaO	SiO ₂	Al ₂ O ₃	Fe ₃ O ₄	MgO	SO ₃	P ₂ O ₅	F	Loss
Cement	62.64	20.94	4.15	2.96	3.10	2.04	–	–	2.44
GGBFS	38.0	37.8	12.8	0.2	7.7	2.0	–	–	1.5
PG	32.31	5.96	0.43	0.32	0.07	46.82	1.20	0.60	11.92

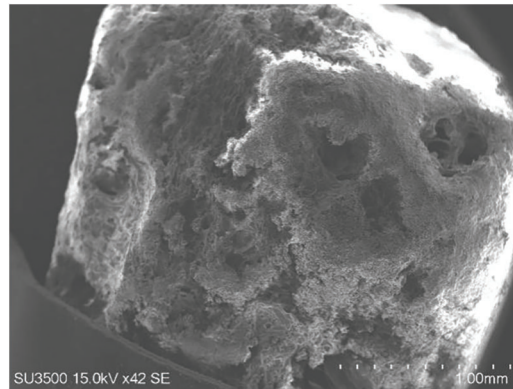


Figure 1: The microtopography of ceramsite sand

2.2 Experiment Methods

2.2.1 Preparation of Mortar Specimens

The mixture proportions of specimens are shown in Table 2. The 24-h water absorption rate of ceramsite sand was tested, and the corresponding extra water was added into the mixing water to pre-wet the ceramsite sand 24 h in advance. The raw materials were weighed according to Table 2, and the replacement of sand by ceramsite sand was calculated by volume according to the apparent densities. The 40 mm × 40 mm × 40 mm steel molds were used to cast mortar. The dosage of the water-reducing agent was adjusted to maintain the fluidity of the mortar at 190 ± 5 mm. The specimens were cured in standard curing conditions (RH ≥ 95%, 20°C).

Table 2: The mixture proportions of specimens

Specimens	Cement (g)	BBGFS (g)	PG (g)	Sand (g)	Ceramsite sand (g)	Water (g)	Extra water (g)	Water-reducing agent (g)	Fluidity (mm)
0.4S20	360	90	0	1350	0	180	0	1.2	187
0.4S16	360	72	18	1350	0	180	0	1.5	186
0.4S12	360	54	36	1350	0	180	0	1.7	185
0.4S12L10	360	54	36	1215	60	180	16.2	1.5	195

(Continued)

Specimens	Cement (g)	BBGFS (g)	PG (g)	Sand (g)	Ceramsite sand (g)	Water (g)	Extra water (g)	Water-reducing agent (g)	Fluidity (mm)
0.4S12L15	360	54	36	1147.5	90	180	24.3	1.3	193
0.4S12L20	360	54	36	1080	120	180	32.4	1.1	190

2.2.2 Fluidity

The fluidity test of the specimens was conducted in accordance with GB/T 2419-2005 by a flow table.

2.2.3 Rheology

The rheological test of mortar is conducted by a MARS40 rheometer (Fig. 2). The shear process is shown in Fig. 3. The shear rate and shear stress in the process from 210 to 270 s are used as the data to analyze the rheological properties of mortar. The Bingham model ($\tau = \tau_0 + \mu\gamma$) is used to analyze the yield stress and viscosity of mortar.



Figure 2: Rheometer

2.2.4 Hydration Heat

The hydration heat is measured by a TAM AIR isothermal calorimeter with 8 channels at 20°C. 30 g mortar is put into each channel, and the instrument starts after 15 min. The test duration is 72 h.

2.2.5 Compressive Strength

The compressive strength test of the mortar is conducted by a microcomputer servo flexural-compressive tester in accordance with GB/T 17671-2021. The compressive strength test is tested at 3, 7, 28, and 60 d, the average value of three specimens is taken as the final compressive strength.

2.2.6 Autogenous Shrinkage

Autogenous shrinkage is recorded by a digital display comparator according to JC/T 603-2004. The mortar specimens are cast in 25 mm × 25 mm × 280 mm molds and demolded after 24 h curing. The mortar specimens are wrapped in plastic films to isolate the exchange of moisture with air. The test is conducted in the curing room (RH = 60 ± 5%, 20 ± 2°C). The test duration is 7 d, and the average autogenous shrinkage of three specimens is taken as the final autogenous shrinkage.

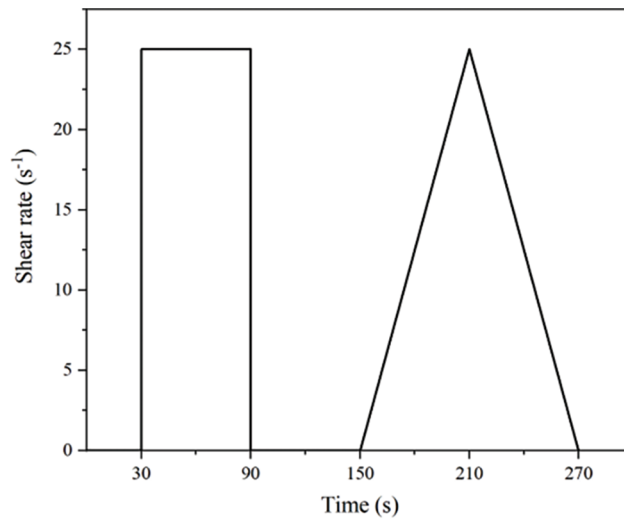


Figure 3: Shear process

2.2.7 Drying Shrinkage

Drying shrinkage is recorded by a digital display comparator according to JC/T 603-2004. The mortar specimens are cast in 25 mm × 25 mm × 280 mm molds and demolded after 24 h curing. The test is conducted in a curing room (RH = 60 ± 5%, 20 ± 2°C). The drying shrinkage is recorded at 1, 3, 7, 14 and 28 d, and the average drying shrinkage of three specimens is taken as the final drying shrinkage.

3 Results and Discussion

3.1 Fluidity of Fresh Mortar Mixture

The fluidity and water-reducing agent dosage are shown in Fig. 4. To avoid the results of subsequent tests being affected by the fluidity of mortar specimens, the fluidity of mortar specimens is maintained at 190 ± 5 mm during the preparation. The influence of PG and ceramsite sand on the fluidity of mortar specimens is represented through the dosage of water-reducing agent. The result shows that the dosage of water-reducing agent gradually increases with the increased content of PG. However, the influence of ceramsite sand shows a contrary effect. The reason is that the monoclinic structure of PG hinders the fluidity of mortars [25], but the part of internal water released from ceramsite sand increases the free water of mortar specimens, resulting in a decrease of water-reducing agent [26].

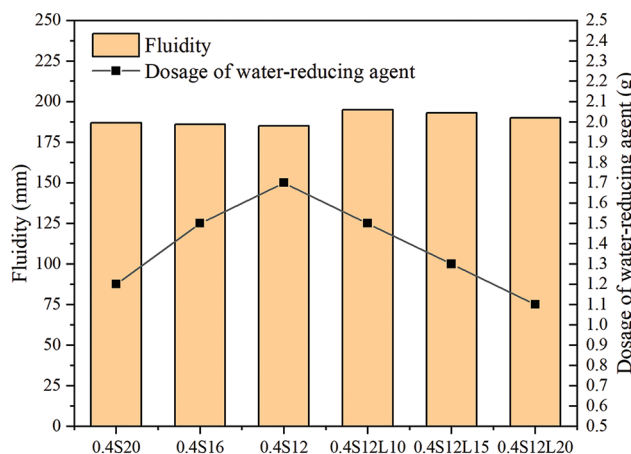


Figure 4: The fluidity and water-reducing agent dosage of mortar specimens

3.2 Rheology of Fresh Mortar Mixture

The rheological curves of mortar specimens are shown in Fig. 5. The yield stress (τ_0) and viscosity (μ) are calculated according to the Bingham model. It can be observed that the yield stress and viscosity of 0.4S20 are 59.48 Pa and 3.22 Pa·s, respectively. With the incorporation of PG, the yield stress and viscosity of 0.4S12 is 56.77 Pa and 4.44 Pa·s. The yield stress of 0.4S12 shows little difference with 0.4S20 but the viscosity is increased significantly, which indicates higher energy is needed to keep its moving status due to the monoclinic structure of PG. However, the addition of internal curing water makes 0.4S12L20 show significantly lower yield stress and viscosity, which are 37.84 Pa and 2.95 Pa·s, respectively. The pre-wet ceramsite sand releases water during the mixing process, the increase of free water significantly reduces the solid phase volume fraction of mortar, which is the main reason for the decrease of yield stress and plastic viscosity of mortar.

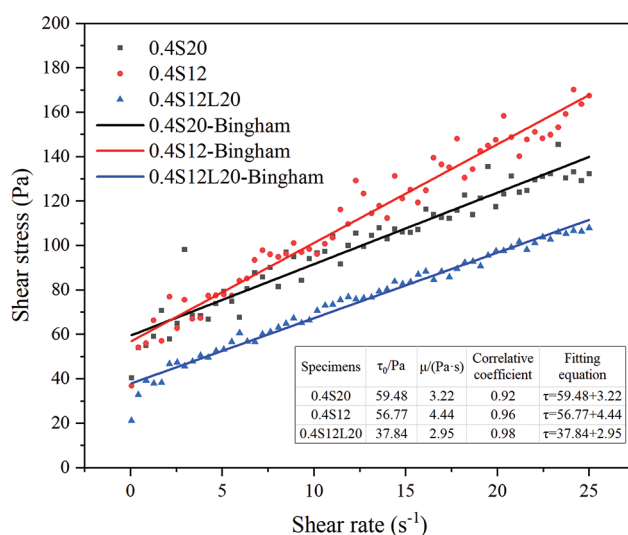


Figure 5: The rheological curves of mortar specimens

3.3 Hydration Heat

The hydration heat of mortar specimens is shown in Fig. 6, the exothermic process can be divided into 5 periods: Early induction period, induction period, acceleration period, deceleration period and stable period. It can be found there are two main exothermic peaks: the first exothermic peak is mainly caused by the heat release of the surface wetting of cement particles and the heat release of C_3A [27], and the second exothermic peak is the heat release peak of C_3S . Comparing the hydration heat curves of three specimens, it can be found that the total heat release is increased with the PG content before 4 h, which indicates a higher first exothermic peak. This is because the increase of PG increases the content of SO_4^{2-} in the liquid phase and promotes the formation of ettringite [25]. The addition of PG greatly delays the second exothermic peak by about 14 h. The soluble phosphorus in PG can form calcium phosphate precipitation on the surface of C_3S to reduce the activation points, and seriously hinder further hydration [28,29]. Although SO_4^{2-} can promote the hydration of C_3S [30,31], this positive effect is weaker than the negative effect of soluble phosphorus. Therefore, the second exothermic peaks of 0.4S12 and 0.4S12L20 are delayed than that of 0.4S20, and the peak intensity is also weaker. With the incorporation of ceramsite sand, it can be found that the exothermic peak occurred in advance slightly and the total heat release of 0.4S12L20 is higher than that of 0.4S20 and 0.4S12, indicating the hydration is accelerated by internal curing [27]. It is worth noting that although the total heat release of 0.4S12 and 0.4S12L20 show

higher compared to 0.4S20, the intensity of the exothermic peaks is much lower, which is beneficial for preventing temperature-induced cracking [32].

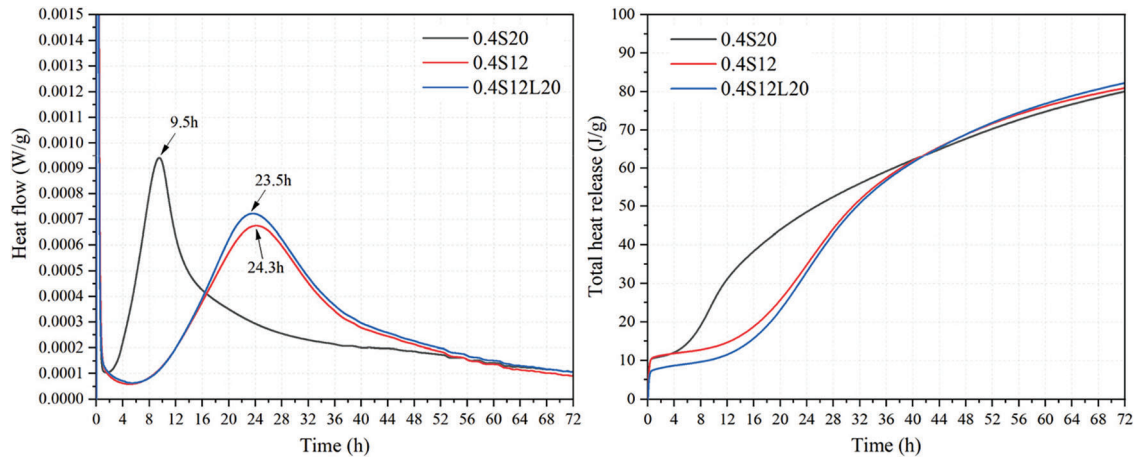


Figure 6: The hydration heat of mortar specimens

3.4 Compressive Strength

The compressive strength of mortar specimens is shown in Fig. 7. It can be found that with the increased content of PG and ceramsite sand, the compressive strength of mortar specimens shows a decreased trend. Since harmful impurities, the free phosphorus and fluoride ions can react with calcium ions to produce calcium phosphate, hydroxyapatite, and calcium fluoride to hinder the hydration, so that the compressive strength is affected negatively at an early stage [19,28,29]. Moreover, the replacement of GGBFS (which has been proven to be highly beneficial to later strength [33,34]) by PG, also leads to a decline in strength in the later stage. It is worth noting that the compressive strength of 0.4S12 at 60 d is lower than that at 28 d, which indicates an overmuch PG content is harmful to the mechanical properties of mortar at later stage. This is because the addition of PG promotes the formation of ettringite which gradually fills the pores of mortar and exerts certain compressive stress on the surrounding area. When the PG content is high, the unreacted PG could generate ettringite to make the compressive stress on the surrounding area over high, leading to the expansion of the matrix and the formation of micro-cracks at a later stage [15,35,36]. With the incorporation of internal curing water, the compressive strength of 0.4S12L10 shows higher than that of 0.4S12 which suggests a positive internal curing effect can endow mortar with a higher hydration degree and compensate for the strength loss brought by PG [37,38]. However, due to the porous structure of ceramsite sand, the increasing content of ceramsite sand decreases the compressive strength of mortar [39,40].

3.5 Autogenous Shrinkage

The autogenous shrinkage curves of mortars are shown in Fig. 8. It can be found that the incorporation of PG and ceramsite sand can reduce the autogenous shrinkage of mortar specimens. Compared with 0.4S20, the autogenous shrinkage of 0.4S16 and 0.4S12 are reduced by 13.6% and 24.1%, respectively, suggesting a higher ability of shrinkage resistance. The reason is that the incorporation of PG produces ettringite in the mortar specimens to compensate for shrinkage [41,42]. In addition, the incorporation of internal curing water makes autogenous shrinkage of mortar specimens fall further, and 0.4S12L15 and 0.4S12L20 show expansion at 2 d. It is well known that the larger water-to-cement ratio presents lower autogenous shrinkage [43]. Compared with 0.4S12, it can be found that the total water-to-cement ratio of 0.4S12L10, 0.4S12L15, and 0.4S12L20 is higher. The internal curing effect can provide extra water to increase the

water-to-cement ratio which has been proven to reduce the self-desiccation effect so that the autogenous shrinkage can be reduced [44]. Moreover, the addition of water to the mortar specimens also promotes the production of ettringite [20].

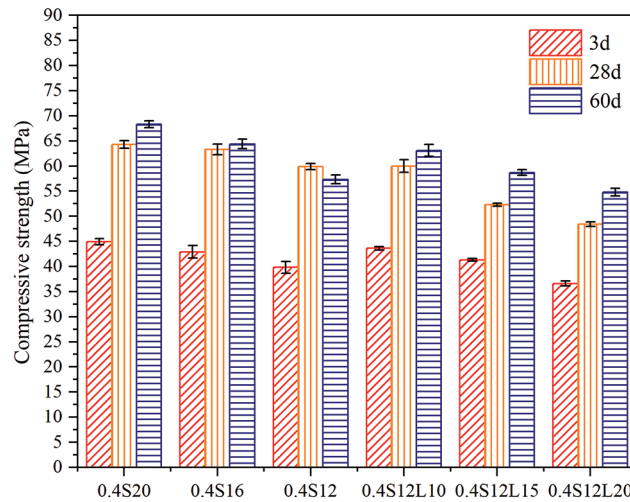


Figure 7: The compressive strength of mortar specimens

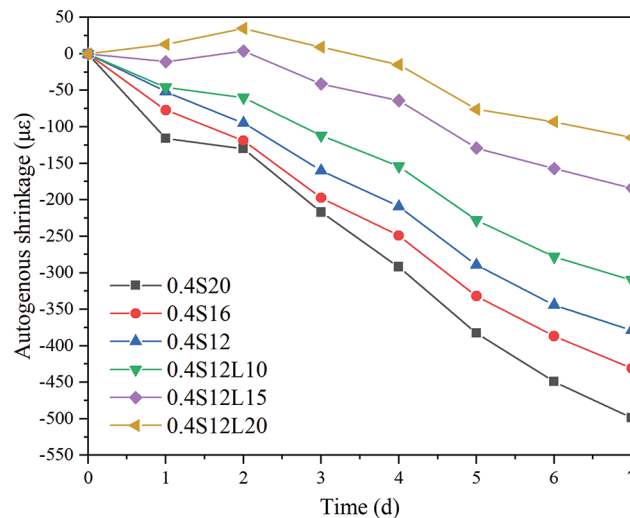


Figure 8: The autogenous shrinkage of mortar specimens

3.6 Drying Shrinkage

The drying shrinkage curves of mortar specimens are shown in Fig. 9. Similar to the result of autogenous shrinkage, the incorporation of PG and ceramsite sand can reduce the drying shrinkage of mortar. However, although the drying shrinkage is decreased with the addition of PG and ceramsite sand, only 0.4S20 shows a significant difference compared with other specimens. The internal curing water seems to play a negative role in the shrinkage process by providing more water to be lost to aggravate drying shrinkage in an exposed environment [44].

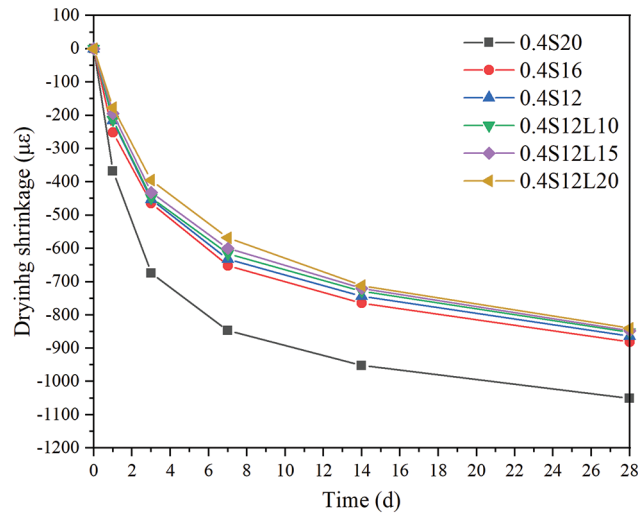


Figure 9: The drying shrinkage of mortar specimens

4 Conclusions

With the study of this research, the conclusions are drawn as follows:

- (1) The fluidity of specimens is represented by the dosage of water-reducing agent. The incorporation of PG increases the dosage of water-reducing agent, but the incorporation of internal curing water shows the contrary.
- (2) The addition of PG makes specimens show higher viscosity, the yield stress and viscosity are decreased due to the release of internal curing water during the mixing process.
- (3) The occurred time of exothermic peak is delayed significantly by the addition of PG, and the intensity of exothermic peaks of 0.4S12 and 0.4S12L20 is much lower compared to 0.4S20, which is beneficial to prevent temperature-induced cracking of concrete.
- (4) The addition of internal curing water can promote compressive strength and compensate for the harmful effect brought by PG at a later stage, but the content of ceramsite sand needs to be limited due to its weak porous structure.
- (5) The synergistic effect of PG and internal curing can endow specimens with autogenous expansive deformation, but the internal curing effect seems to be a negative factor in restraining the decrease of drying shrinkage due to more water being lost in the air.
- (6) A small amount of PG combined with internal curing can significantly improve the shrinkage properties of cementitious materials. For future research, several lines of study are suggested: the influence of different qualities of PG on concrete performance; the development of the long-term performance of concrete doped with PG; the formation, transformation, and decomposition of hydration products in large-volume concrete doped with PG; and the application of PG and internal curing in high-strength structural concrete.

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Availability of Data and Materials: The datasets used or analyzed during the current study are available from the corresponding author upon reasonable request.

Conflicts of Interest: The authors declare that they have no conflicts of interest to report regarding the present study.

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