Synergetic Effect of Expansive Agent (KEA) and Superabsorbent Polymers (SAP) on the Shrinkage, Strength and Pore Structures of Mortars

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Abstract
In this work, the effects of the individual or hybrid addition of superabsorbent polymers (SAP) with varying dosages (0.1%, 0.2%, 0.3%, and 0.6%) and the lime-type expansive agent (KEA) on the length and mass change, compressive strength, and pore structures (MIP) of mortars were investigated. The results showed that the incorporation of SAP can effectively mitigate its autogenous shrinkage and the length change value of the mortar with SAP smaller than reference until 49 days, regardless of the presence of KEA. The hybrid addition of SAP and KEA increases the initial expansion of the specimens as compared with individual addition of SAP, which is a beneficial effect on compensating for the shrinkage of the mortar under drying conditions. Moreover, the addition of SAP seems to delay cement hydration and increase the volume of macropores (greater than 100 nm), thereby reducing the compressive strength of the mortars. The introduction of KEA slightly promoted the formation of micropores, resulting in a slight increase in compressive strength compared with the samples without KEA. Furthermore, in our view, it promotes pore refinement, so as to reduce moisture evaporation.

1. Introduction
Shrinkage cracks in concrete and mortar occur due to evaporation of moisture in the porous, differential settlement, and temperature gradient (Wittmann 1976; Weyers et al. 1982; Cohen et al. 1990; Collinsa and Sanjayan 2000). It is a common phenomenon considered to be a potential factor causing the deterioration of concrete structure durability. Cracks appearing on the surface of concrete structures may allow water, chloride ions, and gas to penetrate, causing corrosion of steel bar, accelerated frost damage, and can cause severe impairment in long-term service and safety of the concrete structure (Cabrera 1996; Otieno et al. 2016). The general way to mitigate early-age shrinkage cracking is to employ an expansive agent (EA) and improve initial curing regimes (Zhang et al. 2019). Among the expansive agents, the two most common and important ones are calcium sulphoaluminate-based and lime type agents (CaO-based), which react with water to produce restrained expansion in reinforced concrete structures and ultimately reduce the risk of shrinkage cracking, especially for CaO-based expansive agents, which can increase the volume by approximately 90% after forming portlandite via reacting with water (Chatterji 1995). Therefore, the presence of water is critical, regardless of the type of expansion agent; at the same time, it also shows the importance of curing regimes.

Conventional curing regimes, such as moisture curing, sheet curing, and permeability formwork curing, are difficult to bring about obvious effects on concrete curing through external moisture penetration due to a relatively low surface porosity of cement-based materials with a low water-cement ratio. Accordingly, internal curing (IC) is considered to be a potential way for the mitigation of autogenous shrinkage and the maintenance of internal relative humidity (IRH) by releasing water/solution of internal curing materials to prevent internal self-desiccation of concrete (Lura 2003). Lightweight aggregate (LWA) and superabsorbent polymers (SAP) are currently thought to be the two most effective internal curing regimes on the mitigation of autogenous shrinkage; they were originally proposed by Philleo and Jensen (1991) and by Jensen and Hansen (2001, 2002), respectively. The effectiveness of eliminating its autogenous shrinkage was then confirmed by other scholars (Zhutovsky et al. 2002; Bentz and Jensen 2004; Cusson and Hoogeveen 2008; Wang et al. 2017a; Oh and Choi 2018; Liu et al. 2019).

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SAPs are some types of cross-linked polyelectrolytes that can absorb tens to hundreds times more than their own weight as contact with water or aqueous solutions (Mechtcherine and Reinhardt 2012). Internal curing by SAP as a “micro-reservoir” may absorb and release liquids inside the concrete, providing a continuous water resource supply for cement hydration, and creating the possibility of long-term curing of concrete (Liu et al. 2017). However, it is interesting that the advantages of SAP internal curing are not limited to the inhibition of autogenous shrinkage at the early age of concrete (Soliman and Nehdi 2010; Kong et al. 2014; Snoeck et al. 2015; Almeida and Klemm 2018; Oh and Choi 2018; Liu et al. 2019a). Moreover, it can be found from a variety of studies that SAPs were used in concrete and mortar to improve resistance to carbonization (Ma et al. 2017), frost damage resistance (Laustsen et al. 2013; Mechtcherine et al. 2016, 2019), and internal relative humidity (IRH) (Shen et al. 2015; Song et al. 2016), change of rheology (Mechtcherine and Reinhardt 2012), as well as to promote self-sealing (Snoeck et al. 2012a; Lee et al. 2016) and self-healing (Snoeck et al. 2012a, 2012b, 2018; Gruyaert et al. 2016). In the view of practical application, however, among many advantages, it seems to be particularly important to control the cracking of the early-age massive concrete through internal curing.

In general, utilizing expansive agents is one of the most effective ways to compensate for shrinkage behavior and reduce the crack risk, with internal curing materials being another. Theoretically, the expansive agent reacts to generate sufficient expansion and needs the aid of abundant water, some of which can be obtained from the internally cured material. Indeed, several studies (Chen et al. 2013, 2016; Li et al. 2017; Wang et al. 2017b) have suggested that the combination of LWA and an expansion agent can effectively improve shrinkage behavior. Wang et al. (2017b) showed that the combination of LWA and expansive agents can effectively decrease the cracking sensitivity of concrete at an early age. Li et al. (2017) showed that the combined use of SLWA and EA exhibits high efficacy in mitigating shrinkage deformation and can limit the reduction of mechanical properties caused by a single EA. However, there is limited information on the combined effect of SAP and EA on shrinkage behavior. Yu et al. (2019) investigated the synergistic effect of MgO-based expansive agent (MEA) and SAP on the long-term drying shrinkage, autogenous shrinkage, internal humidity and mechanical strength of concrete, and experimental results showed that the hybrid system of 0.4% SAP and 6% MEA could not only eliminate autogenous shrinkage but also control the deformation of expansion to a minimum. Hence, it is speculated that the coexistence of lime-type expansion agent and SAP may have a beneficial effect on the shrinkage behavior of mortar. Nonetheless, it is still not clear on the combined effect of lime-type expansive agent and SAP on the mortar.

Furthermore, in the Tohoku region of Japan, infrastructure construction projects have been promoted after the great east Japan earthquake and tsunami disaster in 2011. Lifetime improvement of these concrete structures is the most important issue to reduce future maintenance costs. Blast furnace slag cement (BB), composed of 42% ground granulated blast furnace slag, is employed heavily on the field. Although various safeguards are applied throughout the construction execution process such as expansion agent (EA) added to compensate for shrinkage, optimization of vibration time, control of slump, setting time, and curing conditions (wet cured for 7 days, sheet cured for 21 days). However, the cracking of the RC slab surface has not been sufficiently improved RC (see Fig. 1).

Therefore, in order to ensure that the cracking of the RC bridge slab due to the shrinkage behavior was reduced. The effects of the addition of SAP and lime-type expansive agent (KEA) on the length change, mass change, compressive strength, and pore structures of the mortars were mainly studied. The purposes of this study are to (1) study the effects of various dosages of SAP and with or without KEA on the length change, mass change, and compressive strength of mortars, (2) investigate the influence of various dosages of SAP with or without KEA on the pore size distribution and characteristics of mortars with different ages, and (3) evaluate the effect of pore structure on the drying shrinkage behavior and compressive strength of mortar.

2. Materials and methods

2.1 Materials

In the study, the blast furnace slag cement (BB, provided by Ube-Mitsubishi Cement Corporation) and the lime-type expansion agent (KEA, provided by Taiheiyo Materials Corporation) were employed. Their chemical compositions and physical properties are shown in Tables 1 and 2. Pit sand was used as a fine aggregate with apparent density, fineness modulus, and water absorption of 2.57 g/cm³, 2.52, and 2.96%, respectively. Commercial SAP powder based on a cross-linked potassium salt poly acrylate [particle size 476.6±52.9 μm (n=100)] was used as an internal curing material. The SAP is bulk-polymerized
and consists of irregular particle shapes (Mechtcherine and Reinhardt 2012). The sorption capacity of the SAP powder tested with the tea-bag method (Mechtcherine and Reinhardt 2012; Mechtcherine et al. 2018) at 1, 5, 10, 30, 60 min and at 3 and 24 h was presented in Fig. 2. The SAP powder sorption capacity of using the tap water (TW), the extracted solution of BB cement (W/C=5), and the extracted solution mixed with the expansion agent and BB cement (BB+KEA, W/B=5, BB/KEA=20) were 123.2 g/g, 36.9 g/g, and 34.5 g/g, respectively. It was also noticed that there was an approximately 72% decrease of SAP sorption capacity induced by cement filtrate solution compared with tap water (TW). In addition, in order to facilitate the application of SAP in winter concreting in the future, the adsorption capacity of SAP under the condition of 0°C to 10°C was tested. It can be found that temperature (0°C to 10°C) has little effect on the adsorption capacity of SAP. However, the temperature (0°C to 10°C) has little influence on the sorption capacity of the SAP.

2.2 Mixture proportions and mixing procedure

Mortars with varying SAP dosages (0.1%, 0.2%, 0.3%, and 0.6% by weight of cementitious materials), as well as addition of KEA, were investigated. Adjustment with additional water to ensure that the mortar with and without SAP had a similar slump flow. All mixtures are shown in Table 3. Those named “Ref-KEA” (with KEA) and “Ref” (without KEA) were used as the reference mix proportions. Specimens with the individual addition of SAP were marked as S0.1, S0.2, S0.3, S0.6, with the number following the letter “S” in the specimen name indicating the percentage of SAP by weight of binder. Specimens with the hybrid addition of SAP and KEA were marked as KEA+S0.1, KEA+S0.2, KEA+S0.3, KEA+S0.6. An automatic mortar mixer was used for mixing all materials and the mixing procedure depicted in Fig. 3 (Mechtcherine and Reinhardt 2012; Ma et al. 2017; Yang and Wang 2019). The cementitious material and SAP were equally placed and pre-mixed for 30 s at 140 rev/min in a bowl in order to ensure SAP powder homogeneous dispersion.

![Fig. 2 Sorption capacity of the SAP powder.](image-url)
2.3 Curing conditions
All specimens were prepared in a laboratory to ensure that the curing conditions were consistent with the field. Immediately after casting, specimens were sealed with plastic film for 24 h, and then they were demolded. After demolding, they were cured in water for 6 d, then they were cured sealed with plastic film for 21 d in a constant temperature and humidity chamber (20°C and RH 60±5%). After 28 d of curing, all plastic film was removed, and the surfaces of specimens were exposed to a drying environment (20°C and RH 60±5%).

2.4 Testing methods
2.4.1 Length and mass change
The prismatic mortar specimens with a dimension of 40×40×160 mm were cast for measuring the length change and mass loss. A hand-held strain comparator with an accuracy of 0.001 mm was employed to measure the length at 1 d, 3 d, 5 d, and 7 d, and then every 7 d until 91 d according to the JIS A 1129-2 contact-type strain gauge method (see Fig. 4). Meanwhile, an electronic balance with an accuracy of 0.01 g was used to record the mass of specimens. After demolding and sticking the contact chips, the initial length and mass were recorded. Two lateral surfaces perpendicular to the specimen casting surface were measured and the mean results obtained from every three tested specimens were reported.

The length and mass change at n days was calculated with Eqs. (1) and (2):

\[
\varepsilon_{ln} = \frac{l_n - l_1}{l_1} \times 1000
\]

\[
m_n = \frac{m_n - m_1}{m_1} \times 100
\]

where \(\varepsilon_{ln}\) is length change (μm/m) at the nth day, \(l_1\) is the length (mm) on the first day, \(l_n\) is the length (mm) on an nth day, \(m_n\) is the mass loss (%), \(m_1\) is the mass (g) on the first day and \(m_n\) is the mass (g) on an nth day.

2.4.2 Compressive strength
The compressive strength testing was carried out on mortar specimens with a size of 50 mm in diameter, 100 mm in height according to Japanese Industrial Standard (JIS A 1108). The compressive strength was determined from an average of each batch (three samples) at 7 d, 28 d and 91 d, respectively.

2.4.3 Pore size distribution and characteristics
The pore characteristics of mortar were determined by Mercury Intrusion Porosimetry (MIP) at 7 d, 28 d and 91 d, respectively. The pore characteristics of the specimens were measured using the device (Pascal 140/440) with a pressure capacity of 400 MPa. The parameters used were 0.48 N/m for surface tension and 140 degrees for the mercury contact angle. The mortar specimens were cut into approximately 5 mm block samples and placed into an acetone bath for preventing the cement hydration reaction. The resulting samples were stored for 24 hours in an oven at a controlled temperature of 100°C and ensured that the free water in the pores was completely evaporated, prior to MIP testing.

3. Results and discussion
3.1 Effect of individual and hybrid addition of SAP and KEA on the length and mass change
Figures 5 and 6 show the influence of individual and hybrid addition of SAP and KEA on the length and mass change of mortar specimens for a duration of 91 d. The initial time was set at 24 h after casting. Overall, the length and mass change curves showed the decrease after the initial increase, and a tendency to become stable slowly as the age prolonged. These tendencies may be mainly related to variations in curing methods that were assumed under laboratory conditions. As can be seen in Figs. 5 and 6, after 6 d of water curing, compared with the control specimens (Ref), the addition of KEA and SAP caused mortar specimens expansive behavior to be extended for about one week regardless of individual and hybrid addition. Furthermore, it can be found that the decline rate of the length change between 14 d and 28 d of mortar adding SAP was slower than that without SAP and the slope of the curve gradually decreased with the increase of SAP content. This main reason is thought to be due to the mitigation of autogenous shrinkage. Therefore, it can be noticed that the addition of SAP

![Fig. 4 Specimen and hand-held strain comparator.](image)
reduced the autogenous shrinkage of mortar, whereas the addition of KEA enhanced this effect. **Figure 5(a)** presents the influence of the addition of only SAP on the total length change. As shown in **Fig. 5(a)**, the addition of SAP was not only effective in the reduction of the autogenous shrinkage but also the length change value of the mortar with SAP was smaller than Ref until 49 d. Moreover, for the mortar with SAP, the shrinkage continues for a long time, and the length change value of SAP mortar exceeds that of Ref. Noticeably, the inclusion of SAP decreased the shrinkage magnitude by 5 to 30% before 49 d as compared to the Ref. The reason may be due to the continuous release of water from the SAP particles to compensate for the loss of moisture in the dry environment, and it causes the expansion of the matrix volume in the early stage, and it leads to compensation for the matrix volume contraction caused by the moisture loss under the drying environment (Soliman and Nehdi 2010; Yu *et al.* 2019). As the drying prolonged after 49 d, however, the amount of water released by SAP can no longer meet the evaporative water loss rate and was insufficient to maintain the internal RH of the mortar, thereby weakening its ability to inhibit the shrinkage of the mortar. Hence, there is an adverse effect of SAP on shrinkage under drying conditions at later-age stages (Soliman and Nehdi 2010; Ma *et al.* 2017; Liu *et al.* 2019b). At the 91 d, as can also be seen from the figure, the addition of 0.1%, 0.2%, 0.3%, and 0.6% SAP increased the total shrinkage by 3.6%, 10.2%, 17.1%, and 28.4%, respectively, as compared to Ref. On the contrary, the addition of KEA (Ref-KEA) played a beneficial effect leading to the total shrinkage reduced by around 8.9% as compared with Ref, which can be noticed from **Figs. 5(a) and 6(a)**. **Figure 6** shows the effect of the hybrid addition of SAP and KEA on the total length and mass change. Generally, the trend of length and mass change was similar to that of the individual addition of SAP. As can be seen from **Fig. 6(a)**, owing to the hybrid addition of SAP and KEA, the initial expansion strain peaks increased by 162.3%, 170.9%, 172.8%, and 302.9%, respectively, as compared to Ref [see **Fig. 5(a)**], whereas, compared with Ref-KEA, the initial expansion strain increased by 4.6%, 8.1%, 8.8%, and 60.7%, respectively.
Moreover, it can also be found that the addition of KEA can mitigate the adverse influence of SAP on the drying shrinkage in the later period as well as compared with Ref, the addition of 0.1%, 0.2%, 0.3%, and 0.6% SAP decreased the total shrinkage at 91 d by 10.8%, 14.1%, 15.5%, and 19.8%, respectively (thanks to the hybrid addition of SAP and KEA, the initial expansion of the mortar was increased). In addition, the comparison of Figs. 5(b) and 6(b) shows that the introduction of the KEA reduced the mass loss at 91 d by around 26.1% (Ref-KEA), 35.6% (KEA+S0.1), 11.3% (KEA+S0.2), 9.3% (KEA+S0.3), and 11.9% (KEA+S0.6), respectively. After 28 d, the mortar specimens were completely exposed to the dry environment (RH: 60%, Temperature: 20°C). It can be assumed that after 28 d the samples were almost completely in a drying shrinkage state. Thus, the relative shrinkage at this time can be defined as the total drying shrinkage. The influence of SAP content and with/without KEA on the relative shrinkage and relative mass loss magnitude [see Eqs. (3) and (4) for its definition] between the 28 d and 91 d are presented in Fig. 7. It was worth noting from the results that the relative shrinkage and the relative mass loss increased with the increase of SAP contents under drying conditions regardless of individual/hybrid addition of SAP and KEA. For the hybrid addition of SAP and KEA, when the SAP contents were greater than or equal to 0.2%, its relative shrinkage was smaller compared to the individual addition of SAP. Hence, it can be concluded that the addition of SAP seems to be negative for relative shrinkage, whereas, the addition of KEA seems to improve the adverse effects especially when the SAP content is greater than or equal to 0.2%, the combined effect of SAP and KEA is more prominent.

\[
\text{Relative shrinkage} = L_{91} - L_{28} \tag{3}
\]

\[
\text{Relative mass loss} = M_{91} - M_{28} \tag{4}
\]

where \( L_{91} \) represents the length change (μm/m) on day 91, \( L_{28} \) is the length change (μm/m) on day 28, \( M_{91} \) represents the mass change (%) on day 91 and \( M_{28} \) is the mass change (%) on day 28.

Figure 8 shows the correlation between the length and mass change under drying conditions (from day 7 to day 91). It can be seen that Figs. 8(a) and (b) have similar trends.
correlation can be divided into two phases according to ide (Corinaldesi and Nardinocchi 2016). In addition, this promotion of pore refinement, due to its reaction with water to produce the more voluminous calcium hydroxide (Corinaldesi and Nardinocchi 2016). In addition, this correlation can be divided into two phases according to curing methods. In the first phase from day 7 to day 28 (annotated as phase I, sheet-curing under drying environment), mortar specimens had slight mass loss and shrinkage. In the second phase from day 28 to day 91 (annotated as phase II, air-curing under drying environment), it can be found that initially (above the red dotted line), mortar specimens had a large mass loss but small length change, and then (below the red dotted line) small mass loss but large length. This phenomenon becomes more obvious as the SAP content increases regardless of the presence of KEA. The phenomenon above the red dotted line is mainly related to the SAP content (about before day 42), and the slopes of the curves below the red dotted line are almost similar, indicating that it seems to have nothing to do with the SAP content (about 42 days later). In summary, the results showed that the addition of SAP slowed down the shrinkage of the mortar under the drying environment, whereas the addition of KEA accelerated the shrinkage slightly may relate to the pore refinement. In general, drying shrinkage is mainly caused by moisture evaporation and migration which is related to the formation of surface tension of the liquid and radius of menisci curvature according to the Kelvin and Laplace equations (Kovler and Zhutovsky 2006), meanwhile, it also is related to the micropores volume (Mehta and Monteiro 2006). Consequently, the pores distribution characteristics are significant for shrinkage analysis under a drying environment (Zhang et al. 2015). The results of pores size distribution characteristics and the relationship between drying shrinkage and pores volume are discussed in Sections 3.2 and 3.4.

3.2 Pores distribution characteristics

Mortar samples with the same curing and drying conditions were measured by the MIP technique, the results are shown in Figs. 9 and 10. Overall, mortar samples with SAP have a higher volume of pores than the Ref, and the volume of pores increased with the increase of SAP content, meanwhile, they have similar pore size distribution regardless of presence of SAP and KEA. Furthermore, it can be noticed that mortar samples with SAP show a similar multimodal characteristic, and the shift of the peak point (10 nm to 100 nm) to the right increased with the increase of SAP content. Figure 9 shows the size of the pore distributions and cumulative pore volume of the individual addition of SAP at 7 d, 28 d and 91 d. As can be seen from Fig. 9(a), the peak values gradually decreased and shift slightly to the left as the age increased, especially for the sample with 0.6% SAP. In addition, it can be observed that for the pores in the range of 100 nm to 100 000 nm diameter, the pore volumes with SAP were higher than those of the Ref and that the peak points shift in the range of 10 nm to 100 nm. In addition, when the age was between the 7 d and 28 d, the peak value of Ref was significantly reduced compared to the samples with SAP. In contrast, when the age was between the 28 d and 91 d, compared with Ref, the peak value of the SAP samples significantly decreased. Therefore, the addition of SAP seems to delay the hydration of cement.

Figure 10 shows the pores distribution and cumulative pore volume of the hybrid addition of SAP and KEA at 7 d, 28 d and 91 d. Although the incorporation of KEA seems to have similar trends compared to the samples without KEA, the addition of KEA resulting in the curve shifting to the left more significantly. It may indicate that the addition of KEA promoted pore refinement (Corinaldesi and Nardinocchi 2016), a similar conclusion can also be echoed in Fig. 8. In addition, peak points in the range of 10 nm to 100 nm: when the age was between the 7 d and 28 d, the peak value of Ref-KEA was significantly reduced compared to the samples with SAP. In contrast, when the age was between the 28 d and 91 d, compared with Ref-KEA, the peak value of the SAP samples significantly decreased. This phenomenon is consistent with the results of pore distribution characteristics with individual addition of SAP. Thus, it may mainly be related to the introduction of SAP and not to the addition of KEA. Besides, comparing Figs. 9 and 10, it can be seen that when the age is from 28 d to 91 d, the pore volume, in the range of 0 to 100 nm, of the samples with SAP reduced greater than Ref and Ref-KEA, respectively. It seems to mean that the SAP played a significant role in storing water inside the mortar matrix and promoting further hydration (Wehbe and Ghahremaniezhad 2017; Oh and Choi 2018; Lyu et al. 2019).

3.3 Compressive strength

Compressive strength was determined with the mortar samples under the same curing and drying conditions. Figure 11 shows the effect of the individual or hybrid inclusion of SAP and KEA on compressive strength of mortar specimens at 7 d, 28 d and 91 d, respectively. It can be seen that the addition of SAP has a negative influence on the compressive strength of the mortar at all ages, especially for an early age, and the compressive strength of mortar specimens decreased with the increase of SAP content. Similar results were also attained by some scholars (Kong et al. 2014; Song et al. 2016; Gupta 2018). In this work, the addition of KEA to mortar specimens had little effect on its compressive strength, however, some studies have shown that the use of KEA
causes a slight increase of compressive strength (Gagné 2016). It also seemed that the introduction of SAP had a significant influence on compressive strength, especially for the mortar with 0.6% SAP content, the compressive strength reduced more than 50%. The adverse influence of SAP on the compressive strength of mortar specimens was also reported by others (Kong et al. 2014; Song et al. 2016) and was mainly attributed to SAP, which expands after absorbing water, became a hydrogel, and formed voids in the cementitious materials after water release. Indeed, the distinction in compressive strength of mortar samples with and without SAP decreased over time, indicates that the initial difference in porosity is at least partially compensated by further hydration and reduced porosity due to mixing the SAP (Jensen and Hansen 2002; Hasholt et al. 2012), as manifested as the pore volume from day 28 to 91 caused large reduction due to the addition of SAP (see Figs. 9 and 10).

Fig. 9 Pore distribution characteristics with individual addition of SAP.
### 3.4 Effect of pore structure on the drying shrinkage, mass loss and the compressive strength

After 21 days of sheet curing, the surfaces of the mortar specimens were completely exposed to the drying conditions. Thus, it is considered that the mortar specimens were dominated by drying shrinkage from 28 d to 91 d and assume the relative shrinkage (from 28 d to 91 d) as drying shrinkage. In general, for the drying shrinkage, pores with a size of less than 50 nm have a significant effect (Mehta and Monteiro 2006), whereas, in this work, the most probable pore diameters of mortars are mostly in the 0 to 100 nm diameter range and closer to 100 nm; these results can be observed in Figs. 9 and 10. Therefore, the pores volume (pore diameter<100 nm) at 28 d is a considerable indicator for evaluating drying shrinkage of pores.

![Graphs showing pore size distribution and cumulative pore volume](image)

**Fig. 10** Pore distribution characteristics with hybrid addition of SAP and KEA.
mortars in this study. The relationship among the SAP content, the drying shrinkage, the mass loss, and the pore volume (pore diameter<100 nm) were presented in Fig. 12. As can be seen in Fig. 12(a), the pore volume (pore diameter<100 nm) increased with the increase of SAP content. At the same time, the pore volume (pore diameter<100 nm) of mortar with KEA was slightly higher compared with the non-KEA. A similar result can also be
seen in Fig. 12(d), especially for the samples of S0.6 and KEA-S0.6, although the total pore volume of S0.6 was larger than that of KEA-S0.6, its pore volume (diameter<100 nm) was smaller than KEA-S0.6. Thus, it was verified again that KEA promotes the pore refinement (Corinaldesi and Nardinocchi 2016). Figures 12(b) and 12(c) show the correlation between the drying shrinkage/the mass loss and pore volume (pore diameter<100 nm). It can be found that the drying shrinkage/the mass loss increased with increasing pore volume. Furthermore, it was verified that the high correlation between the drying shrinkage/the mass loss and pore volume less than 100 nm diameter. A similar result was also obtained by Chol et al. (2019). From the results in Figs. 12(c) and 12(d), they also proved the reason that the mass losses of samples with KEA were less than that without the KEA, which are shown in Figs. 5(b) and 6(b). To sum up, the addition of SAP and KEA resulted in an increase in pore volume with pore diameter in the range of 0 to 100 nm at 28 d, especially for samples with KEA, and this result ultimately increased the drying shrinkage. However, for samples with KEA, the moisture evaporation reduced due to that KEA promoted the pore refinement.

A macropore, according to the general classification by the IUPAC (Aligizaki 2005; Zhang et al. 2015), means a pore diameter greater than 50 nm in diameter which is mainly related to the strength. In addition, the fractal dimension and the capillary pore volume are related to the compressive strength as the positive power function and negative power function respectively according to the conclusion by Lin (Jin et al. 2017). Thus, in this study, the compressive strength vs. the pore volume (pore diameter>50 nm) was plotted in Fig. 13. As can be seen from the regression results that the relation between the compressive strength and the pore volume (pore diameter>50 nm) follows a power function with a relatively high correlation regardless of SAP content and with/without KEA, especially for the compressive strength vs. the pore volume at 7 d and 28 d, their regression coefficients were 0.91 and 0.92, respectively. Accordingly, a pore volume greater than 50 nm can characterize the compressive strength regardless of presence of SAP and KEA.

4. Conclusions

Based on the presented experimental results and discussion, the following conclusions could be drawn:

1. The incorporation of SAP can effectively mitigate its autogenous shrinkage and the length change value of the mortar with SAP is smaller than Ref until 49 d, regardless of with/without KEA. Moreover, the hybrid addition of SAP and KEA increases the initial expansion of the specimens as compared with the individual addition of KEA, which has a beneficial effect on compensating for the shrinkage of the mortar under drying conditions.

2. The addition of SAP seems to be negative for relative shrinkage (equivalent to drying shrinkage), whereas, the addition of KEA seems to improve the adverse effects, in particularly, when the SAP content is greater than or equal to 0.2%, the combined effect of SAP and KEA is more prominent. It is because KEA improves the early expansion of the mortar. In addition, the length change vs. mass change has a good linear correlation under the drying environment.

3. The addition of SAP seems to delay cement hydration and increase the volume of macropores (greater than 100 nm), thereby reducing the compressive strength of the mortars. In contrast, the introduction of KEA slightly promoted the formation of micropores, resulting in a slight increase in compressive strength compared to the samples without KEA.

4. The addition of SAP and KEA resulted in an increase in pore volume with pore in the range of 0 to 100 nm diameter at 28 d, especially for samples with KEA, and this result ultimately increased the drying shrinkage. However, for samples with KEA, in view of it promoting pore refinement so as to reduce moisture evaporation.

5. From the regression analysis, the relation between macropore volume (pore diameter>50 nm) vs. compressive strength follows a power function with a relatively high correlation regardless of SAP content and with/without KEA. Therefore, a pore volume greater than 50 nm in diameter can characterize the compressive strength regardless of the presence of SAP and KEA.

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References

Aligizaki, K. K., (2005). “Pore structure of cement-based materials: Testing, interpretation and requirements.”
London & New York: Taylor & Francis.
Almeida, F. C. R. and Klemm, A. J., (2018). “Efficiency of internal curing by superabsorbent polymers (SAP) in PC-GGBS mortars.” 
Cement and Concrete Composites, 88, 41-51.
Bentz, D. P. and Jensen, O. M., (2004). “Mitigation strategies for autogenous shrinkage cracking.” 
Cement and Concrete Composites, 26(6), 677-685.
Cabrera, J. G., (1996). “Deterioration of concrete due to reinforcement steel corrosion.” 
Cement and Concrete Composites, 18, 47-59.
Chatterji, S., (1995). “Mechanism of expansion of MgO.” 
Cement and Concrete Research, 25(1), 51-56.
Collinsa, F. and Sanjayan, J. G., (2000). “Cracking tendency of high-strength concrete.” 
Journal of the Chinese Ceramic Society, 44(2), 189-195. (in Chinese)
Cusson, D. and Hoogeveen, T., (2008). “Internal curing on shrinkage of high performance concrete incorporating a hybrid system of SAP and SRA.” 
Cement and Concrete Composites, 30, 791-798.
Corinaldesi, V. and Nardinocchi, A., (2016). “Mechanical characterization of engineered cement-based composites prepared with hybrid fibres and expansive agent.” 
Composites Part B: Engineering, 98, 389-396.
Cohen, M. D., Olek, J. and Dolch, W. L., (1990). “Mechanism of plastic shrinkage cracking in portland cement and portland cement-silica fume paste and mortar.” 
Cement and Concrete Research, 20, 103-119.
Collinsa, F. and Sanjayan, J. G., (2000). “Cracking tendency of alkali-activated slag concrete subjected to restrained shrinkage.” 
Cement and Concrete Research, 30, 791-798.
Cornish, D. and Hoogeveen, T., (2008). “Internal curing of high-performance concrete with pre-soaked fine lightweight aggregate for prevention of autogenous shrinkage cracking.” 
Cement and Concrete Research, 38(6), 757-765.
Gagné, R., (2016). “Expansive agents.” In: P. C. Aitchinson and R. J Flatt, Eds. Science and Technology of Concrete Admixtures. London: Woodhead Publishing, 441-456.
Gruyaert, E., Debub, B., Snoeck, D., Díaz, P., Arizzo, A., Tziviloglou, E., Schlangen, E. and De Belie, N., (2016). “Self-healing mortar with pH-sensitive superabsorbent polymers: Testing of the sealing efficiency by water flow tests.” 
Smart Materials and Structures, 25(8), Article No. 084007.
Guo, S., (2018). “Effect of presoaked superabsorbent polymer on strength and permeability of cement mortars.” 
Magazine of Concrete Research, 70(9), 473-486.
Hasholt, M. T., Jensen, O. M., Kovler, K. and Zhivotovsky, S., (2012). “Can superabsorbent polymers mitigate autogenous shrinkage of internally cured concrete without compromising the strength?” 
Construction and Building Materials, 31, 226-230.
Jensen, O. M. and Hansen, P. F., (2001). “Water-entrained cement-based materials: I. Principles and theoretical background.” 
Cement and Concrete Research, 31, 647-654.
Jensen, O. M. and Hansen, P. F., (2002). “Water-entrained cement-based materials: II. Experimental observations.” 
Cement and Concrete Research, 973-978.
Jin, S., Zhang, J. and Han, S., (2017). “Fractal analysis of relation between strength and pore structure of hardened mortar.” 
Construction and Building Materials, 135, 1-7.
Kong, X. M., Zhang, Z. L. and Lu, Z. C., (2014). “Effect of pre-soaked superabsorbent polymer on shrinkage of high-strength concrete.” 
Materials and Structures, 48(9), 2741-2758.
Kovler, K. and Zhivotovsky, S., (2006). “Overview and future trends of shrinkage research.” 
Materials and Structures, 39(9), 827-847.
Laustsen, S., Hasholt, M. T. and Jensen, O. M., (2013). “Void structure of concrete with superabsorbent polymers and its relation to frost resistance of concrete.” 
Materials and Structures, 48(1-2), 357-368.
Lee, H. X. D., Wong, H. S. and Buenfeld, N. R., (2016). “Self-sealing of cracks in concrete using superabsorbent polymers.” 
Cement and Concrete Research, 79, 194-208.
Li, M., Liu, J., Tian, Q., Wang, Y. and Xu, W., (2017). “Efficacy of internal curing combined with expansive agent in mitigating shrinkage deformation of concrete under variable temperature condition.” 
Cement and Concrete Research, 145, 354-360.
Liu, J., Farzadnia, N., Shi, C. and Ma, X., (2019a). “Effects of superabsorbent polymer on shrinkage properties of ultra-high strength concrete under drying condition.” 
Construction and Building Materials, 215, 799-811.
Liu, J., Farzadnia, N., Shi, C. and Ma, X., (2019b). “Shrinkage and strength development of UHSC incorporating a hybrid system of SAP and SRA.” 
Cement and Concrete Composites, 97, 175-189.
Liu, J., Shi, C., Ma, X., Khayat, K. H., Zhang, J. and Wang, D., (2017). “An overview on the effect of internal curing on shrinkage of high performance cement-based materials.” 
Construction and Building Materials, 146, 702-712.
Lura, P., (2003). “Autogenous deformation and internal curing of concrete.” Thesis (PhD). Technical University Delft, The Netherlands.
Lyu, Z., Guo, Y., Chen, Z., Shen, A., Qin, X., Yang, J., Zhao, M. and Wang, Z., (2019). “Research on shrinkage development and fracture properties of internal curing pavement concrete based on humidity compensation.” 
Construction and Building Materials, 203, 417-431.
“Effect of initial curing conditions on air permeability and deicing salt scaling resistance of surface concrete.” *Journal of Asian Concrete Federation*, 5(1), 56-64.

Zhang, W., Hama, Y. and Na, S. H., (2015). “Drying shrinkage and microstructure characteristics of mortar incorporating ground granulated blast furnace slag and shrinkage reducing admixture.” *Construction and Building Materials*, 93, 267-277.

Zhutovsky, S., Kovler, K. and Bentur, A., (2002). “Efficiency of lightweight aggregates for internal curing of high strength concrete to eliminate autogenous shrinkage.” *Materials and Structures*, 35(2), 97-101.