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Evaluation of Cement Soundness using the ASTM C151 Autoclave Expansion Test

by Hossein Kabir and R. Douglas Hooton

KEYWORDS

Cement MgO Content, Periclase, Soundness, Autoclave Expansion Test

ABSTRACT

In this research, the expansive behavior and microstructure of cements having different MgO contents and autoclave expansions is investigated. Cements have been found that failed the ASTM C151 autoclave expansion test (AET) limit while still being well below the chemical MgO limit in ASTM C150, while other cement samples from the same source and with similar MgO contents passed the AET expansion limit. While it was first developed to assess cement soundness using an accelerated technique, the autoclave expansion test was never correlated to long term concrete performance. While free lime (F_{CaO}) can also influence expansions in the autoclave expansion test, it was not incorporated as a variable in this study. In the current study, paste, mortar, and concrete prisms were made using these cements then stored at different temperatures for at least one year and little difference was found in their expansions. Based on this, it is concluded that the AET provides an unrealistic basis for assessing volume stability as well as for rejection of portland cements for use in concrete. It is recommended that the AET be dropped from ASTM C150 requirements.

REFERENCE

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INTRODUCTION

The term "soundness" is used to describe cement paste specimens that do not exhibit cracks, disintegration, or other flaws, that result from excessive volume change. While excess quantities of hard burned free CaO also has an influence, one of the main causes of such volume change is the hydration of crystalline magnesia, i.e., periclase (MgO) in cement (Gonnerman et al. 1953, Zheng et al. 1991, He and Huang 2002). A correlation exists between cement MgO content and the long-term expansion of cement pastes moist cured at room temperature (Gonnerman et al. 1953, Chatterji 1995). As a result, a maximum MgO prescriptive limit for cement soundness can be found in most portland cement specifications. The maximum MgO content in cement standards is 5% in China (GB 175 2001), 5% in Europe (BS EN 197-1 2000), 5% in Canada (CSA A3001 2018), and 6% in US (ASTM C150 2019). At ambient temperatures, it was theorised that slow hydration of cements made with "excessive amounts" of periclase could result in deleterious expansion and cracking in concrete structures, although no concrete failures have been attributed to cements failing soundness testing or containing high amounts of MgO in over 60 years (Klemm 2005). Similarly, in countries where the autoclave test is not required, there have been no reported cases of MgO-related soundness failures (Klemm 2005). Expansions associated with higher levels of free lime can be reduced or eliminated as cement ages and it is often difficult to conduct this type of test program with cements of variable free lime contents. Additionally, higher free lime cements or hard-burned free lime in cement are not commonly found in modern day cements due to better efficiencies and process controls in cement plants.

Soundness Testing Methods

In the 1920s, there were some field failures which were attributed to excess free lime in the concrete. A "pat" test was developed to provide an indication of free lime expansion problems. As summarized by Klemm (2005), Lerch (1929) demonstrated that free lime alone was responsible any abnormal expansion, cracking, or distortion in the boiling water pat test. The testing of cements containing 5.0%, 8.0%, and 10.7% MgO showed no unsoundness as measured by the 5-hour steam test (at 100°C). According to Mehta (1978), "at the ASTM Meeting in 1940 the results of a cooperative study on various soundness tests on 221 cements by 30 participating laboratories were discussed. It was shown that the pat test and the Le Chatelier test were not

* Graduate Student and Professor, respectively, Dept. of Civil & Mineral Engineering, University of Toronto, Toronto, Ontario, Canada M5S1A4 (416) 978-5912, hooton@civ.utoronto.ca.

adequate for measuring the potential expansion due to delayed hydration of free CaO and crystalline MgO.” So the pat test was dropped by ASTM in favor of the autoclave expansion test at the same time all the cement types were combined into the new C150 Specification in 1940, even though a ballot on the autoclave expansion test had failed to obtain the required two thirds majority of affirmative votes at the 1939 ASTM meeting (Mehta 1978).

However, the autoclave expansion is sensitive to the periclase to brucite transformation in cement paste bars (Mehta 1978). The test method consists of mixing a cement and enough water make a paste of normal consistency, as per ASTM C187 (2016). That paste is cast into 25 mm × 25 mm × 250 mm (1 in. × 1 in. × 10 in.) bars and moist cured at 23 °C for 24 hours. After the bars are stripped from the molds, lengths are measured and then the bars are placed in an autoclave (pressure and heat) with free water. Over a 6-hour period, the autoclave is pressurized to 2 MPa at about 216 °C (420 °F), held at that pressure and temperature, and then returned to room pressure. The bar is removed from the autoclave and cooled down to room temperature and its length is re-measured to calculate the autoclave expansion.

The two reactions in cement that impact autoclave expansion are periclase (free MgO) that can slowly react to form brucite (Mg(OH)₂) and hard-burned free lime (CaO) that reacts to form portlandite (Ca(OH)₂) (Chatterji 1995). Both reactions could result in expansion. Additionally, the cement C₃A content, SO₃ content, and Blaine fineness have also been reported to have some relationship to autoclave expansion (Gonnerman 1953).

In the ASTM C150 (2019) and CSA A3001 (2018) standards, the maximum autoclave expansion is limited to 0.8 and 1.0% respectively. The usefulness of the autoclave test has been controversial and has not been widely adopted in standards outside of North America (Mehta 1978). The autoclave exposure of 1-day old paste bars is very severe and can lead to unrealistic and excessive expansion (Klemm 2005).

As an alternative soundness test, the Le Chatelier soundness test (EN 196-3) is in use in many countries, including Europe, India and Brazil, to detect expansion mainly from excess free lime. Instead of casting and autoclaving paste prisms, the Le Chatelier method involves casting pastes in a longitudinally split brass cylinder mold as shown in Figure 1. The expansion of the paste is quantified by how much the brass mold deforms, as measured at the tip of needles which are soldered perpendicularly to the mold on either side of the split. The distance between the two needle tips before and after boiling (100 °C) for 3 hours is measured. EN 196-3 has a maximum needle spread limit of 10 mm subsequent to boiling.

One important difference between the 216 °C autoclave expansion test and the Le Chatelier 100 °C boiling water soundness test is that Le Chatelier test has been shown not to be susceptible to MgO-induced expansion, while the autoclave expansion test is susceptible to both CaO- and MgO-induced expansion, since the maximum temperature is 100 °C in the Le Chatelier versus 216 °C for autoclave test (Nokken 2010).

In the late 1800s failures in Europe with cements with greater than 15% MgO resulted in all specifications having MgO content limits in cement which are typically a maximum MgO of 5.0% or 6.0% (ASTM C150, AASHTO M 85, CSA A3000, EN 197). Despite many countries only having MgO content limits and no requirements to measure MgO-induced expansion (i.e. no autoclave expansion test and only Le Chatelier soundness test), no cases of concrete failures due to MgO could be found, except for cases with very high MgO (>15%) in the 1800s

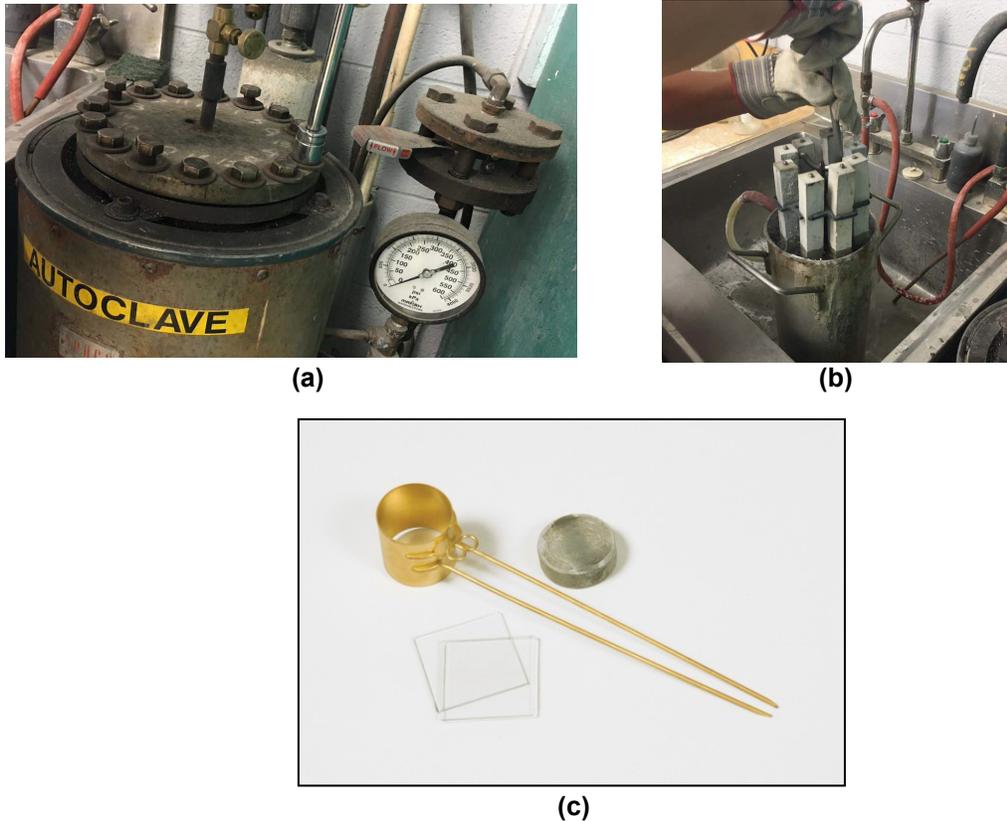


Figure 1. (a) Autoclave chamber, (b) autoclave paste bars (center), and (c) Le Chatelier split ring mold. (Figure 1(c) from <https://www.ele.com/Product/le-chatelier-mould>.)

(Gonnerman 1953, Mehta 1978, Helmuth 1998, Klemm 2005). The extensive literature reviews and subsequent testing documented in those previous reports (Gonnerman 1953, Helmuth 1998, Mehta 1978, Klemm 2005) found a general lack of correlation between autoclave expansion and concrete expansion. Additional literature searches did not result in finding any additional instances of concrete failures due to MgO-induced expansion.

The reasons given for the lack of correlation between autoclave expansion and concrete as well as autoclave expansion and Le Chatelier soundness expansion are related back to the different temperatures that the specimens are subjected to: 216 °C and 100 °C, respectively (Klemm 2005, Mehta 1978). At 23 °C (in-place concrete temperatures), the MgO reactions can take years to occur (Gonnerman 1953). The reason for the high temperature in the autoclave test is to speed up the reactions that lead to expansion. This increase in reaction rate is roughly three to five orders of magnitude higher (6 hours versus years). Another consequence of the high temperature is that the specimens are weakened by changing the main hydration products and the brucite formation is rapid and concentrated. Further, in concrete, creep and shrinkage can alleviate expansive stresses, but do not play a role in the accelerated autoclave test. These behaviors factor into the poor correlation between autoclave expansion of cement paste and concrete performance.

At the high autoclave temperatures, the conversion of the main hydration product, C-S-H to C₂SH, weakens the bar (Gao 2007). When those bars are weakened, they are less able to resist

expansive stress and more expansion can occur. In concrete, that conversion will not happen since they are not exposed to those high temperatures.

When the periclase reaction happens rapidly (within a few hours) in the high autoclaving temperature environment, the local area around the periclase is super-saturated with magnesium ions, which leads to a localized concentration of brucite crystal formation (Helmuth 1998, Chatterji 1995). In concrete at lower temperatures those periclase reactions take years to occur (Gonnerman 1953, Klemm 2005, Helmuth 1998). Because of the slow reaction rate, as in normal concrete hydration, magnesium ions have the opportunity to diffuse throughout the paste matrix, which leads to smaller crystal sizes that are more uniformly distributed. Large crystals develop more stress in the bulk matrix compared to smaller well distributed brucite crystals at the same total brucite level.

Periclase (MgO) has been used as expansive and shrinkage compensating admixtures in cements to compensate for normal drying shrinkage (Mo 2014, Samdariya 2009). Expansive and shrinkage compensating cement concrete make use of similar reaction products (Mo 2014, Samdariya 2009). In addition, since the reaction of periclase is slow at ambient temperatures, any expansive stress in the concrete resulting from brucite formation is reduced if it forms in surrounding pores or is dissipated due to creep.

Recently, at certain cement plants, problems have occurred due to failure to meet the ASTM C150 (2019) autoclave expansion limit of 0.80% (or the CSA A3001 (2018) 1.0% limit) even though these cements had MgO contents well below the 6.0% limit in C150 (or the CSA limit of 5.0%). As a result, the primary objective of this research was to determine if the ASTM C151 autoclave test is serving a useful purpose in protecting the consumer or if the maximum limit on MgO content is sufficient. Cement free lime contents were not measured for all of the cements tested, however, at the plant in question free lime contents measured over several months during the relevant period of production, values averaged 1.4% and ranged from 0.9% to 2.0%.

For this purpose, paste, mortar, and concrete prisms were made with different cements and subjected to various curing conditions and their long-term expansions, as well as their microstructure, were examined and compared with AET results. Since it is possible that the autoclave exposure is too harsh for paste prisms cured for only one day, thus leading to exaggerated expansions, in this study the pre-autoclave curing period was also extended to 2, 7, and 28 days. Additionally, cements that failed the autoclave expansion limit were partially replaced with slag cement to evaluate its impact on autoclave expansion. Additionally, the usefulness of the European Le Chatelier test for detecting cement expansion due to the presence of excessive periclase was evaluated. X-ray powder diffraction (XRD) and scanning electron microscopy (SEM) techniques were used to study anhydrous cements as well as hydrated pastes. With the aid of thermal analysis, the brucite content of paste specimens subjected to different curing conditions was evaluated and results were compared.

EXPERIMENTAL

Cementitious Materials

The chemical compositions of the five ASTM Type I cement samples used are shown in Table 1. These cements had MgO contents well below the 5% or 6% limits specified in CSA A3001 (2018), and ASTM C150 standards (2019), respectively. Nonetheless, three of them passed the

autoclave expansion limit, while two did not. It is worth noting that Cements *B*, *C*, *D*, and *E* were all produced at a single cement plant. Paste prisms, 25 × 25 × 280 mm, with a 250-mm gauge length, were cast at a water to cement ratio that gave a normal consistency as per ASTM C187 (2016). In some mixtures, ground granulated blast-furnace slag (see Table 1) was used to partially replace 25% of the cement, and these pastes were also cast at normal consistency.

Table 1. Composition and properties of portland cements, % by mass unless otherwise indicated.

ID	A	B	C	D	E	Slag
CaO	61.20	60.50	60.85	59.34	59.38	39.51
SiO ₂	19.30	20.32	20.27	20.13	19.63	39.62
SO ₃	4.00	4.17	4.34	4.35	4.82	2.68
MgO	2.60	4.63	4.02	4.44	4.36	10.80
Al ₂ O ₃	5.50	4.07	4.91	4.53	4.37	7.63
Fe ₂ O ₃	2.70	3.67	3.19	3.54	3.57	0.53
LOI	2.50	0.49	0.67	0.35	0.8	-
Total Alkali	0.52	0.96	0.82	0.99	1.06	0.53
CaO _{free}	-	-	-	1.57	1.68	-
Autoclave Expansion (%)	0.03	6.6	0.55	6.80	0.70	0.02
Blaine Fineness (m ² /kg)	376	452	422	407	467	587

Note: Free CaO contents were not measured for all cements, however, at the plant in question over several months during the relevant period of production, values averaged 1.4% and ranged from 0.9% to 2.0%.

Test Specimens

Mortar bars, 25 × 25 × 285 mm, with a 250-mm gauge length, were also cast at a water-to-cementitious materials ratio of 0.485 and a sand-to-cementitious materials ratio of 2.75 as per ASTM C109 (2016). Graded Ottawa sand, meeting the ASTM C778 requirements (2017), was used for mortar mixtures. In addition, 75 × 75 × 285-mm concrete prisms, with a 250-mm gauge length, were cast. Concrete mix designs conformed to that used in ASTM C1293 (2018) with a cement content of 420 kg/m³ and a coarse-to-fine aggregate ratio of 60:40 by mass. The mixtures were cast at water-to-cement ratios of 0.40, 0.50, and 0.60. Non-reactive glacial sand of mixed origin and a crushed limestone coarse aggregate with nominal maximum size of 19 mm were used that met ASTM C33 requirements (2018).

Immediately after casting, samples were covered with saturated burlap and stored in a sealed container over water for 24 ± 0.5 h. After stripping the molds at 1-day, specimens were submerged in water and placed at different temperatures. Using ASTM C490 (2017), the initial lengths of paste prisms were measured on the second day after equilibrating at the intended storage temperature. Mortar and concrete prisms were stored in water for one week prior to taking their initial length measurement.

After 7 days of water curing, the average length change was measured for specimens exposed to drying in a 50% RH chamber. The Le Chatelier soundness test (EN 196-3) was also performed on paste samples mixed at normal consistency. The pastes were cast into split rings fitted with two 150 mm long needles attached on either side of the split opening. At 1 day of age these specimens were boiled for 3 hours and the spread of the needles resulting from expansion of the paste were measured. In addition, three 10 mm × 10 mm × 40-mm miniature paste prisms were cast and also subjected to the same boiling water condition as the Le Chatelier test and the

expansions were measured after 2, 4, 6, and 8 h using a length comparator. The autoclave expansion test was performed as per ASTM C151 (2018) (3 h at 2 ± 0.05 MPa saturated steam pressure at $215.7 \pm 1.3^\circ\text{C}$).

In this study, specimen identifications are as shown in Figure 2 (cement identifications were listed in Table 1).

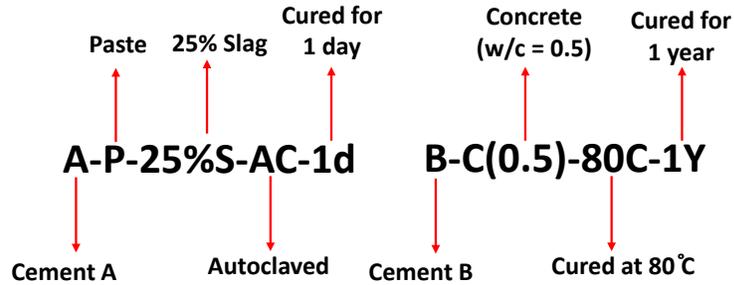


Figure 2. Labeling convention for test specimens. Cements A through E were evaluated with and without slag cement (S), in pastes (P), mortars (M) and concrete (C) in autoclaves (AC) and at 23°C and 80°C .

Sample Conditioning and Analysis Procedures

Prior to sample preparation for SEM analysis, saturated paste bars were immersed in isopropyl alcohol for a week, to stop hydration. They were then placed in a $35 \pm 5^\circ\text{C}$ oven for 28 days at atmospheric pressure to remove the alcohol. Sections of the paste bars were cut and fixed on a glass slide and impregnated with fluorescent-dyed epoxy with a resin to hardener ratio of 4:1 to stabilize the media and fill any gaps and pores. During the sample preparation, kerosene was used as a coolant. As soon as the epoxy began to harden, the samples were placed in a desiccator over silica gel for 24 h.

Diamond polishing pastes were applied to facilitate reaching the desired surface smoothness with successively finer particle sizes from $9\ \mu\text{m}$ to $0.25\ \mu\text{m}$. A mild relief polishing cloth impregnated with a diamond paste was placed on a Buehler Ecomet rotating lap and used to smooth the surface of the thin polished sections. Finally, an ultrasonic device was used to remove dust and slurry from the surface after polishing. To analyze anhydrous cement, a similar procedure was followed, but a column of powder was stabilized with fluorescent dyed epoxy. To avoid charging during the SEM analysis, polished surfaces were coated with a 200-\AA thick carbon layer in a high-vacuum chamber. The SEM analysis was operated in back-scattered electron (BSE) imaging mode at an accelerating voltage of 15 keV with a beam current of 2 mA.

The XRD analysis was performed as per ASTM C1365 (2018). For this purpose, parts of the non-dried pieces of paste were crushed into smaller particles with a hammer and immersed in isopropyl alcohol for a week to stop hydration. Samples were then removed from alcohol and placed inside a 38°C vacuum oven over silica gel and soda-lime pellets. Dried samples were further crushed into powders using a mortar and pestle and passed then through a $45\text{-}\mu\text{m}$ sieve. For quantitative XRD analysis (QXRD), the powder was mixed with 10 wt.% of lithium fluoride as an internal standard, using a McCrone Micronizing Spex Mill. A Philips PW1830 diffractometer with monochromatic Ni-filtered $\text{Cu K}\alpha$ radiation ($\lambda = 1.5418\ \text{\AA}$) running at 40 kV and 30 mA was used for XRD analyses. As well, the XRD patterns of pressed powders were obtained from 2-theta angles of 15° to 65° using a step size of 0.02° , with a dwell time of 2 sec.

Approximately 100 mg of the ground powder prepared for XRD analysis was used for thermogravimetric analysis (TGA). In this test, sample mass loss during the heating process was recorded using a NETZSCH STA 409 DTA/DTG over a temperature range of 30 °C to 1050 °C at a heating rate of 10 °C/min. The atmosphere used was nitrogen gas at a flow rate of 50 mL/min. Differential thermal analysis (DTA) was used to determine the changes in enthalpy as a function of temperature. During the heating process, decomposition (dehydration) of brucite took place as an endothermic peak between 300 °C to 450 °C.

RESULTS

Properties of Anhydrous Cements

The XRD analysis on anhydrous cements *B* and *C* indicated that both their total MgO (found from XRF analysis) and free MgO contents (periclase) were essentially equal (Figure 3). Therefore, the autoclave expansions were not a function of periclase content and were mainly affected by the periclase reactivity.

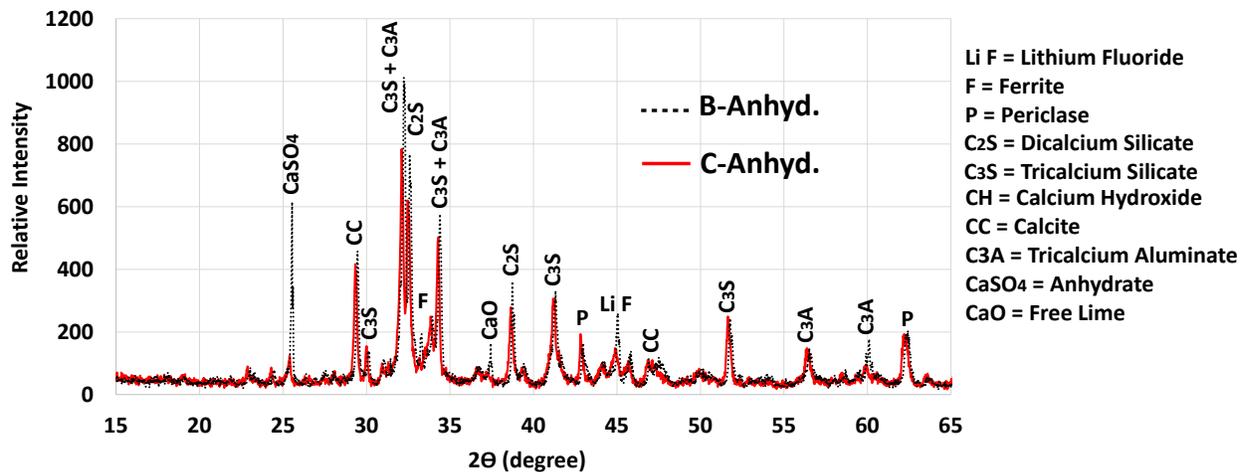


Figure 3. XRD analysis of anhydrous cements *B* and *C* that failed and passed the AET expansion limit respectively.

The periclase reactivity is governed by a major factor, periclase crystal size. In Figure 4, the BSE image of periclase crystals of anhydrous cements *B* and *C* are compared. The periclase crystals in cement *B*, that failed the AET expansion limit, were angular and relatively large, with an average size of 10 μm, hence more destructive. While for cement *C*, which passed the AET limit, these particles were rounded and only around 1 μm in size.

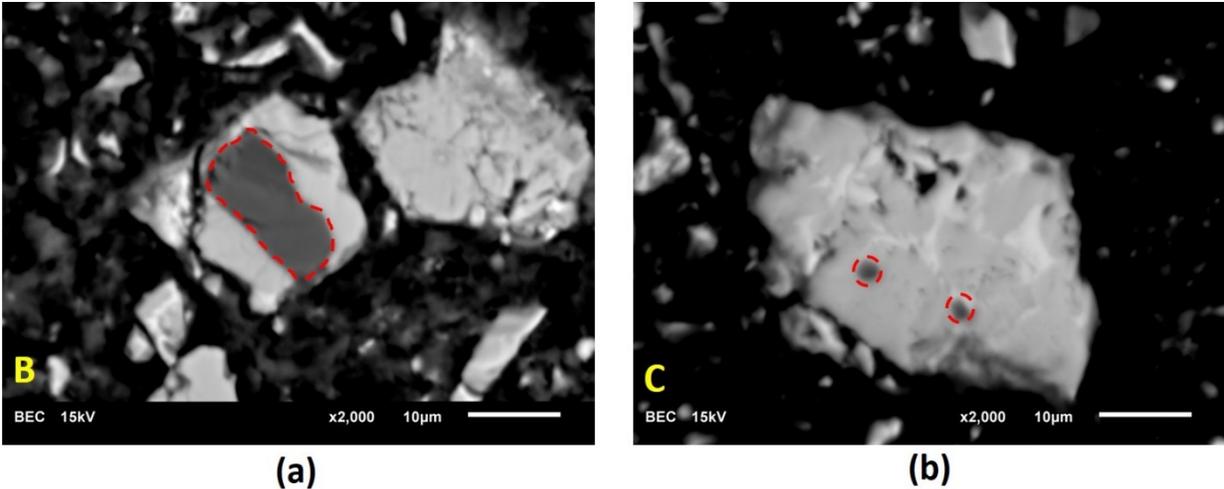


Figure 4. Comparison of the physical properties of periclase crystals (highlighted in red) of anhydrous (a) cement *B* (6.60% expansion in ASTM C151), and (b) cement *C* (0.55% expansion in ASTM C151).

Expansion of Portland Cement Pastes

Expansions of *A*, *B*, *C*, and *E* cement pastes stored at 23, 38, 60, and 80 °C shown in Figure 5. Increasing the curing temperature accelerated the rate of reaction but did not result in any additional long-term volume change. The maximum long-term expansion of cement *A* with MgO content of 2.6% was 0.14% after 450 days of moist curing, which is much higher than its 0.03% autoclave expansion. In contrast, cement *B* with 4.63% MgO expanded 0.33% over the same water storage period but had an average autoclave expansion of 6.6%. Therefore, in one case, the AET underestimated the long-term, ambient temperature expansion of portland cement pastes and grossly overestimated expansion in the other.

Samples of paste specimens made with cement *B* and moist cured at 23 °C and 80 °C were taken at specific ages for chemical analysis. Using thermal analysis (Figure 6), the brucite content versus time was calculated and presented for different temperatures. Figure 6 indicates that the slope of brucite content versus time diagram diminished after six months of storage in water. Although there was a difference in the brucite contents of cement *B* samples, their long-term expansions were similar. It should be noted that the solubility of brucite is very low, so leaching of Mg²⁺ during storage is not considered a concern.

Figure 7a shows the six-month expansions of cement pastes stored in water at 80 °C in relation to their MgO contents. While the number of cements tested here with similar physical properties is small, it is proposed that the MgO content of a cement provides a better indicator of its volume stability than the autoclave expansion. In Figure 7b, paste expansions in 80 °C storage started to stabilize after 7 days, similar to that found by Nokken (2010).

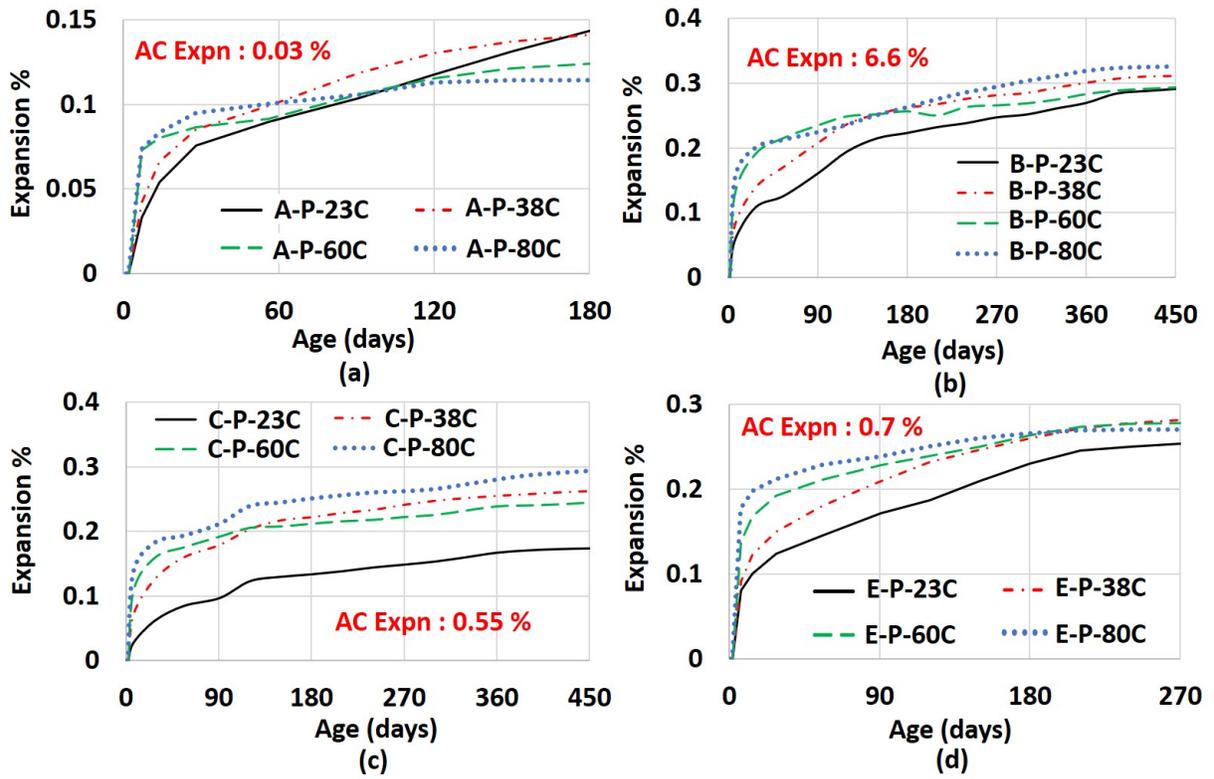


Figure 5. Long-term expansions of cements (a) A, (b) B, (c) C, and (d) E cured at different temperatures.

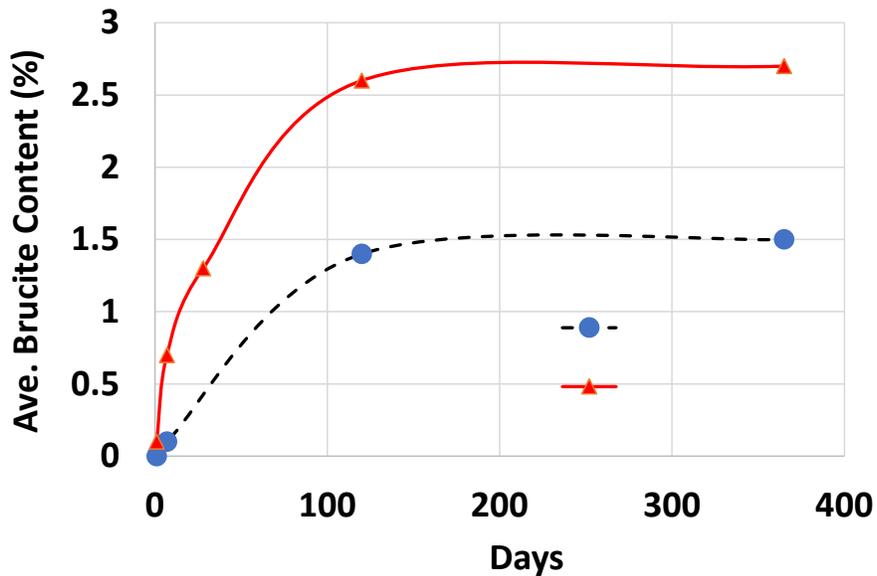


Figure 6. Quantification of brucite content vs. time for cement paste B stored in water at 23 °C and 80 °C using TGA.

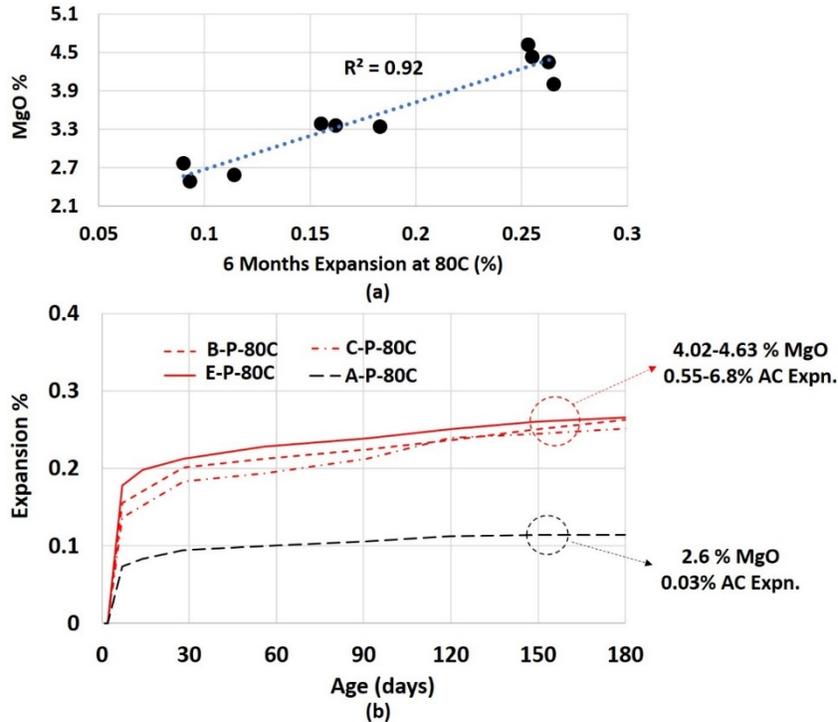


Figure 7. (a) Cement ultimate expansion at 80 °C vs. MgO content. (b) Long-term expansion of cement at 80 °C vs. MgO content.

Figure 8 shows the impact of extending the pre-autoclave curing period in water on the autoclave expansion. Extending the pre-autoclaving period from 1 to 7 days allowed the cement paste prisms to develop more strength and, as a result, the autoclave expansions of *B* and *D* cements were dramatically reduced from 6.6% and 6.8% to 0.6% and 0.5% respectively, well below the CSA A3001(2018) and ASTM C150 (2019) expansion limits. It has been suggested that cements with lower 1-day strengths will be more negatively impacted by autoclave soundness testing. As discussed later, it should be noted that when autoclaved, the strengths of portland cement pastes will be much lower as a result of forming relatively weak $\alpha\text{C}_2\text{SH}$ hydrates. It was also noted that the pre and post-autoclave expansions of paste bars in water at 23 °C were negligible. Figures 8c and 8d compare the length change and visual properties of paste prisms autoclaved at different ages and stored in water before and afterwards. The results indicate that if the pre-autoclave curing period was at least 7 days, all paste specimens remained solid and uncracked. Also, there is negligible expansion in water after autoclaving.

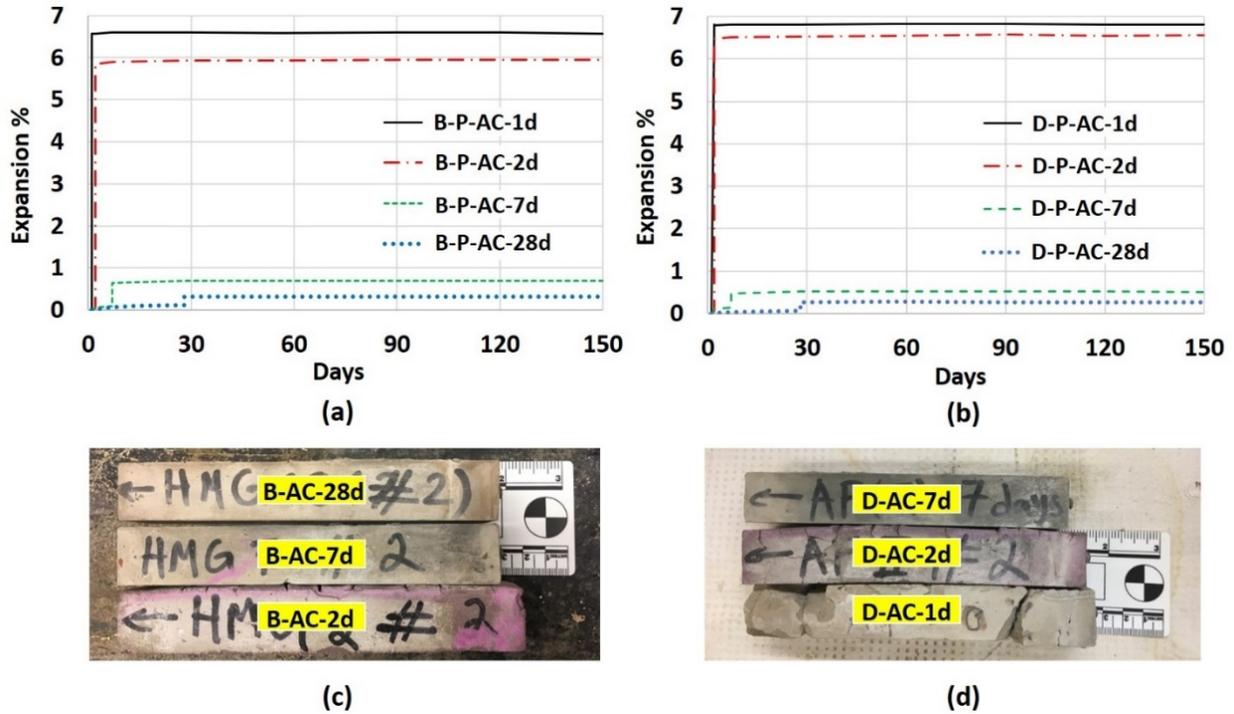


Figure 8. Results of the modified AET for cements (a) B, and (b) D. Visual properties of autoclaved specimens autoclaved at different ages and made with cements that failed the autoclave expansion limit, i.e. cements (c) B, and (d) D.

In Figure 9a, the paste modulus of rupture (MOR) was measured for both B and C paste bars. Results demonstrate that during the first week of curing, the paste MOR approximately doubled. Over the same time period, the compressive strength of paste cubes increased by 22% (Figure 9b). The higher paste tensile and compressive strengths are thought to be one of the main factors limiting the expansion and preventing the formation of cracks.

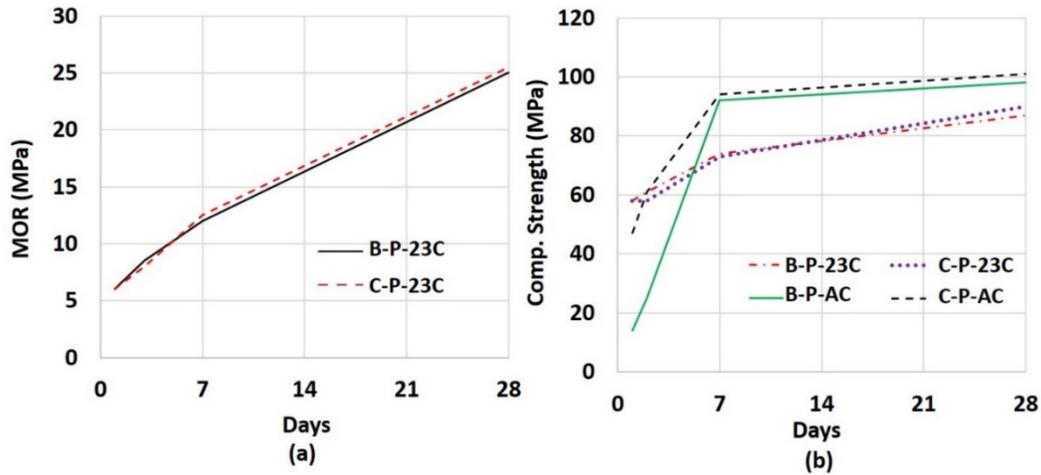


Figure 9. Strength development of cements B and C vs. time.

A BSE image of the 1-day autoclaved paste bar *B*, which failed the AET, is shown in Figure 10, revealing that the matrix was densely cracked and had disintegrated. At the crack interfaces, the MgO grains appeared to be completely converted to brucite (Figure 10b). However, residual periclase crystals were found internal to the cement grains where they were isolated from hydration (Figure 10c).

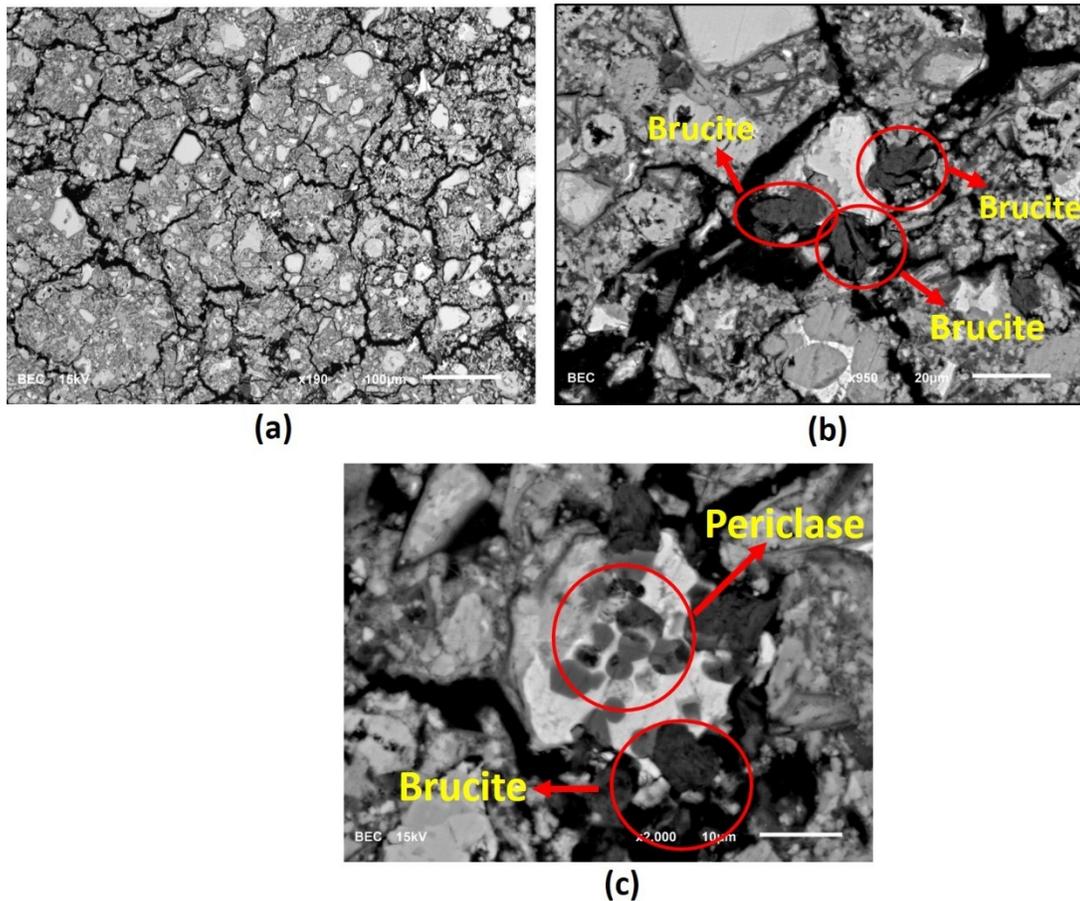


Figure 10. BSE images of cement *B* paste matrix autoclaved at 1-day.

Elemental mapping was done on 1-day autoclaved specimens and an area of 80,000 μm^2 was selected (Figure 11). Brucite (red particles in Figure 11), was located near the cracks (blue zones) and only a small fraction of unreacted magnesium particles, i.e. mostly periclase, were located far from cracks and inside the green zones, see Figure 10a. A possible factor contributing to the excessive expansion of cement *B* could have been the formation of hydrotalcite-type phases. However, there was no overlap of magnesium (red) and aluminum (green) zones so the possibility of hydrotalcite formation in specimens that failed the AET appears to be small (Figure 11c).

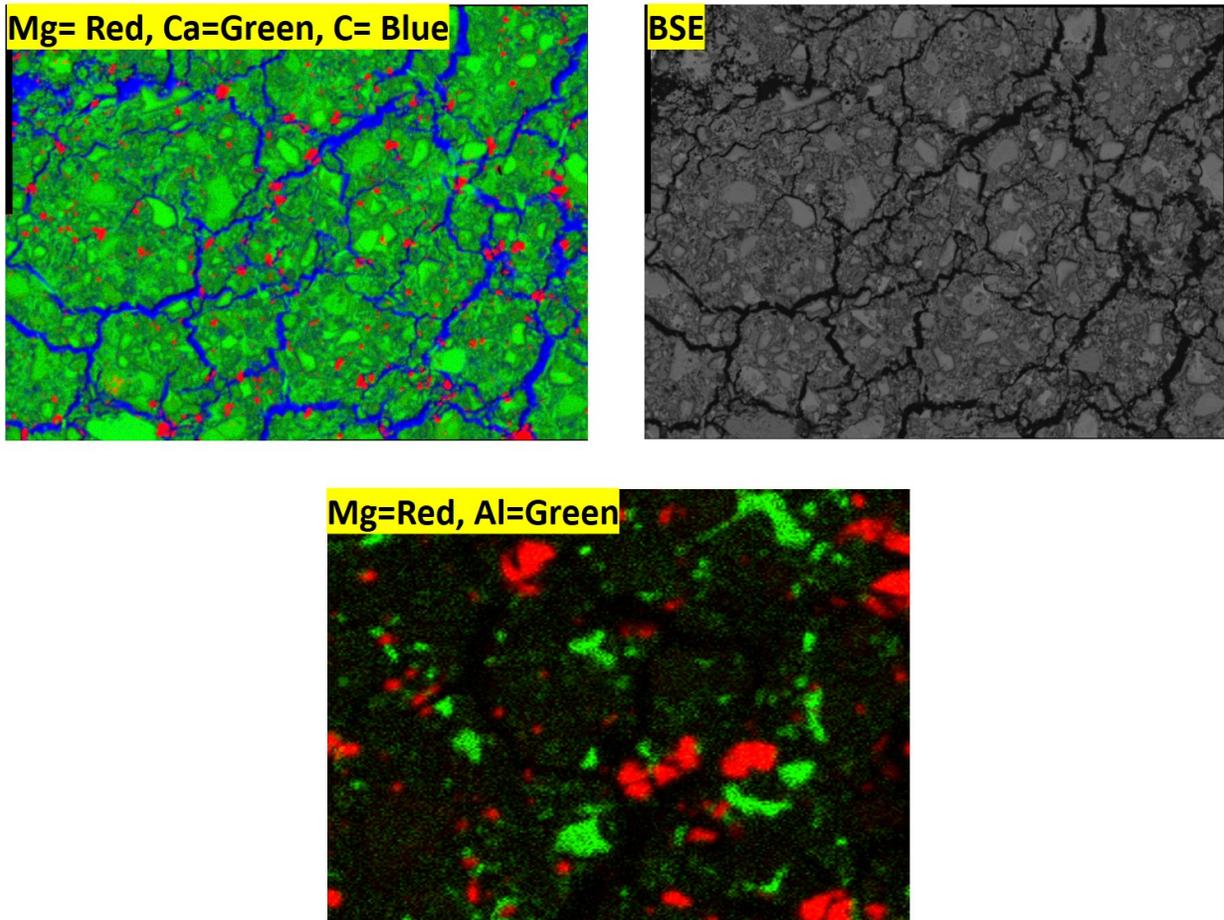


Figure 11. BSE Image and Elemental maps of cement *B* paste autoclaved at 1-day, Al=Aluminum, C=porosity (Carbon from epoxy impregnation), Ca= Calcium, Mg=Magnesium.

In 7-day cured autoclaved paste bars made with cement *B*, the matrix remained sound and solid (Figure 12a). The periclase crystals appear to be partially enveloped by a thin layer of relatively insoluble and impermeable brucite (Figures 12b and 12c). Comparison of Figures 4a and 10b, 10c, and 10d reveals that the volume of hydrated periclase crystals had almost doubled, although this expansion did not induce cracks in the stronger paste matrix. Furthermore, thin layers of impermeable monosulfoaluminate phases (AFm) attached to the periclase crystals also appear to have limited water access to hydrate MgO crystals, see Figure 12d.

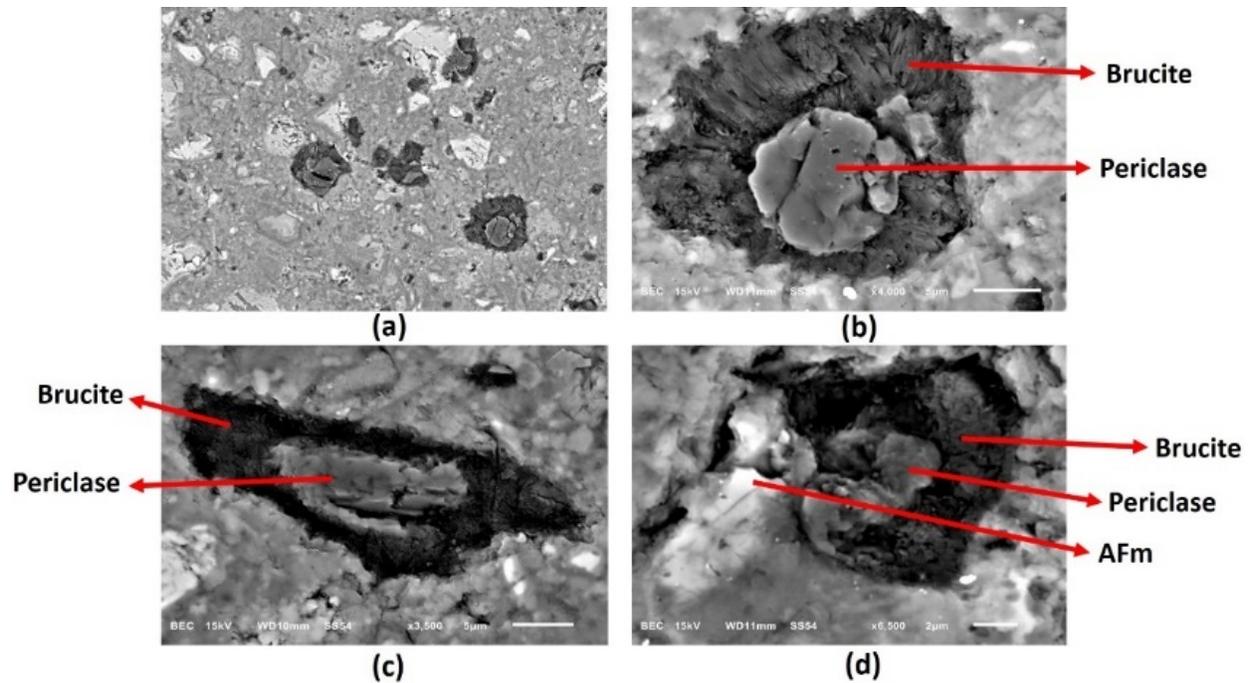


Figure 12. BSE image of cement *B* pastes autoclaved at 7 days.

Autoclaved paste bars made with cement *B* and pre-cured for 28 days had the least expansion, and the matrix remained uncracked after the AET as shown in the low-magnification image in Figure 13a. Furthermore, a relatively thick layer of AFm phases was found to have formed around periclase crystals, isolating them from further hydration (Figures 13b and 13c). Consequently, the periclase crystals were only partially hydrated. It is worth noting that the paste porosity of 28-day autoclaved specimens is relatively low, further limiting water access to the periclase grains.

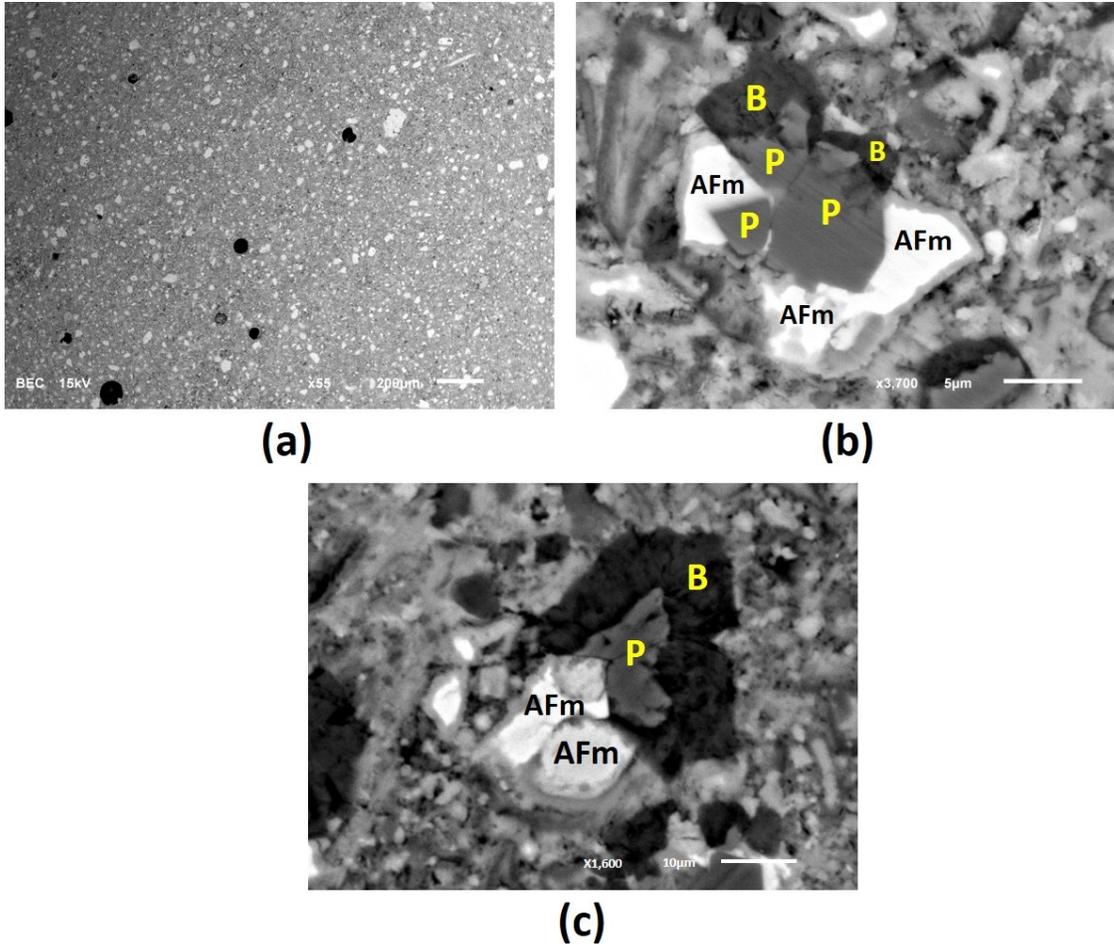


Figure 13. BSE images of cement *B* pastes autoclaved at 28-days.

Chemical properties of cement *B* pastes autoclaved at 28 days were further investigated using elemental mapping. Figures 14b and 14c represent the formation of AFm phases around the periclase crystals. Additionally, brucite did not diffuse away from its original position. Although brucite formation doubled the volume of solid, no cracks formed in the paste matrix and the brucite likely slowly filled capillary pore space. Therefore, for the stronger 28-day paste, the constant stress generated due to brucite expansion resulted in smaller deformation and left the paste matrix sound.

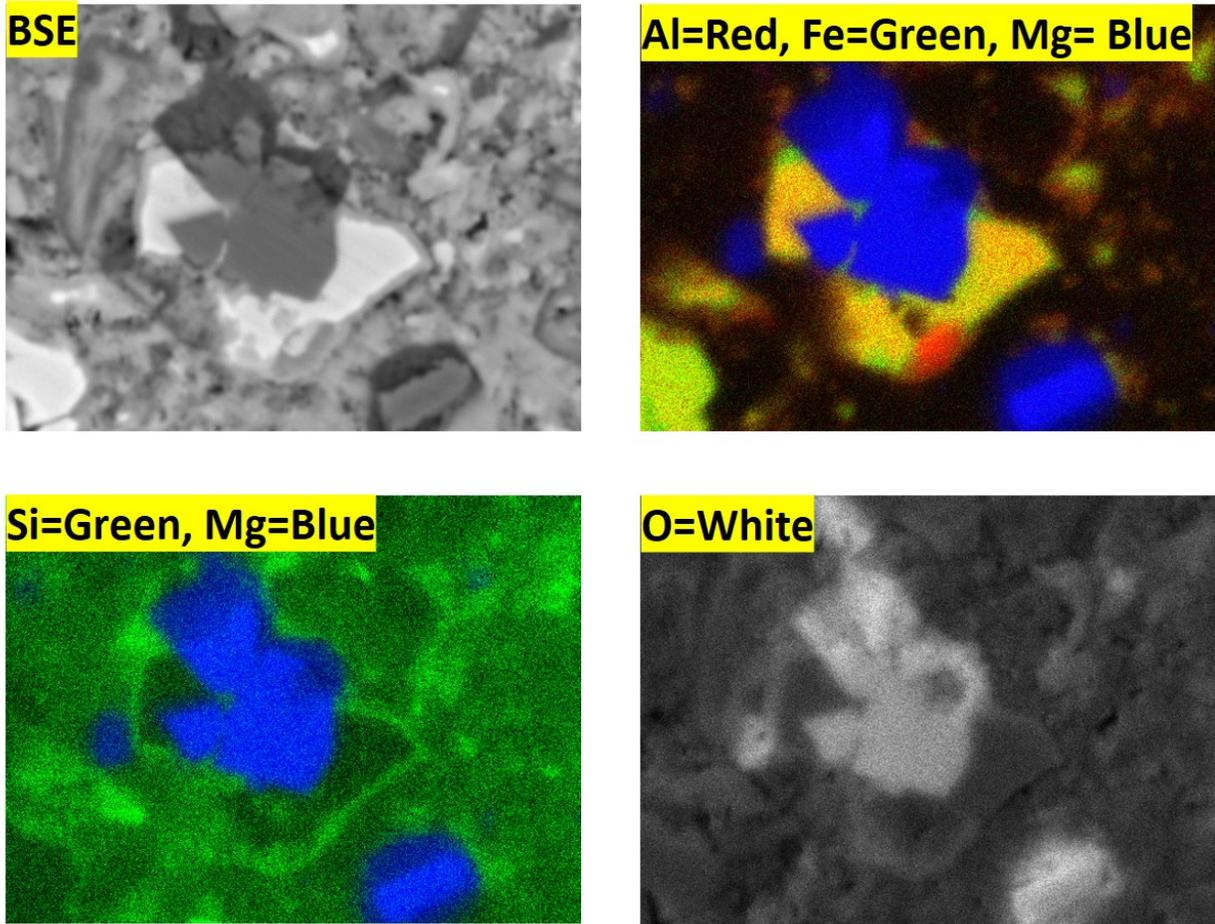


Figure 14. BSE Image and Elemental maps of cement *B* paste autoclaved at 28-days, Al=Aluminum, Fe=Iron, Mg=Magnesium, O=Oxygen, and Si=Silicon.

While it is desirable to be able to assess cement soundness using an accelerated technique, the results need to be representative of the long-term soundness at ambient temperatures. Further investigations revealed that the autoclave expansions of *B* and *D* cement pastes that had been pre-cured for 28 days were almost equal to those cured in water at 80 °C for one year (Figure 15). Also, as indicated in Figure 16, the brucite contents of both of the paste bars made with cement *B* were calculated to be 2.7%.

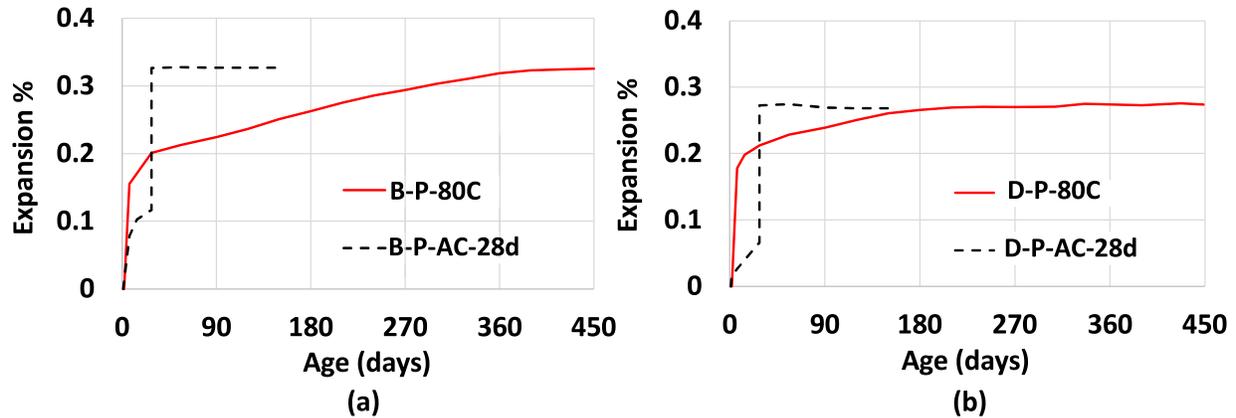


Figure 15. Comparison of expansions of 28-day autoclaved paste specimens with those stored in 80 °C for 1.25 years.

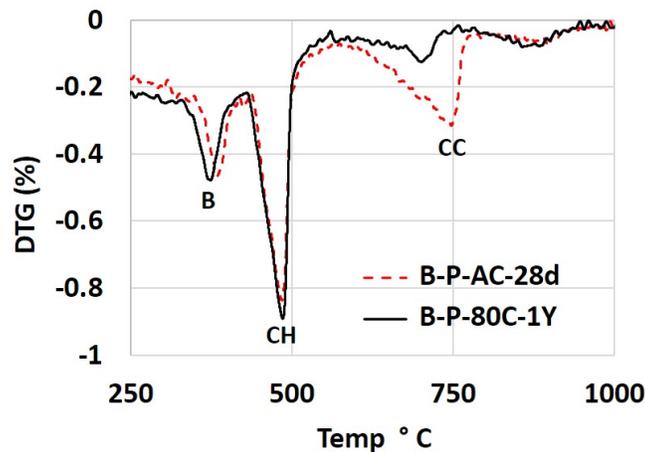


Figure 16. Comparison of the brucite content of cement *B* pastes autoclaved at 28-days with those moist cured at 80 °C for one year, B = Mg(OH)₂, CH = Ca(OH)₂, CC = CaCO₃.

Autoclaved Blended Cement (25% Slag Cement)

The only performance test specified in ASTM C150 (2019) for assessing cement soundness is AET. However, blended hydraulic cements meeting ASTM C595 (2019) and C1157 (2017) are also required to meet the AET limit. To simulate blended cements, cements that failed the autoclaved expansion limit were partially replaced with 25 wt.% slag cement, and their autoclave expansions were monitored. As shown in Table 2, 25% slag cement effectively reduced the 1-day autoclave expansions from 6.6% and 6.8% to only 0.14% and 0.15% for cements *B* and *D*, respectively, far more than would be expected by dilution.

Table 2. Effect of partial cement replacement with 25% slag on autoclave expansion.

Sample ID	Materials	Ave. Autoclave Expansion %
B-P-AC-1d	100% cement <i>B</i>	6.60
B-P-25%S-AC-1d	75% cement <i>B</i> + 25% slag cement	0.15
C-P-AC-1d	100% cement <i>C</i>	0.55
C-P-25%S-AC-1d	75% cement <i>C</i> + 25% slag cement	0.18
D-P-AC-1d	100% cement <i>D</i>	6.80
D-P-25%S-AC-1d	75% cement <i>D</i> + 25% slag cement	0.15

There are two main reasons to explain this phenomenon. Firstly, the dilution effect of the slag cement lowered the periclase content of the mixture (note that although slag cement contains more MgO, none of it is in the form of periclase). Secondly, the strength of the 1-day autoclaved matrix increased from 14 MPa to 93 MPa solely by the 25% slag cement replacement (Figure 17a). Unlike the weak $\alpha\text{C}_2\text{SH}$ that forms when portland cement is autoclaved (Ramachandran 1983; Taylor 1990), the slag cement likely resulted in formation of a more-crystalline 11 Å tobermorite-like C-S-H phase that would densify and strengthen the paste microstructure and reduce its expansion (Figure 17b) (Hooton 1986, Qian et al 1998).

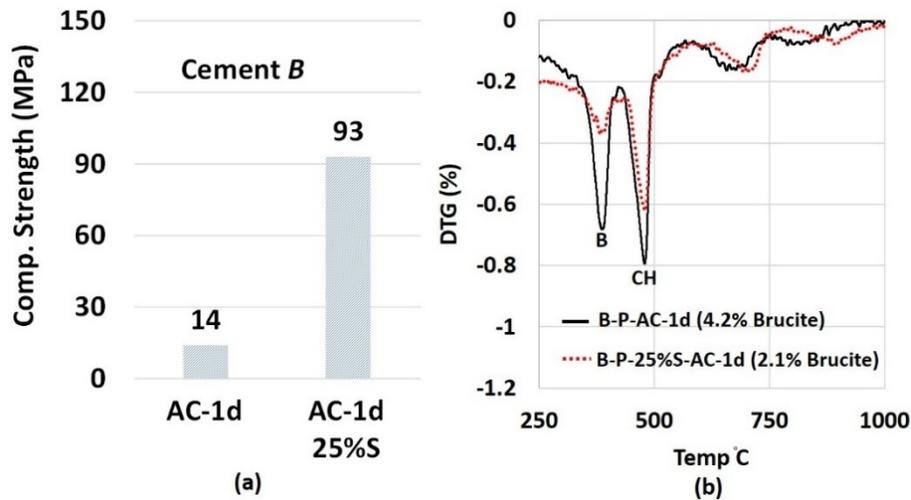


Figure 17. (a) Impact of partial replacement (25%) of cement *B* with slag cement on the paste strength, and (b) DTG analysis of autoclaved samples for brucite quantification.

Le Chatelier Split Ring and Miniature Prism Boiling Water Expansion Tests

Although there was a notable difference in the autoclave expansions of cements *B* and *C*, the Le Chatelier expansion was almost equal for both specimens and well below the 10 mm needle expansion limit, see Figure 18. Companion miniature paste prisms were also exposed to boiling water conditions for periods ranging from 2 h to 8 h, but the results in Figure 18 demonstrate that boiling period made no difference in the expansions of these cements.

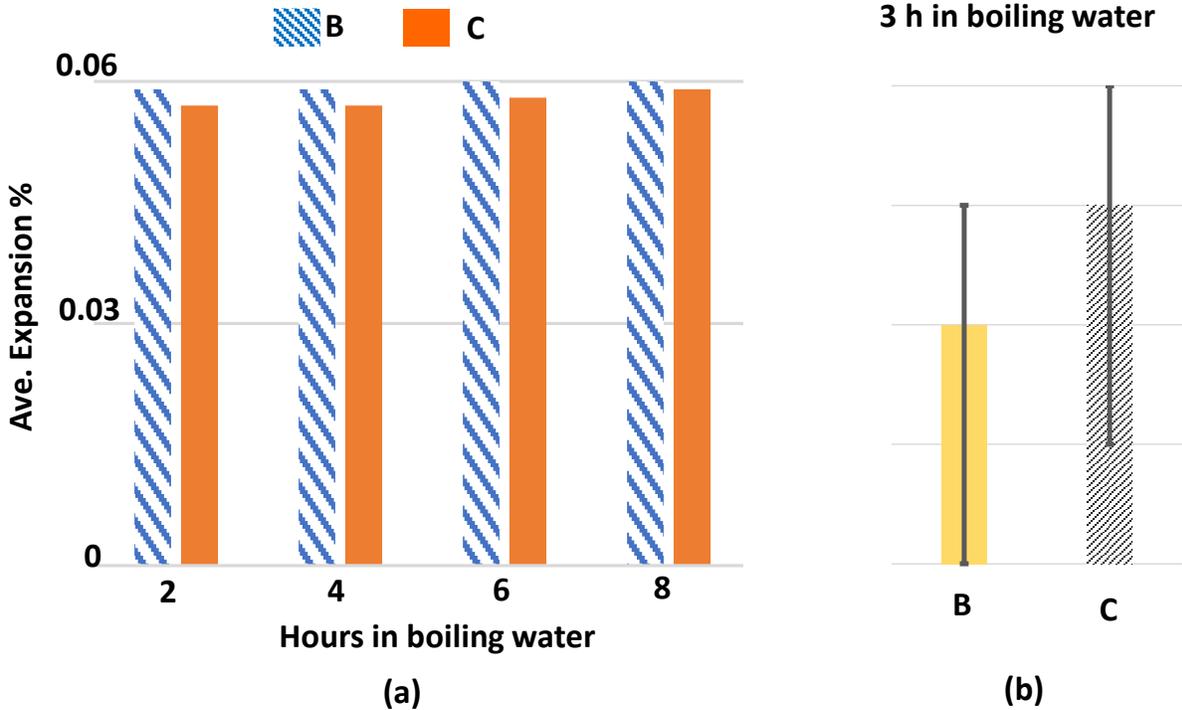


Figure 18. Evaluation of cements *B* and *C* using the boiling water test (a) miniature prism expansions, (b) Le Chatelier expansions. The black bars in (b), indicate the range of expansions.

Further investigation revealed that the brucite content of all the specimens that were boiled from 2 h to 8 h was almost zero (Figure 19). This indicates that the Le Chatelier expansion and companion miniature prism expansion tests are not useful for evaluating unsoundness due to dead-burned MgO reaction. The test was reported to be only suitable for indicating excess free lime in cement and was not sensitive to MgO hydration (Rott et al. 1984). Basically, free lime is intrinsically more reactive than periclase. That is why the Le Chatelier test is best suited to detect the excessive free lime expansion in cement (Chatterji 1995).

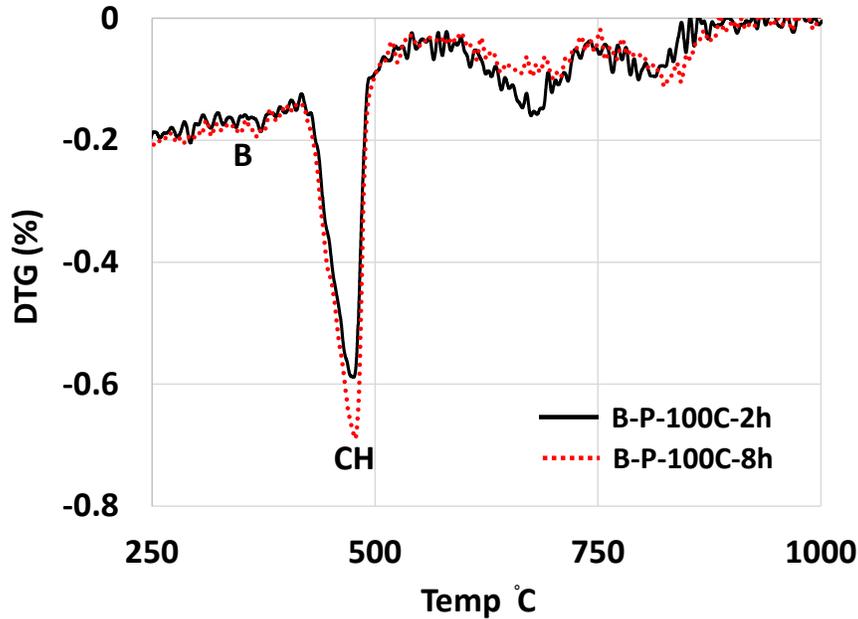


Figure 19. DTG of cement *B* after 2 and 8 h in boiling water, to quantify brucite (*B*).

Expansion of Portland Cement Mortar Bars

Figure 20 shows the expansive behavior of mortar bars made with cements *A*, *B* and *C* stored in water at 23 and 80°C. After 6 months, mortar bars made with cements *B* and *C*, i.e. ones that failed and passed the autoclave expansion limit respectively, were both visually sound and had low long-term expansions, which were well below the 0.10% expansion typically associated with cracking in ASR (ASTM C1260, 2014) and sulfate resistance (ASTM C1012, 2018) mortar bar tests. Mortar bars made with the lower MgO content Cement *A* had lower expansion than the other two cements.

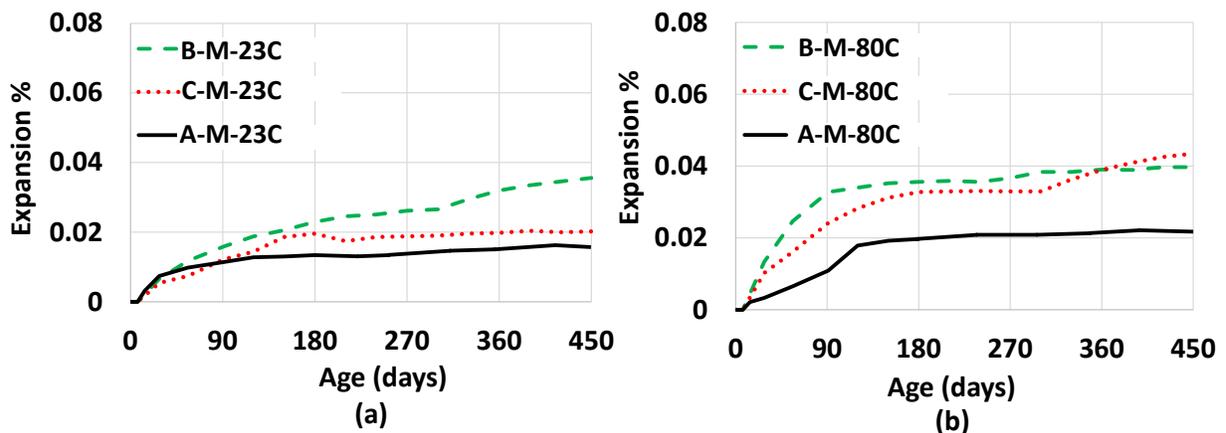


Figure 20. Expansion of mortar bars made with cements *A*, *B*, and *C*, cured at (a) 23 °C and (b) 80 °C.

Mortar bars stored in 23 °C water for 28 days were subjected to the AET and the results were compared to those stored in water for 450 days at different temperatures. In Figure 21, mortar bars made with cements *B* and *C*, that had failed and passed the ASTM C150 autoclave expansion limit respectively, both had similar, negligible long-term expansions.

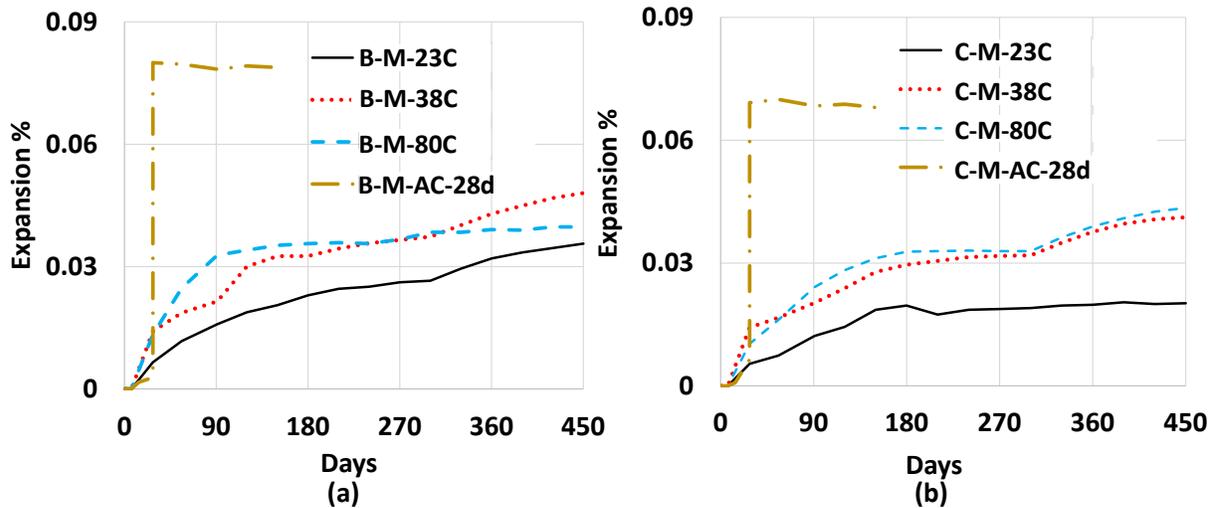


Figure 21. Comparison of the 28-day autoclave expansion of mortar bars with their long-term expansion in water for cements (a) *B*, and (b) *C*.

Expansion of Portland Cement Concrete Prisms

In Figure 22, the long-term expansions of concrete prisms made with cements *B* and *C*, cast at different w/c ratios, and stored in water at 23, 38, 60, and 80 °C are shown. Results show there is almost no difference between the expansions of concretes made with these two cements. Furthermore, at higher temperature water curing, prisms with lower w/c ratios had slightly higher expansions. Increasing the curing temperature from 23 °C to 38 °C did increase the expansion; however, increasing the curing temperature to 60 °C or 80 °C did not have a large impact. Visually, none of the concrete prisms showed any evidence of cracking or damage.

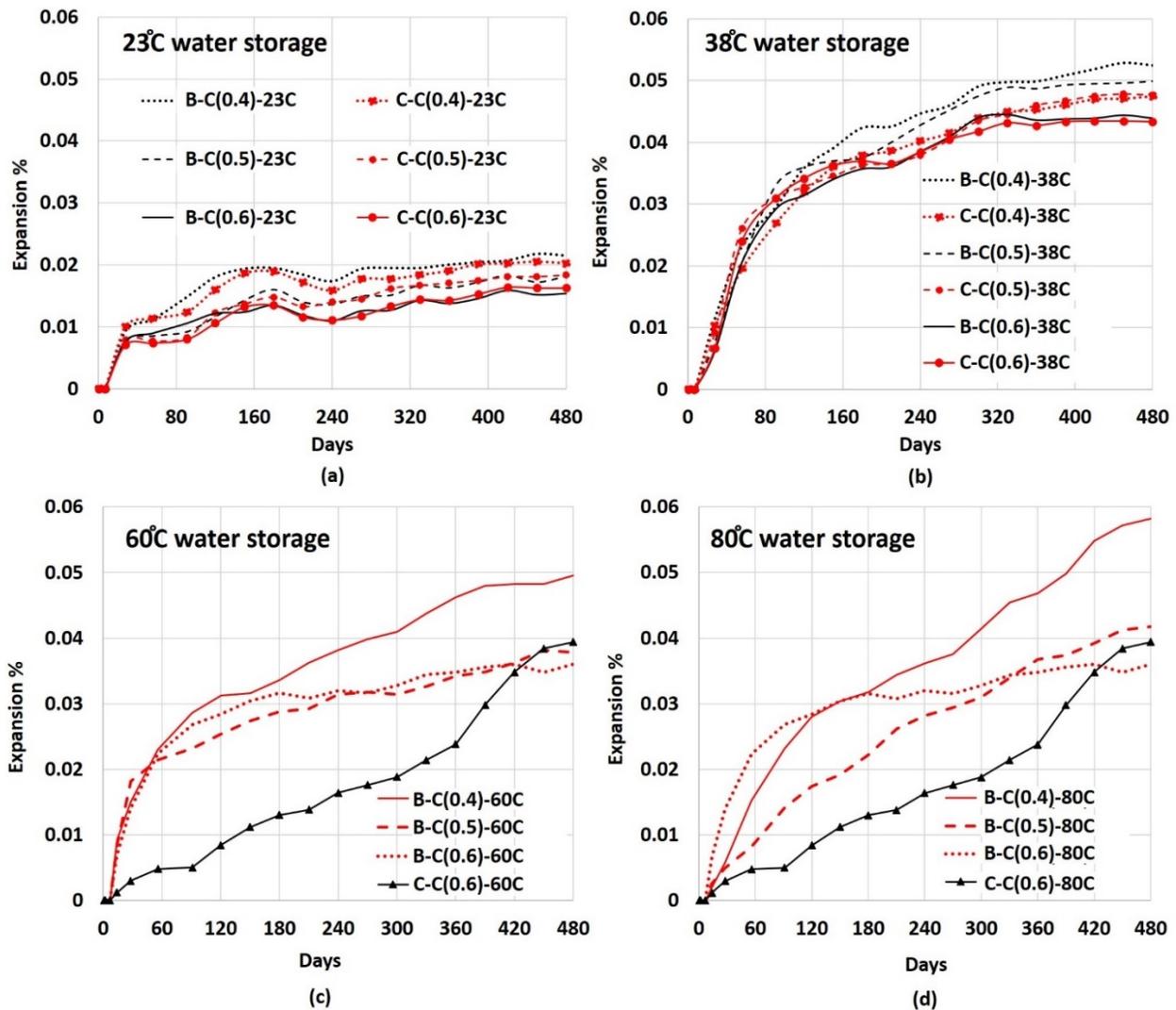


Figure 22. Long-term expansions of concrete prisms made with cements *B* and *C*, stored in water at (a) 23 °C, (b) 38 °C, (c) 60 °C, and (d) 80 °C.

Single concrete prisms pre-cured in 23 °C water for 28 days were also subjected to the AET. This test was done as a potential accelerated technique to assess the long-term soundness of concrete. However, as shown in Figure 23, the autoclave results overestimated the long-term water expansions. Although no visual cracks appeared on the surfaces of the autoclave prisms, all concrete autoclave expansions were higher than 0.10%.

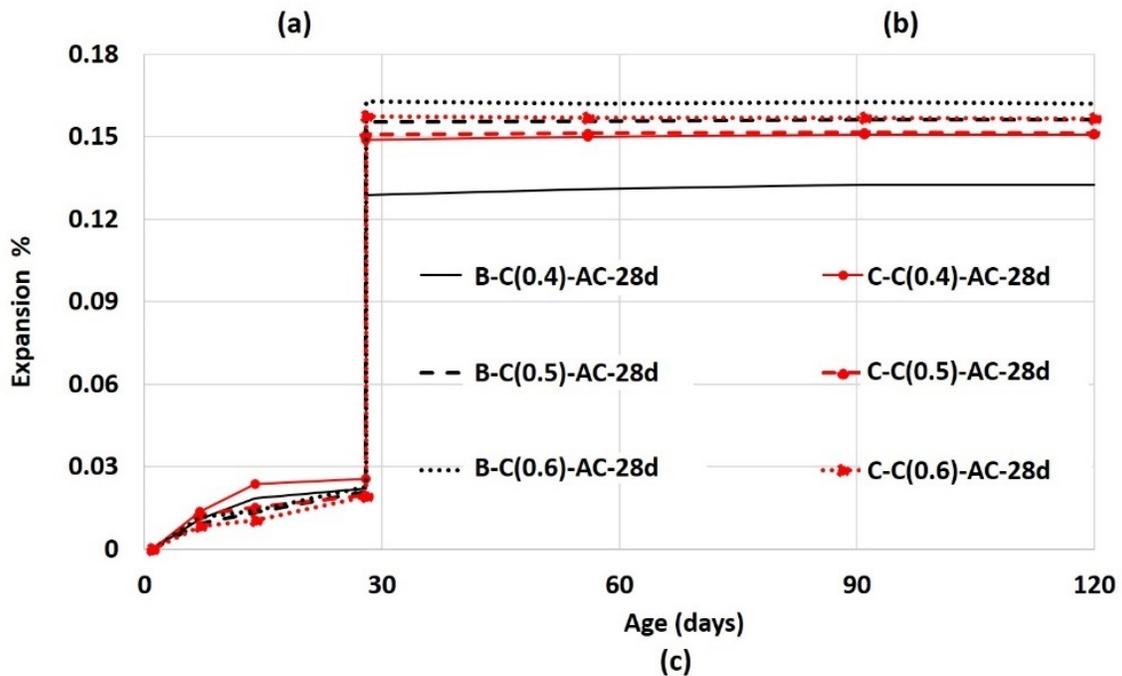


Figure 23. Visual condition and expansion of autoclaved concrete prisms made with cements (a) B, and (b) C.

For mortar bars and concrete prisms made with cements that passed or failed the AET, the 28-day cured autoclave expansions were remarkably higher than the long-term expansions in water. Using optical microscopy, a polished surface of the autoclaved B-C(0.4) specimen was analyzed. As shown in Figure 24, there appear to be gaps between aggregates and the paste matrix possibly due to the aggregates having a different thermal coefficient from the paste. Additionally, no cracks were detected in aggregate particles, eliminating the possibility of ASR formation during the autoclaving condition.

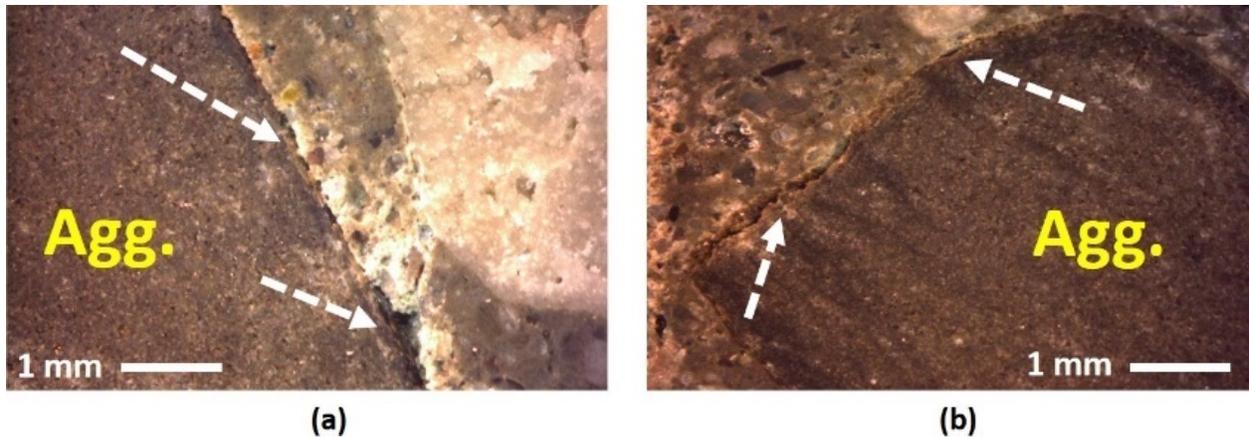


Figure 24. Cracks at paste-aggregate interfaces of C(0.4)-AC-28d, (a) first location, (b) second location.

Drying Shrinkage

Drying shrinkage test results are presented for paste, mortar, and concrete prisms in Figure 25. All specimens were immersed in water for 7 days, then exposed to drying at 50% relative humidity. It was thought that cements with higher levels of MgO might exhibit a level of expansion sufficient to offset some of the normal drying shrinkage. However, after 450 days, there was no difference in the shrinkage of specimens made with cements that failed or passed the autoclave expansion limit, i.e. *B* and *C* respectively. This suggests either a single week of moist curing was not sufficient to react the dead-burned MgO or the level of MgO was not high enough to result in expansion. For concrete prisms, the main factor controlling the extent of shrinkage appeared to be the w/c ratio, i.e. as expected, mixes with higher w/c ratios tended to shrink more.

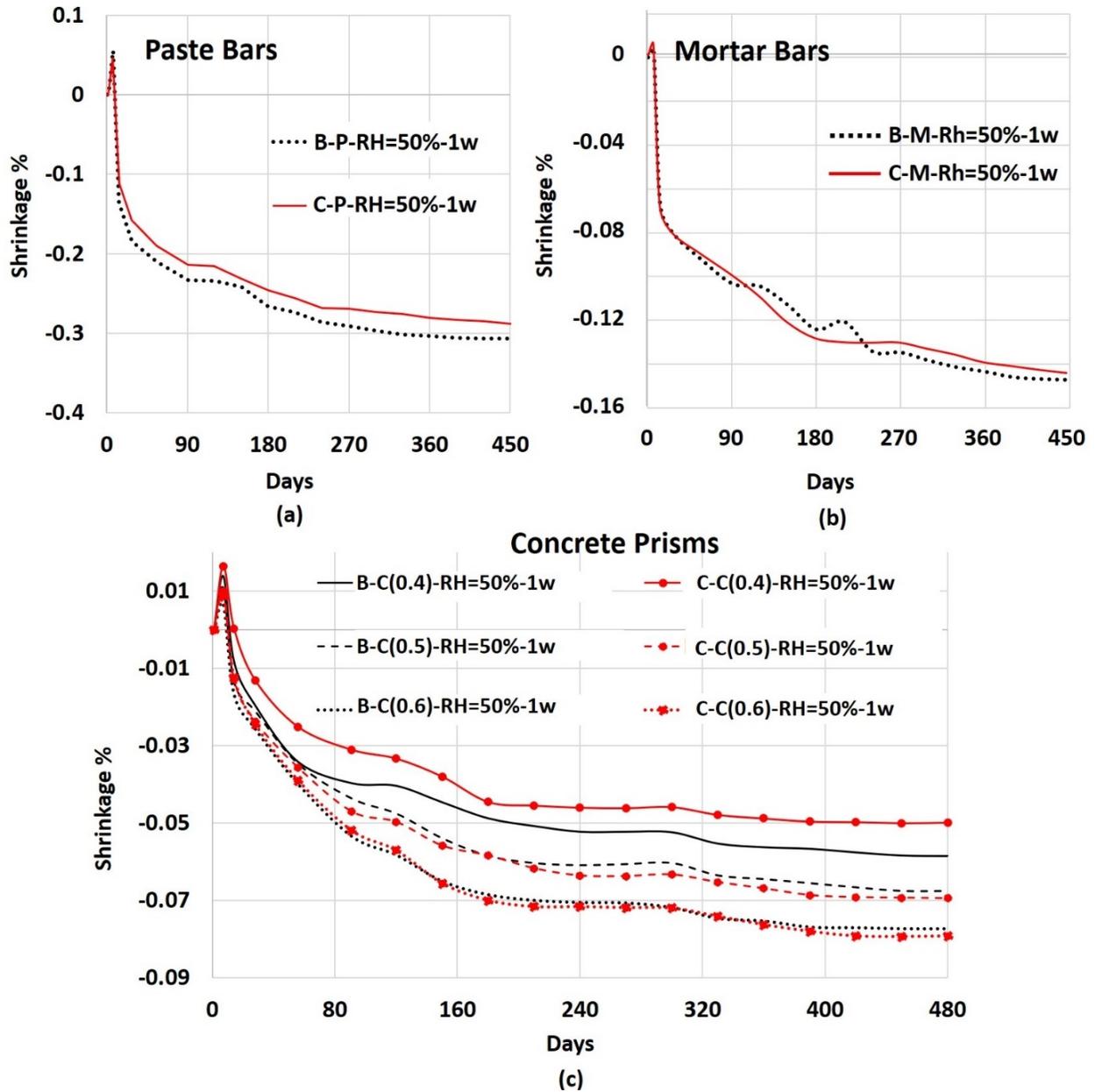


Figure 25. Drying shrinkage of specimens made with cements *B* and *C* for (a) paste bars, (b) mortar bars, and (c) concrete prisms.

DISCUSSION

For cements with similar periclase contents, the factor that influences the extent of autoclave expansion appears to be size of the periclase crystals. Hydration of larger MgO crystals results in larger local volume change (Helmuth and West 1998) and explains the higher autoclave expansion of cement *B* that had larger MgO crystals. However, regardless of large differences in MgO crystal size at ambient temperatures, there was no difference in the long-term expansions in water of cements *B* and *C* with similar MgO contents that had respectively failed and passed the autoclave expansion limit in ASTM C150. For companion paste prisms stored in water, increasing the water storage temperature from 23 °C to 80 °C did not change the 1-year

expansions but accelerated the reaction of periclase to brucite and increased the rate of expansion at earlier ages.

The AET appears to be overly harsh, and the strength of the pastes at one day are too weak to resist the extremely rapid volume changes induced by the AET (Tazawa et al. 2000; Kabir et al. 2019). The paste expansion is a result of competitive effects between the cement matrix strength and the stresses due to brucite expansion (Grasley and Lange 2007) Increasing the pre-autoclave curing period from 1 to 7 days resulting in higher strength, significantly reduced the paste autoclave expansion. Formation of monoaluminate phases (AFm) was increased by extending the cement curing period before the AET, which appeared to partially encapsulate the unreacted periclase crystals and limit further reaction. Extending the pre-autoclave curing period from 1 to 28-days prevented excess expansion and more realistically represented the brucite content of cement *B*, which had originally failed the AET.

For cement *B* cured for a year in water at different temperatures, there were differences in their brucite contents, but this did not cause additional volume change. During long-term curing, the crystalline MgO either remained inert or reacted so slowly that either brucite filled in capillary pore space, or creep was able to provide sufficient stress relief and prevented the brucite from causing disruptive expansion. It was also observed that the rate of periclase hydration decreases with time. It is believed that much of the crystalline MgO had either partially hydrated, was encapsulated by AFm phases, or remained inert in the cement phases when stored in water (Gao et al. 2008).

There are two possible explanations for the dramatic reduction in the AET expansion of blends made with 25% slag cement. Firstly, the dilution effect of the slag cement lowered the periclase content of the mixture by 25%. Secondly, the cement expansion was likely limited due to the enhanced matrix strength resulting from the more crystalline 11 Å tobermorite-like phase formed (Taylor 1990, Qian et al. 1998, Hooton 1981).

It is believed that in the Le Chatelier test, the boiling period is not sufficiently long to be able to hydrate much of the dead burned MgO crystals and hence may only be useful for evaluating the presence of free lime, in agreement with Helmuth and West (1998).

The long-term expansion of paste specimens in water appears to be mainly a function of cement MgO content, independent of the autoclave expansion. Instead of the AET, water storage of cement pastes at 80 °C could provide a more realistic estimation of long-term expansion (Mo and Deng 2007).

CONCLUSIONS

Based on the findings of this study, it is concluded that:

- The ASTM C151 autoclave expansion test (AET) test does not correctly predict the long-term expansion of cement paste prisms containing different levels of periclase when stored in water.
- The one-day cured AET specimens were too weak to resist the rapid expansive forces of brucite formation during the AET. When the AET was modified by extending the initial curing period before autoclaving to 2, 7, and 28 days, the autoclave expansions specimens were dramatically reduced. The amount of brucite formed in paste bars that were autoclaved at 28 days of age was similar to the amount formed in companion bars stored in water at 80 °C for one year.

- When paste prisms were stored in water at temperatures ranging from 23 °C to 80 °C, the higher storage temperatures expedited periclase hydration, and increased the initial rate of expansion but did not result in additional long-term expansion. After more than one year, no deleterious expansion was measured but long-term paste expansion was mostly a function of cement MgO content.
- In the literature it is reported the formation of hydrotalcite-type phases may also contribute to the expansion of cements in the AET. However, results of the elemental mapping using back-scattered electron imaging did not show evidence of formation of these phases.
- It is concluded that the autoclave expansion limit in ASTM C150 be removed. This conclusion is in agreement with those in previous reviews by Mehta (1978), Helmuth and West (1998) and Klemm (2005).
- Results from the Le Chatelier test for cement soundness used in some other countries indicated that, while it may be useful for detecting excess amounts of dead-burned free lime, this test does not appear to be capable of detecting unsoundness due to the hydration of periclase. As an alternative test, since free lime reacts with water over a short time period, it is also likely that excessive 14-day expansion in the ASTM C1038 test would be able to detect any concerns.
- Partial substitution of a cement that failed the AET limit with 25% slag cement dramatically reduced the autoclave expansion, far more than expected by dilution of the cement. It is suggested that this is likely due to the different nature of the hydrates formed by blended cements when cured at autoclave temperatures. This also calls into question the applicability of the ASTM C151 test for testing blended cements and supplementary cementitious materials.

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