

Admixture compatibility and freeze-thaw resistance of concrete prepared using commercially
produced limestone calcined clay cements

by

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Abstract

Admixture compatibility and fresh, hardened, and durability properties of mortars and concrete prepared using commercially produced limestone calcined clay cements (LC3s) were evaluated during this study. Incompatibility was evaluated for commercially available high range water reducers (HRWRs) and air entraining agents (AEAs) by studying the impact on flow diameters and initial setting time of mortars. Polyethylene-based HRWR was the least suited, whereas vinsol-based AEA was the most suited for use with LC3 cements. The high fineness of LC3 demanded a significantly higher dose of HRWR than control to achieve similar flow for mortars and slump and air content for concretes. Fresh properties (like slump, unit weight, and air content), mechanical properties (compressive and flexural strength) and durability properties (drying shrinkage and freeze-thaw resistance) were documented for LC3 concretes using standardized testing procedures. While all concrete mixtures produced satisfied the compressive and flexural strength requirements for paving concrete, concrete produced using one LC3 showed higher compressive and flexural strength than the control, whereas the concrete produced using another LC3 had lower compressive strength. Air-entrained LC3 concrete demonstrated adequate freeze-thaw resistance. Furthermore, LC3 concrete experienced lower drying shrinkage than the control. While LC3 concrete showed satisfactory mechanical and durability properties for paving application, the primary challenge was to achieve the desired fresh properties, like slump and air content.

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Chapter 1 - INTRODUCTION

Approximately 60% of the carbon emitted during cement production is due to the calcination of limestone, while the remaining 40% is due to the energy required for grinding, and other ancillary cement production processes. Partial replacement of cement with supplementary cementitious materials (SCMs) has been used to produce durable concrete with lower embodied CO₂. However, the local availability of SCMs satisfying ASTM C618 (1) specifications has become strained due to changing (a) energy production (low reliance on coal-burning power plants), (b) environmental regulations (specifically alterations to scrubbing procedures), and (c) location of industrial production (globalization moved production of iron and steel outside the US thereby restricting slag supply in the US). Furthermore, worldwide production of blast furnace slag is capped at 5 – 10% of cement, and although fly ash is available in larger quantities, its variability restricts quantities suitable for use in concrete as specified in ASTM C618 (1). Further reduction in embodied carbon in concrete, therefore, requires identification of alternatives to traditional SCMs. Limestone is abundantly available around the world, but replacement of more than 10% of ordinary Portland cement (OPC) with limestone could result in concrete with undesirable fresh, hardened, and durability properties (2). The addition of calcined clay to the system containing OPC clinker and limestone has shown promising results. The resulting cement, namely, limestone calcined clay cement (LC³), has been shown to be a viable alternative to OPC. In particular, clays with a kaolinite content of more than 40%, which are easily accessible, when calcined and used in combination with limestone and OPC, have been shown to produce mortars with mechanical properties similar to OPC mortars (3–5). Furthermore, production of LC³ can utilize the same manufacturing infrastructure and technologies as are currently being used to produce Type I/II (90% + OPC clinker content) or

Type II (between 5 – 15% limestone content) cements. Commercially produced LC³ is available in markets outside of the US (France, India, etc.), and there are ongoing attempts to commercially produce LC³ in the US. The work presented in this manuscript uses LC³s, which were produced during a large-scale trial production at a US-based cement manufacturing company.

LC³ is typically produced by intergrinding 20-40% calcined clay, 5-15% limestone, 5% gypsum, and 40-60% OPC clinker. Numerous studies have shown that concrete produced using LC³ has the desired strength development needed for paving applications. Although compressive strength was slightly lower due to dilution of the very reactive clinker phase with less reactive (calcined clay) or non-reactive (limestone) phases, flexural strength increased due to the formation of a higher volume of aluminate hydrates in LC³ concrete. Furthermore, using LC³ produces concrete with increased resistance to chloride ingress and sulfate attack (6, 7). Additionally, LC³ has a lower alkali content, thereby reducing the risk of alkali-silica reactions in concrete structures (8, 9).

Despite these advantages of LC³, there are some challenges that need to be addressed to facilitate widespread adoption. The high surface area of calcined clays increases the water demand and thereby reduces the workability of concrete produced using LC³ (10, 11). Hence, to maintain workability similar to Type II and/or Type I/II concretes, superplasticizers are required (3, 12). Furthermore, the cations on clay particles have the tendency to bind molecules of the water reducing admixture, thereby requiring a higher dosage of admixture to produce the desirable workability (3). For applications requiring freeze-thaw durability, like paving concrete, air entraining agents (AEA) are used in conjunction with water reducers to achieve the desired flow and air void parameters. Since literature on the admixture incompatibility for commercially

produced LC³ cement is sparse, this study will document incompatibilities between commonly used plasticizers/superplasticizers and AEAs when used with LC³ cements.

This study evaluates the possibility of using commercially manufactured LC³ to produce durable paving concrete. Admixture incompatibility was identified by comparing the flow and initial setting time of mortars prepared with LC³ to the control mortars (i.e. mortars prepared with Type II cement). Four widely used high-range water reducers (HRWR) and four different AEAs were included in the study. Fresh and hardened properties of paving concrete mixtures prepared using LC³ were compared to control concrete mixture (i.e. concrete prepared using Type II cement). Furthermore, since the literature on freeze-thaw durability of LC³ concrete produced using LC³ is limited, the freeze-thaw performance of air-entrained LC³ concrete was compared to that of Type II concrete.

Chapter 2 - Materials

1. Cements

One Type IL cement and two different LC³ cements, labelled LC³-1 and LC³-2, were utilized during this study. The Type IL cement satisfied the requirements specified in ASTM C595(13). Table 1 summarizes the chemical and physical properties of all three cements as obtained from the producer. The Type IL cement contained 11.8% (by weight) of limestone (92% CaCO₃). The LC³-1 cement was produced by intergrinding 60% Type I/II clinker with 30% (by weight) calcined clay, 5% (by weight) limestone, and 5% (by weight) gypsum, whereas LC³-2 cement contained 50% Type I/II clinker, 30% calcinated clay, 15% limestone, and 5% gypsum.

Table 1 Chemical and physical properties of cements

Chemical Properties			
Oxide	Type IL	LC ³ -1	LC ³ -2
SiO ₂	22.35	30.68	32.55
Al ₂ O ₃	4.91	12.20	7.81
Fe ₂ O ₃	3.37	3.15	3.20
CaO	65.64	46.94	43.45
MgO	1.85	1.12	1.60
SO ₃	0.60	2.38	2.66
Na ₂ O	0.19	0.10	0.20
K ₂ O	0.75	0.73	0.91
TiO ₂	0.30	0.63	0.53
LOI	N.A.	2.88	6.85
Limestone Content	11.80	5.05	13.85
Physical Properties			
Specific Gravity	3.05	2.90	2.87
Blaine Fineness (cm ² /g)	5007	6979	7198

2. Admixtures

All four water reducers and AEAs utilized during this study were widely available products. All water reducers utilized were HRWRs as defined by ASTM C494(14). The water reducers were labelled WR1, WR2, WR3, and WR4, whereas the four AEAs utilized were labelled AE1, AE2, AE3, and AE4. WR1, WR2, and WR3 were polycarboylate-based HRWRs whereas WR4 was polyethelyn-based HRWR. Three of the four AEAs were synthetic products, whereas AE2 was a vinsol-based AEA.

3. Aggregate

Two different coarse aggregates (3/4" and 1/2" nominal maximum size) and one fine aggregate were obtained from local quarries in Kansas. The specific gravity and absorption (provided by the aggregate supplier) for all three aggregates are summarized in Table 2. Particle size distribution was determined following the procedure described in ASTM C136(15). The two coarse aggregates were mixed together to achieve the desired gradation for aggregates as per KDOT requirements for paving concrete with the fine, coarse and intermediate aggregates making up 55%, 20% and 25% of the total aggregate mass, respectively. As shown in Figure 1, the particle size distribution for both coarse and fine aggregates fall within the size required by KDOT specification for paving concrete(16).

Table 2 Aggregate Type and Properties

Type	Specific Gravity	Absorption %
Coarse Aggregate 1 (3/4" nominal size)	2.56	4
Coarse Aggregate 2 (1/2" nominal size)	2.56	2.8
Fine Aggregate	2.61	0.5

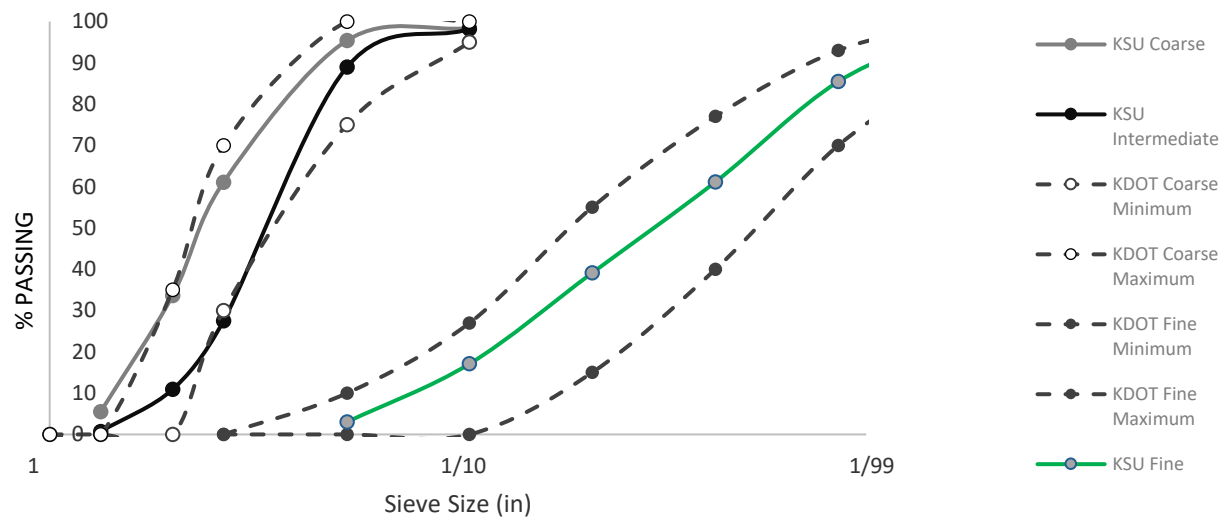


Figure 1 Particle Size Distribution of Coarse and Fine Aggregates

Chapter 3 - EXPERIMENTAL METHODS

Due to the limited availability of LC³ cements, mortars were used to assess the incompatibility of LC³ with HRWRs and AEAs. Admixture incompatibility was determined by quantifying the impact of HRWR and the combination of HRWR with AEAs on the flow and initial setting time of mortars. The incompatibility with HRWR was determined by quantifying the dosage needed to achieve a similar flow diameter (± 10 mm) as the control mortar (i.e. mortar prepared using Type II cement). The incompatibility of LC³ cement with each HRWR was perceived to be higher if the dosage needed to achieve a similar flow diameter (compared to the control) was significantly higher. Significant impact on initial setting time while maintaining the same flow will also point towards potential incompatibility. While evaluating incompatibility with AEAs, the dosage for each HRWR was maintained at the same value as that needed to achieve a similar flow (± 10 mm) to the control determined while evaluating compatibility with HRWR. The change in the flow diameter and initial setting time when AEA was added to mortar containing HRWR was utilized to identify incompatibility. The dosage of AEA utilized was arrived at using a trial and error method and recommendations provided by the manufacturer.

Only select combinations of AEA and HRWRs were utilized to produce concrete due to the limited availability of LC³. Fresh and hardened properties of concrete prepared using LC³ were compared to those prepared using Type II cement. Furthermore, freeze-thaw durability of concrete produced using commercially produced LC³ was determined.

3.1 Mortar Testing

3.1.1 Mortar Mix Design and Mixing

The mortars were batched following the procedure described in ASTM C305(17). A water to cementitious ratio of 0.40 and sand to cement ratio of 2.75 were utilized as recommended in

ASTM C109(18). 90% of the mixing water was added to a Hobart mixer bowl prior to adding cement over a period of 30 seconds while the mixer operated at 140 ± 5 rpm. Following that, an appropriate amount of sand was added over 30 seconds. After the addition of the sand, the paddle mixer was set to 285 ± 10 rpm, and the remaining water was added along with the appropriate HRWR and AEAs.

3.1.2 Mortar Flow Table

The procedure described in ASTM C1437(19) was followed to determine the flow after 10 and 30 minutes of mixing. The flow recorded after 30 minutes was primarily conducted to document the slump loss experienced between the 10 and 30 minute flow readings. The mortar was confined and consolidated into a conical brass mold as shown in Figure 2 and then subjected to twenty five half-inch drops after removing the confinement. The average diameter of the resulting circular cake was obtained by measuring along four different diameter segments as shown in Figure 3. Mortar flow was reported as the percent change in diameter from the initial diameter of 4" (from the brass mold). Three replicates were tested for each combination of cement and admixtures.



Figure 2 *Flow Table*



Figure 3 *Mortar after 25 half-inch drops*

3.1.3 Initial Setting Time of Mortars

Initial setting time was measured following the procedure outlined in ASTM C191(20) using a 1 mm Vicat needle and a consolidation rod weighing 300g. Immediately after batching the mortar,

molds were prepared and stored in a 100% relative humidity environment for 30 minutes. Henceforth, the samples were tested every 10 minutes using the Vicat needle until the initial setting time was determined. The mold utilized was conical in shape with a height of 40 mm, and top and bottom diameters of 40 mm and 70 mm, respectively.

3.2 Concrete Testing

3.2.1 Concrete Mix Design and Mixing Procedure

Table 3 shows the mixture design used to produce concrete. The water-to-cementitious ratio was maintained at 0.40 for all three mixtures. The volumetric content of aggregates was kept constant ($0.7 \text{ yd}^3 \text{ per yd}^3 \text{ of concrete}$) and the mass of cement was changed based on the density of the cementitious materials utilized in each mixture. HRWR was not needed for the control mixture, but a significant dosage of HRWR was utilized, as shown in Table 3, to produce workable LC³ concrete.

Coarse and intermediate aggregates were added to the mixing drum along with a small quantity of mix water to start the mixing process. Following that, fine aggregate was poured into the drum along with some more mix water. The aggregates were allowed to be mixed for 30 seconds prior to adding cement. All admixtures were mixed with the remaining mix water before adding it to the mixing drum. Concrete was mixed for three minutes, allowed to rest for three minutes, and then mixed again for two minutes before measuring fresh properties.

Table 3 Concrete Mixture Design

Material	Control Concrete	LC ³ -1 Concrete	LC ³ -2 Concrete
Cement	518.29	521.5	519.1
Water	207.3	208.6	207.6
Coarse Aggregate	616	616	616
Intermediate Aggregate	770	770	770
Fine Aggregates	1694	1694	1694
AEA (AE2)	1.5 fl oz/lb of cement	1.5 fl oz/lb of cement	1.5 fl oz/lb of cement
HRWR (WR2)	Not Used	7.0 fl oz/lb of cement	11.0 fl oz/lb of cement
All values specified in lb/yd ³			

3.2.2 Slump and Unit Weight

Slump cone tests were performed in accordance with ASTM C143(21) on freshly produced concrete within two minutes of completing mixing. The sample was consolidated in three layers prior to lifting the cone to measure the slump. KDOT specification for pavement concrete requires the slump to be less than 2.5”(16). The unit weight was determined by recording the weight of concrete consolidated into a 0.25 ft³ pot and dividing the recorded weight of concrete by the volume of the container, as specified in ASTM C138(22).

3.2.3 Fresh Air Content

Air content of fresh concrete was determined using the pressure method outlined in ASTM C231(23). KDOT specification for paving concrete requires the air content to be between 5 – 8% (by volume).

3.2.4 Compressive and Flexural Strength

The procedure described in ASTM C39(24) and ASTM C78(25) was followed to determine the compressive strength and flexural strength, respectively. For each type of concrete produced, three replicates of 4” by 8” cylinders were tested after 7 and 28 days of curing to determine compressive strength. A loading rate of 35 psi/s was applied until failure.

Flexural testing was performed on 16” long beams with a cross-section of 5” by 5”. Three replicates were tested for each mixture after 7 and 28 days of curing in a 100% relative humidity environment. The maximum load was recorded during testing and utilized to calculate the modulus of rupture using Equation 1, which was obtained from ASTM C78(25). Figure B.1 in Appendix B shows the beams after performing the flexural strength test.

$$R = \frac{PL}{bd^2} \quad \text{Equation 1}$$

where

R = modulus of rupture, psi

P = maximum applied load recorded by the testing machine, lbf

L = span length, in.

b = width of specimen, in.

d = depth of specimen, in.

3.2.5 Freeze-thaw testing

Methodology described in ASTM C666(26) was followed to determine the freeze-thaw durability of concrete samples. Three replicates of 16” long beams with a cross-section of 3” by 4” were tested for each concrete mixture. The freeze-thaw chamber was programmed to undergo approximately eight freeze-thaw cycles per day. The concrete specimens were frozen in air and thawed in water (i.e. ASTM C666 Procedure B). ASTM C215(27) procedure was followed to measure the fundamental transverse frequency of the concrete beams after approximately every

36 freeze-thaw cycles. The relative dynamic modulus of elasticity (RDME) was calculated for each sample using the equations 2 below and as described in ASTM C666(26).

$$P_c = \left(\frac{n_1^2}{n^2} \right) * 100 \quad \text{Equation 2}$$

Where:

P_c = relative dynamic modulus of elasticity, after c cycles of

freezing and thawing, percent,

n_1 = fundamental transverse frequency at 0 cycles of freezing and thawing, and

n = fundamental transverse frequency after c cycles of freezing and thawing.

3.2.6 Shrinkage

The unrestrained length change was determined following the procedure described in ASTM C157(28) to quantify drying shrinkage experienced by 3"x3"x11 3/4" concrete beams. Concrete beams were cast, and the initial length was measured after 28 days of curing in lime water.

Following initial length measurement, the beams were stored in a 50% relative humidity environment until the end of testing. Measurements were made after 4, 7, 14, and 28 days as shown in Figure 4. The change in length was normalized with the initial length of the sample and reported as a percentage shrinkage value. Two replicates were tested for Type IL concrete and LC³-1 concrete. LC³-2 concrete specimens were not tested because of the limited availability of LC³-2 cement.



Figure 4 *Length change measurement*

Chapter 4 - RESULTS AND DISCUSSION

4.1 Mortar Testing

4.1.1 Impact of HRWR on flow diameter and initial setting time

This testing was conducted to document the dosage of commercially available HRWR needed to achieve similar workability as control mortars (i.e. mortars prepared using Type IL cement).

Mortar prepared using Type IL cement (without any admixtures) had a flow diameter of 6.9" and 6.33" after 10 minutes and 30 minutes of mixing, respectively. The manufacturer-recommended dosage of HRWR admixtures (3.782 fl oz/lb of cement) was added to control mortar to produce mortars referred to as control-WR mortars, except for when WR2 was used (where a 6.82 fl oz/lbs of cement was utilized). For example, control-WR1 mortar refers to mortar produced using Type IL cement and WR1 as the HRWR. Flow diameters of 7.91", 7.68", and 7.71" were recorded for control-WR1, control-WR2, and control-WR3, respectively, after 10 minutes of hydration. As expected, lower flow diameters of 7.24", 7.16", and 6.85" were recorded for control-WR1, control-WR2, and control-WR3, respectively, after 30 minutes of hydration. The slump loss for the control mortar (i.e., mortar without any admixture) was 8% between 10 and 30 minutes, whereas that for control mortars containing WR1, WR2, and WR3 were 8.4%, 6.6%, and 11%, respectively. The dosage of WR4 needed to achieve the target flow was very high, and therefore, disregarded from further investigation due to a clear incompatibility issue.

To identify the incompatibility of LC³ cements with each HRWR, the dosage needed to achieve the same flow diameter (after 10 minutes) as the corresponding control mixture (i.e. Type IL mortar prepared using the same HRWR) was determined. Figure 6 shows the amount of HRWR needed (as a percentage of the corresponding control) to achieve a similar flow as the control-WR mortar. As expected, all mortars prepared using LC³ needed a significantly higher dosage of HRWR to achieve the targeted flow diameters (i.e. flow diameters achieved by Type IL mortar prepared with the same HRWR). This could be attributed to the higher Blaine fineness of LC³s compared to the Type IL cement. Following along the same explanation, mortars prepared using LC³-2 required a higher dosage of HRWR than mortars prepared using LC³-1. Between the four HRWRs tested, WR3 was the most efficient one, while still needing 140% and 250% dosage to achieve the same flow diameter as the control-WR3 mortar. On the other hand, WR4 was the least efficient HRWR for dispersing LC³. The polyethylene-based HRWR, i.e. WR4, was

determined to be incompatible with LC³s because it needed up to 600% of the control-WR4 dosage to achieve similar flow. This finding corresponds well with other published studies suggesting that polycarboxylate-based HRWRs are best suited to be used with LC³ cements(12). However, further testing needs to be conducted to explain the observed differences in the flow diameters between the different polycarboxylate-based HRWRs. Given this limitation of polyethylene-based HRWR, all further testing was performed exclusively using polycarboxylate-based HRWR.

Table 4 shows the slump loss experienced by all mortars between 10 and 30 minutes. Percent slump loss was determined by dividing the difference in the flow diameters measured at 10 and 30 minutes by the 10-minute flow diameter. Since the dosage of HRWR was arrived at by targeting the flow diameter at 10 minutes, the slump loss between 10 and 30 minutes could highlight which HRWRs experienced the largest slump loss. Despite LC³-1 being coarser than LC³-2, LC³-1 mortars showed higher slump loss with and without all HRWRs. This could be due to the higher OPC clinker content of LC³-1. Type IL and LC³-1 cement show the highest slump loss with WR3, whereas LC³-2 shows the highest slump loss when mixed with WR1. LC³-1 shows a narrow slump loss range (between 9.7% and 10.9%) with or without HRWRs, and therefore are not considered incompatible based on slump loss. LC³-2 shows a high slump loss with WR1 compared to other HRWRs; however, it's in the same range as those demonstrated by Type IL mortars. Therefore, no incompatibilities with HRWR were identified based on the slump loss during this study.

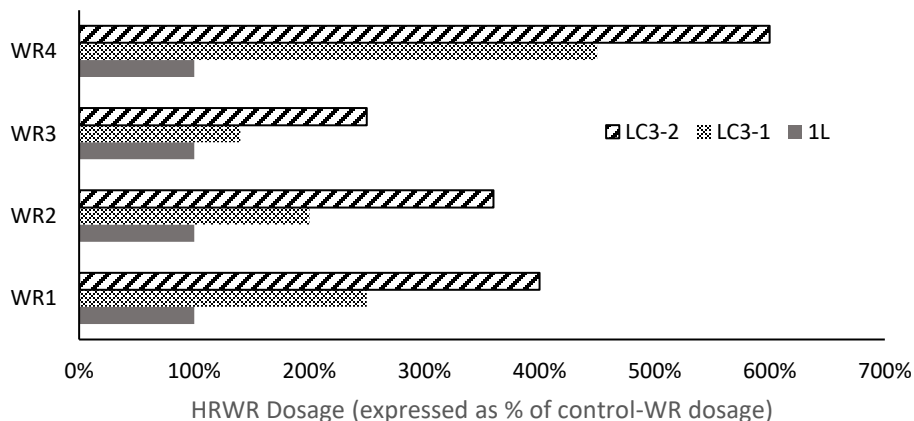


Figure 5 Dosage of HRWR needed to achieve the target flow diameter

Table 4 Slump loss between 10 and 30 minutes for all mortars

Type of Cement	HRWR			
	<i>None</i>	<i>WR1</i>	<i>WR2</i>	<i>WR3</i>
<i>Type IL</i>	8.0%	8.4%	6.6%	11.0%
<i>LC³-1</i>	9.9%	10.4%	9.7%	10.9%
<i>LC³-2</i>	6.8%	10.0%	7.6%	8.6%

Figure 6 shows the impact of HRWR on the initial setting time of mortars. The dosage of HRWR utilized was the same as that needed to match the flow of the corresponding control-WR mixture. As expected, the initial setting time increases for all mortars when polycarboxylate-based HRWRs are used. The highest increase was observed when using WR2 with all three cements, while this increase was the smallest for LC³-1. Despite the higher dilution of OPC clinker in LC³ cements compared to Type IL cement, LC³ cements had lower initial setting time with and without HRWR. Furthermore, all LC³ mortars (except LC³-2 WR2 and LC³-2 WR3) had lower initial setting time than the control mortar with the lowest initial setting time (i.e. Type IL mortar without any HRWR). This could be due to the significantly higher surface area of LC³ (see Table 1). This impact of fineness on initial setting time disappears when comparing LC³-1 and LC³-2 because, despite being finer, LC³-2 has a higher initial setting time than LC³-1 when HRWR are used. HRWR seems to alter the initial setting time of LC³-2 mortars but the initial setting time values are still lower than that for the control and therefore, no incompatibilities could be identified based on initial setting time. Further testing needs to be conducted to understand the disparate impacts of different polycarboxylate-based HRWRs on the initial setting time of LC³ cements.

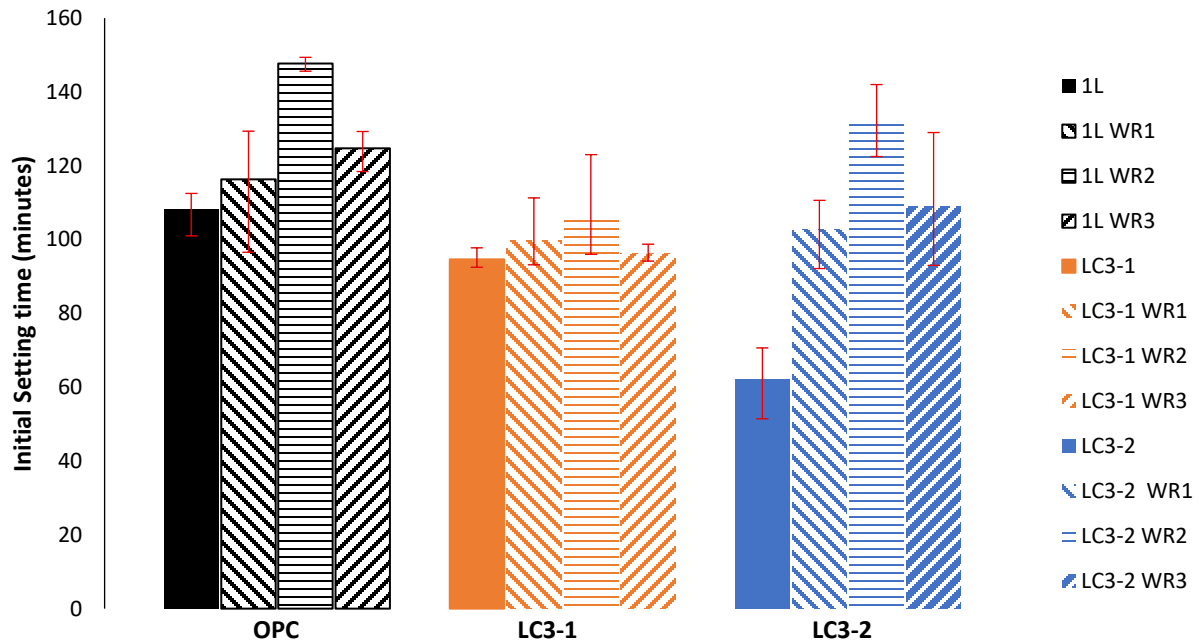


Figure 6 *Impact of adding Water Reducers on the setting time of cements*

4.1.2 Impact of AEA on flow diameter and initial setting time

AEA dosages for control mortars were determined based on manufacturer recommendations. Control mortars experienced a slight increase in flow diameter (10 minutes after mixing) for all different AEA tested. The AEA dosages for LC³ mortars were arrived at by attempting to match the flow diameter (after 10 minutes) with the corresponding control mortar. Despite using 3-5 times higher dosage for AEA for LC³ mortars, Figures 7 a, b, and c show there was little or no change observed in the flow diameter (after 10 minutes) when different AEAs were added to both LC³ mortars. As shown in Figure 8a, for control mortar containing WR1, there was a measurable increase in flow diameter when AEA was added, and that increase was different for different AEAs. Entraining air is expected to increase workability; therefore, these

observation points towards a potential reduction in the efficiency of commercially available AEA when used with LC³. This hypothesis needs further investigation with an air void analyzer (AVA) to confirm the low efficiency of commercially available AEA with LC³ systems.

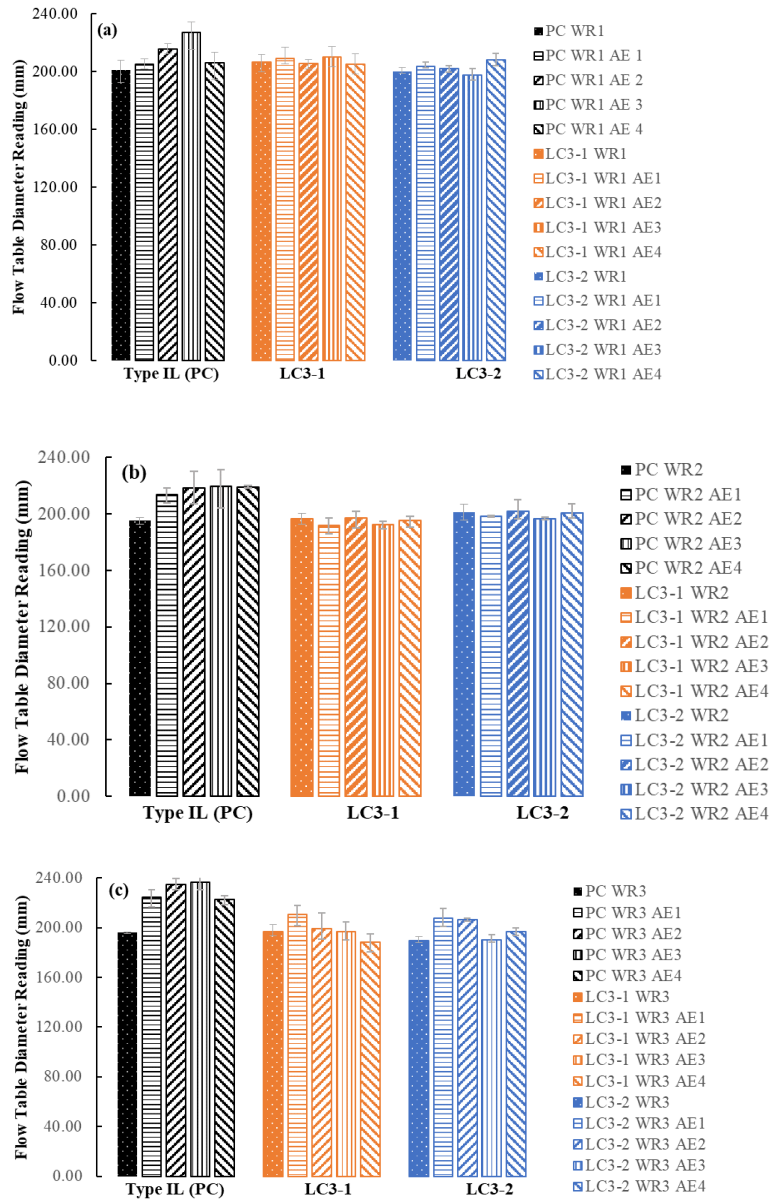


Figure 7 Impact of AEA on flow diameter of mortars containing (a)WR1, (b) WR2, and (c) WR3

The addition of AEA caused a noticeable increase in the initial setting time for most mortars with a few exceptions. While only results containing WR2 are shown in Figure 8. Significant increases in initial setting time for control mortar containing WR2 were observed when using AE1 (51%) and AE2 (47%) as AEAs. Larger increases in initial setting time were observed for LC³-1 (79%) and LC³-2 (109%) mortars when using AE3 as the AEA. Vinsol-based resin (AE2) had the lowest impact on the setting time of mortars containing WR2. Similarly, low impact on initial setting time was observed in mortars prepared using WR1 and WR3 (data not included in this manuscript). This points towards better compatibility between vinsol-based AEA and polycarboxylate-based HRWR when used in LC³ cementitious systems. On the other hand, the addition of AE3 led to a notable increase in the initial setting time, particularly for LC³-2, across all tested HRWRs. This effect was more pronounced compared to that for control and LC³-1 mortars, indicating incompatibility between AE3 and the LC³-2 cementitious system.

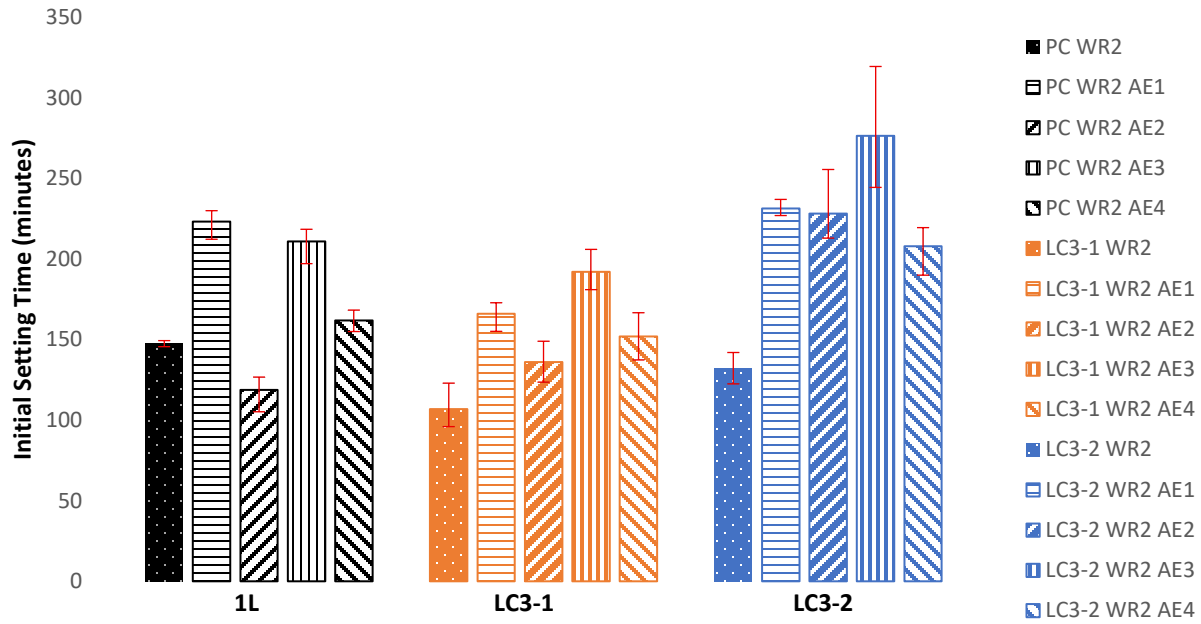


Figure 8 *Impact of adding Air Entrainers on the setting time of cements*

4.2 Concrete Testing

4.2.1 Fresh Properties

The slump, air content, and unit weight of all concrete mixtures are shown in Table 5.

Despite using high dosage of HRWR in LC³ mixtures (see Table 3) the slump of LC³ concretes was lower than that for control concrete (which did not contain HRWR). During trial batching, it was challenging to achieve the 6% targeted air content for LC³-1 concrete without using HRWR.

The non air-entrained LC³-1 concrete had an air content of 3.5%.

Table 5 Fresh Properties of Concrete

Property	Control	LC ³ -1 Concrete	LC ³ -2 Concrete
Slump	1"	0.8"	0.5"
Unit Weight	145 lb/ft ³	140 lb/ft ³	n/a
Air Content	6.0%	6.5%	n/a

4.2.2 Compressive Strength

Figure 9 shows the compressive strength values for control and LC³ concrete mixtures. At an early age (7 days), concrete prepared using LC³ is expected to be weaker due to the high dilution factor (i.e. replacement of reactive clinker with less (or not) reactive calcined clays and limestone). However, that was only true for LC³-2 concrete (6.6% lower than the control). LC³-1 concrete, on the other hand, had a 21.6% higher compressive strength than the control after 7 days. The higher fineness of LC³-1 could have been sufficient to overcome the impact of dilution on strength development. While LC³-2 is finer than LC³-1, it also has a higher replacement of OPC clinker with limestone and calcined clay, and therefore was not as strong as the control.

A similar trend was observed in the strength measured after 28 days of curing. The compressive strength increased at a faster rate between 7 and 28 days for the control mixture (40% increase in strength) than both LC³ mixtures (27% and 23% increase in strength). A slower strength gain at later ages is expected for finer cements as more of their surface area is accessible for reaction at early stages compared to a coarser cement. Regardless, as shown in Figure 9, all three concrete mixtures achieved the KDOT-specified 28-day compressive strength for paving application. Control and LC³-1 concretes were able to achieve the target value before 7 days, whereas LC³-2 concrete exceeded the target by only 400 psi after 28 days of curing. This is less than the 1200 psi buffer required by KDOT for laboratory produced concrete with a specified design strength. LC³-2 did however gain strength towards the later stages of the curing process. Further research is needed to study the strength gain of LC³-2 beyond 28 days of curing.

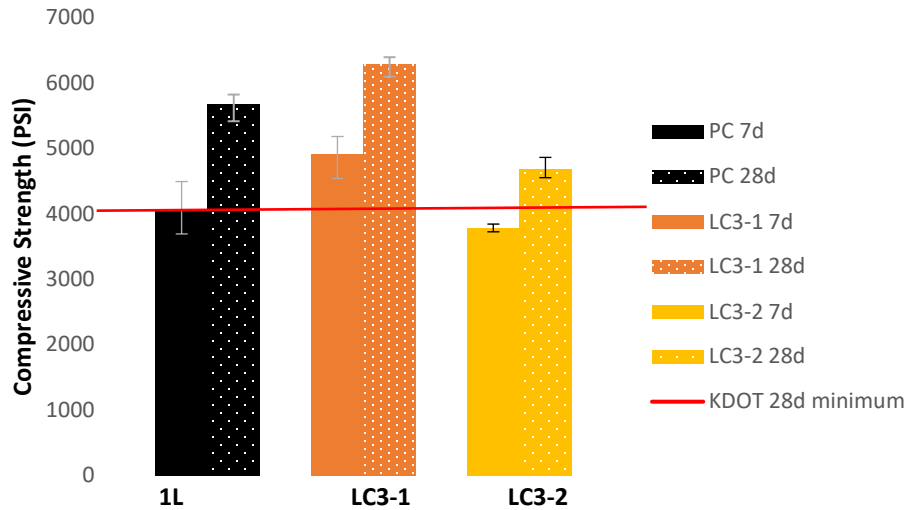


Figure 9 Compressive Strength of concrete

4.2.3 Flexural Strength

Due to the limited availability of LC³-2 cement, only control and LC³-1 concrete beams were produced for flexural testing. Furthermore, the limited availability of LC³-1 restricted flexural testing to only be performed for one specific age, i.e. after 7 days of curing. Control LC³-1 concretes had a modulus of rupture of 533 psi and 638 psi, respectively. The higher flexural strength of LC³-1 concrete follows the same trend as observed for compressive strength. Extrapolating from this, it could be safe to assume that LC³-1 concrete would show a higher modulus of rupture than the control concrete after 28 days of curing. This higher modulus of rupture exhibited by LC³-1 concrete indicates enhanced flexural performance, which could make it a suitable option for paving applications. Further mechanical testing, particularly split tensile testing, is needed to confirm enhanced behavior for LC³-1 concrete.

4.2.4 Drying Shrinkage

Figure 11 shows the % change in length for the control and LC³-1 concrete specimens. Despite the faster strength gain and higher fineness, the LC³-1 specimen showed lower shrinkage compared to the control concrete specimen. Control specimens approached a plateau after drying for 28 days in a 50% relative humidity environment. On the other hand, LC³-1 specimens experienced unrestrained length change at a slower rate in the early phases of drying, but were approaching the shrinkage experienced by the control specimen by 28 days. Further testing is needed to quantify drying shrinkage up to one year.

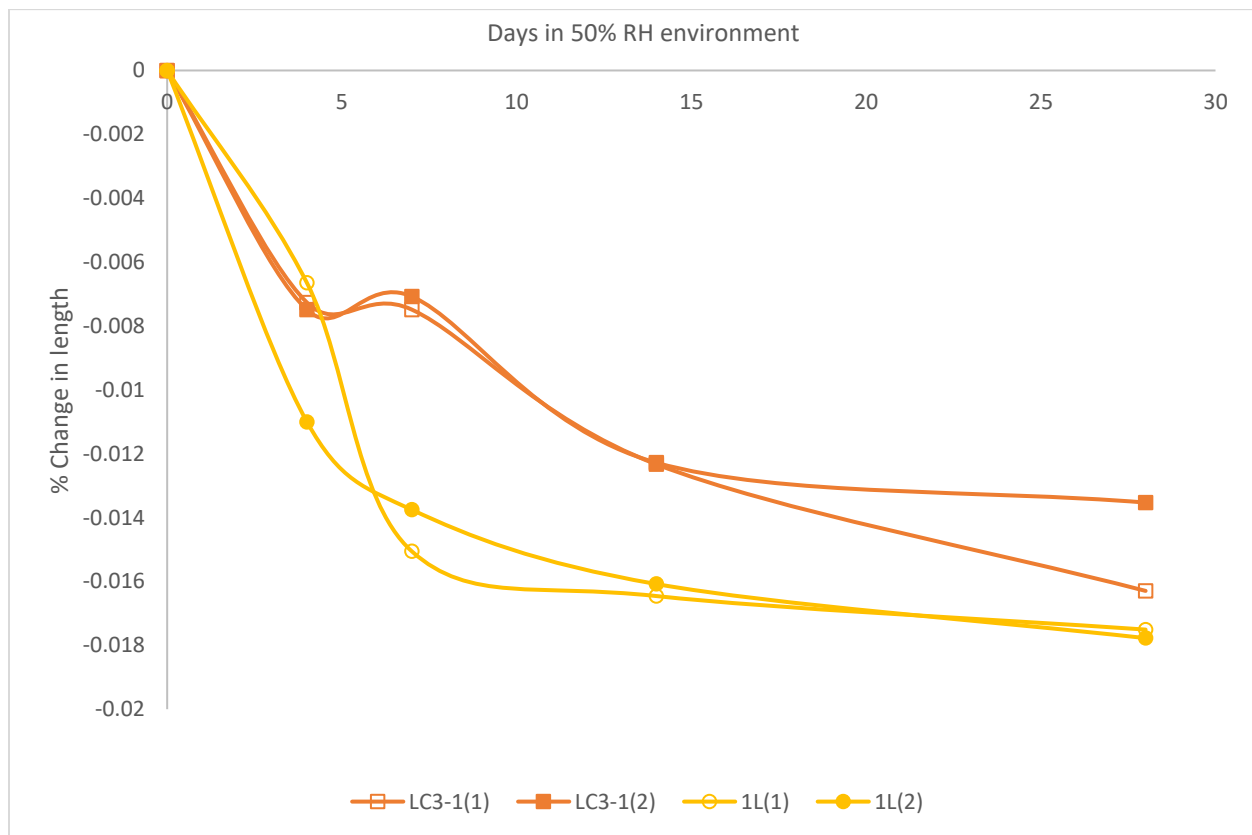


Figure 10 Shrinkage of control and LC³-1 concrete

4.2.5 Freeze-thaw Resistance

The relative dynamic modulus of elasticity is plotted against the number of freeze-thaw cycles in Figure 11. Air-entrained Type 1L control (labelled as PC-AE in Figure 11) and LC³-1

(labelled as LC³-AE in Figure 11) concrete specimens showed little to no signs of deterioration after experiencing 300+ freeze-thaw cycles. To observe failure, LC³-1 concrete was prepared without air entrainment. As shown in Figure 12, RDME was reduced to 0.9 after 300 freeze-thaw cycles, which still satisfies the KDOT specifications for paving concrete. Chips, cracks, and edge spalling were observed at the end of 300 freeze-thaw cycles on type 1L and non-air-entrained LC³-1 concrete specimens as shown in Figures 12 and 13. Despite these observed surface damages, there was little to no impact on the measured transverse frequency.

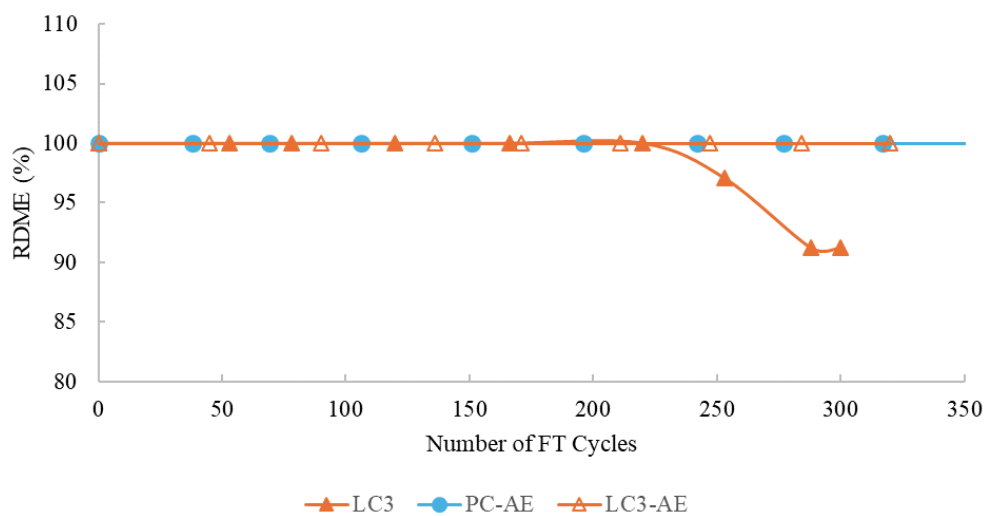


Figure 11 RDME for concrete subjected to freeze-thaw



Figure 12 TYPE 1L beam 1 showing some damage to the surface



Figure 13 LC³ beam showing small damage at the corner of the beam

Chapter 5 - CONCLUSION

Mortars and concretes were prepared using two different LC³ cements that were produced in a trial run at a US-based cement manufacturing plant. Admixture incompatibility of these cements, with commercially available AEAs and HRWRs, was evaluated by comparing the impact on flow diameters and initial setting time of mortars prepared using LC³ to that of mortars prepared using Type IL cement. Furthermore, the freeze-thaw durability and drying shrinkage potential of paving concrete produced using LC³ cements were evaluated. The following observations were made during this study:

- LC³ mortars required significantly higher dosage of HRWR to achieve the same flow as mortar prepared using Type IL cements. Up to 600% dosage of HRWR was documented for LC³ to achieve the same flow Type IL mortar.
- Polycarboxylate-based HRWR were more efficient in dispersing LC³ cements than polyethylene-based HRWR. Different polycarboxylate-based HRWR performed differently when used with LC³s. More research is needed to determine the cause of this observed difference among different commercially available polycarboxylate-based HRWRs.
- When sufficiently dosed with HRWR to achieve target flow diameters, the initial setting time more than doubled for one of the LC³ mortars, while there was little impact on the initial setting time (up to 12%) for the other LC³ mortar.
- Addition of AEA to Type IL mortars containing HRWR increased their flow, but it had no impact on the flow of LC³ mortars, which contained HRWR. On the other hand, the addition of AEA increased the initial setting time for all mortars that contained HRWR. Vinsol-based AEA performed best when used with LC³ mortars.

- As was observed with mortars, LC³ concrete had a lower slump despite being dozed with HRWR.
- Concrete produced using LC³-1 showed higher compressive and flexural strength than the control whereas concrete produced using LC³-2 showed lower compressive strength than the control, primarily because of 10% lower clinker content.
- Concrete produced using LC³ showed similar freeze-thaw resistance as the control mixture.
- The rate of unrestrained length change due to drying shrinkage was slower in LC³ concrete than in the control.

Further testing needs to be conducted to identify the cause of the differences observed in the flow diameter and setting time when using different commercially available polycarboxylate-based HRWR. In addition, the efficiency of commercially available AEAs in generating desirable hardened air parameters in LC³ concrete needs further evaluation. While durability parameters like freeze-thaw resistance and drying shrinkage appear to be acceptable for LC³ concrete, further research is needed to evaluate these properties over a longer period of time and under more strenuous conditions (like deicing salt exposure).

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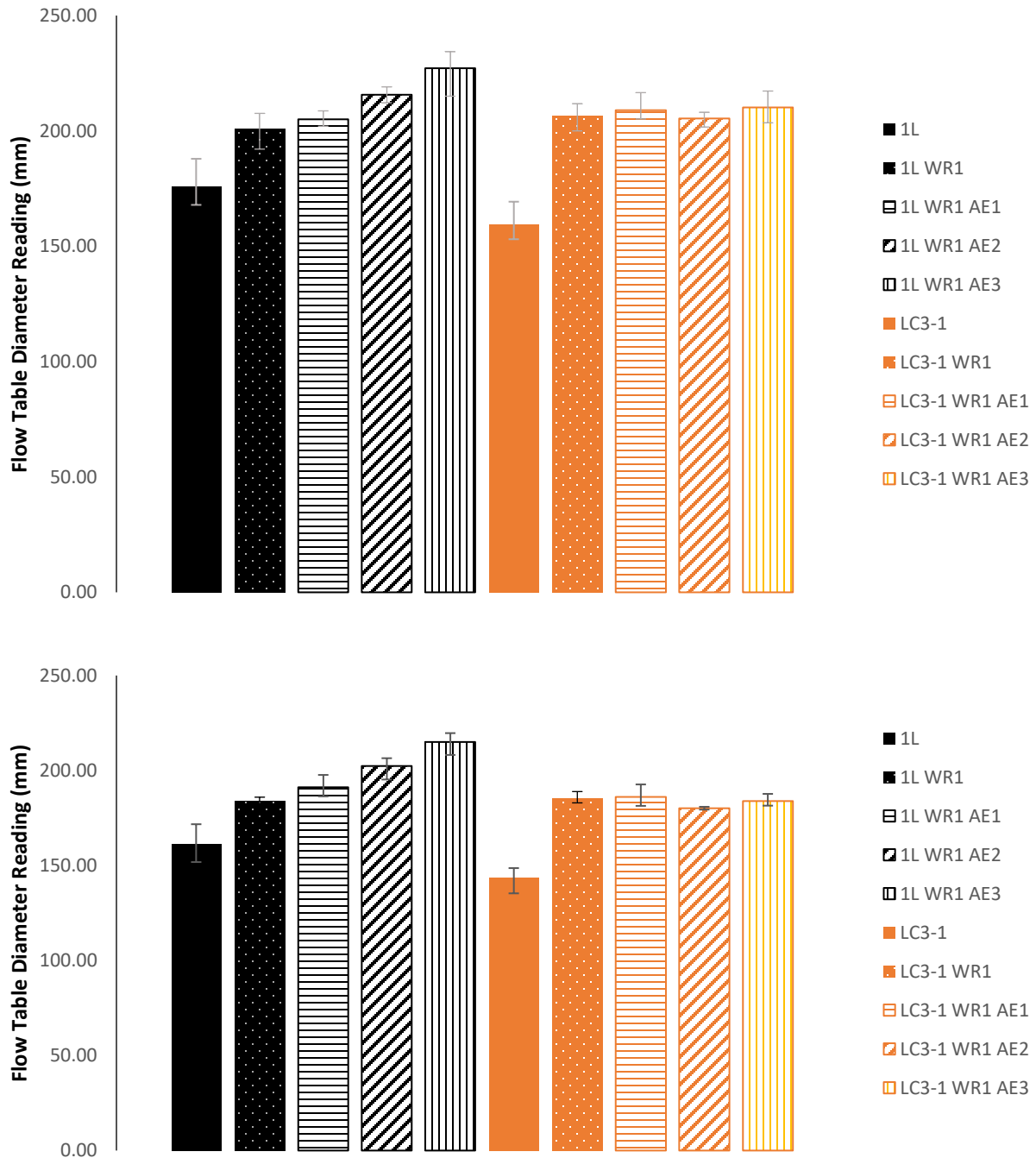
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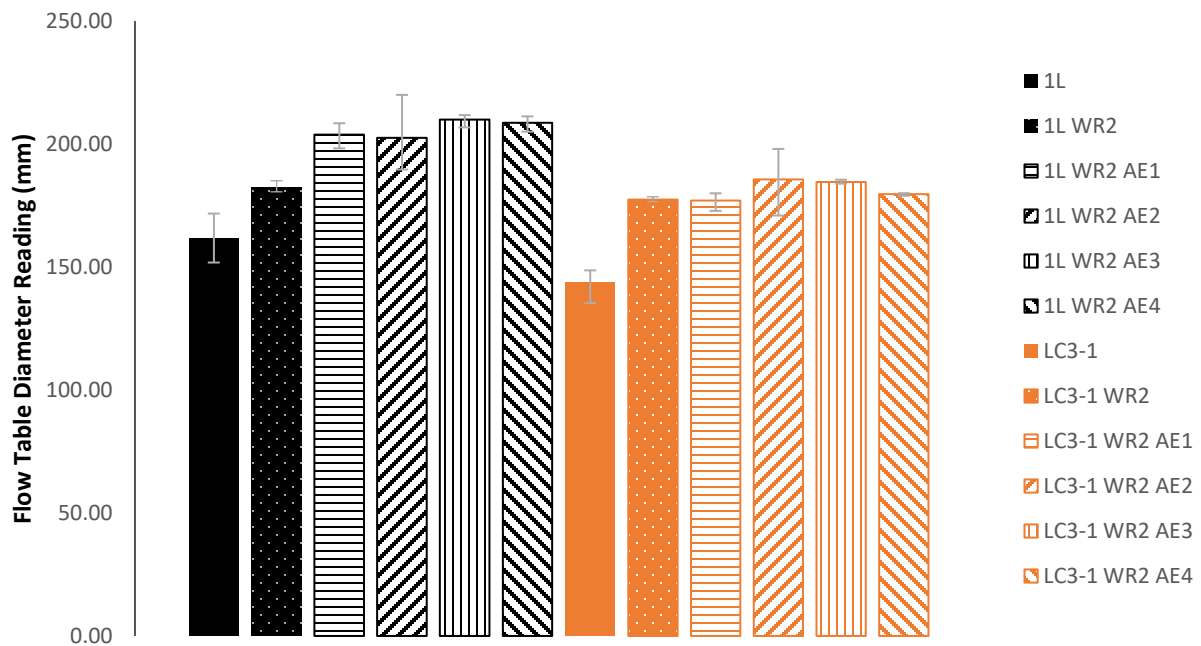
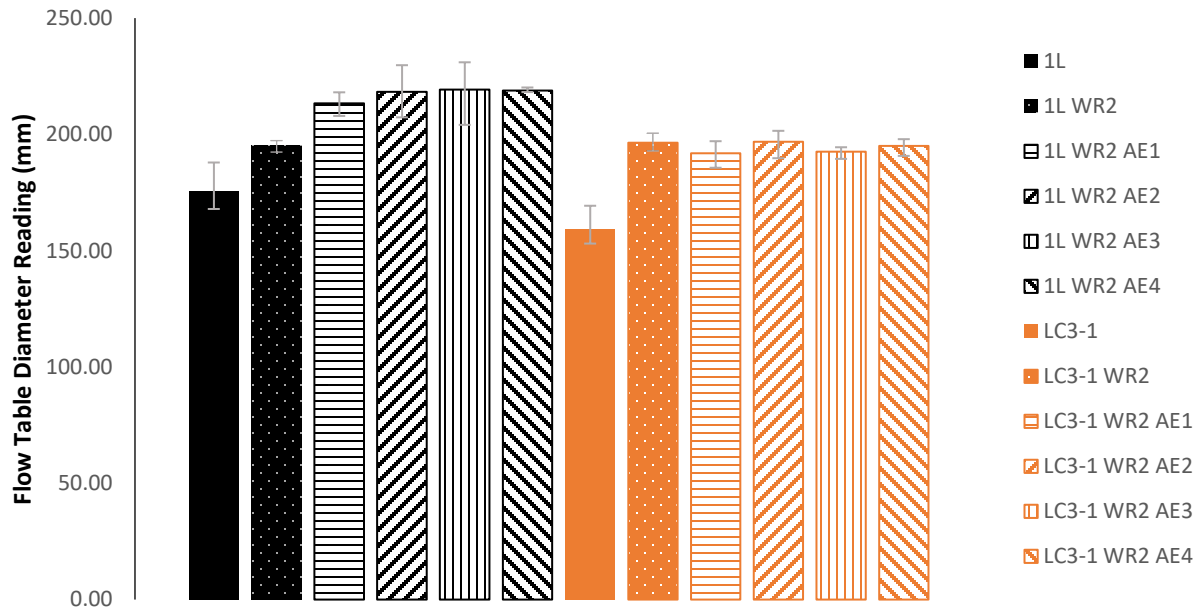
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Appendix A - Mortar Flow and Setting Time Charts

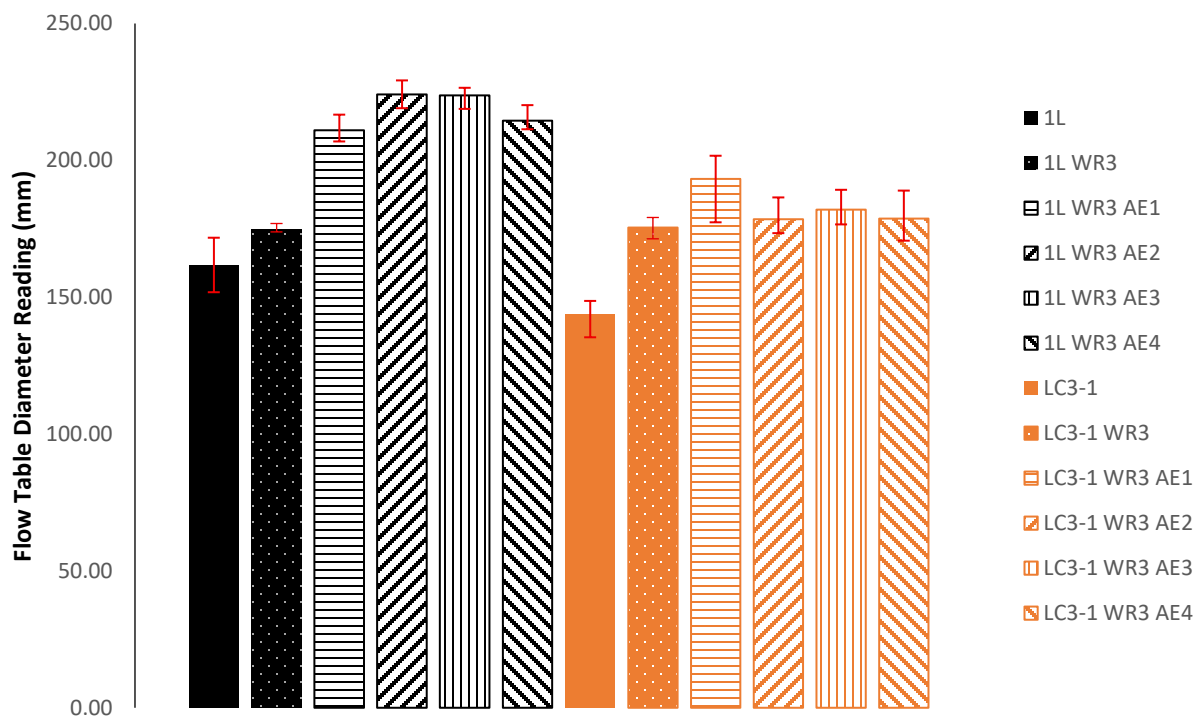
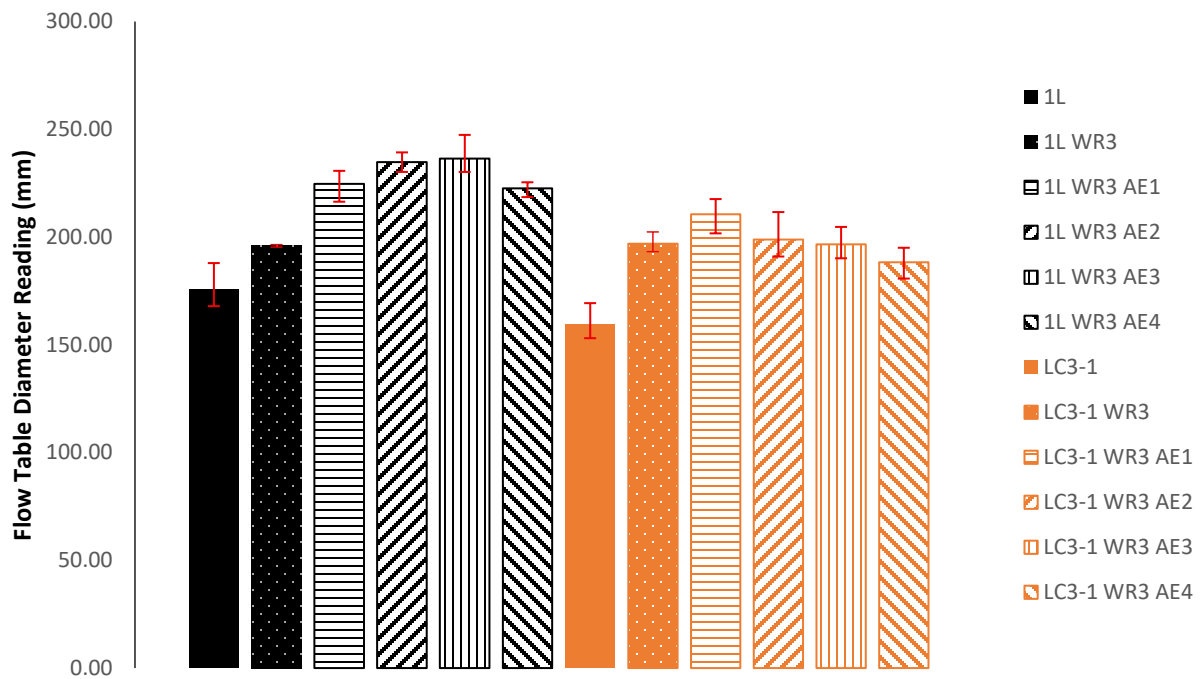
Appendix Figure A.1. 10 and 30 minute Flow of TYPE 1L vs LC³-1 with WR1 and AEs



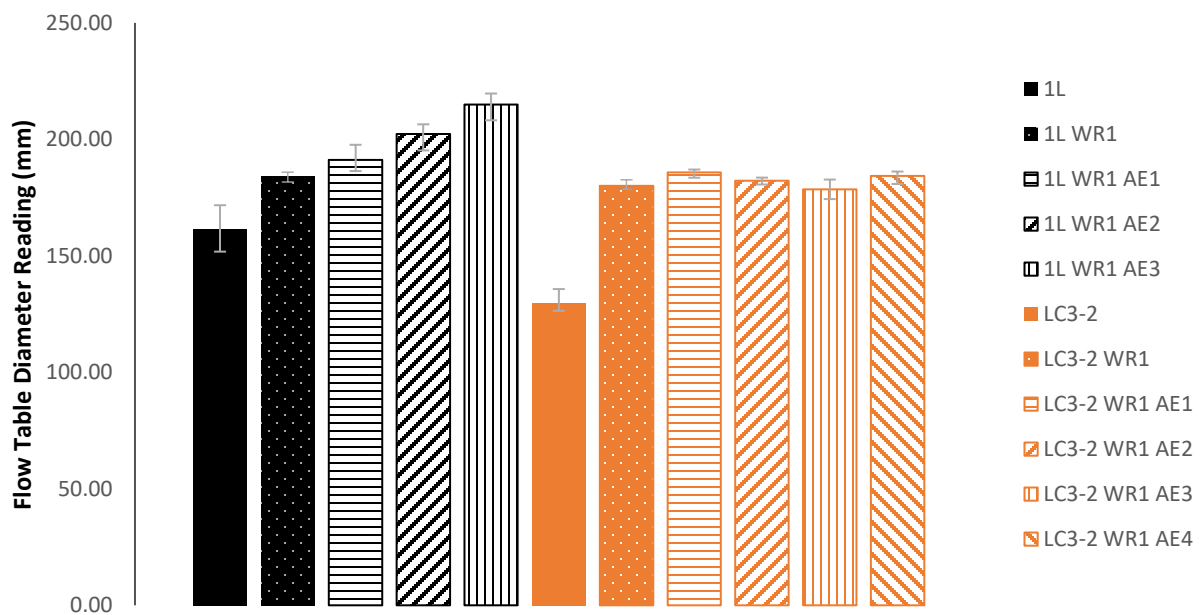
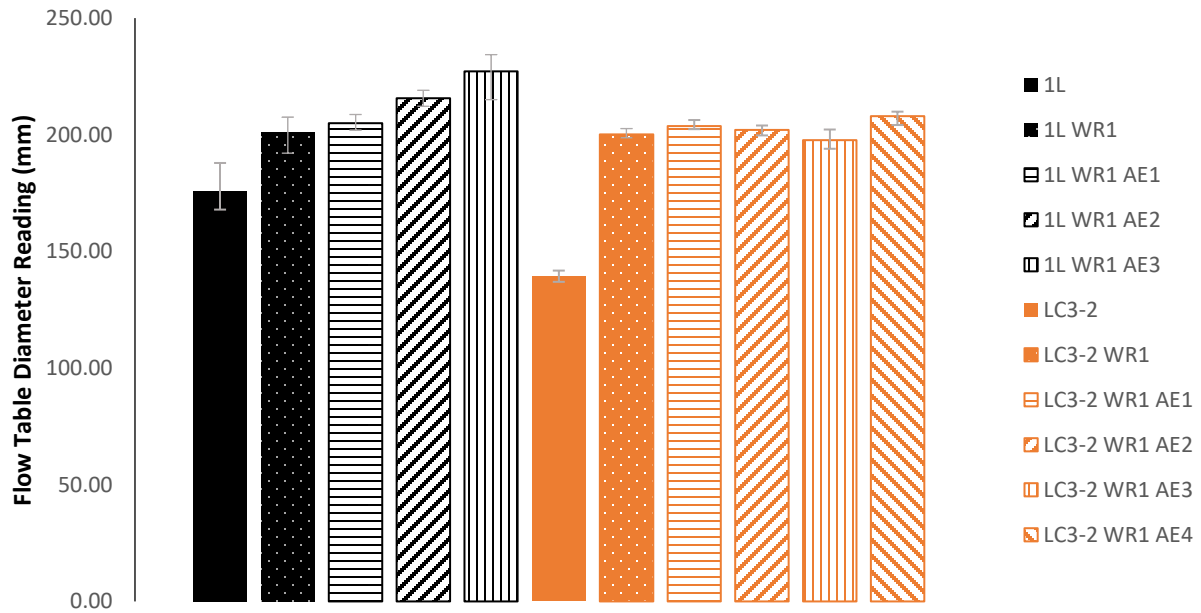
Appendix Figure A.2. 10 and 30 minute Flow of TYPE 1L vs LC³-1 with WR2 and AEs



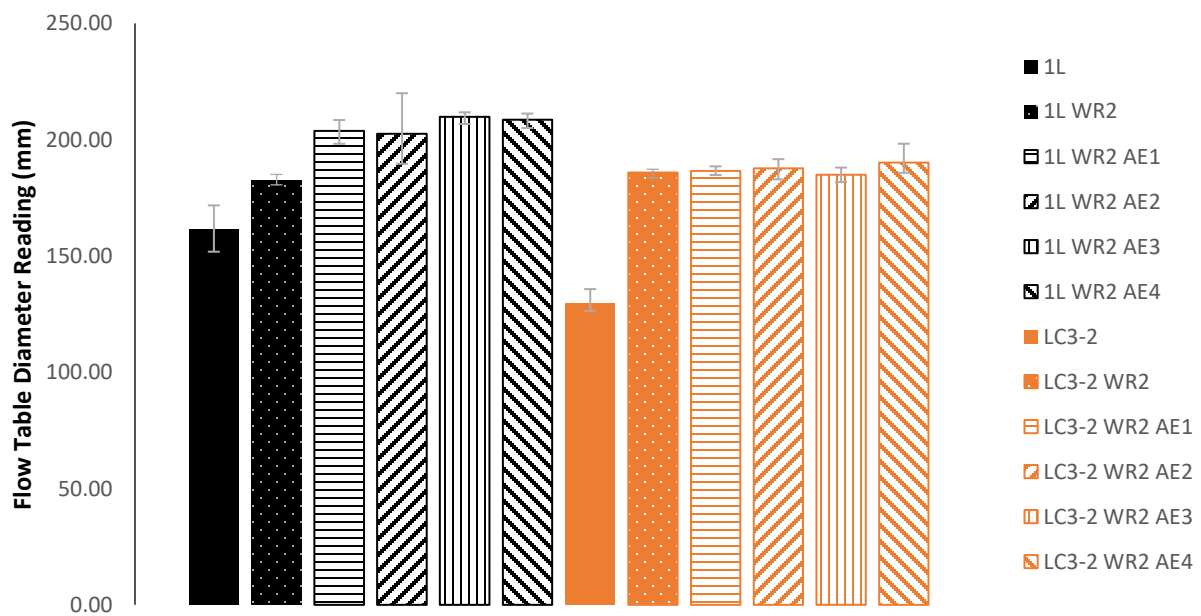
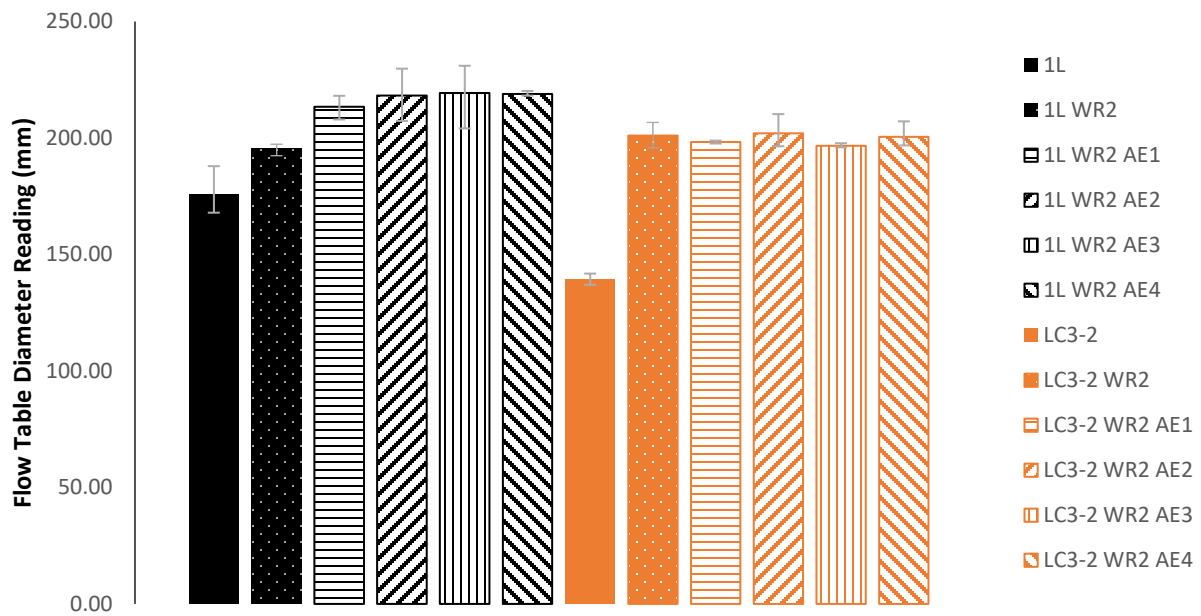
Appendix Figure A.3. 10 and 30 minute Flow of TYPE 1L vs LC³-1 with WR3 and AEs



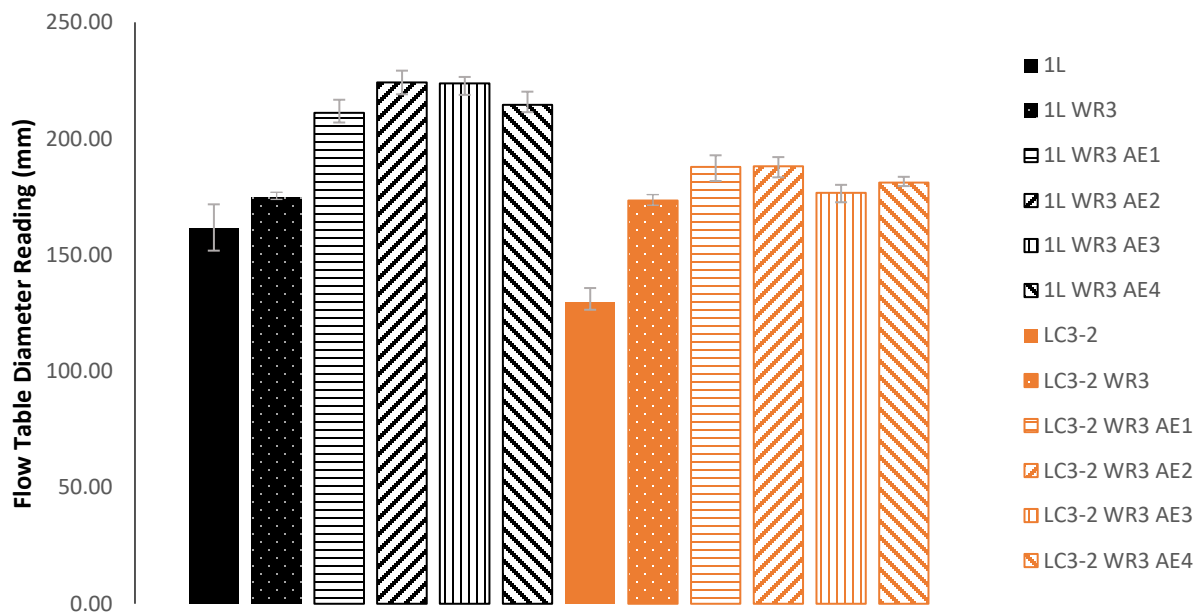
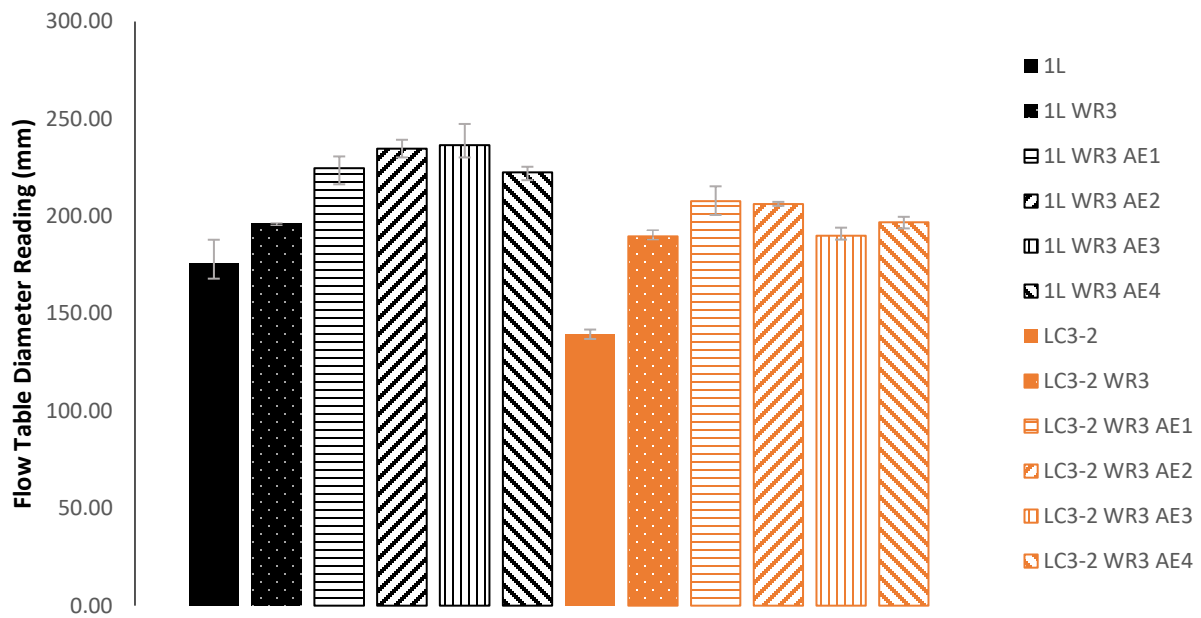
Appendix Figure A.4. 10 and 30 minute Flow of TYPE 1L vs LC³-2 with WR1 and AEs



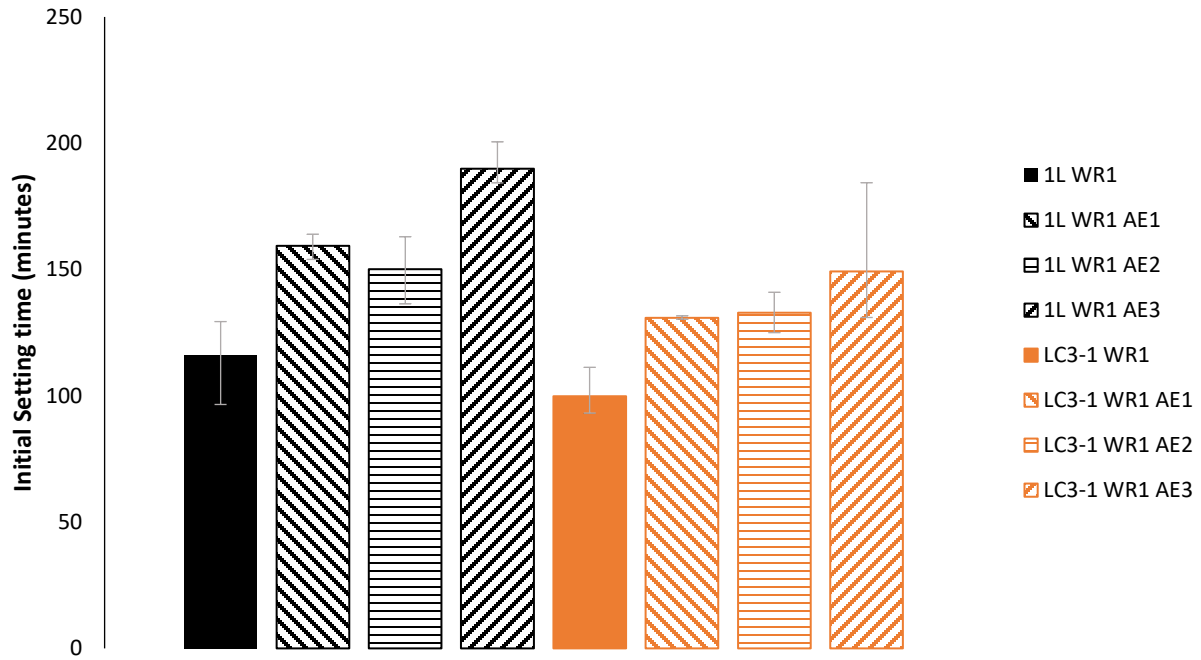
Appendix Figure A.5. 10 and 30 minute Flow of TYPE 1L vs LC³-2 with WR2 and AEs



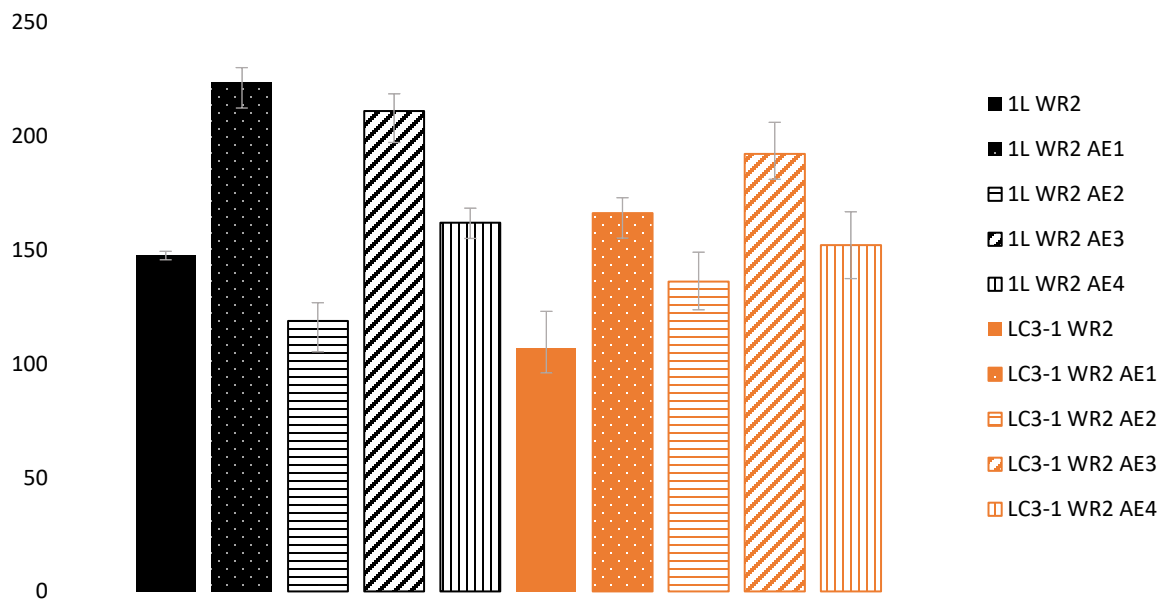
Appendix Figure A.6. 10 and 30 minute Flow of TYPE 1L vs LC³-2 with WR3 and AEs



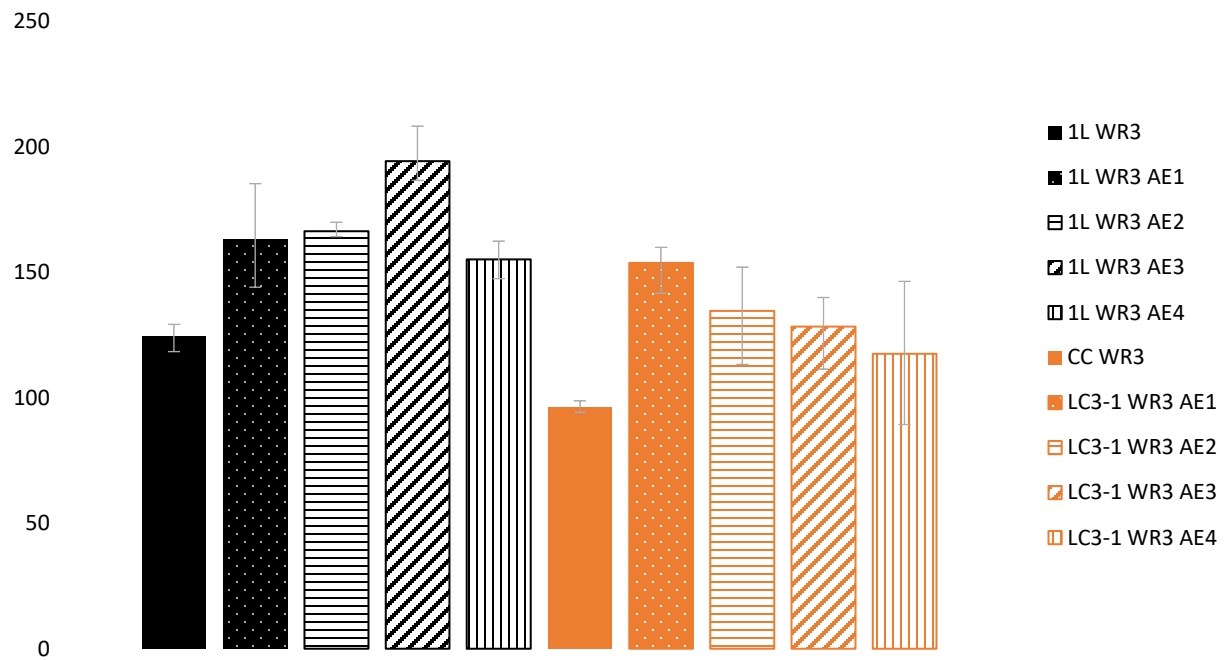
Appendix Figure A.7. Setting time of TYPE 1L vs LC³-1 with WR1 and AEs



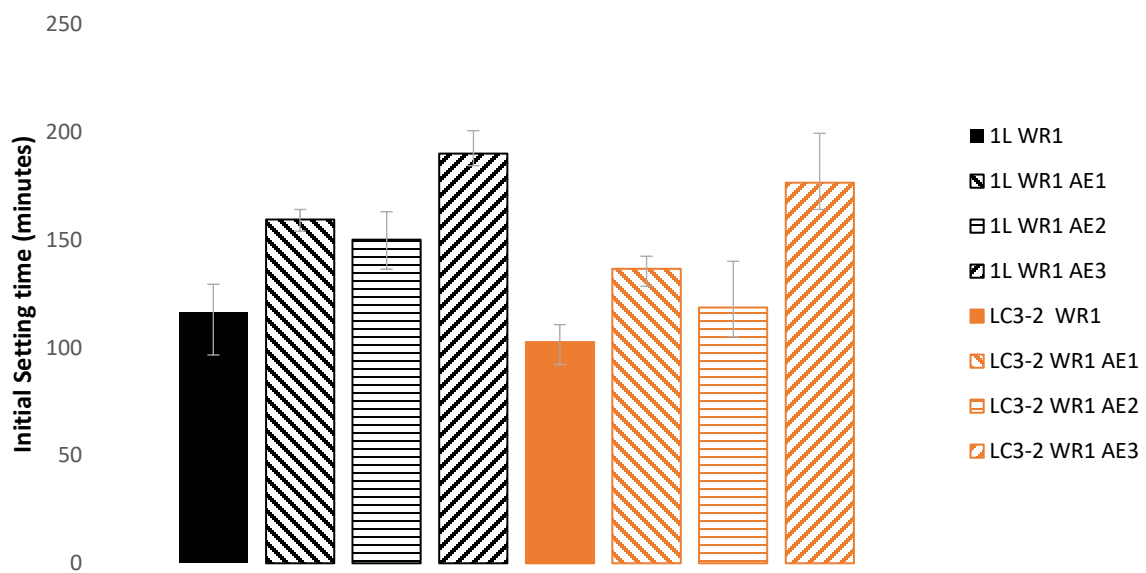
Appendix Figure A.8. Setting time of TYPE 1L vs LC³-1 with WR2 and AEs



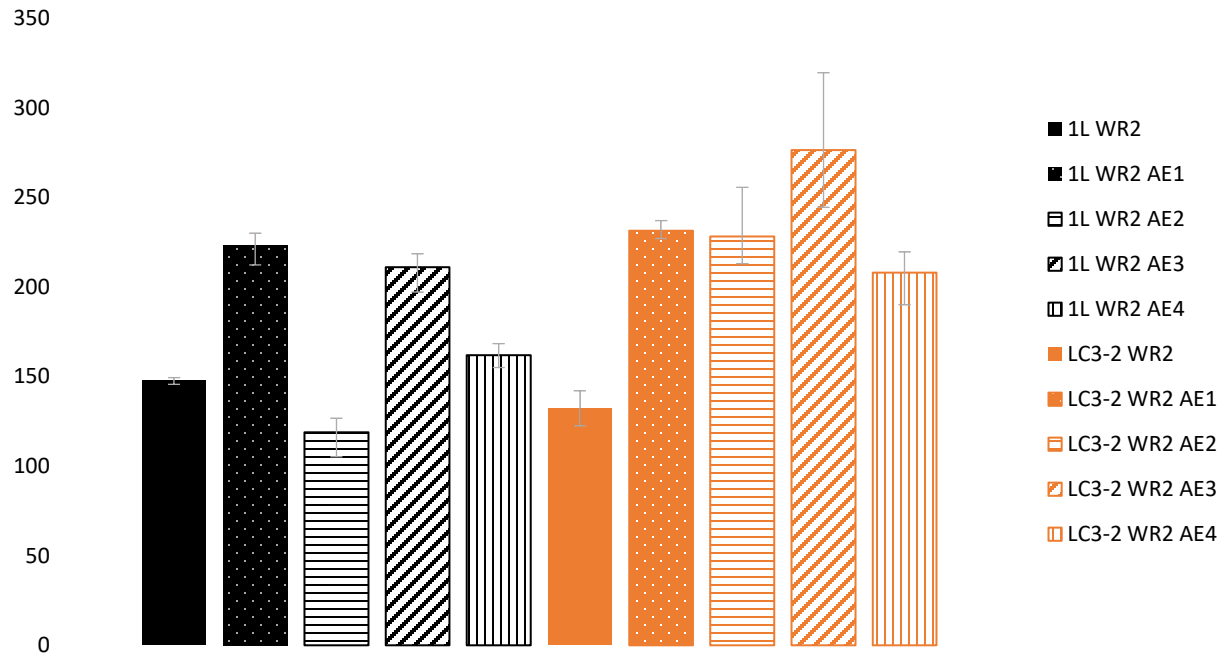
Appendix Figure A.9. Setting time of TYPE 1L vs LC³-1 with WR3 and AEs



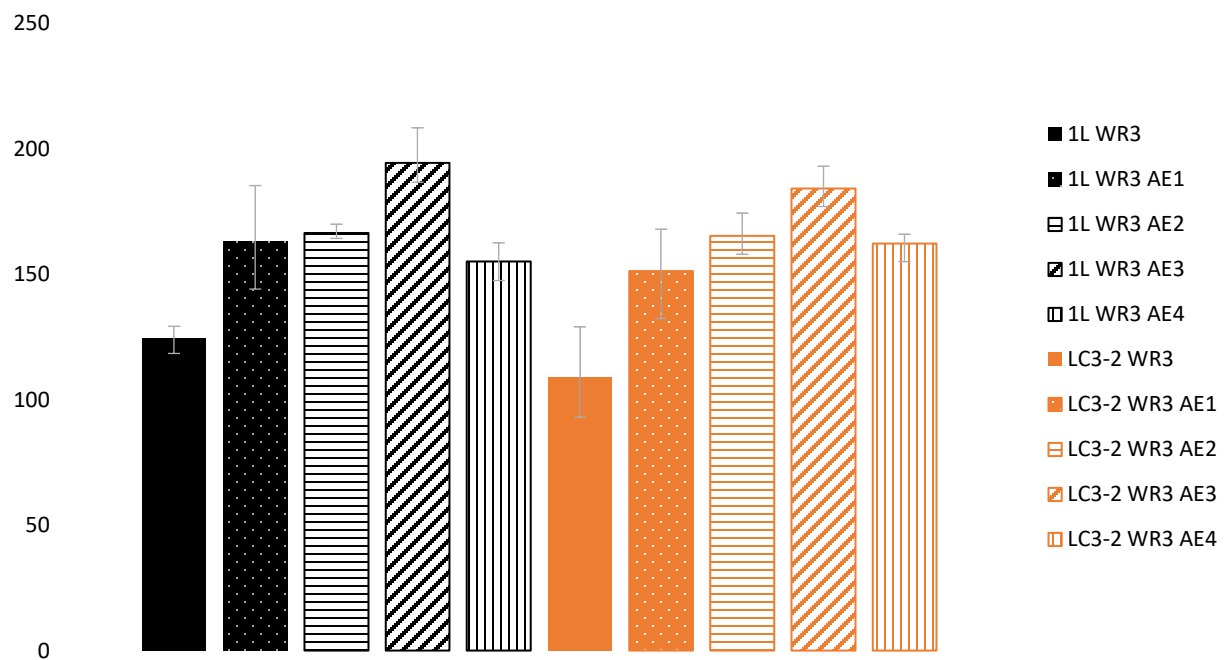
Appendix Figure A.10. Setting time of TYPE 1L vs LC³-2 with WR1 and AEs



Appendix Figure A.11. Setting time of TYPE 1L vs LC³-2 with WR2 and AEs



Appendix Figure A.12. Setting time of TYPE 1L vs LC³-2 with WR3 and AEs



Appendix B - Additional Charts

Figure B.1 Flexural Strength beams breaking within the middle 1/3rd



Appendix Figure B.2. Weight of concrete beams over # of cycles

