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Influence of bleed water reabsorption on cement paste autogenous deformation

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ABSTRACT

The effects of bleed water reabsorption and subsequent early age expansion on observed autogenous deformation are investigated in this research. Bleeding was induced by varying superplasticizer and shrinkage-reducing admixture dosages and by increasing the water-to-cement ratio. This research revealed that significant early age expansion occurs with increasing chemical admixture dosages and higher water-to-cement ratios, as expected, due to increasing bleeding of those samples. When samples were rotated, negligible early age expansion was observed. Thus, bleed water reabsorption is shown to be the primary mechanism causing initial expansion in sealed autogenous deformation samples. Thermal dilation and ettringite growth appear to have a minimal influence on the observed expansion. Rotating the samples during setting eliminates the potential for bleed water reabsorption and is recommended for all autogenous deformation testing.

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1. Introduction

With the advent of high performance concrete containing low water-to-cement ratios and typically silica fume, early age shrinkage cracking of concrete has occurred with greater frequency. Early age cracking (primarily due to autogenous shrinkage) significantly compromises the durability of the concrete. Since the use of high performance concrete is increasing, there is a growing need for accurate assessment of autogenous deformation.

As such, there is a growing body of literature concerning autogenous deformation measurements. However, there are varied measurements techniques, primarily divided into two categories: (1) volumetric and (2) linear. The volumetric test method generally consists of buoyancy measurements of a sealed cement paste in water. The major drawback for this test method is that it has been shown [1] that, upon self-desiccation, water will penetrate through the membrane, possibly leading to underestimations of autogenous shrinkage. Linear measurement techniques, particularly the corrugated tube method developed by Jensen and Hansen [2], have the potential to more accurately measure autogenous deformations.

However, previous research [3–6] showed that samples made by the corrugated tube method developed by [2] often exhibit early age expansion. There have been several proposed mechanisms for this observed early age expansion [7–10], primarily: (1) reabsorption of bleed water after setting, (2) formation of hydration products such as ettringite and calcium hydroxide, and (3) thermal dilation. It has been suggested that bleed water reabsorption is the main source of the observed expansions [7]. The contribution of bleed water reabsorption has been subjected to limited investigation, but the research by Bjontegaard et al. [11] is very promising. In those tests, the bleed water was physically removed from the samples or additional water was added to the existing bleed water. However, the subsequent effects of removing or adding water on the water-to-cementitious material ratio of the mix may be contributing to differences in the observed autogenous deformation [7].

The effects of thermal dilation may also contribute to the observed early age expansion noted in some research. The influence of thermal effects is expected to be more of a contributing factor as the surface area-to-volume ratio of a sample decreases. Furthermore, thermal effects can also be accounted for in the data analysis of autogenous deformation samples. By measuring the difference between the internal sample temperature and the surrounding air temperature, the strain due to thermal expansion can be estimated and autogenous deformations corrected. One potential drawback of this correction occurs in the assumption of the sample coefficient of thermal expansion, which changes rapidly at the early ages of interest in this research.

The initial autogenous expansion exhibited in the literature is a cause for concern and was investigated in this research for two reasons: (1) are the test methods accurately measuring autogenous shrinkage strain, and (2) to what extent is initial expansion an artifact of bleeding? Thus, the objective of this research was to investigate the extent to which bleed water reabsorption influences initial expansion and subsequent net autogenous shrinkage. In addition, a proposed method for eliminating bleeding and maintaining a constant w/c, even in highly fluid mixes, is presented and assessed. Bleeding was induced by (1) increasing the dosage of superplasticizer and (2)

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varying the *w/c*. Paste samples containing shrinkage-reducing admixtures were also investigated for the influence of rotation on the observed autogenous deformation.

2. Experimental study

2.1. Materials and sample preparation

To assess the influence of superplasticizer dosage on autogenous expansion, a water-to-cement ratio (*w/c*) of 0.30 was used. Glenium 3200 high range water reducer (superplasticizer), obtained from BASF, was added at dosage rates of 0, 0.62, 1.85, 3.09, or 4.32 µL/g cement. All dosage rates were below the manufacturer's maximum dosage rate of 5.2 µL/g cement, such that there should be negligible effects on cement hydration. Samples containing shrinkage-reducing admixtures were also examined at a *w/c* of 0.30. Tetraguard AS20, obtained from BASF, was used at dosage rates of 1% and 2% by mass of cementitious materials (10.1 and 20.2 µL/g cement). The water content of the admixtures was compensated for by decreasing the relative amount of mix water necessary to maintain a *w/c* of 0.30. To assess the effect of varying *w/c* on autogenous deformation, another set of cement paste samples were prepared without any chemical admixtures and with a *w/c* of 0.30, 0.35, 0.40, or 0.45.

High performance cement pastes were prepared using ASTM Type I/II portland cement and deionized water (resistivity of 18.2 MΩ m). Oxide analysis and Bogue potential composition for the cement are listed in Table 1. Samples were prepared by mixing the cement and water according to ASTM C 305 [12] in a 1.5 L-capacity planetary mixer. If chemical admixtures were used, they were added to the water prior to mixing. Eight corrugated tube samples were prepared for each mix.

2.2. Sample rotation and autogenous deformation

Autogenous deformation was measured by the technique described by Jensen and Hansen [2] and as shown in Fig. 1. This technique involves taking frequent linear deformation measurements of cement pastes sealed in a rigid polyethylene mold with low friction.

Measurements began at final set, as determined by Vicat needle penetration, ASTM C 191 [13]. The initial measurement was taken at final set to exclude plastic deformation. The final set times for all samples are given in Table 2. Though the Vicat test is empirical, it has been shown to correlate well with the deviation of chemical and autogenous shrinkage, as measured by other techniques [14]. Manual measurements were taken periodically with a dilatometer, accurate to 0.001 mm (approximately ± 5 µε), as often as possible for the first day, followed by once per day up to ten days.

Table 1
Oxide analysis (percent by mass) and Bogue potential composition for Type I/II portland cement.

Oxide	Type I/II portland cement
SiO_2	19.70
Al_2O_3	4.33
Fe_2O_3	3.66
CaO	63.41
MgO	3.30
Na_2O	0.07
K_2O	0.68
TiO_2	0.23
P_2O_5	0.06
SrO	0.05
Loss on ignition	1.40
C_3S	65.18
C_2S	7.65
C_3A	5.29
C_4AF	11.12

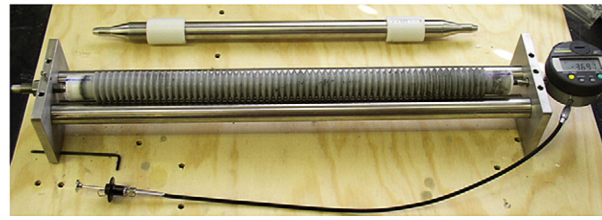

Fig. 1. Autogenous deformation dilatometer.

A rotational device shown in Fig. 2, which rotated the samples at approximately 45–60 rpm was used to limit the amount of bleeding and subsequent reabsorption. Of the eight samples made for each mix, four were rotated for 24 h and four were placed in an environmental chamber. All samples were maintained at 23 ± 2 °C.

Between final set and the end of rotation, the device was stopped and samples removed for manual measurements. All measurements were conducted within approximately 5 min before the samples were returned to the device and rotation resumed. Over the ten day period, samples were visually inspected prior to each measurement and any samples that cracked due to mishandling were discarded. A minimum of three samples was required for each variable. If more than one sample was discarded in a particular set, the entire set was redone.

Maximum strain differential (MSD) was defined as the difference between the maximum expansive strain and the shrinkage strain at a given time. In this research, the maximum strain differential was taken after 10 days (MSD_{10d}), as illustrated in Fig. 3. It has been noted that storage of the corrugated tubes in an ambient environment may lead to diffusion of water through the plastic tube, increasing shrinkage [6]. Expansive and shrinkage strains are denoted as positive and negative strains, respectively. The maximum strain differentials are given as negative values to represent accumulated shrinkage after peak expansion.

This data analysis has been applied previously to compensate for the early age expansion on the overall amount of autogenous shrinkage. However, the use of the maximum strain differential values may not adequately capture the strain behavior of non-rotated samples. If early age expansion is observed, it is expected that the use of a maximum strain differential may slightly to moderately underestimate the actual autogenous shrinkage of non-rotated samples compared to those rotated. Though, it is an improvement compared to using the observed autogenous shrinkage without regard to expansive strains, which may significantly underestimate shrinkage strains if the observed expansions are large.

In other words, if early age expansion is observed due to non-rotation and bleed water reabsorption, maximum strain differential data analyses effectively ignore the initial period of time during which autogenous shrinkage is critical. This concept is represented in Fig. 4. In this example, the maximum expansion is normalized to zero strain. Thus, compared to the rotated sample, approximately 6 h of autogenous

Table 2
ASTM C 191 (Vicat) final setting times.

w/c	Superplasticizer dosage (µL/g cement)	SRA dosage (percent by cementitious mass)	Final set (h)
0.30	0	0	4.5
0.30	0.62	0	5.25
0.30	1.85	0	5.25
0.30	3.09	0	5.83
0.30	4.32	0	6.0
0.30	0	1	5.5
0.30	0	2	6.0
0.35	0	0	4.75
0.40	0	0	4.75
0.45	0	0	5.0

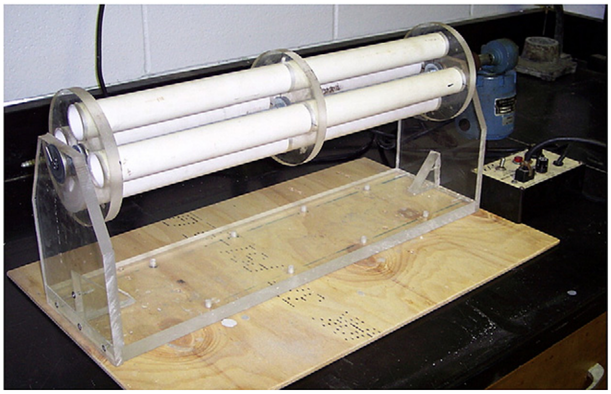

Fig. 2. Rotational device for autogenous deformation tubes.

shrinkage measurements are “lost”. These critical hours may represent a significant amount of shrinkage for certain cementitious mixtures and should not be neglected. Further discussion is found in the following sections.

3. Results

In this research, early age autogenous expansions were investigated via the corrugated tube method. Bleed water reabsorption was hypothesized to be the primary cause of the observed expansions. Bleeding was induced and/or amounts of bleeding were modified in order to evaluate the extent of bleeding on autogenous deformation. Samples were rotated to minimize bleeding and subsequent reabsorption, providing a means for evaluating the device's effectiveness compared to complementary, non-rotated samples.

In some cases, bleeding was very substantial and at a maximum, occupied up to approximately 1/3 of the corrugated tube volume prior to reabsorption. Bleeding was observed for all samples that exhibited early age expansion. Increased expansion was associated with increased bleeding. The reabsorption of bleed water into the sample correlated with the time of observed expansion. In other words, expansions occurred during bleed water reabsorption. Maximum expansions were noted at the approximate time of complete reabsorption. Once all the bleed water was reabsorbed, expansion stopped and shrinkage was observed.

3.1. Effect of superplasticizer on expansion

Fig. 5 illustrates the effects of increased superplasticizer dosages on the autogenous deformation of non-rotated samples. It can be seen that as the superplasticizer dosage increased, the observed expansion increased while the average autogenous deformation at 10 days decreased. However, the maximum strain differential for all samples,

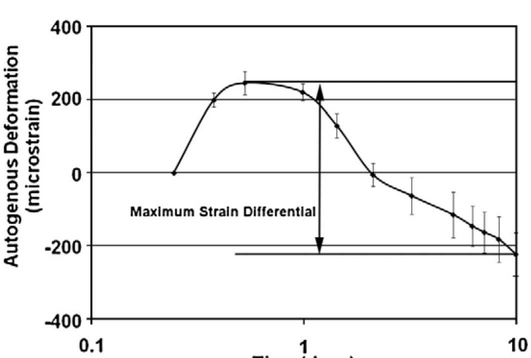

Fig. 3. Illustration of maximum strain differential.

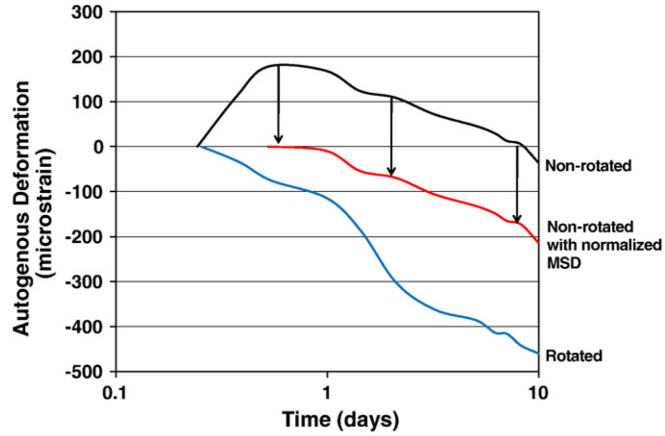

Fig. 4. Comparison of maximum strain differential for non-rotated samples to autogenous deformation of rotated samples.

except that containing 4.32 μL superplasticizer/g cement, was fairly consistent. The non-rotated samples containing the highest superplasticizer dosage (4.32 μL/g) were still expanding at ten days with a maximum autogenous expansion of 565 microstrain (Table 3), even though the dosage rate was below the manufacturer's maximum recommended dosage. The maximum expansions observed are found in Table 3.

When rotated, all samples containing superplasticizer, regardless of dosage, exhibited similar autogenous shrinkage (−394 to −468 με) and maximum strain differential values (−416 to −487 με) at ten days (Fig. 6 and Table 3). Minimal or no initial expansion was seen for all rotated samples. The early age expansions for the rotated samples varied from 0 to 46 με. When the maximum strain differential was calculated for the non-rotated samples containing superplasticizer, it was found to be similar to that of the rotated samples (−445 to −503 με), except at the highest superplasticizer dosage (4.32 μL/g), which was still expanding at ten days (i.e., the maximum strain differential was zero).

In this research, any expansion observed for the rotated samples may be attributed to mechanisms other than bleed water reabsorption, for example, thermal dilation and/or ettringite formation. It would be expected that in certain cases, such as with the use of a low C_3A cement producing less ettringite, early age expansions would be further minimized. On the other hand, cement with a higher C_3A content than that used in this research (5.29%) would potentially exhibit increased early age expansion even with rotation. Certain chemical admixtures may also significantly influence the formation of ettringite at early ages, thus potentially influencing the amount of

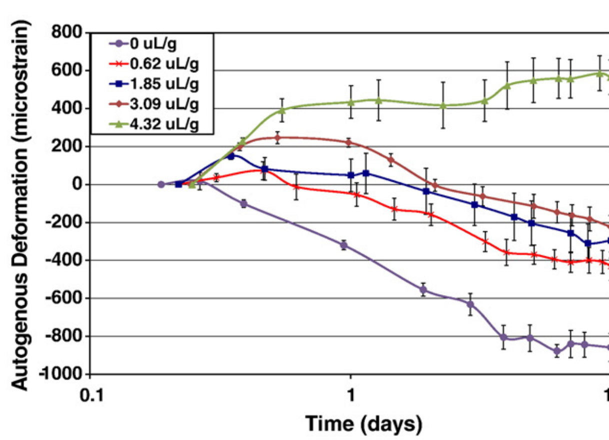

Fig. 5. Autogenous deformation of non-rotated cement pastes containing superplasticizer.

Table 3
Maximum strain differential for various superplasticizer dosages ($w/c = 0.30$).

Superplasticizer dosage (μL/g cement)	Rotation	Average maximum expansion (με)	Average deformation at 10 days (με)	Average maximum strain differential (με)
0	None	18 (46)	−858 (74)	−876
	24 h	0.0	−837 (27)	−837
0.62	None	73 (47)	−431 (75)	−504
	24 h	21 (19)	−394 (13)	−415
1.85	None	150 (39)	−295 (40)	−445
	24 h	46 (26)	−417 (26)	−463
3.09	None	246 (32)	−223 (59)	−469
	24 h	27 (13)	−460 (17)	−487
4.32	None	565 (92)	565 (92)	0
	24 h	0.0	−468 (53)	−468

bleeding. Isothermal testing would also be expected to minimize or eliminate any thermal dilation effects.

Furthermore, as stated in Section 2.2, these values may represent a lower limit on observed early age expansions due to the measurement intervals. If continual measurements (for example, via wireless LVDTs) were possible during rotation, it is anticipated that all rotated samples would experience similar expansive strains at early ages. For the type of rotational device used in this research, it is therefore suggested that manual measurements be conducted at least every hour within 6 h after final set.

There was a notable difference between the autogenous shrinkage for all samples containing superplasticizer compared to the control sample without superplasticizer. The addition of superplasticizer, regardless of dosage, appeared to decrease autogenous shrinkage at ten days by 44–53% (−394 to −468 με) compared to the control sample without superplasticizer (−837 με) for those samples rotated. Given a constant w/c, autogenous deformation for each sample should be similar if the influence of the superplasticizer on the pore solution surface tension and cement particle flocculation is neglected.

Research indicates that the addition of a polycarboxylate superplasticizer, such as that used in this research, will decrease the pore solution surface tension, similar to that of commercially available shrinkage-reducing admixtures [15]. Thus, it can be inferred that the addition of superplasticizer reduced the surface tension of the pore solution in these samples as indicated by similar decreases in the autogenous shrinkage of all samples containing superplasticizer compared to the control sample without superplasticizer. Furthermore, increases in the superplasticizer dosage appear to cause negligible reductions of the pore solution surface tension as the samples containing superplasticizer all exhibited similar rates of deformation and autogenous shrinkage at ten days.

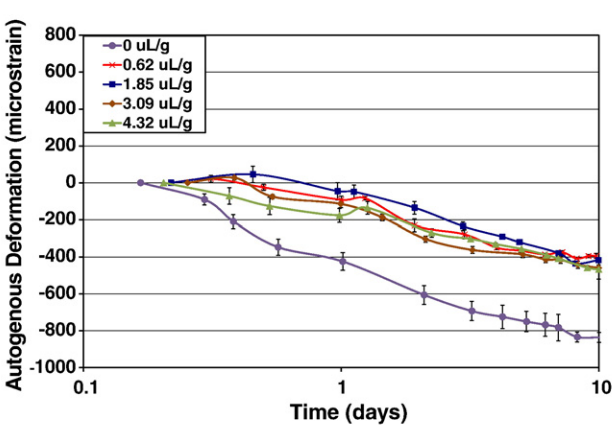

Fig. 6. Autogenous deformation of cement pastes containing superplasticizer rotated for 24 h.

Furthermore, the reduction in the pore solution surface tension due to superplasticizer addition presents a competing effect to cement particle deflocculation. It is expected that without the use of a superplasticizer (i.e., a control sample), autogenous deformation would occur at a slower rate and possibly result in less autogenous shrinkage at ten days. Without superplasticizer, water would be held by the cement particle agglomerations, reducing the availability of water for hydration reactions. Subsequently, slower hydration rates would be expected and as hydration is slowed, the rate of autogenous shrinkage would decrease proportionally at the water-to-cement ratio used in this portion of the research.

However, this does not appear to have an effect in this research, as the use of superplasticizer decreases autogenous deformation by approximately 50% compared to the control sample. This may be due to laboratory mixing procedures or the reduction in pore solution surface tension outweighs the flocculation effect. Additional research investigating superplasticizers for their respective surface tension effects and shrinkage-reducing capabilities appears warranted.

3.2. Effect of shrinkage-reducing admixtures on expansion

Rotation of samples containing shrinkage-reducing admixtures (SRAs) was also performed to further investigate the influence of early age expansion on the observed autogenous shrinkage. SRAs are also expected to influence bleeding. Thus, the effectiveness of the rotation was also examined as seen in Fig. 7 and Table 4.

With rotation, the difference between the two SRA dosages was negligible. Both the 1% and 2% dosages by mass of cementitious materials experienced minimal early age expansion and similar autogenous shrinkage at ten days. Compared to the control sample without SRAs, the use of SRAs reduced autogenous shrinkage at ten days by 40–45% (−464 to −498 με for the SRA samples compared to −837 με for the control).

Without rotation, the samples containing 1% and 2% SRA appeared to reduce autogenous deformation by 79% and 94%, respectively, compared to the non-rotated control. However, early age expansion was observed for both these samples. The 1% SRA sample did not experience shrinkage strains until approximately 36–48 h after casting. The 2% SRA sample begins to exhibit shrinkage strains between four and five days, after reaching an expansive strain of approximately 150 με. Thus, it is imperative that rotation of samples be performed to accurately assess the effect of SRAs (and other chemical admixtures) on autogenous shrinkage mitigation. Furthermore, rotation should also be applied when internal curing is used to also accurately investigate the shrinkage-reducing effects of these materials.

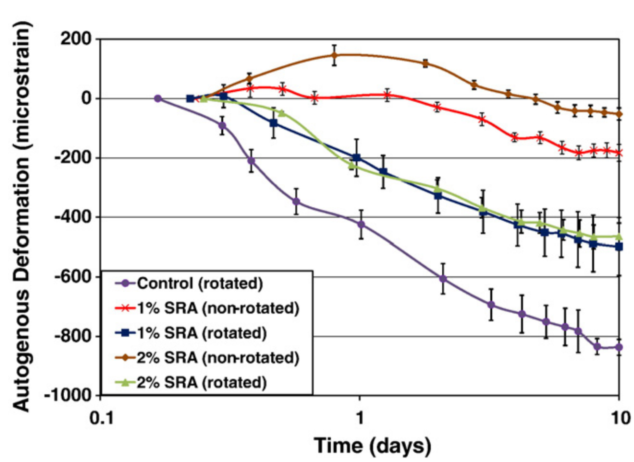

Fig. 7. Autogenous deformation of cement pastes containing shrinkage-reducing admixture.

Table 4
Maximum strain differential for various shrinkage-reducing admixture dosages ($w/c = 0.30$).

SRA dosage (percent by cementitious mass)	Rotation	Average maximum expansion (με)	Average deformation at 10 days (με)	Average maximum strain differential (με)
0	None	18 (46)	−858 (74)	−876
	24 h	0.0	−837 (27)	−837
1	None	34 (30)	−183 (28)	−217
	24 h	8 (39)	−498 (98)	−506
2	None	146 (34)	−52 (21)	−198
	24 h	0.0	−464 (45)	−464

It is interesting to note that both the superplasticizer (Section 3.1) and SRAs reduced autogenous shrinkage at ten days by comparable amounts. With rotation, the use of superplasticizer and SRAs reduced autogenous shrinkage by an average of 48% and 43%, respectively, compared to the control without chemical admixtures. The reduction in autogenous shrinkage due to the SRA is expected but was not anticipated with the use of the superplasticizer. As stated previously, evidence in the literature indicates that a polycarboxylate superplasticizer decreases the pore solution surface tension similarly to SRAs. As this was unexpected, further research is needed to validate the effect of various commercially available water reducing chemical admixtures for their influence on the pore solution surface tension and subequent effect on autogenous deformation.

3.3. Effect of water-to-cement ratio on expansion

The intent of varying the w/c was to allow for a comparison between rotation and non-rotation at the different w/c. As the w/c increases, increases in bleeding would be expected along with less autogenous shrinkage, regardless of early age expansion. Figs. 7 and 8 illustrate the autogenous deformation results for cement pastes without chemical admixtures and with water-to-cement ratios (w/c) varying from 0.30 to 0.45 (in Fig. 8, samples have not been rotated and in Fig. 9, samples have been rotated for 24 h). It was observed that all samples that were not rotated expanded within 24 h, while the rotated samples did not expand or expanded negligibly. As expected, as the w/c increased, autogenous shrinkage at ten days decreased for all samples. Table 5 provides the maximum expansion autogenous shrinkage at ten days, and the maximum strain differential for the non-rotated and rotated samples with varying w/c.

However, it can be seen in Table 5 that there were some apparent difference between samples rotated and those not rotated. The influence of rotation on the samples with a water-to-cement ratio of 0.30 was negligible, as those samples experienced minimal bleeding. Differences due to rotation were noted as the w/c was increased to

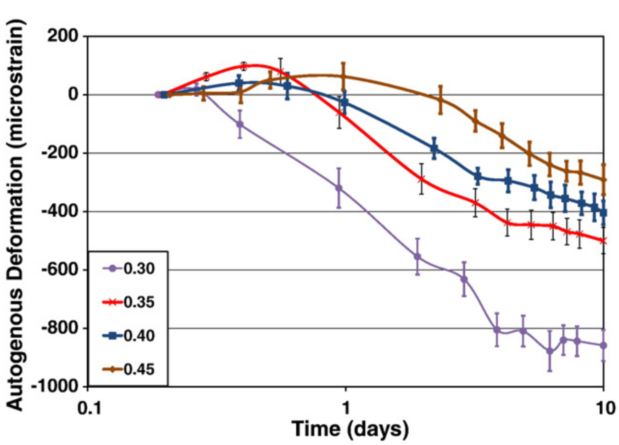

Fig. 8. Autogenous deformation of non-rotated cement pastes at various w/c.

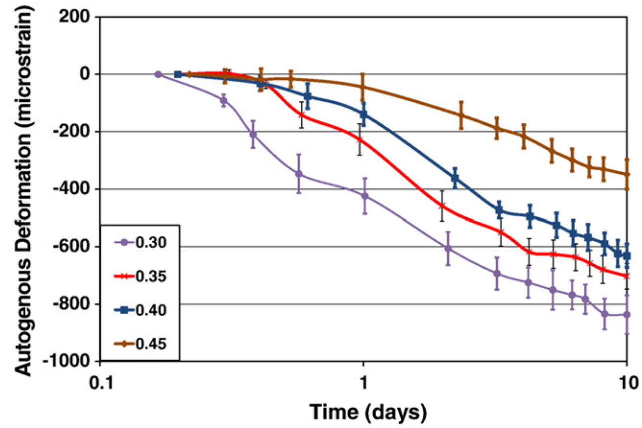

Fig. 9. Autogenous deformation of cement pastes at various w/c rotated for 24 h.

0.35 and higher. In addition, it appears that as the w/c increases, early age expansions did not continually increase for the non-rotated samples, indicating that bleeding did not occur as much as expected for the higher w/c samples. This could be potentially due to improved mixing action with the higher w/c samples leading to decreased flocculation. Thus, the increased cement particle surface area available for reaction would be a possible cause for the observed bleeding rates leveling off.

Though the 0.30 w/c sample in this research did not exhibit bleeding, there may be situations (e.g., presence of internal curing materials) where samples at this w/c would potentially exhibit expansion. In addition, the presence of supplementary cementitious materials, for example, may necessitate the use of water reducing admixtures. Subsequently, bleeding may occur and rotation would be necessary.

It is interesting to note that the 0.45 w/c rotated samples still experienced autogenous shrinkage of approximately 350 με at ten days, indicating that self-desiccation still occurs in these samples at higher w/c. Generally, it is assumed that self-desiccation does not occur at this w/c; however, it appears that even these higher w/c mixes could benefit from internal curing and/or shrinkage-reducing admixtures. Furthermore, the effects of autogenous shrinkage should not be neglected for any cementitious mixture. Thus, it is therefore suggested that rotation be conducted on all autogenous deformation samples regardless of mixture composition or proportions.

As stated previously, by not rotating samples, the observed autogenous shrinkage is expected to be underestimated at ten days. The reabsorption of bleed water presents a competing effect to the autogenous shrinkage at early ages. By rotating the samples, this influence is effectively mitigated (as clearly evidenced in Fig. 7). Furthermore, the use of the maximum strain differential values, as seen in Table 5, does not adequately capture the strain behavior of non-rotated samples, though it is an improvement compared to using the observed autogenous shrinkage of non-rotated samples. However,

Table 5
Maximum strain differential for various w/c.

w/c	Rotation	Average maximum expansion (με)	Average deformation at 10 days (με)	Average maximum strain differential (με)
0.30	None	18 (46)	−858 (74)	−876
	24 h	0.0	−837 (27)	−837
0.35	None	98 (14)	−500 (45)	−597
	24 h	2 (10)	−703 (6)	−705
0.40	None	40 (26)	−404 (41)	−444
	24 h	0.0	−632 (14)	−632
0.45	None	62 (46)	−291 (51)	−353
	24 h	0.0	−349 (68)	−349

in Section 3.1, the maximum strain differential (Table 3) appeared to be a relatively effective means to describe the shrinkage behavior of non-rotated samples. Though, with the different *w*/*c* samples, differences in the maximum strain differential between the rotated and non-rotated samples were found to be over 100 με in some cases (Table 5).

Thus, this clearly illustrates that the application of maximum strain differentials is not sufficient and rotation of samples must be performed on all samples in order to accurately observe autogenous deformation and eliminate the effects of bleed water reabsorption. Without rotation, the effectiveness of chemical admixtures and internal curing may be overestimated. As such, rotation is vital to capturing the autogenous deformation behavior of cementitious samples, regardless of *w*/*c* and chemical admixtures.

3.4. Implications of bleed water on autogenous deformation in practice

In this research, the influence of bleed water reabsorption on autogenous deformation was investigated using the sealed corrugated tube method. It was shown that the reabsorption of bleed water was the primary factor causing early age expansions of non-rotated samples. Thermal dilation and/or ettringite formation were shown to play a minor role in the observed expansions.

However, in field applications, the corrugated tube method is not applicable. Bleeding is ubiquitous in practice and is beneficial for the prevention of drying and plastic shrinkage. Furthermore, in larger samples (i.e., smaller surface area-to-volume ratio), thermal dilation may induce more expansion than that observed in this research. Therefore, it is important to note that test methods replicating more realistic field conditions should be more thoroughly investigated and developed in order to assess the true contribution of autogenous deformation to overall concrete shrinkage in field applications.

4. Conclusions

In this research, the effect of early age expansion due to bleed water reabsorption on autogenous deformation at ten days was evaluated. Varying amounts of bleeding were induced by increasing superplasticizer and shrinkage-reducing chemical admixture dosages as well as increasing the water-to-cement ratio. Rotation of samples for the first 24 h was performed to eliminate bleeding to assess differences in the samples due to bleed water reabsorption or lack thereof compared to samples that were not rotated. From the autogenous deformation testing, the following conclusions may be drawn:

- Bleed water reabsorption is the likely cause for the majority of initial expansion exhibited in the linear shrinkage measurements. Rotating the samples as they set greatly reduces, and in some cases, eliminates the initial expansion. Thus, sample rotation is recommended as a necessary technique that should be incorporated into any test method regardless of mix composition or proportions.
- Data analysis and reduction via maximum strain differential calculation was not effective at accounting for early age expansions compared to sample rotation techniques.
- With rotation, all superplasticizer and shrinkage-reducing admixture dosages decreased autogenous shrinkage by similar values compared to their respective controls without superplasticizer or shrinkage-reducing admixtures.
- Both the superplasticizer and shrinkage-reducing chemical admixtures reduced autogenous shrinkage by comparable percentages compared to the control without any admixtures. It is suggested that this decrease is due to a reduction in the pore solution surface tension for both admixtures.
- As expected, increasing water-to-cement ratio led to decreased autogenous shrinkage. Without rotation, bleed water reabsorption caused early age expansion at water-to-cement ratios greater than 0.30.

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