Using early age concrete properties to determine setting time

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THESIS SUMMARY

USING EARLY AGE CONCRETE PROPERTIES TO DETERMINE SETTING TIME

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Abstract:

When fresh concrete sets, it changes from a Bingham fluid to a solid, with strength and stiffness. Changes in concrete volume however starts taking place from the moment when water comes into contact with the cement, starting the hydration process. The chemical reaction between the water and the cement cause shrinkage because the resulting product is smaller than the two reactants. The reaction is very slow in the beginning, but when it speeds up it causes an increased temperature that can result in an expansion of the mix. These volumetric changes increase in high strength concrete due to the reduced water/cement ratio, the increased cement content and the use of High Range Water Reducing Agents (HRWRA). It is important to know the exact setting time of concrete as volume changes that takes place after setting, will cause internal stresses, which could exceed the early age strength, resulting in the formation of cracks. Existing setting time test methods do not take into account the effect of changes in water/cement ratio and concrete composition on the setting time. The aim of this study was to determine a setting time for concrete based on the point in time when the concrete starts behaving as a solid. A variety of test methods were used to determine and compare early age properties of concrete. The point in time when a rapid change in behaviour was observed was used as point from where early age shrinkage would have an effect on the stresses or strains that develops in

the concrete. The measured shrinkage results were recorded from as soon as possible after casting but the influence of the shrinkage should be considered from this time onwards.

The results of this investigation confirm that neither the initial nor final setting time typically recorded are representative of the time when the concrete starts behaving as a solid. The time that it takes concrete to change from a viscous liquid to a solid is not a constant, but depends on w/c ratio, specimen size, temperature, inclusion of admixtures and the type of test used. The use of HRWRA retards the temperature increase caused by heat of hydration as well as the initial strength and stiffness development, but this trend is reversed within 24 hours of casting.

A comparison between the setting times calculated from load application (such as penetration testing) and other setting time methods (such as derivatives of heat of hydration measurements) show that HRWRA have an influence on the setting times. The mixes without admixtures gave similar setting times, when the average for the different tests were used whether calculated from load test data or from other setting time test methods. The use of HRWRA resulted in differences of almost 2 hours for w/c ratios below 0.45 between the different types of setting time measurements. This confirms that when load tests are used in mixes containing admixtures, the time when concrete change from a Bingham fluid to a solid can be wrongly estimated. This incorrect assumption of setting time could cause an over estimation of the early age shrinkage that can cause stresses or cracking in high strength concrete.

DECLARATION

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Date: **13 February 2024**

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LIST OF ABBREVIATIONS

American Standard Test Methods	(ASTM)
Autogenous Deformation	(AD)
British Standards	(BS)
California Bearing Ratio	(CBR)
Condensed Silica Fume	(CSF)
Cone Penetrometer Test	(CPT)
Dynamic Cone Penetrometer	(DCP)
Falling Cone Method	(FCM)
Fly-ash	(FA)
Ground Granulated Blast Furnace Slag	(GGBFS)
Heat of Hydration	(HoH)
High Range Water Reducing Admixture	(HRWRA)
Linear Variable Differential Transducer	(LVDT
Modules of Elasticity	(MoE)
Modules of Rupture	(MoR)
Poly Carboxylate Ethers	(PCE)
Radius of Inside Steel	(RIS)
Radius of Outside Concrete	(ROC)
Radius of Outside Steel equals Radius of Inside Concrete	(RIC)
Radius of Outside Steel	(ROS)
Relative Humidity	(RH)
South Africa National Standards	(SANS)

Thermal Coefficient of Expansion	(TCE)
Ultra-Pulse Velocity	(UPV)
Water/Cement	(w/c)
X-ray Fluorescence spectroscopy	(XRF)

1 INTRODUCTION

1.1 BACKGROUND

Significant progress has been made in concrete development since Joseph Aspdin patented Portland hydraulic cement in 1824. As the use of cement changed and developed over the years, the testing of cement and concrete developed as measurement of different properties became necessary. Early advancements include the development of the Fuller and Thompson curves for aggregates in 1907 and the well-known Abram's law for water/cement ratios, developed by Duff Abrams in 1918 (Domone and Illston, 2010). In recent years, computer programs are mostly used to analyse concrete structures with Finite Element Analysis and this topic has been extensively researched. There are also significant recent developments in concrete technology with regard to the placement of concrete on site. Concrete with strengths in excess of 100 MPa is being placed on site and strengths exceeding 200 MPa have been obtained in some laboratories. Some researchers speculate that it is possible to obtain concrete with a strength up to 800 MPa (Richard and Cheyrezy, 1995). All of this should be very exciting for researchers, but how are these strengths measured and what input does the Finite Element Analysis program need to be able to predict the failure of high strength concrete structures? Structural designers use compressive strengths, E-value, Poisson's ratio, and tensile strength (that could be either a split cylinder or a modulus of rupture strength) from the laboratory. Compressive strength is based on strength of specimens tested after 28 days of water curing at a specific temperature. The direct tensile strength is calculated by multiplying the indirect tensile strength with a factor, because the standard test method for measuring direct tensile strength, CRDC 196-92 need special equipment and is difficult to perform (Bamforth et al., 2008, Roziere et al., 2014).

The question remains whether the tests as developed and used over the years for different materials and concrete are valid for high strength concrete? With higher strength concrete, the behaviour of the structures over a long period of time may not be as expected, as these high strengths have not been in use for that long in structures. One of the concerns with the use of high strength concrete is the shrinkage associated with the use of finer materials required to obtain higher concrete strength. Detailed research on one specific shrinkage called "Autogenous" shrinkage has been ongoing since 1990, although the effect has been known from the early 1900s, identified first by Le Chatelier. This type of shrinkage occurs when the chemical reaction that happens in concrete, called hydration, starts to withdraw water from the capillary pores. This is called self-desiccation and causes shrinkage. Autogenous shrinkage causes a volumetric change, but it is measured by various methods as a linear deformation (Jensen and Hansen, 2001). This shrinkage causes concern that only becomes a measurable problem in high strength concrete and especially in concrete with a water/cement (w/c) ratio below 0.42 (Holtz, 2001).

1.2 PROBLEM STATEMENT

Because of the potential increase in the use of high strength concrete, normally achieved by lowering the w/c ratio, it has become essential to measure the properties of the concrete correctly. For designing structures, the properties required are normally the compressive strength, the Modulus of Elasticity (MoE) (E-Value), the tensile strength from split cylinders or the Modulus of Rupture (MoR) and occasionally Poisson ratio, drying shrinkage and creep. These measured values are used in computer programmes to predict the behaviour of complex structures, determine the dimensions required for structural elements, or to accurately calculate stresses and strains in the structure. All these material tests are conducted on hardened concrete, using test specimens cured according to prescribed methods. Concrete properties are mostly measured after 28 days of water curing. The drying shrinkage and creep are however measurements over a period of time, with measurement time after casting and curing differing between test methods and countries. The size of the test specimens also varies (SANS 6085:2006, ASTM C157 2017, RILEM TC 129-MHT 2000), for example, in these three shrinkage test methods, the specimen size for the SANS specification is a 100 x 100 x 300 mm prism (depending on the maximum aggregate size), the ASTM specification is a 70 x 70 x 300 mm prism and the RILEM specification is a 70 mm diameter x 300 mm long specimen. The curing and treatment of these specimens over the testing period also differs between test methods. These differences will cause different results for concrete with similar properties and one has to ask the question, what test is relevant to what really happens in the structure?

To calculate the drying shrinkage, engineers should consider so many variables that it is hard to believe that the calculations will correlate with the real shrinkage. Drying shrinkage is a property measured since the early 1900s and from the beginning researchers looked at factors that influenced the shrinkage of concrete the most. Those factors seem to be the water content, water-cement ratio, type of cement, type of aggregate and the coarse-fine ratio of the aggregate (Zhang et al., 2013). Research on shrinkage includes the effect of the use of materials that are by products of other industries, also called cement extenders, or additions. These include Fly-ash (FA) a byproduct from coal power stations, Condensed Silica Fume (CSF) a byproduct in the manufacturing of non-ferrous metals and Ground Granulated Blast Furnace Slag (GGBFS also called Slagment) that is a byproduct from steel manufacturing, admixtures and recycled aggregates. With the use of admixtures and additions, the ultimate strength of the concrete increased. During all this, the test methods used to determine drying shrinkage stayed the same in most specifications, together with the equations used for calculating the predicted shrinkage in structures.

The increase in the early age shrinkage associated with the higher concrete strengths required new methods to test specimens at earlier ages, even before the concrete can be demoulded. One of the arguments that has not been resolved is when to start the data recording. It was agreed by most researchers that early age shrinkage that can cause cracking, can only occur after the end of the liquid phase of the concrete, when the concrete setting is complete and the concrete starts to act as a solid material. That point is called "initial setting" or as some researchers refer to it, "Time Zero". There is a test method to determine that point in the hydration process in cement pastes. The test is known as the Vicat needle test. The Vicat needle test came from Louis Joseph Vicat developing a test for the hydraulic setting of limes mixed with clay in 1817. In 1877 Henry Reid described the test with a machine to do the test with bigger weight and only in 1904 the test as we know it today was included in BS 12 (Gromicko and Shepard, 2013). The disadvantages of this method are not only that it can only be used for cement setting times, with the inclusion of aggregate distorting the results, but the test was also developed for a constant consistency of the cement or mortar and not to a constant w/c ratio. Therefore, the Vicat needle test cannot be used to determine high strength concrete setting times.

There are various researchers that used different methods to determine the setting time of high strength concrete, but they try to link it to the standard needle penetration test results. In this study, various test methods that can be used on early age concrete material, were compared with the aim of finding a single point where the concrete changes from a liquid to a solid.

1.3 OBJECTIVES

The aim of this study was to determine when the concrete changes from a liquid to a solid, by using and comparing results obtained from various test methods. This aim was met by meeting the following objectives:

- establishing whether one test method can be used to determine setting time of concrete, regardless of paste strength (w/c ratio), by using as many early age property measurements as possible,
- determining whether actual early age shrinkage can be measured from a new defined point called time zero,
- establishing whether results from a test method where only one experimentally determined setting time is obtained can replace the currently used initial and final setting time when evaluating early age shrinkage.

1.4 METHODOLOGY

A literature review was conducted showing the history of testing and to establish the current state of knowledge on factors affecting setting time and early age shrinkage. Various tests were conducted on mixes with different w/c ratios to find the time after casting required for the concrete to change from a liquid to a solid. These tests include the heat of hydration, suction, restrained ring test (similar to the ASTM C1581-18 method), the needle setting time test according to ASTM C403-16, an in-house cone penetration test, compressive strength, an in-house direct tensile test and split cylinder tests. The effect of w/c ratio from 0.25, (typical for high strength concrete) to 0.55 (typical for normal strength concrete), admixtures, sample size and curing temperature on concrete setting time and early age strength development, were studied experimentally. Results from a large variety of concrete tests were compared to find a test method that can be used to measure the setting time, governing early age shrinkage.

All the results were compared with each other and also with the standard test currently in use to find a suitable point in time from where the early age shrinkage can be measured.

1.5 SCOPE OF RESEARCH

Only one type of cement and aggregate was used in the experimental study. Early age concrete properties were studied for the first 48 hours after casting and the variables studied were:

- The effect of w/c ratio and the influence on workability, heat of hydration, setting time and strength gain both with and without admixtures.
- The effect of High Range Water Reducing Admixtures (HRWRA).

- The water content in all the mixes were kept the same at a water content that would be reasonable for a concrete mix with a maximum aggregate size of 5 mm.

The following variables and effects were not studied:

- The effect of cement extenders such as Fly Ash, Silica Fume and Ground Granulated Blast Furnace Slag.
- Long term concrete properties such as compressive strength, split cylinder strength, modulus of elasticity and drying shrinking.
- The effect of different types and sizes of aggregates on setting time and shrinkage.

1.6 ORGANISATION OF THE REPORT

This report consists of the following chapters:

- Chapter 1 gives the background of the research and the order in which the work was planned.

- Chapter 2 is the literature review on the current knowledge in the field of the research

- Chapter 3 provides the test methods used to conduct the research, and where the method varies from the specified method the new method with explanations on why it was opted for is also given.

- Chapter 4 presents the results of all the tests with discussions on the results.

- Chapter 5 provides the comparison between the results of the different tests to determine if there is a comparison between the various tests showing a similar behaviour at the same point in time.

- Chapter 6 provides the conclusions and the recommendations for further study.

- List of references.

2 LITERATURE REVIEW

2.1 INTRODUCTION

The literature review focused on the history of tests developed over the last two centuries to measure the properties required for the use of concrete in new applications. It shows how concrete developed from a material that was only used as a mortar with rocks and later bricks to a point now where the strength of concrete becomes the same as that of a weak metal. This changes the properties of the concrete during mixing, casting, setting and strength gain. High strength concrete mixes require additional materials, to not only get to these high strengths, but also assist with managing the brittle failure. This study however only concentrates on tests and properties of concrete in the first 48 hours after casting.

2.2 HISTORY OF CONCRETE

2.2.1 History of concrete test methods

One of the first tests used in concrete was developed by Louis J. Vicat in 1817 as shown in Figure 2-1. It is called the Vicat setting time tests for Roman cements, but it was only included in the British Standards (BS) specifications in 1904 by using an apparatus that still looked like the original (Bye, 2015; Currently BS EN 196-3 2016; ASTM C191 – 21; SANS 50196-2006). Strength tests were developed in 1836 and these included a compressive and a tensile strength test. It was only prescribed for use in contracts in the 1850's by Henry Reid. The first official standards committee was that for the British Standard (BS) that was established on the 22nd of January 1901. America followed with American Standard Test Methods (ASTM) on the 17th of January 1905. From the 1920's most counties having ties with the UK followed by adopting the BS although they have their own numbering systems (Gromicko and Shepard, 2017).

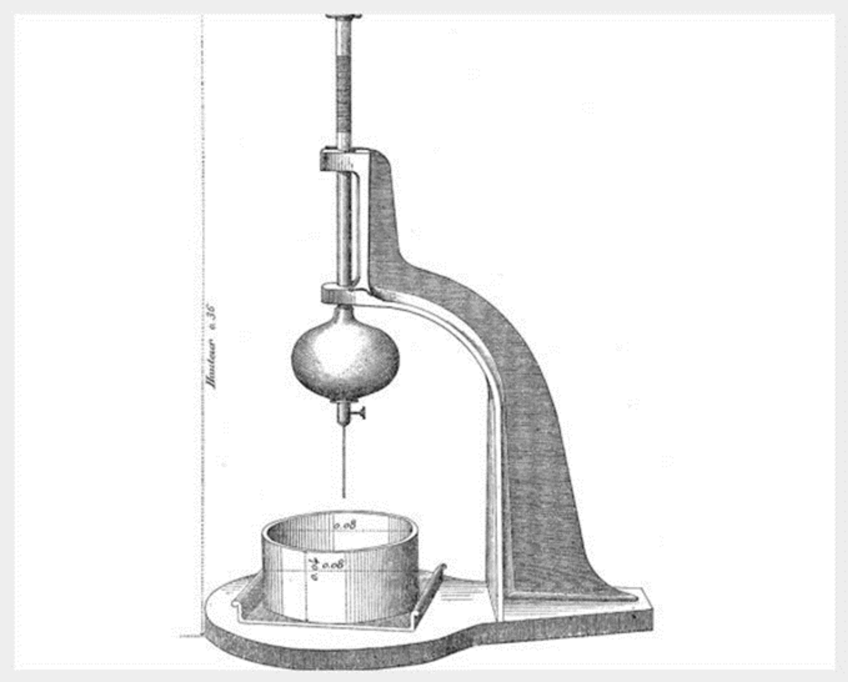


Figure 2-1: The first Vicat apparatus from JL Vicat in 1817 (VICAT, 2017)

Although Portland Cement (PC) as we know it today, was patented in 1824 by Joseph Aspdin. The manufacturing was changed from vertical stationary kilns to rotating horizontal kilns with a slight slope. The specification of setting time for cement was followed by the strength specification covered by a standard. The start was slow for concrete structures and although reinforced concrete was patented in 1867, structural concrete was only used from about the turn of the century (1900), and it was then mostly used in industrial buildings (Potter, 1894).

In 1904 the first high rise building with 16 floors was built in Cincinnati in America, followed by a bridge spanning about 100 meters in Rome (Italy) in 1911. This was followed by ready-mix concrete in 1913. In 1917 the National Bureau of Standards and the ASTM set a standard for the chemical composition of Portland cement. The patent for pre-stress concrete was awarded to Eugène Freyssinet, a French engineer, in 1928. In 1930 the discovery was made that entraining air into concrete used for roads in very cold areas, improved the durability of the concrete. This principle of concrete casting with the inclusion of air to improve the durability of pavements is still in use in cold countries where de-icing salts are used to remove the snow (Gromicko and Shepard, 2015).

2.2.2 Heat of hydration

Two dams, namely the Hoover dam and the Grand Coulee dam were built around 1935-1942. Before the building of these dams commenced, research was carried out to reduce the heat of hydration. The aim was to prevent the dams from cracking to pieces by heat expansion and contraction. To help with the heat of hydration reduction three things were done. Firstly, the use of the newly developed low heat cement was prescribed, secondly the maximum aggregate size was increased to 250 mm (10 inches) causing a reduction in overall cement content and then lastly the dam wall was filled with a maze of cooling pipes through which chilled water was constantly pumped to reduce the heat in the mass concrete (Riding, 2007).

2.2.3 High strength concrete

In 1970 the development of steel fibre reinforcing was introduced and all other types of fibres followed. Then in the 1980's superplasticizers were developed, with the potential to reduce the water content, resulting in higher concrete strengths. This led to taller concrete structures and in 1992 a 305-meter-high reinforced concrete building was built in Chicago, Illinois called by its street name 311 South Wacker Drive. The Petronas twin towers were finished in 1998 and this was the tallest building at 351.8 meters until 2004. It was also a landmark regarding the use of concrete strengths above 80 MPa. To achieve that Condensed Silica Fume (CSF) was used in the concrete mix design (Kribanandan, 1995).

During the last 20 years numerous researchers published work aimed at reducing the environmental impact of concrete by not only increasing the use of cement additions such as FA and CSF, but also reducing the volume of material required for the construction of infrastructure by increasing the strength of concrete (Kearsley and Mostert, 2009). This is possible with reduced water/cement ratios and increased use of superplasticizers (Brooks, 2000; Orosz, 2017).

2.2.4 Design codes

Design codes of practice do not contain information on suitable material properties and behaviour of high strength concrete (Compressive strengths > 120 MPa). Current Eurocode 2 (EN1992, 2014) only made provision for characteristic cube strengths up to105 MPa (Cylinder strengths of 90 MPa). Research is required to determine whether existing design codes can be used to design high strength concrete structures. Determining the hardened properties of high strength concrete is not a problem, but care must be taken to make sure the testing equipment is big and strong enough to test the specimen to failure. What does become a problem is that for strengths above about 80 MPa, the failure of the specimen is sudden and can damage equipment with the sudden release of energy at failure. The standard tests that are normally conducted include the compressive, and indirect tensile strengths like the split cylinder or the Modules of Rupture (MoR) on a beam (SANS 6253:2006).

2.3 WATER/CEMENT RATIOS AND ADMIXTURES USED IN CONCRETE

In concrete with strengths from 25 MPa to 45 MPa, which in the past were the typical strengths used in the day-to-day structural building and bridge designs, no special materials or admixtures are required to get good workable and place-able concrete. So, when structures with long spans and deep sections were required, no serious measures were taken to lower the heat of hydration during the first couple of days after casting. There were no problematic expansions caused by heat of hydration or early age shrinkage which took place in any well-designed concrete structure. This can however not be guaranteed with high strength concrete. For concrete with a compressive strength above 105 MPa, the cementitious content could be more than 500 kg and very low w/c ratios would be required (Neville & Brooks, 2010).

Shrinkage of concrete starts as soon as the placement of the concrete is done and the compaction process stopped. Researchers started labelling the different shrinkage processes by giving names to the process. Chemical shrinkage can be measured from directly after casting. This is the shrinkage caused by the chemical reaction between water and cement where the reaction product volume is smaller than that of the two original products. Chemical shrinkage in the concrete, however starts as soon as the water makes contact with the cement (Kheir et al., 2021). This theoretically means that the volume of concrete cast is already less than the volume that started in the mixer. It is possible to calculate the difference in volume between the original water and cement and the final products and that is found to be about 10%. That will only be if all the cement hydrated and it is only 10 % of the cement content not the concrete volume in total (Tazawa et al., 1995). Chemical shrinkage will take place as long as there is water available for the reaction to take place, and that could be as long as the concrete exists. Properties of the materials that are used that can influence this type of shrinkage include type and amount of cement, water/cement (w/c) ratio, the amount of water, aggregate type that can have an influence on the heat of hydration, ambient and curing temperature, shape and size of the concrete structure and the ratio of all different materials that form the concrete matrix (Chen et al., 2013; Eppers, 2011; Huang and Ye, 2017). Chemical shrinkage is measured by either the buoyancy method or the density method (ASTM C1608, 2017). Both methods are questionable because too many things can influence the result, such as material type or the volume of the material used in the test (Zang et al., 2013).

Plastic settlement shrinkage takes place but this is not a big concern because the concrete is still in a semi liquid form known as a Bingham fluid. A Bingham fluid is a material that has a yield strength and a stress needs to be applied before it moves and becomes a viscous material. Good site practise is to re-vibrate the placed concrete after the plastic settlement took place. Together with the plastic shrinkage right after casting, the chemical shrinkage (Holtz, 2001) also starts. Plastic shrinkage is caused by the settlement of the heavier material to the bottom of the cast and movement of the lighter material to the top. It depends on the amount of water used in the mix, the w/c ratio of the mix, and the grading of the non-cementitious material that are used (Brits, 2021). When some of the cement is replaced with cement addition materials, also known as mineral admixtures, the fines and the shape of these materials as well as the water absorption and adsorption of all the different materials used in the mix play a role in the magnitude and duration of plastic settlement shrinkage (Brits 2021, Nehdi and Soliman, 2011 Soliman, 2011). There is no standard test method to measure plastic settlement shrinkage but ASTM C1579-21 gives a test method to measure and compare early age shrinkage cracks between different mixes of concrete. No shrinkage value is however measured.

It is known that at w/c ratios below about 0.38 to 0.42, early age shrinkage becomes a problem (Holtz, 2001; Eppers 2010). The early age shrinkage that researchers talk about is the normal chemical shrinkage that happens in all concrete from the moment water is added to the cement, but at the w/c ratios below 0.42 another shrinkage also starts to have an influence on the total early age shrinkage and that is autogenous shrinkage. If the magnitude of these two types of shrinkages is added to drying shrinkage, then this can cause problems in structures. However, the biggest concern lies in the early age shrinkage results published to date.

To compare research results is almost impossible. Figure 2-2 contains the results from a round robin test by 10 different institutions, conducting 30 tests, using a "Dilation Rig" in a horizontal position to obtain the same result (Bjøntegaard et al., 1995). The institutes participating all used the same identical mix. From these results it is clear that significant work on the test method for early age shrinkage is still required. During these tests the laboratories all seem to use the same starting time after casting as time-zero. In this case it was 8 hours as can be seen in Figure 2-2. In the research paper they published there was no indication what motivated this chosen time-zero. The true shrinkage readings should be taken from a fixed determined point in time used by all researchers to compare concrete properties all over the world and most probably this time should be called time-zero.

Early age cracking of concrete can only be prevented if the early age tensile strengths of the concrete is known. The strength must resist the stresses caused by the early shrinkage and thermal deformation of high strength concrete. Although there is a standard test method for autogenous shrinkage of paste and concrete with fine aggregates (ASTM C1698-19 and Bjøntegaard et al., 2006), there is ongoing research all over the world to develop better test methods for use on complete concrete mixes including coarse aggregate. Significant recent work took place to develop a test method or device to determine early age strength and stiffness (E-value) of the concrete for use in shrinkage crack prediction models (Delsaute, et al 2016; Holz, 2001).

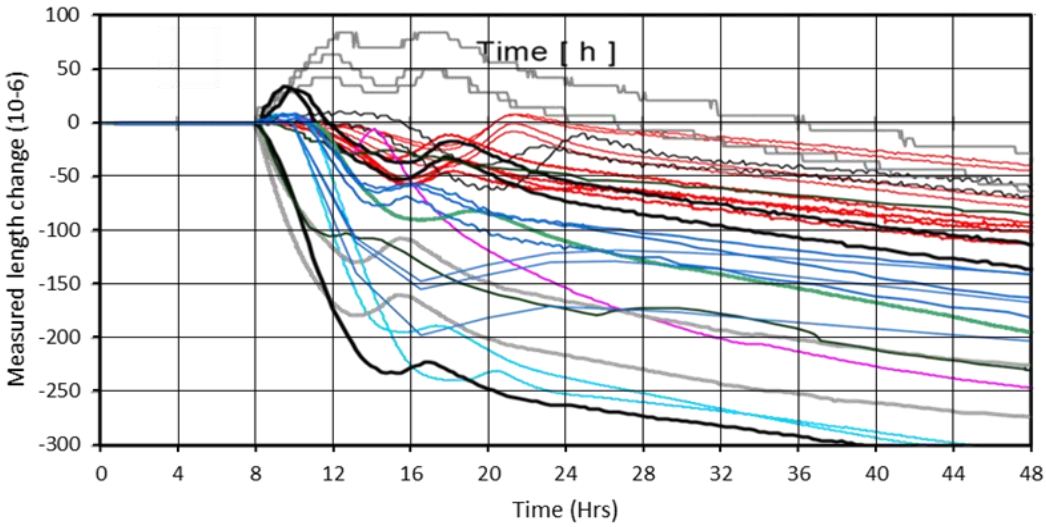


Figure 2-2: Results of a round robin tests (Bjøntegaard et al., 2006)

In this literature review factors affecting early-age concrete properties were discussed before focusing on test methods that can be used to determine when wet concrete turns into hardened concrete, thus being able to develop internal stresses, caused by volume change that can result in crack formation.

2.4 EARLY AGE PROPERTIES OF CONCRETE

Early age properties are those properties of the concrete changing while the concrete goes through phases changing from a liquid to become a solid. The factors that influence the tempo at which the process takes place include the following:

2.4.1 Workability

Workability of concrete is defined by ASTM C125-14 as the ease fresh concrete can be placed and worked of without losing homogeneity. Cement type and content, w/c ratio, use of admixtures, aggregate shape and size of aggregate affect workability (Demone and Soutsos, 2018).

2.4.2 Heat of hydration

Heat of hydration in concrete is caused by the chemical reaction that takes place when cement and water get in contact with each other. The temperature can be measured using various methods but the semi-adiabatic method is used to determine setting times. In Figure 2-3 a heat of hydration graph is shown (Prasad, 2021). On the graph the 5 stages that is normally used to described the temperature that occur from casting till a time where the temperature stabilises. On the graph two setting times are also indicated, determined by using the heat of hydration results.

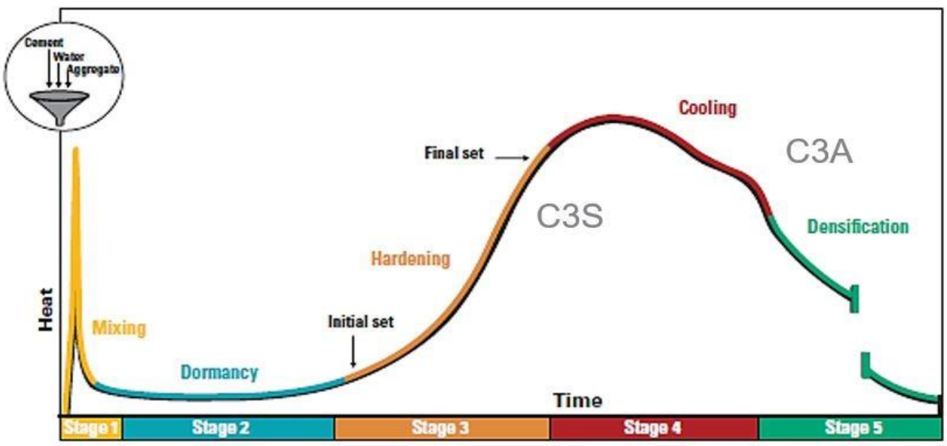


Figure 2-3: Heat of hydration curve showing the five stages during hydration (Prasad, 2021)

2.5 EARLY AGE SHRINKAGE

There are numerous methods used by researchers to measure early age shrinkage, with almost as many different outcomes in the results. The three types of shrinkage that are named to have an influence in wet concrete before set is Plastic settlement, Chemical and Autogenous shrinkage. For plastic settlement there is no standard test method, and for Chemical and Autogenous shrinkage, each one has its own different ASTM test method, but research by Sant et al. (2006), showed that these two different shrinkages are the same in magnitude till time zero. Each one of these shrinkages has its own conditions that influence the particular shrinkage. Each type of early age shrinkage with problems in measuring it, is explained separately.

2.5.1 Thermal deformation

Thermal deformation is one of the most difficult deformations to measure correctly even in hardened concrete. In fresh concrete before final setting, this is even more difficult. Before setting, concrete is a Bingham fluid and any deformations can take place without physical damage to the structure in the concrete. There are no recognised tests methods that can be used on the liquid material to test the influence of temperature alone. There are researchers that used the ASTM C1698 − 19 corrugated pipe test that was developed by Jensen for measuring autogenous shrinkage (Jensen and Hansen, 1995). They also measured the temperature development during the test. The test is then altered by applying a negative temperature to the test specimen to keep the specimen at a constant temperature (say 25ºC). By then deducting the two shrinkage graphs from each other, the influence of the difference in temperature on the concrete can be established. The problem with this method is, that by keeping the temperature constant, the setting time is now later. The measurement of early age shrinkage should start at setting time (time-zero), so strictly speaking these two measurements cannot be linked.

In Figure 2-4 the influence of temperature on the density of water is shown (Xu et al., 2009). What is also shown is that it is not a linear function with the highest density at 4ºC. If this fact is taken into consideration the test method where the temperature of the test specimen for early age shrinkage is kept constant the influence of the much higher water expansion is eliminated. The problem however is still that the influence of the change in measured setting time on the concrete specimen performance.

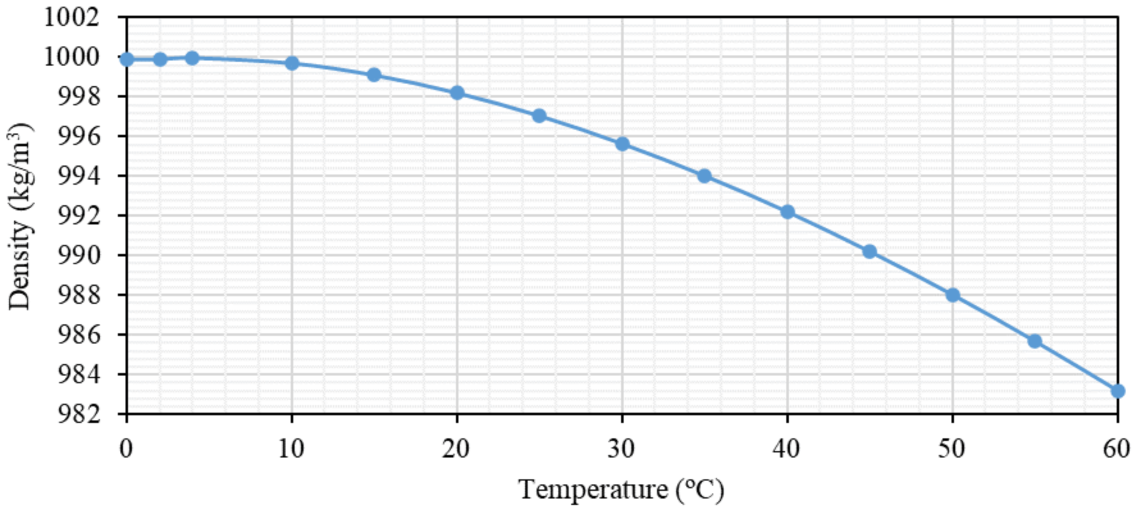


Figure 2-4: Density of water at different temperatures

From the graph in Figure 2-5 it can be seen that the Thermal Coefficient of Expansion (TCE) for water ranges from 1 x 10^{-4}/ºC to 5.5 x 10^{-4}/ºC (Thakur 2019), where-as the TCE for most natural aggregates used in concrete as shown in Table 2-1 (Alexander, 2021), is a linear coefficient and varies between 6 x 10^{-6}/ºC and 12.2 x 10^{-6}/ºC. To explain the influence of the water a bit more we should look at the volume of water in most concrete mixes that varies between 15% and 25% of the volume of the mix. If the mixing temperature of all material was kept at 25ºC and a specimen was allowed to hydrate freely, the temperature of concrete with a w/c ratio below 0.4 should rise by at least 10ºC, resulting in the expansion coefficient of water changing from 2.57 x 10^{-4}/ºC to 3.50 x 10^{-4}/ºC. In contrast, for a mix that is kept at a constant temperature the water expansion has no influence. This explanation indicates that the thermal deformation of wet concrete cannot be estimated using a method where the temperature of the concrete is kept constant.

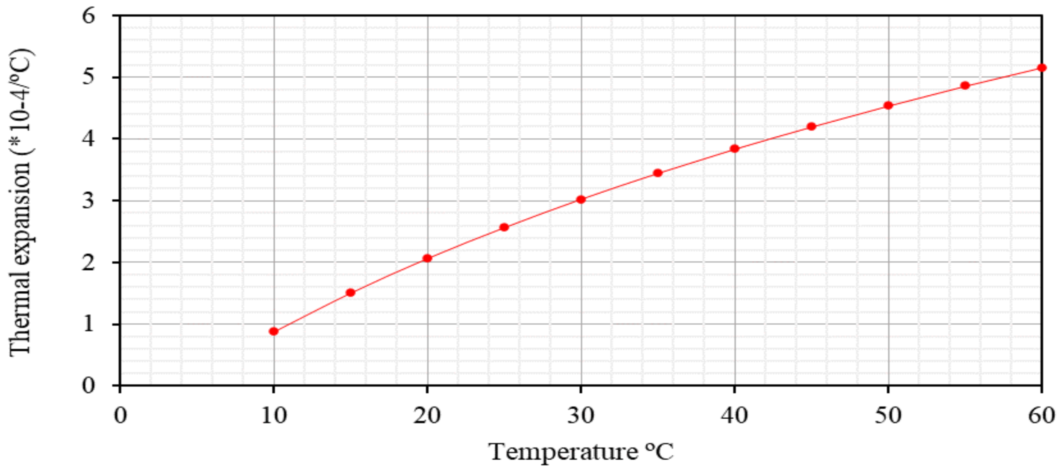


Figure 2-5: Thermal expansion coefficient of water (Zhutovsky,2015)

The values for TCE of aggregates shown in Table 2-1 is for the aggregates widely used in South Africa (Alexander, 2021) and differ from results for some aggregates shown by researchers from other countries (Karagüler and Yatağan, 2018).

Table 2-1: Thermal coefficient of expansion of natural aggregates in South Africa.

Temperature	TCE Aggregates
°C	x 10^{-6}/°C
Andesite	7.4
Dolerite	6.0-8.1
Dolomite	7.5-9.2
Felsite	9.2
Granite	6.4-9.7
Greywacke	10.9
Quartzite	9.4-12.2
Tillite	6.6

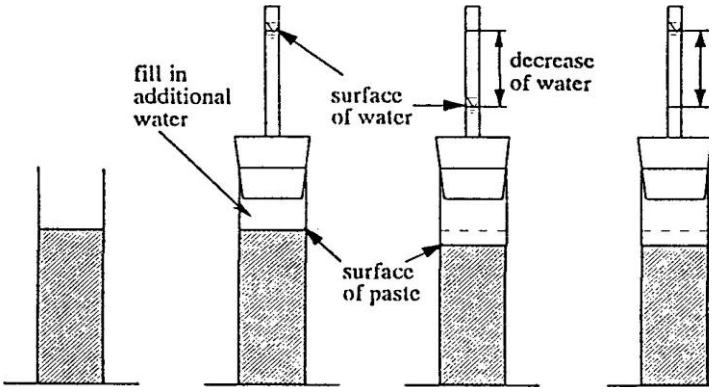
From the data in Table 2-1 it is clear that the type of aggregate has an influence on thermal deformation of the concrete, but the amount of water and the temperature during the heat of hydration will have a bigger role to play in early age concrete before final set. The lack of knowledge on thermal deformation is most probably still a big concern in the casting of big concrete pours, especially where there are different thicknesses involved in the same structural element such as T-beam bridge deck or coffer slab designs.

2.5.2 Plastic settlement shrinkage

In work done by Tian and Jensen in 2008 they showed that material grading, w/c ratios and even the direction (horizontal or vertical) of measurement play a role in the amount of deformation measured before setting. They did conclude when the concrete has set, the measurement direction does not have an influence furthermore. For the tests they used the corrugated pipe method for both horizontal and vertical measurements and in both cases the test specimens were completely covered in water at a constant temperature. The setting time of final set, using ASTM C403 test method from where autogenous shrinkage readings were used. In this work they showed the measured shrinkage in all different conditions and even for corrugated pipes made from different materials and stiffnesses. They also showed that the autogenous shrinkage measured after final set under all conditions were similar but the part before set, that included plastic settlement, was not comparable at all. When the mix has a lot of bleed water the horizontal and vertical test results were non-comparable.

2.5.3 Chemical shrinkage

Chemical shrinkage is the reduction in volume caused by the reaction between the water and the C_3S in the cement. The product of the hydration process is smaller than the two components before the reaction (Tazawa et al, 1995). In their research they also showed that the specimen for which the chemical shrinkage is being measured, should be completely submerged in water. They further showed in a calculation that the shrinkage is about 11% of the C_3S content of the cement. Their calculations (Tazawa et al, 1995) also indicated that the old Le Chatelier method, (rubber bag method) only gave accurate readings until the bleed water was adsorbed and thereafter the measurement actually was the autogenous shrinkage. The preferred method is shown in Figure 2-6 (ASTM C1608-17). The method consists of a glass tube that is filled with the cementitious paste or concrete, whereafter the tube is then fully filled with water. A plug that seals the tube is placed on top with a smaller tube going through the plug. Water is then placed in the small tube to a certain level close to the top of the tube. A moist sponge is placed in the mouth of the tube to prevent evaporation. The decrease of the water in the tube is measured at certain time intervals. The tube can be refilled for continuous measurement over langer periods or till no or very little decrease is measured over the planned period of measurement.



addition of water for continuous measuring.

Figure 2-6: Measuring chemical shrinkage of cement paste (Tazawa et al. 1994)

Another method to measure chemical shrinkage is the buoyancy method as shown in Figure 2-7 as performed by Sant et al. in 2006. The method makes use of a flat open container and is partially filled with the concrete sample and then filled with water. The sample is attached to the underside of a scale and place in a bigger container filled with paraffin oil. This container is then placed in a bigger container filled with water and kept at a constant temperature.

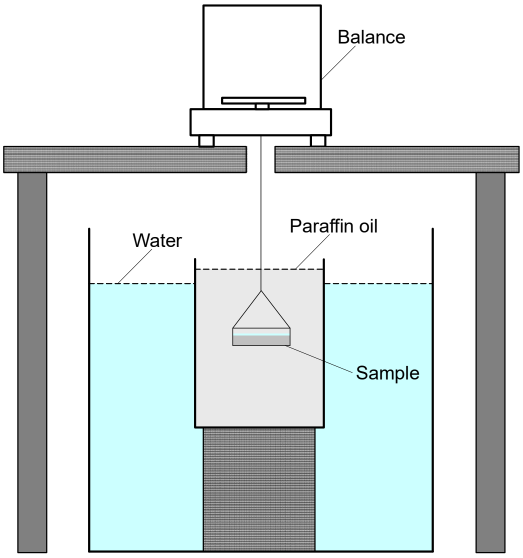


Figure 2-7: Measuring chemical shrinkage with the buoyancy method (Sant et al. 2006)

In Figure 2-8 (Chen et al, 2013), the difference in the measurement of chemical shrinkage is shown when the thickness of the cement paste is altered to allow more water to enter the paste per volume while the chemical shrinkage is measured. This test method allows the shrinkage to be measured directly after casting, and the way it is set up (Figure 2-8) plastic settlement should not have an effect on the reading, because the test is done under water. The readings can be taken manually by reading it off a calibrated scale and writing it down, by a camera set to take pictures at fixed time intervals, or by Laser deformation measurement and continuous logging. Chen et al. (2013), made use of the buoyance method shown in Figure 2-7 and by changing the thickness of the concrete in the container that was covered with water, they produced the results in Figure 2-8 showing that the thinner the layer the more it shrinks. This shows that the volume change method measuring chemical shrinkage can result in inaccurate answers because it also measures plastic settlement.

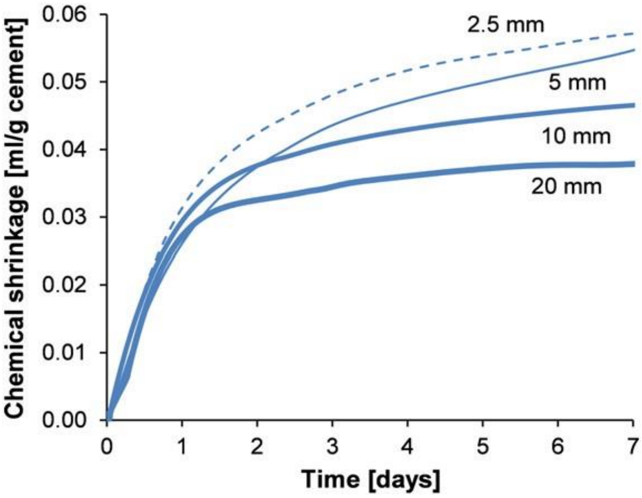


Figure 2-8: Measuring chemical shrinkage of different cement paste thicknesses (Chen et al. 2013)

2.5.4 Autogenous Deformation

Autogenous Deformation (AD) cannot take place if chemical shrinkage stops and as long as there is water available chemical shrinkage will continue. The definition of AD is the isothermal macroscopic volume change during hydration of cementitious materials after initial setting, in a sealed system (Zhutovsky and Kovler, 2015). The inaccuracy of determining the AD is because researchers do not have a test method to accurately determine initial setting time and to do the test at a constant temperature is not representative of any structure and should thus rather be called an extension of chemical shrinkage when the free water in the mix is used up by the hydration process. It is then that the C_3S in the cement starts using the free water from the capillary pores. As the water is taken from the pores with a smaller diameter the suction in the pores becomes higher causing increased shrinkage (also called self-desiccation). The mechanism is not yet 100% understood (Lura et al, 2003) but the relative humidity in the concrete plays a significant role in what happens during early age deformation. There are researchers that believe that a drop in pore humidity indicates the correct moment to start measuring AD. Huang and Ye (2017) showed in various ways that the final set time results obtained with the Vicat apparatus are far from the “knee-point” that they recorded for a drop in pore humidity inside the concrete. They also confirmed that the Ultra-Pulse Velocity (UPV) test results, when the change in the rate of the pulse velocity is used to indicate time-zero, are close to the Relative Humidity (RH) time-zero (Huagh and Ye, 2017).

Measurable autogenous deformation that is causing concern, happens only in high-strength concretes, in other words, concrete with a low water/cement ratio. In most of the research, the w/c ratio mentioned is between 0.38 and 0.42 when autogenous deformation starts having an influence. Holtz (2001) in her research used the number 0.42. Her research mainly aimed to show the influence of the water/cement ratio on shrinkage. The influence of both cement replacement materials and admixtures on shrinkage was also investigated. Not much research was done to show the influence of aggregate types, possibly because the influence of that is well documented for “normal” strength concrete. One of the problems is that most research on high-strength concrete development is done with fine materials and very little of the autogenous shrinkage research is done on concrete that also contains coarse aggregate. Most early age shrinkage tests are developed for cement pastes and mortars. The second problem is that not all researchers start the measurement of the autogenous shrinkage at the same time after mixing. The third concern for accurate reporting is the type of specimen and the direction of measuring the AD. Tian and Jensen (2008) looked at one test type (Figure 2-9, ASTM - C1698) and changed the material and size of the mould and then they changed the test from the horizontal test in the specification to a vertical test. None of the results were comparable, which should be of concern too.

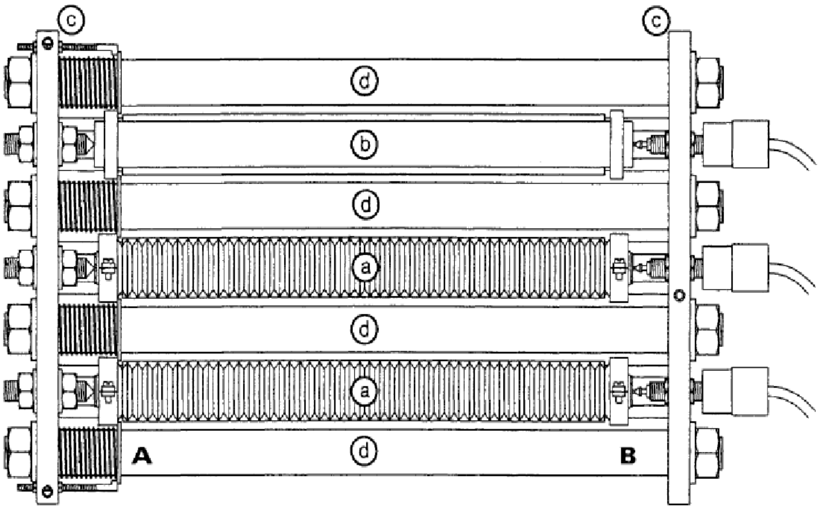


Figure 2-9: The dilatometer frame developed by Jensen and Hansen (1995)

In a paper by Bouasker et al. (2018) on improved methods to measure autogenous shrinkage it was mentioned that a vertical method that also used a corrugated pipe is under development.

In Figure 2-10, Sant et al. (2006) show the difference in Autogenous shrinkage of a w/c ratio mix of 0.3, when measured using two different methods (ASTM C157 and ASTM C1698). The ASTM C157 shrinkage is a concrete prism that is cast in a mould and measurements can only start the next day after the specimen is removed from the mould thus readings were started after 24 hours. The graph produced from this measurement were then moved to a corresponding time on the graph produced by the corrugated tube method as described in ASTM C1698. The same deformation is assumed for the ASTM C157 showing that the shrinkage is almost identical after 24 hours.

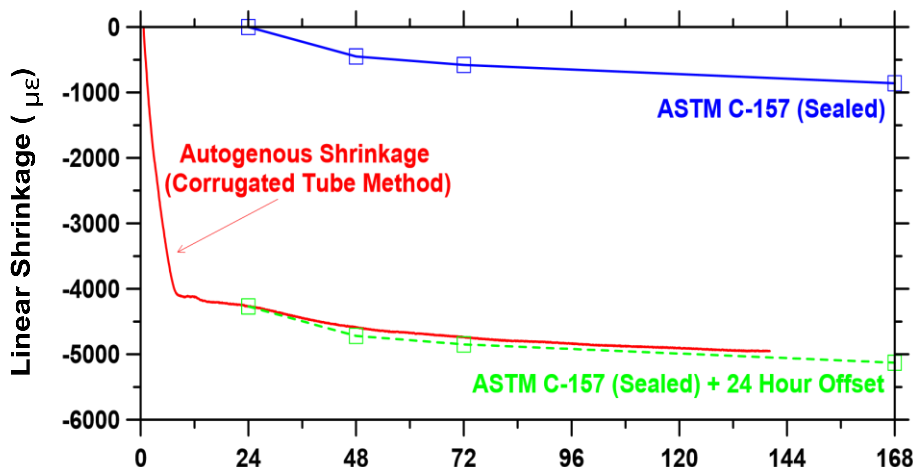


Figure 2-10: Measuring of Autogenous shrinkage using two methods (Sant et al. 2006)

Another method of measuring Autogenous shrinkage is also a buoyancy method where the paste is placed in a balloon and the weight change over time is measured. Figure 2-11 shows the experimental set-up of Sant et al. (2006). This setup is very similar to the chemical test method but the concrete is placed in a sealed balloon and then placed in paraffin to prevent water leakage through the rubber balloon.

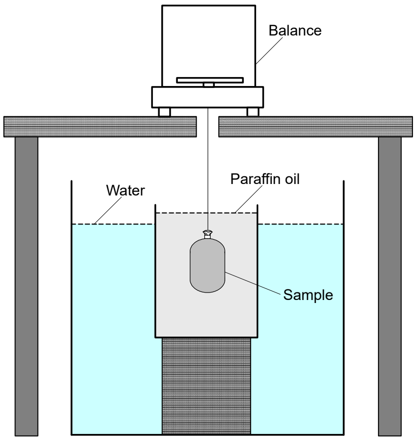


Figure 2-11: Measuring autogenous shrinkage with the membrane method (Sant et al., 2006)

In Figure 2-12 the results from the two buoyancy methods measuring chemical and autogenous shrinkage can be seen from casting. These measurements with the buoyancy method results in volume measurement and to converted to linear shrinkage, the volume change should be divided by 3 according to the paper by Sant et al. (2006). The result shows that the two shrinkages are almost identical which is the shrinkage representation of the concrete still in a Bingham liquid form. From where the lines diverge and the chemical shrinkage increases and the autogenous shrinkage stays almost constant. This result shows that chemical shrinkage and not autogenous shrinkage causes the problem with early age shrinkage. This result shows that autogenous shrinkage is not the main problem for low w/c ratio concrete at early-age shrinkage.

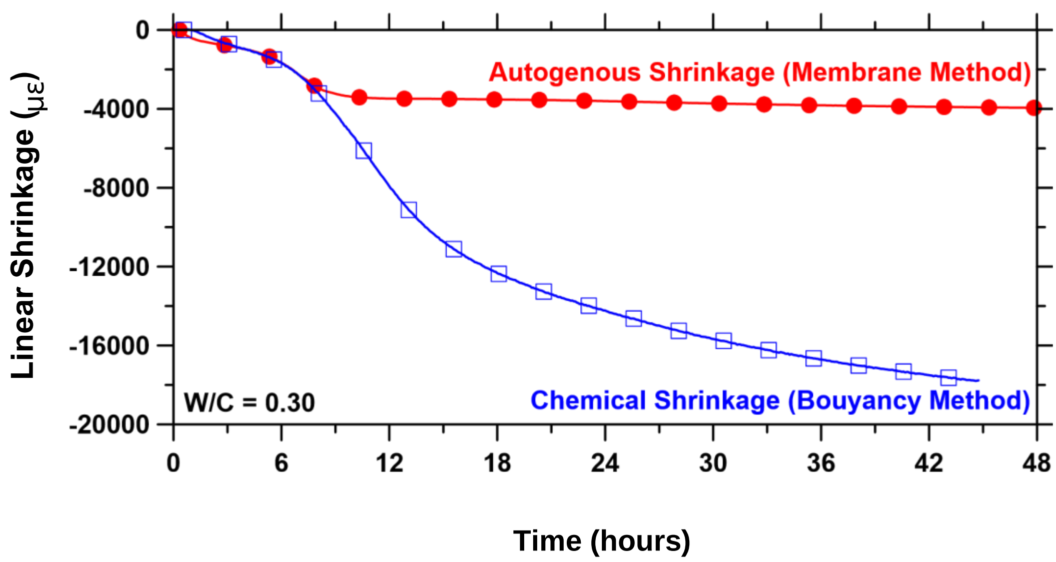


Figure 2-12: Comparing chemical and autogenous shrinkage from start (adopted from Sant et al., 2006)

Table 2-2 shows the results of where the different researchers start measuring autogenous deformation. The table only shows results up to 2017. In the period after, researchers started concentrating on other test methods to get a single point from which AD can be measured. The disadvantage of this research is that it is still compared to either the initial or final setting time achieved by using either ASTM C191-16 or ASTM C403-16 (Huang and Ye, 2017; Filho, 2019).

Further, the research started to concentrate on prediction of the early-age shrinkage using computer models and early-age properties like concrete stiffness, poison ratio, capillary tension, creep, and humidity level in concrete (Hu et al., 2019; Lu et al., 2023) The disadvantage of this research is that the ASTM C191-16 method is still used to compare their measured with their predicted models.

The use of Super Absorbent Polymers (SAP) and restrained concrete with the inclusion of steel fibers and reinforcing and sand became highly studied variables and all have an influence on the setting times or the measuring of thereof (Kawashima and Shah, 2011; Filho et al., 2019; Lu et al., 2021).

Table 2-2: Researchers and type of setting time used to start measuring AD

Author Names	Year	Time assumed as Time Zero	Paper title
Jensen O and Hansen P	1995	1 hour	A dilatometer for measuring autogenous deformation
Yang Y, Sato R, Kawai K.	2001	Initial set	Evaluation of Autogenous and Drying shrinkage based on bound water content of cementitious materials.
Bjøntegaard Ø, and Sellevold EJ	2001	11 hours	Interaction between thermal dilation and autogenous deformation in HPC
Lura P	2003	Setting linked to internal stress	Autogenous Deformation and Internal Curing of Concrete
Barceloa L, Moranvilleb M, Clavauda B	2005	9 hours after mix-ing	Autogenous shrinkage of concrete: a balance between autogenous swelling and self-desiccation
Bjøntegaard Ø and Ham-mer TA	2006	8 hours after mix-ing	RILEM TC 195-DTD: Motive and tech-nical content
Lura P and Jensen OM	2007	20 minutes after mixing	Measuring techniques for autogenous strain of cement paste.
Viviani M, Glisic B, Smith IFC	2007	Final set	Separation of thermal and autogenous deformation at varying temperatures us-ing optical fiber sensors
Tian Q and Jensen OM	2008	Final set	Measuring Autogenous strain of con-crete with corrugated moulds.
Thomas W A, Moham-mad D, South W	2010	Initial set	Autogenous Shrinkage Interpretation from Experimental Shrinkage Measure-ments
Hu Z, Shi C, Cao Z, Ou Z, Wang D, Wu Z, He L	2013	Initial set	A Review on Testing Methods for Au-togenous Shrinkage Measurement
Gao P, Zhang T, Luo R, J Wei J, Yu Q	2014	Initial set	Improvement of autogenous shrinkage measurement for cement paste at very early age
Ghafari E, Ghahari SA, Costa H, Júlio E, Portu-gal A, Durães L	2016	24 hours	Effect of supplementary cementitious materials on autogenous shrinkage of ul-tra-high-performance concrete
Wu L, Farzadnia N, Shi C. Zhang Z, Wang H	2016	Initial set	Autogenous shrinkage of high-perfor-mance concrete: A review
Wyrzykowski M, Hu Z, Ghourchian S, Scrivener K, Lura P	2019	1 hour after mix-ing	Corrugated tube protocol for autogenous shrinkage measurements

2.6 RESTRAINED DEFORMATION

The restraint deformation test on concrete can be done by using ring-test (ASTM C1581–18) or a linear test (Figure 2-13). The use of either test is to determine if the concrete will crack in the period or the condition the concrete is exposed too. There are no specifications for linear tests, but various methods have been used over the years as mentioned by Weiss (2022) in his paper on guidance to reduce shrinkage and restrained shrinkage. According to him, the biggest problem is to secure the concrete at the ends thus allowing for restrained shrinkage. A vast majority of methods are used, but the most popular is the method where the end of the specimen is bigger and full of reinforcing that connects it to the mould and the middle section is tapered into a narrower section where the measurements take place either internally by different types of strain gauges, or on the surface with LVDT measurements. This is shown in Figure 2-13 **(**Weiss, 2022).

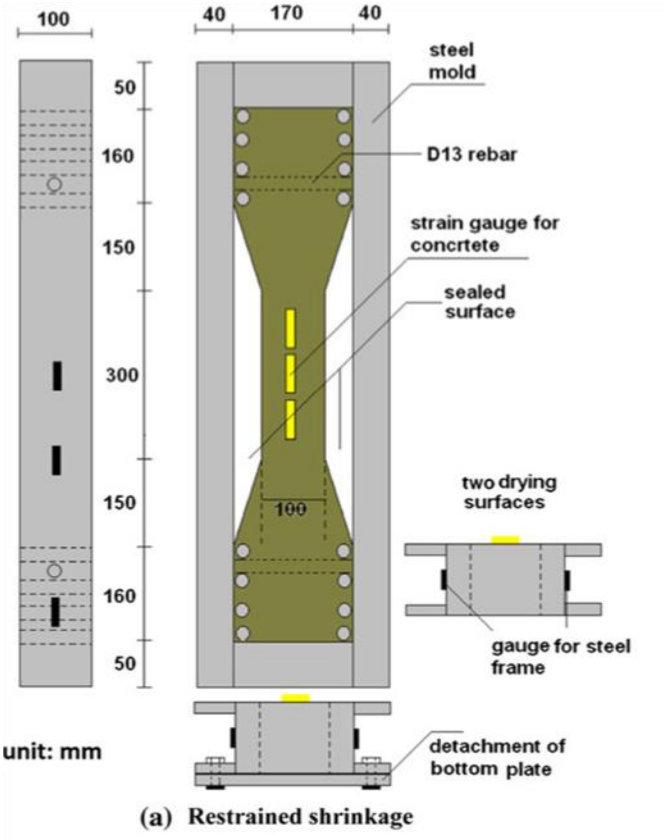


Figure 2-13: Test equipment for linear restrained shrinkage (Weiss, 2022)

The ring test is also seen as a restrained shrinkage test. Khaleel Younis mentioned in his PhD thesis of 2014 on restrained shrinkage that the method was developed by Carlson during WWII (1939) to determine drying shrinkage cracking potential from concrete mixes. This is also mentioned by Grzybowski and Shah (1990). It however took over 70 years to be added as a recognized concrete test in the ASTM (2004) and also AASHTO (2008). The ring test is however mostly used to measure the strain in drying concrete and predict cracking in concrete. This is done by opening up the surface of the concrete after a pre-determined time, mostly 24 hours. The outer ring can be removed and the top and bottom surfaces covered with an impermeable medium (Weiss, 2022). In Figure 2-14 the layout of the components of the ring test is shown (Kanavaris et al., 2019). Dimensions were given as in ASTM C1581-18, but researchers can change the dimensions to suit their required research. What is not shown is the instrumentation of the inner ring which should be full bridge strain gauges to eliminate temperature influence on the strain measurement.

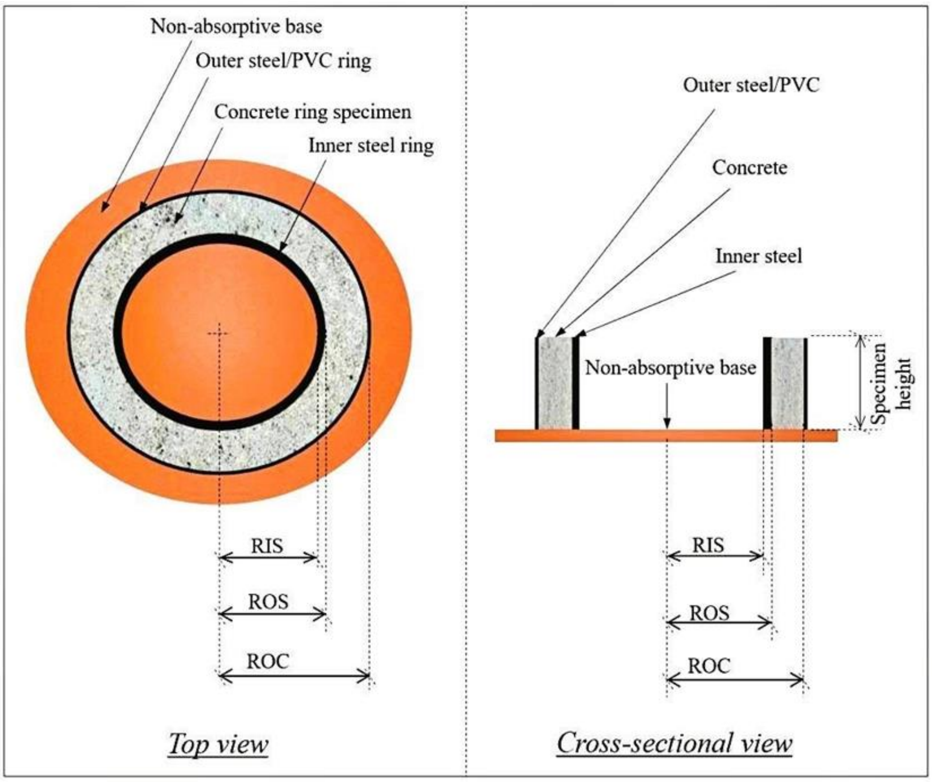


Figure 2-14: Ring test (Kanavaris et al., 2019)

In Figure 2-15 the results of 8 identical mixes are shown and the repetitive nature of the test when it is done under a controlled environment is clearly evident. Although the coefficient of variance seems to be high during the first eleven hours, the standard deviation seems to be small. However, what is visible in these results is the fact that the point where the strains start to develop are all more or less at the same time. This fact was however not mentioned by Eppers but seems to be a potential change in rate of shrinkage or knee point of the shrinkage of early age concrete (Eppers, 2010).

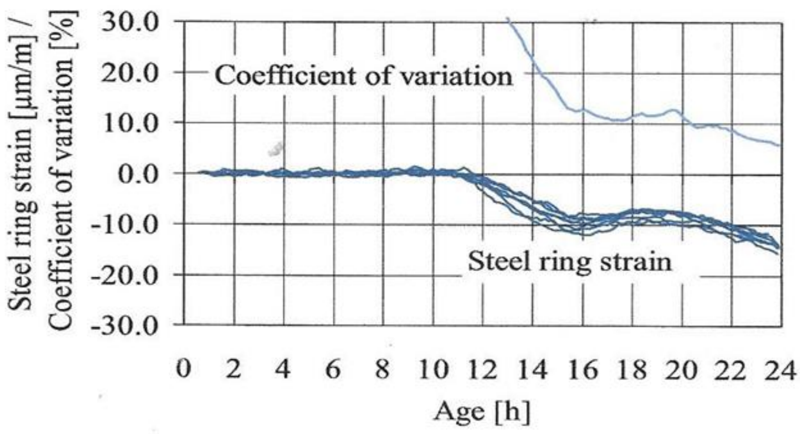


Figure 2-15: Results of ring test strain (Eppers, 2010)

2.7 EARLY AGE STRENGTH

The mechanical properties of high strength concrete after 24 hours can be measured using standard test methods for compressive strength, tensile strength tested with either a split cylinder test or a Modules of Rupture (MoR) (SANS 6254-06). The Modules of Elasticity (MoE) test combined with the Poisson's ratio can be determined from a cylinder (ASTM C469/C469M-22) before the split cylinder strength is determined (SANS 6253-06). None of these tests would tell us what happened during the first 24 hours after casting, the period when the bulk of the early age shrinkage takes place and the tensile strength of the concrete is at its weakest. It is during this period that a test is required to be able to determine if the concrete mix is strong enough at that stage to resist the tensile stresses caused by early age shrinkage. Therefore, none of the standard indirect tensile strength test methods used on hardened concrete are applicable.

2.7.1 Direct tensile strength

There is no standard test method for measuring early age tensile strength development. It is for this reason that some researchers developed a test frame to do direct tensile tests at different times after casting to test the tensile strength at very early ages (Dippenaar, 2015; Dao, et al., 2009; Hannant et al., 1999).

One of the concerns is to have a frictionless surface supporting the wet concrete. An air bearing box was developed and used by researchers (Dippenaar, 2015; Nguyen, 2020; Dao, 2009; Hannant et al. 1999). The test that was developed by Hannant in 1999 was also adopted into the geotechnical side of civil engineering but they used a shear box frame and a different type of mould as can be seen in Figure-2-16 (Nahlawi et al., 2004).

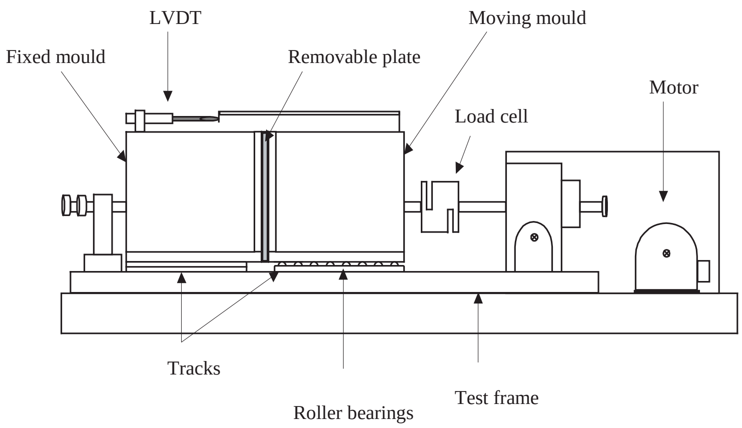


Figure 2-16: Direct tensile apparatus (Nahlawi H, 2004)

2.8 SETTING TIME TEST

2.8.1 Definitions

Concrete is currently seen to has an initial setting time and a final setting time. These two times are defined by the American Concrete Institute (ACI, 2013) as following:

"*The paste formed when cement is mixed with water remains plastic for a not very long time. It stiffens and sets. The setting process is arbitrarily divided into stages, time of initial setting and time of final setting, depending on the resistance to penetration by a probe. Before the time of initial setting, it is still possible to disturb the concrete and remix it without injury. Later revibrating can be beneficial, but as the reactions between the cement and water continue, the mass loses its plasticity. At the time of final setting, the concrete has become rigid and fractures rather than flows as increasing stress is applied*."

As the setting times measured using standard test methods are arbitrary values and according to the definition provided above, the concrete has not really set at the initial setting time, a third time, called time zero is investigated by researchers from where shrinkage of concrete should be measured (Sant et al. 2006 and Filho et al., 2022. This was defined as the divergence point between the chemical and autogenous shrinkage curves or the point where the autogenous shrinkage become zero.

2.8.2 Test methods

The only two methods that have a specification for setting times, is the Vicat needle test (ASTM C191-21) on cement pastes and ASTM C403-23 on concrete passing through a 5 mm sieve This test became a specification in 1957 (Pessiki and Carino, 1987). When testing pastes or mortars, some researchers such as Holt (2001) and Filho (2019) used the Vicat needle tests developed for cement paste setting times (ASTM C191) as shown in Figure 2-17. Some researchers then used the initial setting time and some the final setting time, as shown in Table 2-2, as the start point for measuring autogenous shrinkage and this time is referred to as "Time-Zero". Eppers, in 2010 showed in his PhD thesis, the difference between the same Vicat setting time test using 2 different specifications. The ASTM C 191-21 and EN 196-3 (EN 196-3:2005 is the same as SANS 50196-3:2006) are both called the Vicat test but there are differences in the equipment and the testing procedure. Table 2-3, from his work, shows the difference between the two specifications. The final setting in both standards is when the needle does not leave a complete impression on the surface anymore.

Table 2-3: Vicat needle penetration tests: (Eppers, 2010)

Parameter	**ASTM C191-08**	**EN 196-3-08**
Needle diameter	1 mm (0.785 mm^2)	1,13 mm (1.003 mm^2)
Initial set	penetration of needle ≤ 25 mm (sample hight 40 mm)	penetration of needle 31 - 37 mm (sample hight 40 mm)
Final set	Not making a 5 mm diameter ring mark	Not making a 5 mm diameter ring mark
Curing climate	23°C @ 95% RH	20°C kept under water

When calculating the area of the two needles (0.785 mm^2 and 1.003 mm^2) the difference is about 22%. The second difference is the penetration depth that differs by 12 mm into a 40 mm sample and lastly, the curing conditions differ in temperature and humidity. These differences between the curing, penetration depth, and needle size, will cause these two tests not to yield the same result. Eppers (2010) then mentioned that in both tests the pastes are prepared to have a specified consistency and when testing pastes to determine the initial setting time, from where the autogenous shrinkage should be measured, the current consistency of the paste must be used. This procedure provides a time zero that may or may not be the actual time zero for the concrete mix composition under investigation.

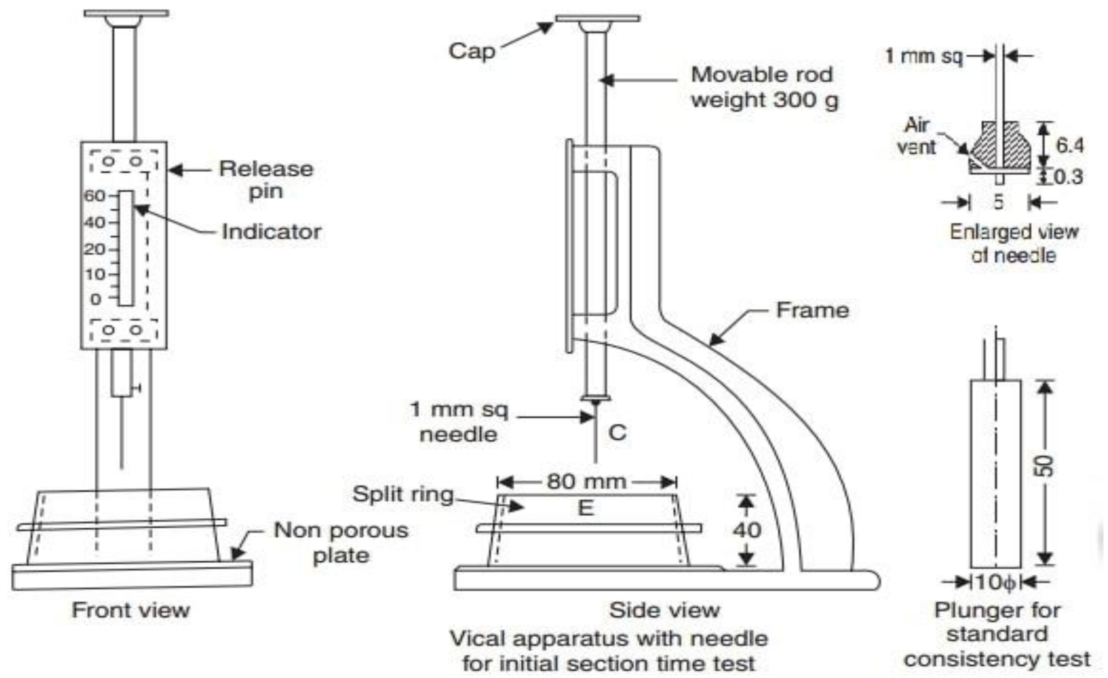


Figure 2-17: Vicat needle apparatus (ASTM C191-21, 2021)

When testing concrete some researchers used the ASTM penetration test for concrete to determine concrete setting times (ASTM C403-2023) as shown in Figure 2-18. Some researchers used either the initial or the final setting time, while other researchers used a fixed time (Table 2-2). Assumed starting times found in the literature were from 1 hour after casting up to 24 hours after casting. Many of the researchers agree that the two setting time tests are arbitrary numbers selected to define the two setting times (Sant et al, 2006, Lara, 2008).

Figure 2-18: Setting time tester for concrete ASTM C403-23 (Humboldt 2016)

In Figure 2-19 it can be seen that the initial set is defined at a resistance strength of 3.6 MPa (500 PSI) and the final set at a resistance strength of 27.5 MPa (4000 PSI) measured at a penetration rate of 25 mm in 10 seconds. The ASTM uses the PSI values which are rounded numbers that do not represent a precise change from one condition to the next.

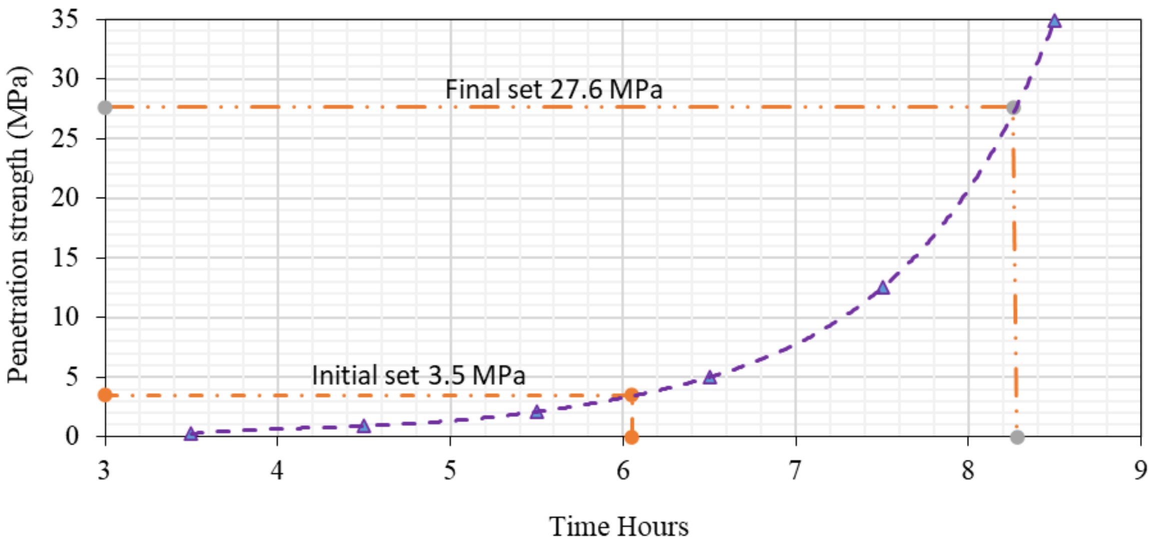


Figure 2-19: ASTM C403-23 concrete setting time results

A further problem with the setting time test is that different diameter needle ends are specified in areas of the needle end in the specification ASTM C403-23 (650, 325, 160, 65, 32 and 16 mm^2). To select the correct size needle to start the test and then change diameter as needed, requires experience and sometimes good guesswork. The speed of the test is also very fast, 25 mm in 10 seconds and a hand-operated instrument can take significant practice before mastering. In Figure 2-20 a specimen after testing according to ASTM C403-23 is shown (Civiconcepts, 2019). In this figure, the difficulty in selecting the correct needle is shown. The one test using the 650 mm^2 needle did not go the required depth of 25 mm and a change in needle size was needed to do the test for this time slot. This phenomenon is a common occurrence, every time a new type of cement or blend is tested especially with the first penetration where it can either be too early or too late. When admixtures are added it becomes even more difficult to get the timing of the first test right. A further problem with the concrete test is that the material for the penetration test must all go through a 4.75 mm sieve according to the ASTM C403-23 test. The test method was amended in 2016 to allow for an automated test setup (ASTM C403/403M-23). The sieve size for the test to run on the automatic testing machine was changed to 2.0 mm making the sieving even more difficult. This causes a problem because of the time it takes to get the fine aggregate and cement paste through the sieve. Up to now, most, if not all high-performance concrete work was done with fine materials, because of the belief that coarse aggregate reduces strength. This can be true but is not cost-effective.

Figure 2-20: Concrete setting time (Civiconcepts 2019)

Work done by Holt and Leivo (1996) showed that by changing the admixture % in HPC, the setting time changed dramatically. They also showed that different types of cements react differently with the use of the same admixtures at the same dosages. This can be seen in Table 2-4 from their work on mixes with a w/c = 0.45.

Table 2-4: Concrete setting time with a superplasticizer (Holtz and Leivo, 1996)

SP Dosage (%)	Setting Time (hrs.: min)	
	White Cement	Gray Cement
0	04:10	03:40
0.5	04:20	03:50
1	06:00	04:30
1.5	07:30	05:00

Holtz and Leivo (2000) showed that the temperature of the environment also plays a major role in the final setting time of concrete (see Table 2-5). This can result in more deformation taking place before setting when the ambient temperature is low. This can also cause more cracking during plastic settlement.

Table 2-5: Concrete curing temperature effect on setting (Holtz and Leivo 2000)

Temperature (°C)	Setting Time (hrs.: min)
5	11 +
20	05:30
30	04:20

In Figure 2-21 the initial and final set lines obtained from the Vicat method are compared with autogenous and chemical shrinkage, determined by using the volume method. In the paper by Sant, it was mentioned that to convert volume to linear, the measurements should be dividing by 3. That could however not be correct, to chance volume to a linear measurement the volume result should be converted by calculating it to the power of 0.333. The time at which the two types of shrinkage split, is defined as time-zero (Sant et al., 2006). In this case, it is very close to the measured final setting time**.** The test results in Figure 2-21 for both the chemical and autogenous shrinkage were measured using the buoyancy methods proposed by Sant et al. (2006).

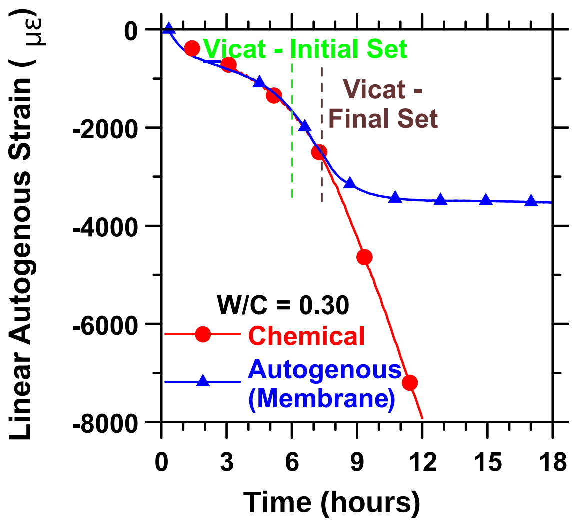


Figure 2-21: Deviation between chemical shrinkage and autogenous strain as a procedure to determine time-zero (adapted from Sant et al., 2006)

2.9 OTHER SETTING TIME TESTS DEVELOPED

As researchers realized that the tests for determining setting times are not really applicable to determine the real time from where the early age shrinkage should be measured, they started looking for other methods of determine time zero.

The following are some of the methods developed by researchers:

- The use of the heat of hydration graph where two different percentages (21% for the initial set and 42% for the final set) of the temperature change, between the baseline and maximum was used as seen in Figure 2-22, called the fractions method (Wang, 2007; Kim et al., 2009; Lora, 2013; Kopecskó and Baranyi, 2022).

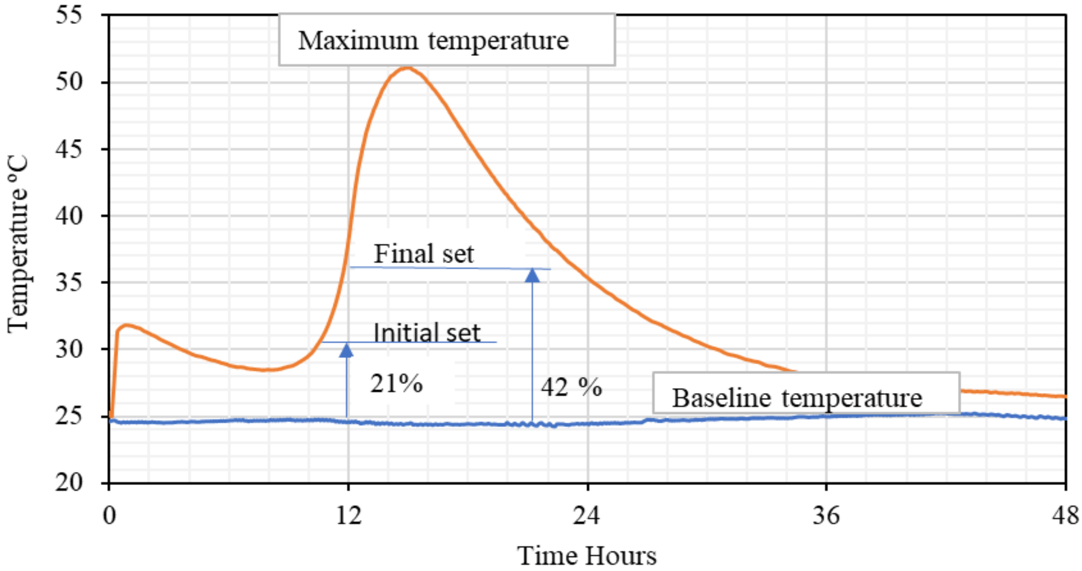


Figure 2-22: Fractions method to determine initial and final set (adapted from Kopecskó and Baranyi, 2022)

- The adiabatic heat of hydration temperature graph is used and the final setting time is calculated using the first derivative of temperature and the initial set is the second derivative of the temperature as shown in Figure 2-23. The values are approximately 25% of the temperature increase for initial setting time and 50 % for final setting time (Loura, 2013; Wang et al., 2015).

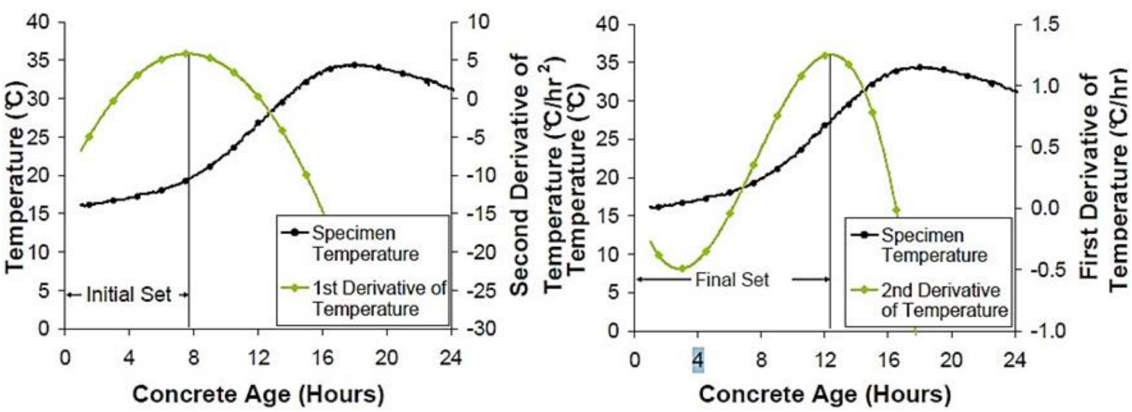


Figure 2-23: Derivative method to determine initial and final set (Loura, 2013)

- The use of capillary pressure to monitor the suction in concrete and set limits to prevent early age cracking (Slowik et al., 2013 and Tian et al., 2012). As can be seen in Figure 2-24 the graphs produced by the equipment in Figure 2-25 measuring the suction in concrete slabs. In the paper by Slowik et al., (2013), the difference between cured and uncured concrete is shown, indicating how to prevent cracking. For the concrete covered with plastic, it can be seen that the graph suddenly changes slope at about 520 minutes and that point should also be investigated with other methods for determining time-zero.

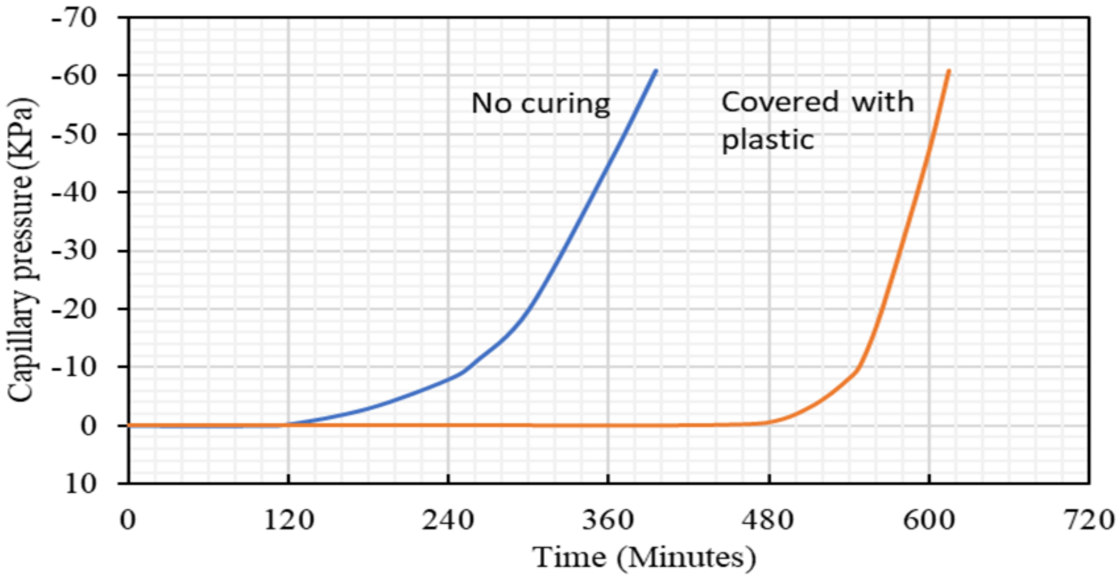


Figure 2-24: Capillary pressure versus time, measured in cured and in uncured concrete specimens (Slowik et al., 2013)

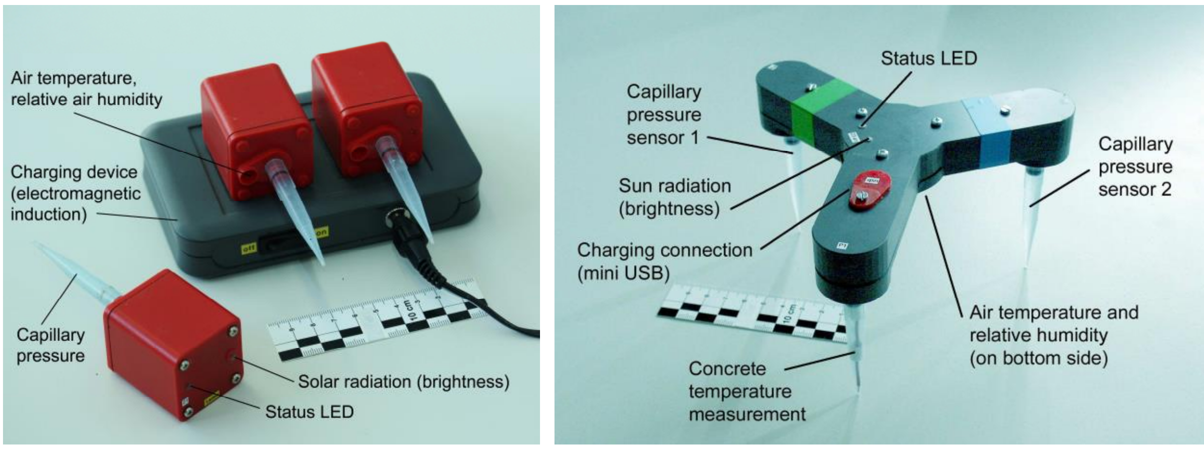


Figure 2-25: Prototypes of enhanced capillary pressure sensors (Slowik et al., 2013)

- Time-zero determined by the measuring of the moisture content drop in early age concrete (Huang and Ye, 2017). In their paper of 2017 Huang and Ye used different test methods to establish the correct time-zero. Results for two of the tests are shown in Figure 2-26 showing that the two methods do not completely coincide. They also used the UPV test (discussed later) and the heat of hydration but their results differed from the change in slope for the graph supplied.

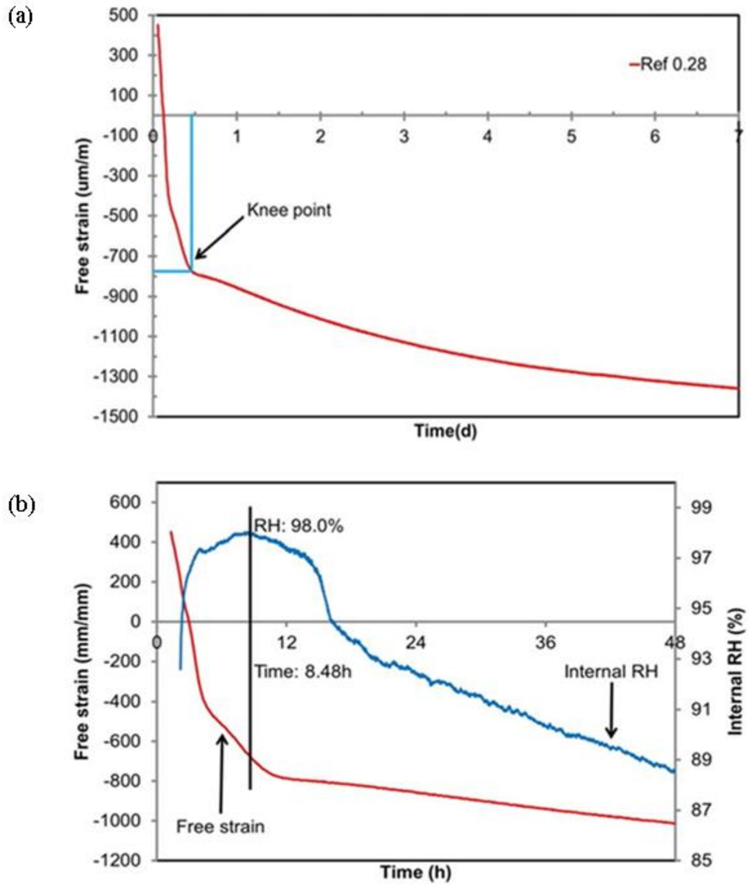


Figure 2-26: Time-zero (Knee point) indicated by free strain and relative humidity tests (Huang and Ye, 2017)

- The use of a Rheology meter to determine time zero (Sant et al., 2008; Bentz 2017). Rheology is mostly used to determine the fluidity of a liquid but it is used in cement matrixes to determine the ease with which the material can be pumped. It is especially used in mining where cementitious slimes are pumped back into the mine as backfill. Concrete is a Bingham fluid as shown in Figure 2-27 and a measurable force is required to move the material from the start. At a certain point the same mix starts requiring more force and that point in time is used in this work as time-zero.

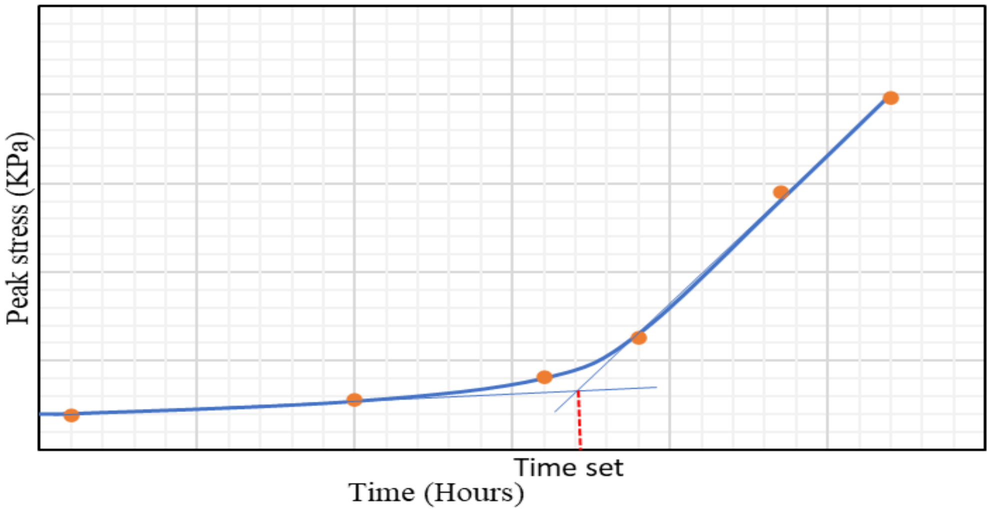


Figure 2-27: Rheology stress measurements on cement matrix (Bentz and Ferraris, 2017)

- The use of Electric resistivity to measure the setting time (Liao and Wei, 2013; Ghoddousi et al., 2015). is only used for laboratory research but this can definitely be a method to find the time-zero. The setup is shown in Figure 2-28 and the result for using the method to determine the influence of admixture is shown in Figure 2-29 and it also shows a clear turning point in time that can be used as the time-zero.

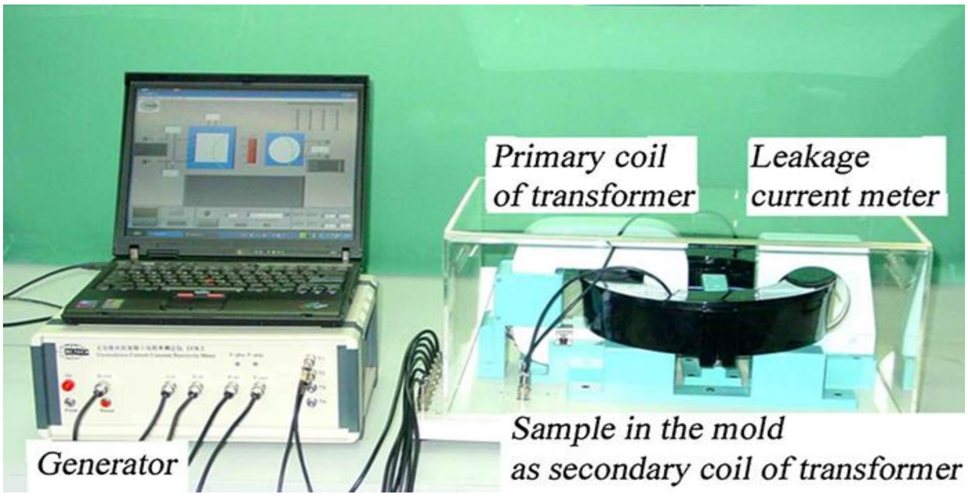


Figure 2-28: The measuring setup to measure resistivity in concrete (Liao and Wei, 2013)

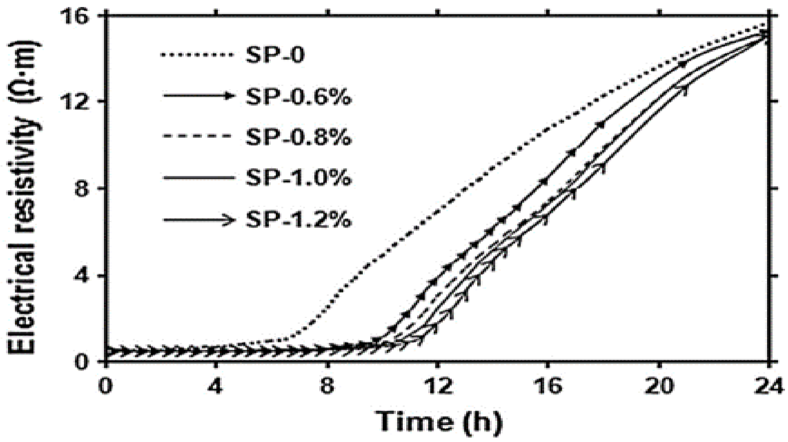


Figure 2-29: The results of electric resistivity on concrete (Liao and Wei, 2013)

- Ghoddousi et al. (2015) used a method where a plate was placed in the concrete whereas Li et al., (2007) used a method where they measured the current flow in the concrete supplied by a transformer as in normal electricity current measurement.
- The Ultrasonic Pulse Velocity meter (UPV) has been used in concrete since the 1940's. The main use of the instrument is to calculate the dynamic E-value of concrete. The use of the UPV is covered under ASTM C597-16. It can also be used to determine the thickness of concrete slabs, or the difference in densities between pours in slabs and to find voids in concrete. The use of the UPV to determine concrete setting times was studied by Wei et al., (2014) and Sant et al., (2006). The data in Figure 2-30 (Filho et al. 2019) is compared to the Vicat penetration test (ATM C191-16) as shown in Figure 2-30 as initial and final set Ryu et al. (2020) used the UPV to produce a similar S-shaped result and determined the intersections of the lines as the time for initial and final set, to compare the results for air entrained mixes.

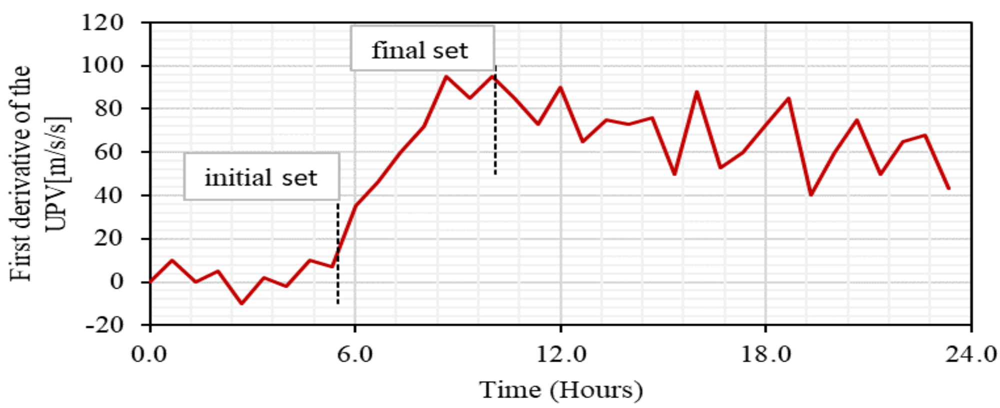


Figure 2-30: Determining the initial and final setting using the UPV (Filho et al., 2019)

- The use of cones in Civil engineering is not new and cone penetration tests are used to determine numerous material properties in civil engineering. The Dynamic Cone Penetrometer (DCP) is covered under ASTM D6951-16. The test was developed in Australia in 1956, but the shape of the cone (30 to 60 degrees) and the weight of the hammer (8-10 kg) varies between countries. The DCP is mostly used in road construction where the values are converted to California Bearing Ratio (CBR) values.
- Another cone test is called the Cone Penetrometer Test (CPT covered under ASTM D5778-20). The test was developed in 1932 with a 60-degree tip, which is still today the preferred tip angle. The test was developed to determine geotechnical material properties and depths of 50 meters and more can be investigated. This test is one of the most developed tests since its initial use and with the arrival of the computer more and more data can be gathered to deep depths at a fraction of the cost of other methods like core drilling and sampling (Robertson 2010).
- The third soil test where a cone is used is to determine the liquid limit in the fine particles smaller than 425 microns in diameter in soil, (Ibrahim et al., 2019; ASTM D4318-17). Ibrahim compared the ASTM test for liquid limits with the cone drop test to determine the same limits (BSI- BS 1377-2,1990). The cone test is the drop depth over a period of 5 minutes of an 80-gramme cone with a 30° angle into the wet material and then drawing a graph of penetration depth and water content. The required penetration depth is 20 mm.

- There is a test where a cone is used in concrete but that is to determine the workability of the concrete directly after mixing. Sachan and Kameswara Rao (1988) developed a test with a cone to test the workability of fibre reinforced concrete. The test is done by calculating the volume displacement of a 30-degree cone loaded with different weights as can be observed in Figure 2-31. In this test, different types of aggregates were tested with the maximum aggregate size of 19 mm (Metwally et al. 2014). The fact is that Sachan and Kameswara Rao (1988) developed a cone penetration test to use on fiber reinforced concrete to replace the standard workability test methods. The reason being that the normally used methods do not work very well for fibre reinforced concrete. The fact that Metwally (2014) used a cone to determine workability for concrete containing aggregates with different surface textures, made this the inspiration to develop a cone penetration test for concrete. With further research and development, a cone penetration tests can replace the current ASTM penetration test to determine setting time and possibly find a more defined point where the concrete change from a Bingham flued to a solid., This single point can be compared with "time-zero" as defined by other researchers rather than two setting points that does not really define any changing point in wet concrete.

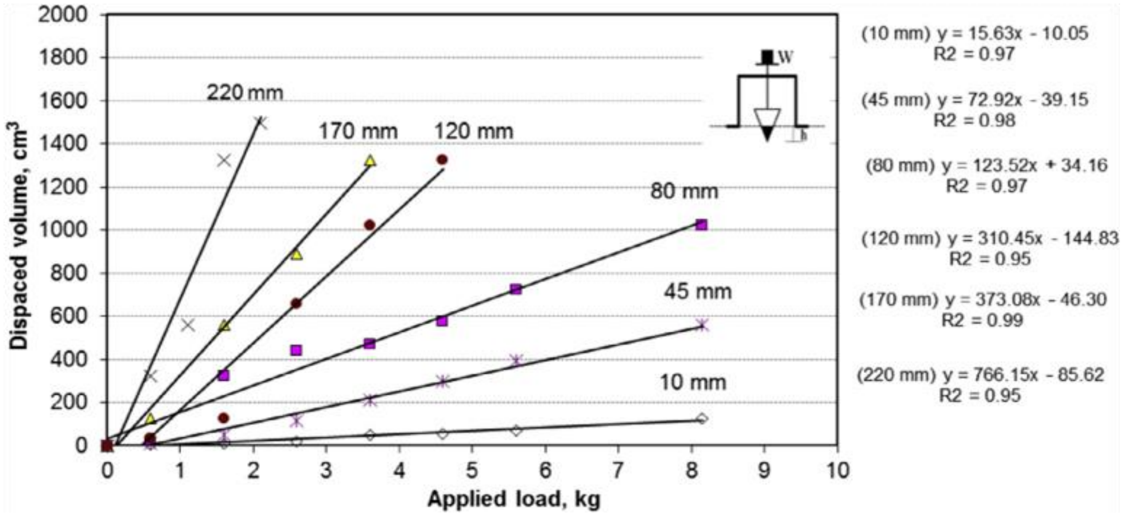
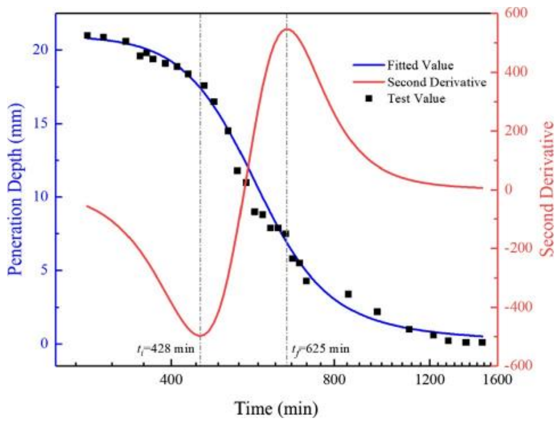
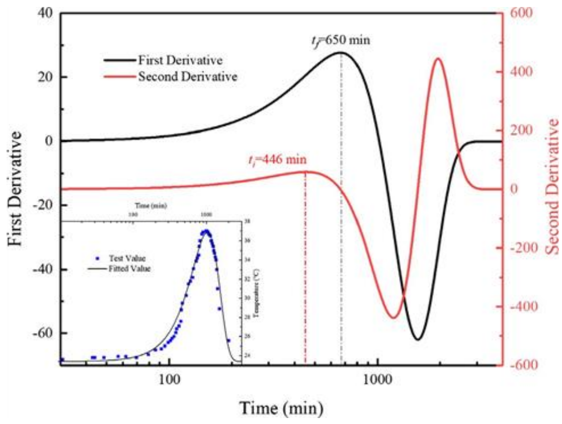


Figure 2-31: Slump versus displaced volume rate, (Metwally et al. 2014)

- Kang et al. (2020), used the DIN ISO/TS 17892-6 test method that is used for determining the undrained shear strength of soils, to determine concrete setting time properties. The Falling Cone Method (FCM) with 60º cone weighing 60 grammes was used to produce Figure 2-32. The S-curved result was used to develop an equation to fit the data from the FCM test results shown in the first graph in Figure 2-32. By then using the equation and determining the second derivative they produced the curve also shown in Figure 2-32 (a). The lowest point on the graph is then used as the initial setting time and the highest point the final set-in time. In Figure 2-32 (b) the authors measured the semi adiabatic temperature using the first and second derivative results, determined from the semi-adiabatic calorimetry graph. The authors used the same cementitious mix to determine initial and final setting time with the Vicat method to compare the results with each other.



(a)



(b)

Figure 2-32: Falling cone test results (a) Heat of hydration test results (b) (Kang et al., 2020)

2.10 SUMMARY

Based on the literature reviewed there is still confusion about the measurement of early age deformation. The main problem is, to specify the correct time to start measuring the deformation. A standard test method for measuring the early age deformation is required. The method adopted by ASTM - C1698 (2019) has flaws when different mould materials and sizes are used. Another concern is that the researchers try to link their new type of setting time to the times that come from the ASTM C403 (2016) method although most researchers agree that the initial and final set time obtained when using this method are arbitrary numbers. There are however a number of researchers that are searching for a single turning point called the "time-zero" to start deformation measurement.

Both ASTM methods C191 (2016) and C403 (2016) for mortar, paste and concrete have two setting times, an initial and a final setting time. These setting times do not represent the time when the cementitious material changes from a Bingham fluid to a solid material. To quantify autogenous shrinkage that will cause internal stresses that can result in cracking, researchers need one point in time from where the shrinkage should be measured. This would also make it possible to compare results from different researchers.

The aim of this study was to determine a single-point result from a test method, which can be used from where the early age shrinkage can be measured. The point in time from this method will have to indicate a clear slope change in the test result behaviour. This point in time will be called "time zero". Early age concrete properties were used to determine the point in time when the concrete starts acting as a solid material in which strains can develop due to shrinkage. To eliminate the effect of aggregate size on the observed trends, only fine aggregate (sand < 5mm) was included in the experimental study.

3 EXPERIMENTAL PROGRAMME

3.1 INTRODUCTION

The main focus of the research is to determine an applicable setting time from where early age shrinkage can be measured. To be able to do that a large variety of concrete properties were determined during the first 24 hours after casting, to identify a change in the behaviour of the mix. That change in behaviour should make it possible to find a more accurate method for determining the point from where autogenous shrinkage, that was identified as a problem occurring in high strength concretes, should be measured. From the literature research it became clear that not all researchers use the same value from either of the two setting time tests to start the early age shrinkage measurement. The existing setting time tests were not developed to determine a specific point on the early age property graphs, but rather to determine arbitrary values as chosen when those tests were developed. Some researchers believe that a "Knee-point" exists where the concrete changes from a Bingham liquid to a solid (Sant et al, 2008. Huang and Ye, 2017). The experimental programme of this project was planned to look at possible early age concrete properties that could give an accurate indication of the time when concrete changes from a liquid to a solid in which stresses and strains can develop. The aim is to find one point in time that can easily be determined in a concrete laboratory, which can be taken as the moment the concrete starts acting as a solid material. This point in time can then be used as time zero when measuring early age deformation.

3.2 MATERIALS AND MIXES

Throughout the research a Cem I 52.5N cement from the same source, with the properties as shown in Table 3-1, was used. The X-ray Fluorescence spectroscopy (XRF) chemical analysis of the cement can also be seen in Table 3-1. The cement had a relative density of 3.14 for both of the two batches used. The four compounds for the cement used were obtained by using XRF determined oxides and the Bogue formula to calculate the compounds. The bottom part of the table shows the results from the standard cement mortar test (SANS 50197, 2006) to determine the strength of the two batches of cement used in the tests. Standard setting time and mortar prism strength tests were performed over the span of the research to make sure that the cement properties did not vary significantly. The results of 2018 and 2019 were from the same batch at the beginning and end of the first batch. Each strength result is the average of 6 specimens tested. The cement of both batches was stored in air tight containers over the whole period of testing. Typical mortar prism strengths can be seen in Table 3-1. The C_3S is very high giving the cement an earlier setting time and reduced the difference between the 7- and 28-day strengths. The setting time tests were conducted according to the standard Vicat test as adopted by the EN 196-16 and the ASTM C191-08 test method respectively shown in Table 3-1.

Table 3-1: Cement XRF and strength results.

XRF chemical analysis of the cement		Chemical analysis by using Bogue equations			
Oxides	**Percentages (%)**	**Compound**		**Percentages (%)**	
SiO_2	21.5	**C_3S**		68.7	
Al_2O_3	5.2	**C_2S**		9.8	
Fe_2O_3	2.8	**C_3A**		9.0	
CaO	66.6	**C_4AF**		8.5	
MgO	1.0	**Total**		**96.0**	
K_2O	0.6	**Mortar prism strength test results (MPa) (SANS 50196)**			
Na_2O	0.2	**Mix date**	**2 Day (STDEV)**	**7 Day (STDEV)**	**28 Day (STDEV)**
SO_3	1.0	06/18/2018	33.0 (0.67)	49.3 (1.18)	55.2 (1.91)
Free Lime	1.1	03/25/2019	34.5 (1.08)	46.9 (1.95)	52.7 (0.94)
Total	**100.0**	06/13/2021	33.6 (1.15)	48.9 (0.64)	52.4 (1.64)
Month tested	**Blaine**	**Vicat initial setting time**		**Vicat final setting time**	
06/2018	442	234		381	
03/2019	448	252		401	
06/2021	431	245		414	

The fine and coarse aggregate used were from the same quarry and is known in South Africa as a Dolomitic rock, while it could be called Limestone in other countries. The relative density of the aggregate was 2.84. For the main test programme only the material smaller than 5 mm (fine aggregate) was used with the various w/c ratios, with and without a Polycarboxylate Ether (PCE) admixture to do the bulk of the tests. The particle size distribution of both the cement and the fine aggregate can be seen in Figure 3-1.

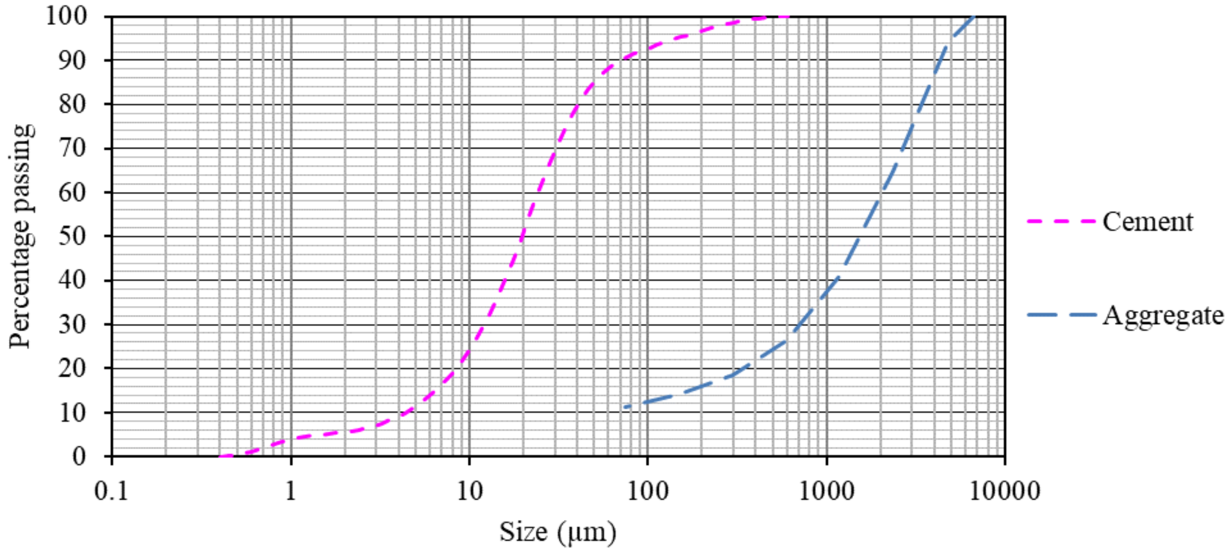


Figure 3-1: Particle size distributions of the cement and crushed sand

3.2.1 Trial mixes

Before the mix designs for the whole project was finalized, trial mixes were made using the selected materials to get the right amount of water and admixture for an almost constant workability while keeping the HRWRA dosage intervals between mixes with different w/c ratios constant. Once the mix designs were selected, all the materials for the different mixes were placed in a temperature-controlled room. The mix sizes as planned for all casts were batched and mixed according to the planned sequence.

The workability of each mix was determined and enough cubes were cast to conduct standard cube strength tests at concrete ages from one day right through to 28 days. This was only done once because the aim for the project was not to investigate the repeatability of cube strength or workability. The mixes without admixtures were added at a later stage to highlight the influence of the admixture on the concrete properties before setting as shown in this project. The trends observed for the cube strengths can be seen in Figure 3-2. Polynomial trendlines were fitted for each preliminary testing age, with the R^2 values shown in the legend.

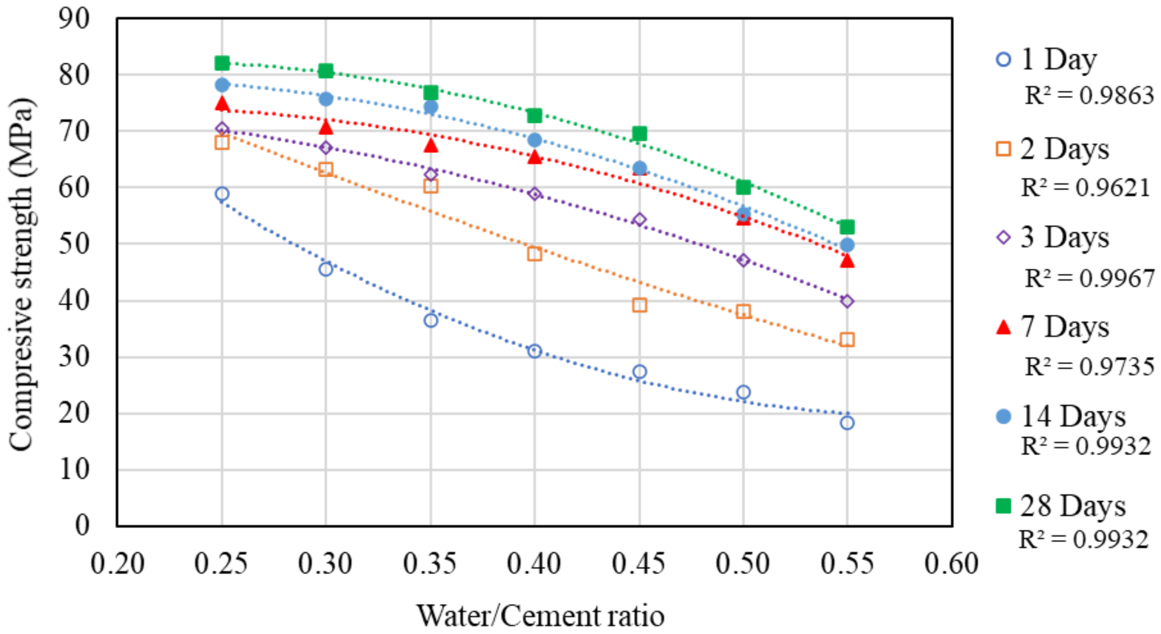


Figure 3-2: Trial mix compressive strength results from 1 to 28 days

3.2.2 Final mix designs

The concrete mixes were designed with the aim to keep the water content constant and admixtures were used to keep the workability as similar as possible for the different w/c ratio mixes as indicated in Table 3-2. This was done because in the vicat test the water content is also changed to arrive at a prescribed consistency. Mix 0.35 na to Mix 0.50 na (na is no admixture) were added later to the programme but all the relevant tests were completed on the mixes. These added mixes were similar to Mix 0.35 to Mix 0.50 but contained no admixture. The water content was altered to compensate for the water in the admixture (which is 55% of water and 45% solids according to the supplier). Although the w/c ratios shown seem to be different than for the mixes with admixture, the free w/c ratios are the same. The colour code shown for the different mixes were kept the same in all graphs throughout this document.

Table 3-2: Mix designs with constant water content

Colour Code		W/C ratio	Water/m^3	Cement/m^3	Aggregate/m^3	Admixture/m^3	Admixture %
Mix 0.25	-------	0.250	225	900	1315	18.0	2.00
Mix 0.30	-------	0.300	225	750	1472	12.5	1.67
Mix 0.35	-------	0.350	225	643	1584	8.6	1.33
Mix 0.40	-------	0.400	225	563	1668	5.6	1.00
Mix 0.45	-------	0.450	225	500	1734	3.4	0.67
Mix 0.50	-------	0.500	225	450	1783	1.5	0.33
Mix 0.35 na	-------	0.358	230	643	1584	0.0	0.00
Mix 0.40 na	-------	0.405	228	563	1668	0.0	0.00
Mix 0.45 na	-------	0.454	227	500	1734	0.0	0.00
Mix 0.50 na	-------	0.502	226	450	1783	0.0	0.00
Mix 0.55 na	-------	0.550	225	409	1829	0.0	0.00

3.3 MATERIAL PREPARATION AND MIXING

All the materials were batched the day before casting and then stored with the mixer pan inside a 25°C controlled temperature room. The dry materials were first mixed in a double action pan mixer for 30 seconds before the water was added. The time of mixing for each mix from the time that all the water was added, was five minutes for all the mixes in the programme. The HRWR was added to the mixing water for the mixes where required. The size of the mixes was also kept to approximate 50 % of the mixer capacity. After casting, all the specimens went into a 25°C curing room, covered with a double layer of plastic. All specimens cast were kept in a constant temperature room for the first 24 hours after casting. After 24 hours (SANS 5861-3:2006) cubes and cylinders were demoulded and placed in a 25°C water curing bath up to the age of testing.

3.4 SPECIMENS CAST FOR TESTING IN THE FIRST 48 HOURS

The importance of this part of the research was to cast as many as possible different types of test specimens, and test them at regular time intervals to determine the change in concrete properties over the first 48 hours after casting. Nearly 1000 kg cement was used to cast over 900 specimens tested as part of this study. Where multiple specimens were tested, average values are reported in the thesis.

The specimens cast for the different tests were as follows:

- Cylindrical specimen of various sizes (between 100 mm and 300 mm in height) for comparative temperature measurements.
- 105 x 300 mm cylinders to measure early age shrinkage (minimum of 2 per mix design).
- 105 x 105 mm cylindrical specimens for measuring setting times (5 per mix and was repeated twice).
- 105 x 105 mm cylindrical specimens for measuring of cone penetration test (5 per mix and was repeated 3 times).
- 365 mm diameter ring test for measuring shrinkage forces (one per mix design).
- 30 mm diameter cups for measuring development of suction (this test was only done on the mixes with admixture and repeated 3 times with different weights in cups).
- 100 mm cubes for early age strength development tests (2 specimens per testing time starting 6 hours after casting every 2 hours up to 24 hours and 48 hours, 7 and 28 days for the mixes with admixtures).
- Direct tensile strength from a dog-bone shape specimen (only 12 tests were done).
- 100 x 200 mm cylinder tests for E-value and split cylinder strength (2 cylinders per mix for every mix design).

3.5 WORKABILITY OF CONCRETE MIXES

The workability tests were conducted according to ASTM C1437 - 20: standard which is a small flow-table tests as described in ASTM C230 -14. The apparatus is normally used for cement pastes and mortars. The result is an average diameter of flow on the table after a specified number of drops through a fixed height. In Figure 3-3 the difference between similar mixes with and without admixture can be seen.

Figure 3-3: ASTM C 1437 flow table (Flow 100 – 200mm)

3.6 HEAT OF HYDRATION

Heat of hydration was measured using specimens cast in plastic pipes, where the height and the diameter of the specimens varied. No standard specification for adiabatic or semi-adiabatic heat tests were used. The aim was to test specimens with sizes equal to the early age shrinkage specimens, as well as the cubes and setting time cylinders cast in this study. The results of the heat graphs could then be compared with shrinkage, setting time for penetration resistance, ASTM C403 -16 needle penetration, cone penetration, cube strength, restrained ring test, direct tensile test and split cylinder strength.

Figure 3-4 shows the specimen with the same volume of concrete than the cubes and the setting time specimen used. Thermocouples were placed in the middle of each specimen and the temperature was recorded every minute for the first 48 hours after casting. The temperature of both the 1 litre mix in the pipe and the Semi Adiabatic (SA) flask (see Figure 3-5) were measured at the same time.

Figure 3-4: The test specimen for testing the heat of hydration

Semi adiabatic heat of hydration was measured using the “Langevant” flasks containing the standard containers developed with the flask. The advantage was that both heat of hydration measurements could be measured with the same data logging system at the same time making comparison of the results much easier. The two flasks in Figure 3-5 show the size and also the thermocouple wires coming out of the Langevant flasks.

Figure 3-5: The Langevant test flask for testing the semi adiabatic heat of hydration

3.7 EARLY AGE SHRINKAGE TESTS

The early age shrinkage was not measured using any standard specification. The measurement was done by using a 110 mm diameter and 310 mm high plastic pipe, lined with a single layer of 250-micron Teflon. Similar dimensions were used in a vertical shrinkage test method by Hammer et al. (2002). A lid was placed at the bottom of the pipe section and sealed with grease to prevent any loss of water at the bottom when the specimen was cast. The concrete was cast in a continuous process during vibration up to about 10 mm from the top. A 2 mm thick steel plate with a diameter 2 mm less than the specimens inside diameter was placed on top of the concrete. In the centre of the plate was a bolt that was screwed through the plate and vibrated into the top of the concrete to a distance of 10 mm into the concrete. The Linear Variable Differential Transducer (LVDT) measured movement of the top of the smoothed bolt head. This setup gives a specimen length of 290 mm. Once placed in position the top surface was covered with oil to prevent evaporation and measurements were started. The shrinkage measurement started immediately after casting, as soon as the specimens were set in position. Two specimens were tested for each mix. The shrinkage measurement typically started about 10 minutes after the first water got in contact with the cement. The rate of data gathering was a reading every 50 seconds (maximum time interval for data capturing equipment). The shrinkage measurement setup can be seen in Figure 3-6.

Figure 3-6: Early age shrinkage measurement setup.

3.8 RING TESTS

The ring test was used in current research to serve as a restrained ring test. The ring-test apparatus is a steel ring with thickness of 10 mm, 60 mm high and an outer diameter of 255 mm. The outside measurement of the ring was the inside diameter of the concrete and the outer diameter of the concrete was 365 mm, resulting in a 55 mm concrete thickness and 60 mm high concrete ring as can be seen in Figure 3-7. The steel ring was instrumented with 4 half bridge strain gauges placed at 90 degrees apart on the ring in the middle on the height of the ring. This is similar to the ASTM C1581 –18 but the specimen dimensions differed from the ASTM prescribed size. The strain gauge configuration allowed for temperature compensation and the assumption was made that the heat expansion coefficient of steel and concrete is about the same. Therefore, only the temperature compensated horizontal strain on the inside was used to calculate the strain on the contact surface of the concrete assuming the E-value of the steel is 200 GPa. To calculate the stress in the concrete the cross-sectional area of the concrete ring was used, and from the calculated stress in the steel ring, the average stress in the concrete was calculated by using the calculated force in the steel ring and dividing that by the cross-sectional area of the concrete. The purpose of the test was not to calculate the stress in the concrete accurately through the cross section as other researchers did, but rather to follow the strain development on a graph against time to find possible correlations to other tests in determining the time when a change in behaviour can be observed. The second use of the ring was to determine whether the different w/c ratios could cause cracking in the first 48 hours after casting when kept sealed, correlating to autogenous and chemical conditions. The test specimen in the ring test was cured by keeping it in the constant temperature room at 25°C while covered with a double layer of plastic.

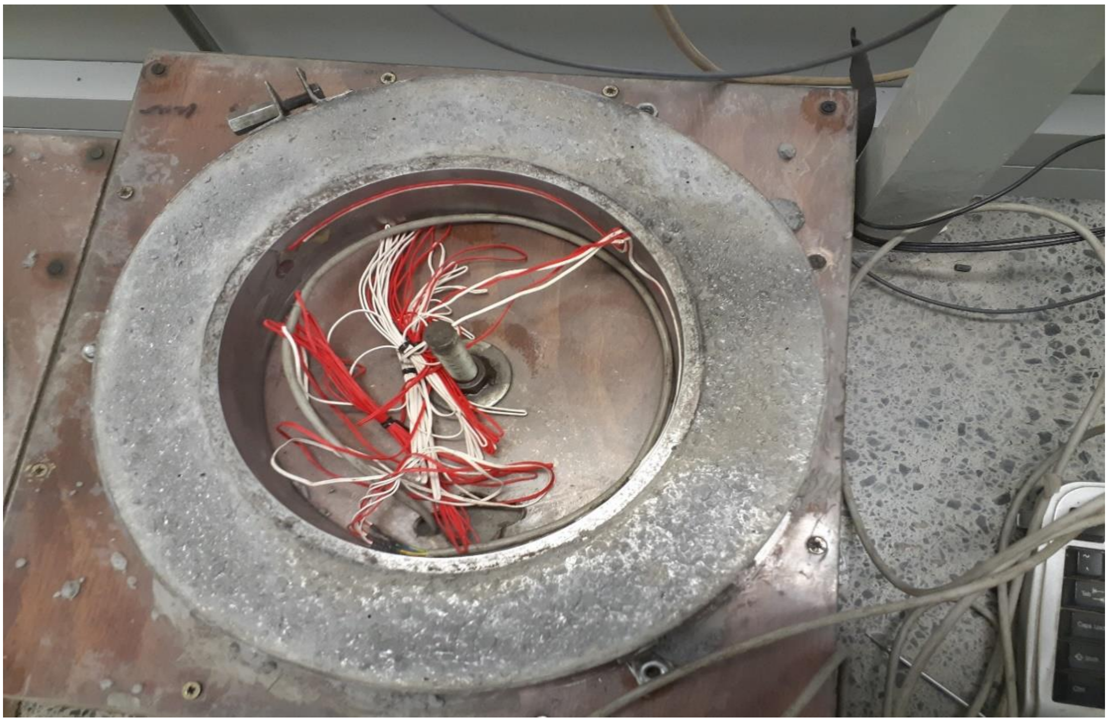


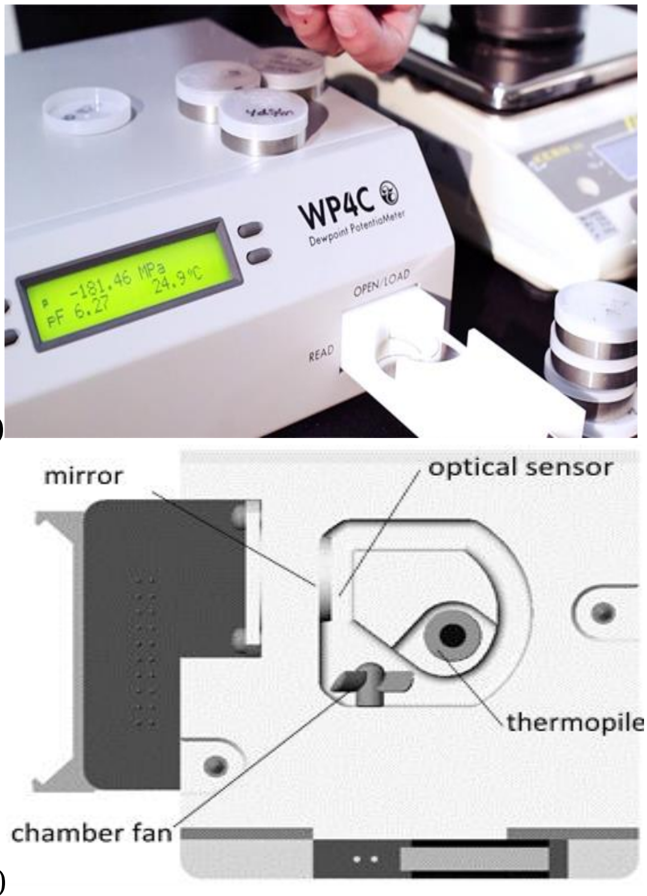
Figure 3-7: Ring test setup showing the strain gauge positions

3.9 SUCTION IN CONCRETE

Suction tests were conducted using a WP4 apparatus. Typically, the WP4C water potentiometer is used for soils, soil-less substrates, plant material and seeds, but it is equally applicable to substances as diverse as fruit, bricks and oil. In Figure 3-8 a photo of the apparatus is shown with the stainless-steel cups and the watertight plastic lids. The loading bay that is open in the photo, can also be seen where the cups without the lid can be loaded (WP4 Operators manual 2018).

The apparatus uses chilled mirror dew point technology and was developed for use in the geotechnical field to provide input for drawing the water retention characteristic curves in soils as indicated in ASTM D6836-16 under method D (WP4 Operators manual 2018). The chilled mirror dew point hygrometer apparatus was developed for low water content and high suctions. The measured suctions can vary from 500 kPa to 100 MPa in the ASTM method whereas the WP4C can go up to 300 MPa. There was work done by Vilasboas et al. (2016) to measure the water retention curves of mortars and cement samples by using the filter paper method also covered in ASTM D6836 -2016. The container sizes for the WP4C apparatus are 30 mm diameter and 15 mm high plastic or stainless-steel cups, which are filled halfway to contain about

20 grammes of wet material. The specimens were covered with a lid (also part of the equipment) and left in the constant temperature room with the other specimens. The testing started immediately after casting with the disadvantage that the specimen must be cooled down to about 24°C before the testing can commence. It is also good practice to measure the weight of the samples directly after every measurement to determine the amount of water lost.



(a)

(b)

Figure 3-8: (a) WP4C apparatus open. (b) Chamber inside the apparatus with all the working parts.

3.10 NEEDLE TEST FOR SETTING TIME

Cylindrical specimens were used to determine the setting time. The mould used was plastic pipe with a 110 mm diameter but the specimens were only 110 mm high to give the same approximate heat generating volume than the 100 mm cubes. The cylinder mould was sealed at the bottom and after casting a lid was placed on top to make sure no evaporation could take place over the intended curing period. The standard needles specified in ASTM C403-23 were used but the specimens tested were the same size than those used for the cone penetration test.

3.11 CONE PENETRATION TEST FOR SETTING TIME

Cone penetration test is an in-house test that was compared to the ASTM C403-23 needle test. This test is done the same way as the ASTM C403-23 test but instead of using of the different size needles a cone with an angle of 20^{o}s was manufactured. By using a cone, the test would not need changing of needles and mixes with aggregates up to 10 mm can be tested without sieving.

The cone penetration test is not a standard setting time test but a potential test that can be used in future to replace the ASTM needle test for concrete. The 20^{o} cone and the cylinder setup can be seen in Figure 3-9. The idea of using cone penetration to determine strength is not new in Engineering. It is used in soil testing to determine the range between the liquid limit and the plastic limit when determining the plastic index of soils. A cone is used in the Dynamic Cone Penetration (DCP) test in road construction to test the hardness of layers or the quality of compaction. Also, Piezo cone penetration is used world-wide in tailings dams to determine moisture content and settlement of materials to predict and prevent potential failure of these dams. The idea for this test however came from the workability test that was developed by using the cone penetration and then linking that to a slump. The workability test however uses a constant weight to give the penetration. The disadvantage of the test is that it can only be done in a well-equipped laboratory with presses that can apply load at fixed movement rates and record both the displacement and the load required to calculate the shear strength. Speed of penetration was also the 150 mm per minute as for the needle test in ASTM C 403 while the penetration depth was 50 mm and the tempo of recording both penetration depth and load were every second for the duration of the test. To calculate the shear strength the load applied was divided by the outer circumference area of the cone in touch with the concrete resulting in a shear stress in MPa.

Figure 3-9: Cone penetration setting time test setup.

3.12 COMPRESSIVE STRENGTH

The cubes used to do the strength tests during the first 48 hours were 100 mm in size according to SANS 5860-2006. Cubes were tested at 2 hours intervals from the time when demoulding was possible. The mixing, sampling and casting was done according to SANS 5861: 1-3 2006, with a slight change in the mixing duration that was changed from 90 seconds to 5 minutes to allow for the admixture used to reach its full potential. The filling of the moulds was done in a continuous casting while the vibrating table was left on. This procedure was used to mix and cast concrete for all the specimens cast during this project. The cubes cast for the first hours of testing (up to 8 hours after casting) were cast in steel moulds that can be demoulded by using hand tools without damaging the cube at very early stages after casting. The cubes for the later tests (in the first 48 hours) were cast in plastic moulds, making the demoulding faster and allowing for the curing and testing to take place closer to the selected times. The early cube strengths were determined on different testing machines to obtain more accurate results for low strengths. Cubes were tested using deflection control as the testing machine stability in load control was not good enough, causing shock loading at the low strengths recorded during the first 24 hours after casting the concrete. All specimen tested after more than 24 hours from casting, were cured in water at 23-25^{o} C up to the testing date according to SANS 5861-3. The cubes were weighed in water and in air to determine the density of each mix cast.

3.13 DIRECT TENSILE STRENGTH

Because autogenous shrinkage in high strength concretes causes early age cracks it was felt necessary to build a direct tensile test apparatus to test the early age direct tensile strength development of the concretes and compare these with the stress development measured in the ring test. The mould-box in this apparatus run on low friction linear bearings. This test set-up can be seen in Figure 3-10.

The direct tensile test was conducted only on mixes 0.25, 0.35, 0.45 (with admixture) & 0.55 and only at 8, 10 and 12 hours after casting. This was the time span where an E-value, a cube strength and the direct tensile strength test was possible on all the mixes without running out of capacity on the direct tensile testing apparatus. A custom-made mould producing a concrete specimen in the shape of an "I" with the dimensions of the portion of the specimen in direct tension 100 mm long, 100 mm deep and 70 mm wide was manufactured to conduct the direct tensile test on. The mould can be seen in Figure 3-10b.

The apparatus has a load cell on both sides to assist with the measuring of possible friction between the mould and the roller bearing bed. The measurements between the two load cells did not reveal any difference in the load on the specimen on either side. The design of the mould was also such that the two sides of the narrow middle section could be removed once the mould was in place and fastened to the load cells.

The same logging setup used to measure the shrinkage was also used to record the load experienced by the load cells, measuring from as soon as possible after casting. The idea was to measure possible forces on the load cells caused by shrinkage. However, no measurable forces were recorded.

When the concrete gained enough strength, the indirect tensile strength was determined with the standard split cylinder test covered in SANS 6253:2006.

Figure 3-10: (a) Direct tensile strength apparatus and (b) Mould for direct tensile specimen

3.14 MODULES OF ELASTICITY

The cylinders used to determine the MoE (Modules of Elasticity) and then split cylinder tensile strength tests in the first 24 hours were 100 mm diameter and 200 mm high, according to ASTM C469-2014. The specimens were cast in steel moulds and demoulding only commenced when sufficient strength was reached to do the loading up to 40% of the cylinder strength for the MoE test. Some of the cylinders were then tested to failure while measuring the deformation over two thirds of the middle (133/200) of the specimen while the rest of the cylinders used for MoE testing were then prepared and used for split cylinder strength according to SANS 6253 (2006).

The MoE tests were only conducted on 4 of the different w/c ratio mixes at different times after casting because the E-value is mainly influenced by strength and type of aggregate (Alexander, 2021). In this research only one type of aggregate was used and strength testing was conducted at various times to determine the influence of HRWRA and w/c ratios on the early age strength development.

3.15 ULTRASONIC PULS VELOCITY MESUREMENTS

Ultrasonic Pulse Velocity (UPV) tests were only done on two mixes because of the shortfall of the available equipment. The equipment gave a reading “Overflow” when the concrete was in liquid state The equipment was also not compatible with any automatic logging equipment, causing the testing to be recorded manually. The results recorded did however show definite trends that could be compared with other setting time tests. As seen in the literature study, this test shows potential to be used as a setting time test.

Figure 3-11 is a sketch of what the set up looked like. The mould was manufactured from timber with two holes the size of the receivers drilled directly opposite each other. The Mould was lined with thin plastic and the holes were plugged with the same piece that was drilled out. The size of the cube mould was 150 mm. After casting the top was also closed with timber and the measurements were taken.

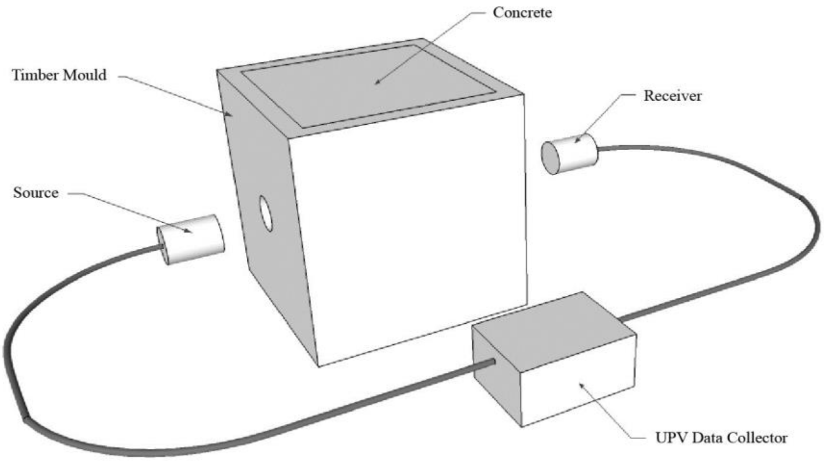


Figure 3-11: Sketch of the UPV test setup used

3.16 SUMMARY

As testing was completed over a span of more than two years it was important to make sure that the methods, materials, mixing and curing conditions were kept similar over the whole period. This was important as the results required came from the first 48 hours and during this period any differences could result in completely different results. Some of the tests were conducted not using standard equipment, but the methods and equipment was the best locally available.

4 EARLY AGE PROPERTIES

4.1 INTRODUCTION

This chapter contains the results obtained and it follows in the same chronological order in which the tests were conducted for each concrete property, taking place from mixing, to 24 hours. Some of the tests were extended to 48 hours (such as temperature, shrinkage and cube strengths). In this chapter results for all the different tests are provided for all the mixes that were tested.

4.2 WORKABILITY OF CONCRETE MIXES

The workability tests were done using the ASTM C 1437 – 15 standard which is a small flow-table test normally used for cement pastes and mortars. The test was not compared to the standard slump test although the mix included aggregates up to 5 mm. The ASTM C 1437 flow-table test was done on all mixes, using identical batch sizes. The materials were stored in a 25ºC climate room for 24 hours before casting. From these batches, only cubes were cast and the flow-table test conducted so as not to waste time that could influence the results of the tests.

The flow test was done to get the right admixture dosage in the trials to give a similar workability or consistency result for all the mixes. The workability was kept the same because the Vicat needle test uses a method where the mix that is tested first has to be prepared to have a specified consistency. The consistency will mean that a standard amount of cement is used and water is added to get a specified flow or consistency. This implicates different w/c ratios for different fineness of cement or blended cements.

The results of the flow test can be seen in Figure 4-1. The flow of the 0.25 w/c ratio mix is lower, it however was still workable enough to get a good compaction. Also shown in the figure are the results from the mixes made without admixture. As expected, an increase in the w/c ratio resulted in an increase in flow. Mix 0.35 to mix 0.50 without admixture were added to compare with the mixes with admixture but the free water was kept constant at 225 litre per cubic meter of concrete so as not to change the total water content. That is the reason why the workability of those mixes was lower.

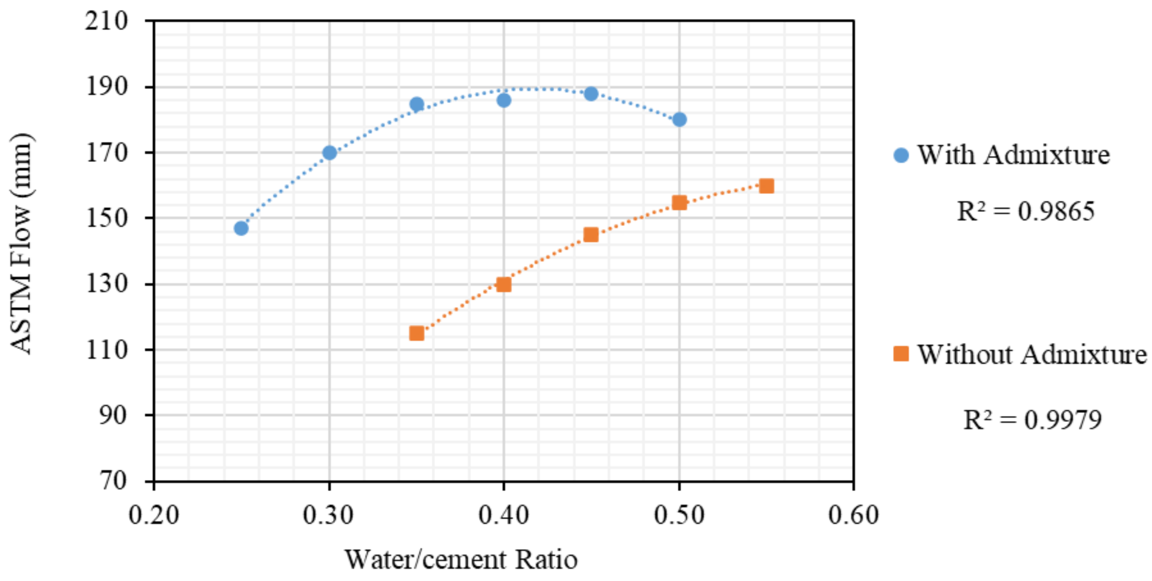


Figure 4-1: Workability results from mixes according to ASTM C1437-15 flow test

All of the mixes were workable enough to be place in the moulds and compacted using a vibrating table. The cubes cast were all weighed in air and under water to determine how close the compaction came to full compaction for the various mixes.

Figure 4-2 shows the comparison between the calculated densities, using the relative densities of the materials compared to the measured densities of all the mixes cast. The trend in the calculated densities is the opposite of the measured densities from the cast mixes, as a slight increase in density was recorded as the w/c ratio decreased. This is to be expected as the relative density for the cement (3.14) is slightly higher than that of the aggregate (2.82) and with the water content kept the same the cement became less as the w/c ratio is increased. The opposite trend is depicted for the measured densities. This trend is to be expected and could be caused by two factors. It is known that admixtures entrain a little bit of air and with the dosage increasing from 0 % in the mix with the w/c ratio of 0.55 to 2.0% in the mix with the w/c ratio of 0.25. In the mixes without admixture the workability reduced as the w/c ratio decreased and that could have resulted in entrapped air preventing full compaction.

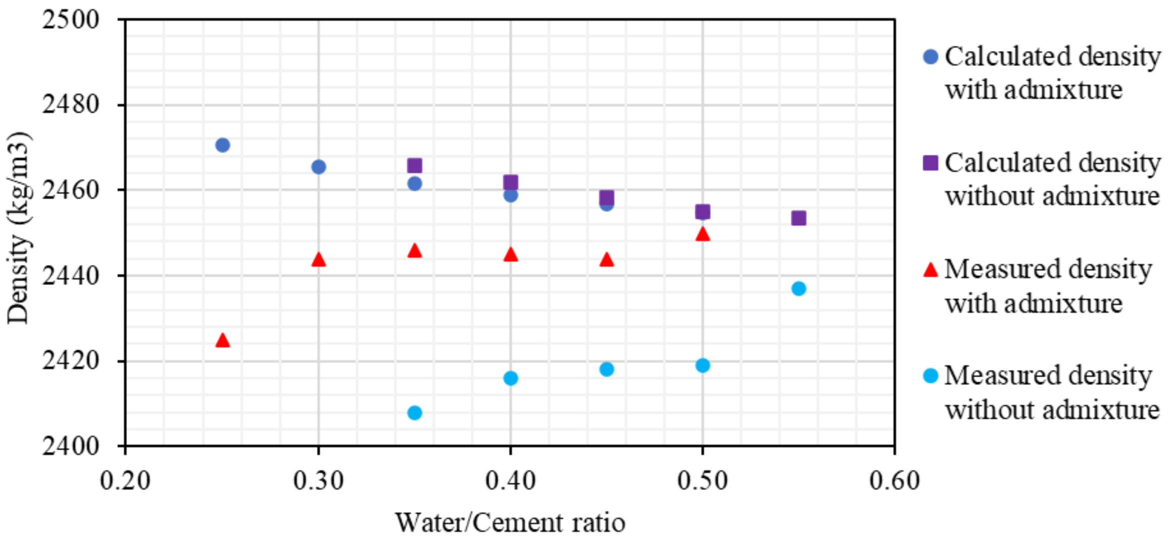


Figure 4-2: Calculated density compared to measured density from all mixes

Table 4-1 shows the results from Figure 4-2 and include the standard deviation calculated from the sample size (number of samples weighed) for each mix.

Table 4-1: Average density per mix, the standard deviation and the sample size

Mix no	Calculated density kg/m^3	Average density kg/m^3	Standard deviation kg/m^3	Sample size
Mix 0.25	2471	2425	21.0	35
Mix 0.30	2465	2444	6.3	40
Mix 0.35	2462	2446	6.0	20
Mix 0.40	2459	2445	11.1	15
Mix 0.45	2457	2444	8.1	15
Mix 0.50	2455	2450	13.0	15
Mix 0.35 no admix	2466	2408	5.1	16
Mix 0.40 no admix	2462	2416	7.0	15
Mix 0.45 no admix	2458	2418	10.2	16
Mix 0.45 no admix	2455	2419	10.6	15
Mix 0.55 no admix	2454	2437	19.1	15

4.3 HEAT OF HYDRATION

The measured heat of hydration shown in Figure 4-3 is for the 1 litre specimen (105 mm diameter by 105 mm high). The admixture percentage is shown in the legend after the mix name.

For all the heat of hydration results, only four different w/c ratio mixes shown in this figure were used to illustrate the trends. The fact that the maximum temperature was reached longer after casting as the w/c ratio reduced can be linked to the increase in superplasticizer as a percentage of the cement in the mixes used. It is a well-known fact that most superplasticizers retard the setting time and then cause the heat of hydration to be at its maximum at a later stage (You, 2019). This fact can be seen in Figure 4-3 with the maximum temperatures shown with a single point on the heat of hydration graph. What is also clear from these results is, that although the superplasticizer retarded the heat of hydration, the maximum temperature was still higher when the w/c ratio was reduced. In the mix design with a constant free water content, it resulted in the cement content to be higher. It is also noticeable that for a constant increase in the w/c ratio, the reduction in maximum temperature is almost constant between 5.5°C and 6.5°C for every 0.1 the w/c ratio was reduced from 0.55 down to 0.25.

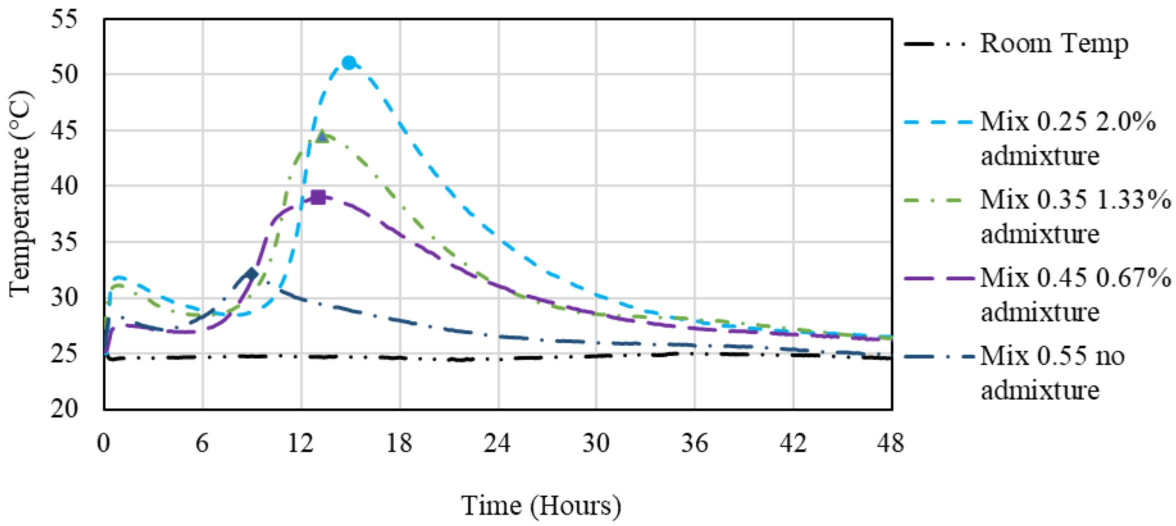


Figure 4-3: First 48-hour temperature graphs for 1 litre specimen

In Table 4-2 the results for the one litre containers of all mixes are shown . Included in the table are the semi adiabatic maximum temperatures as well as times from casting for these temperatures to occur. Although the semi adiabatic temperatures were more than 20ºC higher than the one litre temperatures, it took about 5 hours longer to reach the semi adiabatic maximum temperatures.

Table 4-2: Showing the results and times of the temperatures of the mixes in Figure 4-3

	Semi adiabatic		**One litre**	
Mix no	**Maximum temperature (°C)**	**Time from casting (hours)**	**Maximum temperature (°C)**	**Time from casting (hours)**
Mix 0.25	78.40	19.83	41.10	16.17
Mix 0.30	70.10	20.67	37.90	15.00
Mix 0.35	64.30	18.17	37.20	14.50
Mix 0.40	61.40	19.17	35.10	14.00
Mix 0.45	59.20	17.50	34.40	11.67
Mix 0.50	57.20	16.50	36.00	9.50
Mix 0.35 na	69.50	14.33	40.70	6.67
Mix 0.40 na	65.10	14.33	38.30	7.00
Mix 0.45 na	60.90	15.50	38.10	7.67
Mix 0.50 na	56.70	16.00	36.10	8.83
Mix 0.55 na	58.10	17.00	34.70	8.17

In Figure 4-4 the influence of the admixture is illustrated in the comparison between the same w/c ratio mixes with and without HRWRA (marked mix with na). The influence of the admixture over the first 48 hours when cured in semi-adiabatic conditions can be observed. The inclusion of HRWRA not only delayed the increase in temperature caused by hydration, but also reduced the maximum temperature reached. The data on this graph shows that the maximum temperature between the low w/c ratio mixes differed by about 5ºC and the time to reach that peak temperature differed by about 5 hours. These two differences then reduced as the w/c ratio increased and the mixes at w/c ratio of 0.5 showed almost no difference between the mix with admixture and the one without admixture. The difference in maximum temperature reached could be caused be the reduction in cement content with increased admixture content

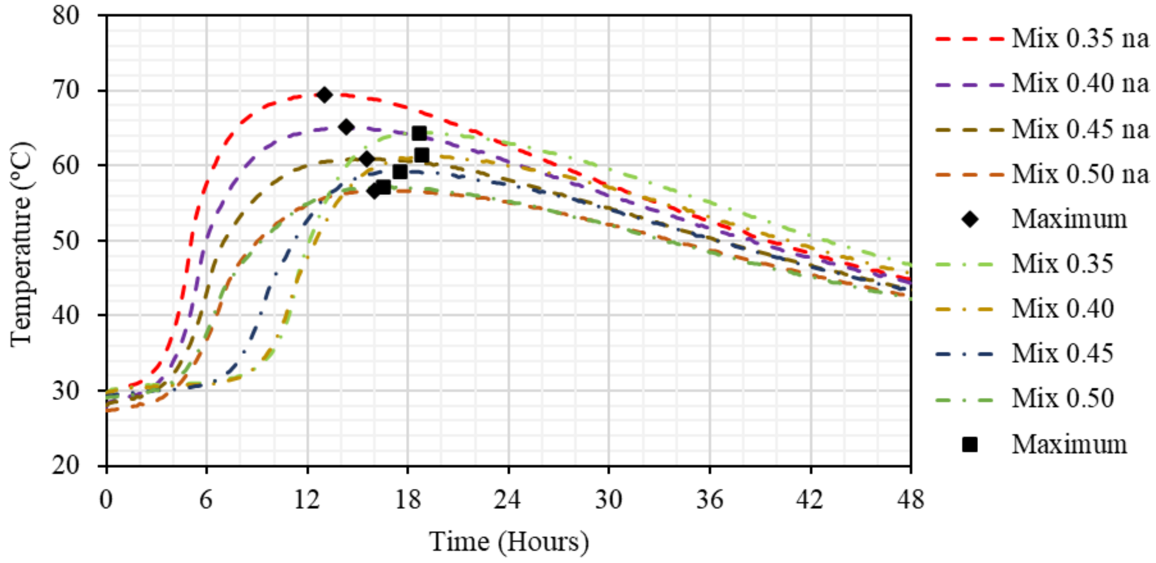


Figure 4-4: First 48-hour temperature graphs for semi-adiabatic specimen.

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4.3.1 Heat of hydration for concrete of different volumes.

The following section on the temperature measured shows the results for different shapes and volumes of specimen. The reason for this part of the research was to be able to correlate the setting time (that will be discussed later) with the type of specimen that was tested to determine concrete properties in the first 48 hours after casting. The 105 mm diameter by 300 mm high cylinders (2.65 litre) were used to measure the early age shrinkage. A 152 mm diameter by 150 mm high (2,7 litre) cylinder is the approximate volume used to measure the setting time of the concrete (ASTM C403-16 uses a 150 mm cube not completely filled). The 105 mm diameter by 105 mm high specimens (0.91litre) were used for the two different types of setting penetration tests and it is also the approximate size of the cubes (1.0 litre) that were tested for strength determination throughout the research. In Figure 4-5 the results for the 0.25 water/cement ratio mix are shown for different volumes of containers of the same material. This mixture was used as it would be one of the mixes where the effect of heat of hydration would be the most noticeable. In this figure the results show that when the volume increased, the temperature also increased and that was the reason why the temperatures were measured for different concrete volumes, because this might have an influence on the setting time. What was interesting from these results, is that the turning points in the graphs are at the same time, although the minimum and maximum temperatures were different for the different volumes of concrete.

In Figure 4-5 the two containers with nearly the same volume of material, the 105 mm diameter by 300 mm high and the 152mm diameter by 150 mm high cylinders gave identical heat of hydration results and only one line is visible on the graph. This was the case with all the different w/c ratio mixes where temperature was measured in different volume containers. This result was a bit confusing because the temperature probe in the one specimen is 52.5 mm from the nearest edge and in the other specimen 76 mm. This might not be significant, but results indicate that the volume of concrete in a mould plays a bigger role in terms of the heat of hydration than the shape of the specimen cast. Further research should be conducted to confirm this observation.

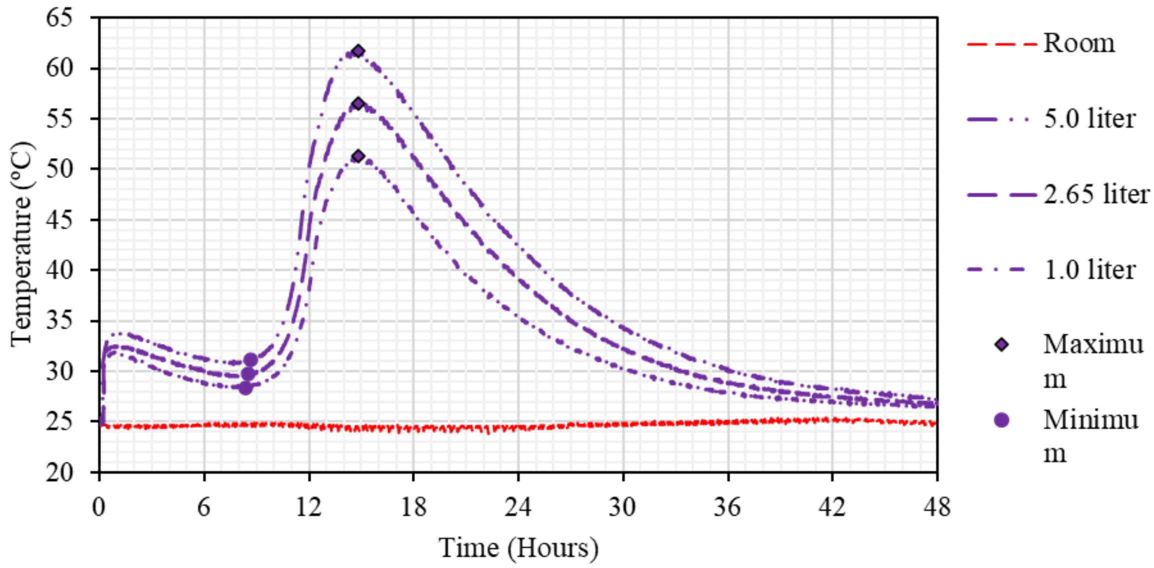

Figure 4-5: First 48-hour temperature graphs mix 0.25 for various size containers

4.3.2 Heat of hydration for concrete cast in different mould material type.

In Figure 4-6 the influence of the mould material on the maximum heat of hydration is shown. This test was conducted to determine whether the material of the container used for the setting time test can have an influence of the measured heat of hydration and perhaps the measured setting time. Again here, similar to the test with the different volumes, it shows that even if the hydration temperature was significantly different between the container types, the maximum heat of hydration stayed at the same point in time from when the water was added to the mix. The steel mould had a significantly lower peak temperature. This could have a negative influence on the strength development of the concrete over the first 24 hours and might have a positive influence on the final 28-day strength. Although this influence was not investigated, it also could have an effect on the final setting time when the ASTM C403 test is used. From the results of the plastic moulds used, there is only a small difference between the single and the double cube mould temperature and that should not have any significant influence on the cube strengths. In the double cube mould, there was no difference between the centre of the cube and the side of the same cube in the mould.

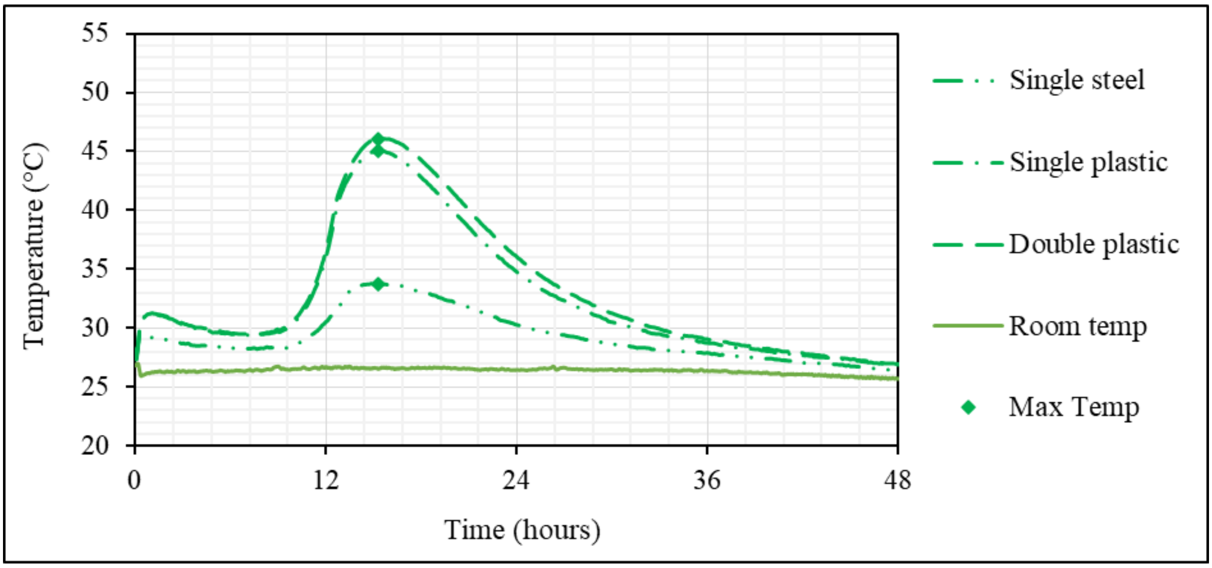

Figure 4-6: Effect of mould material type

4.3.3 Setting time linked to the heat of hydration curve

In Figure 4-7 a typical heat of hydration graph is shown. This graph is based on the results recorded for the mix with a w/c ratio of 0.35 that contained admixture. The period where the plastic shrinkage can take place is during Stages 1 and 2. The initial and final set in this figure is calculated by using 21% and 42% of the total temperature gain of the heat of hydration curve from the baseline (Rolo, 2013, Kopecskó and Baranyi, 2021). The initial and final set is the time corresponding to the temperature gain at those two points. It is however known that the percentages used to determine the temperature, vary between 20 and 25% for initial set and 40 to 50 % for final set (Wang et al. 2015), when the base temperature is the mixing temperature or the room curing temperature and the maximum temperature of the mix is at the end of Stage 3. The determination of the values is linked to the initial and final setting times from the ASTM C403 setting time test.

According to the hydration graph setting takes place in Stage 3 where the temperature change is caused by the heat of hydration and the material is now out of the plastic stage. It should make sense to use the heat of hydration curve to select the time from where the early age shrinkage can be measured, but a standard test method is required.

Kopecskó and Baranyi (2022) used the HoH data and determined the first derivative and called the peak value the final set and the second derivative peak value the initial set. This method produces good results for the final set but the initial set gave erratic answers. The results were compared with the ASTM C403 results. The values Kopecskó and Baranyi (2022) suggested are 25% and 50% of the rise in temperature from the baseline temperature to the maximum temperature of the semi-adiabatic heat of hydration The graph in Figure 4-7 is from the one litre sample for mix 0.35 with HRWRA. The initial and final setting time values are as indicated at 9.1 hours and 11.1 hours after casting respectively in Figure 4-7.

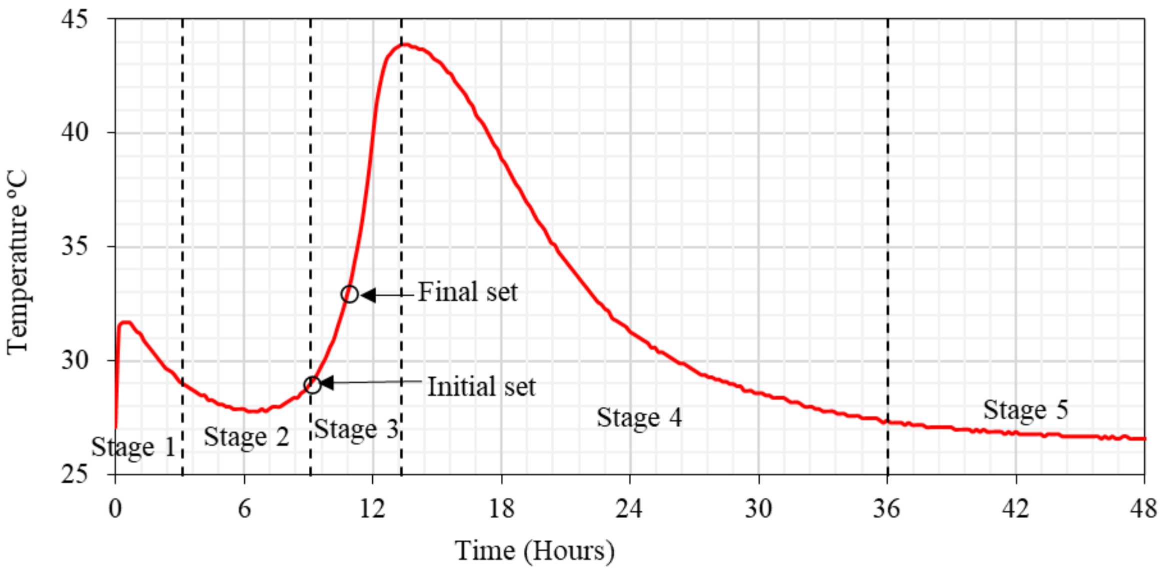

Figure 4-7: Typical heat of hydration curve (Mix 0.35 with admixture)

4.4 EARLY AGE SHRINKAGE TESTS

The early age shrinkage measurement setup can be seen in Figure 3-5 where the cylinder length change was measured. The early age shrinkage data over the first 48-hours from casting is shown in Figure 4-8 for mixes containing admixture. Recording started immediately after casting and the time to set-up the measuring equipment, thus measuring the plastic settlement shrinkage. From this graph and also the graph in Figure 4-9, for mixes with no admixture it became clear that shrinkage cannot accurately be measured from directly after casting. The method used to measure the shrinkage was a 300 mm by 105 mm pipe.

From the two figures, the concrete containing HRWRA gave lower total shrinkage after 48 hours than the mixes without HRWRA. A possible reason could be that the admixture that was used reduced the bleeding and the plastic settlement. The difference in the first six hours between the mixes with and without admixture is clear. The mixes without admixture had a much higher settling, or call it plastic shrinkage, indicating that the admixture changed the early age behaviour in plastic concrete. The exact cause of this influence is beyond the scope of this research.

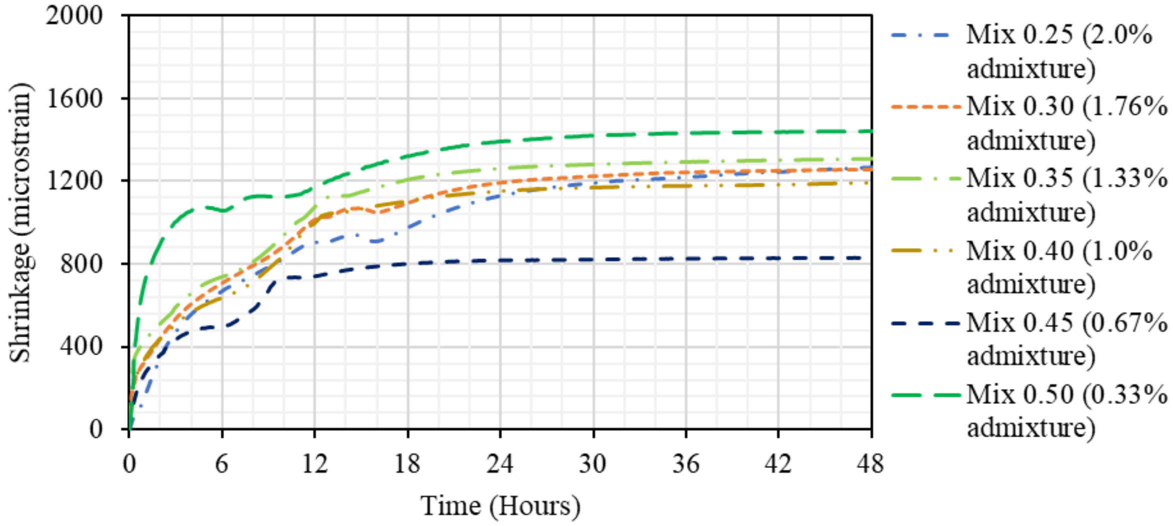

Figure 4-8: First 48-hour shrinkage for various w/c ratios with HRWRA

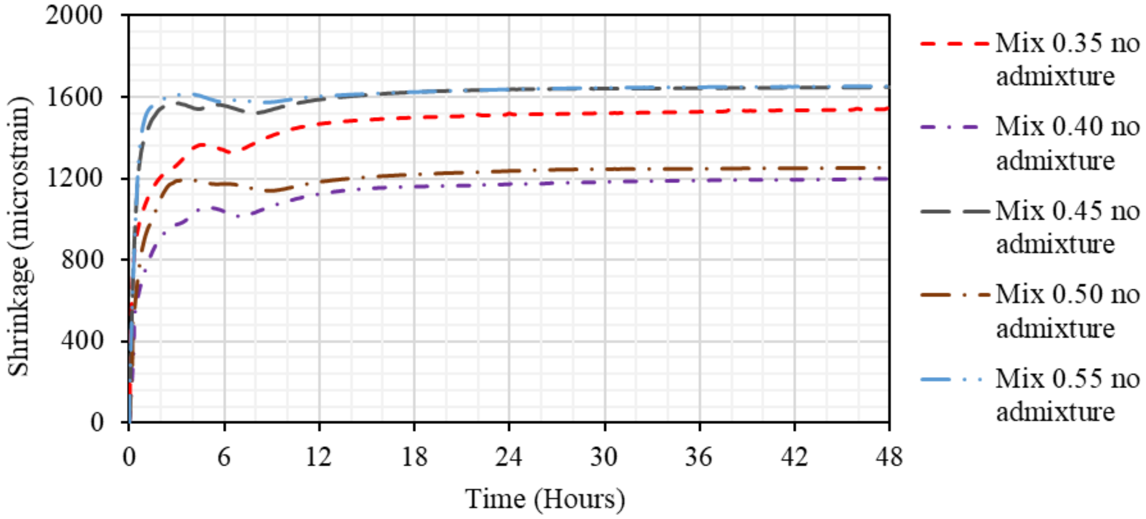

Figure 4-9: First 48-hour shrinkage for various w/c ratios without HRWRA

4.5 RING TESTS

The results from ring testing can be seen in Figure 4-10 for the mixes with admixture and in Figure 4-11 for the mixes without admixture. There are two things clearly visible in the results. The results for all mixes indicated close to zero stress on the ring from the start of the test. Then at a certain point in time the stress started to increase but showed no sudden change as it would for when the concrete would crack. For mixes containing HRWRA lower w/c ratios resulted in higher stresses, but these stresses started developing from a later point in time.

When considering the setting time of the material, based on the current setting time tests that use arbitrary values from a needle test, it seems that the point where stress started developing in the ring test could be a better indicator for when the mixes change from a liquid to a solid that can handle tensile stresses without failing. This test is compared to more results found from other types of tests and will be discussed in more detail in Chapter 5.

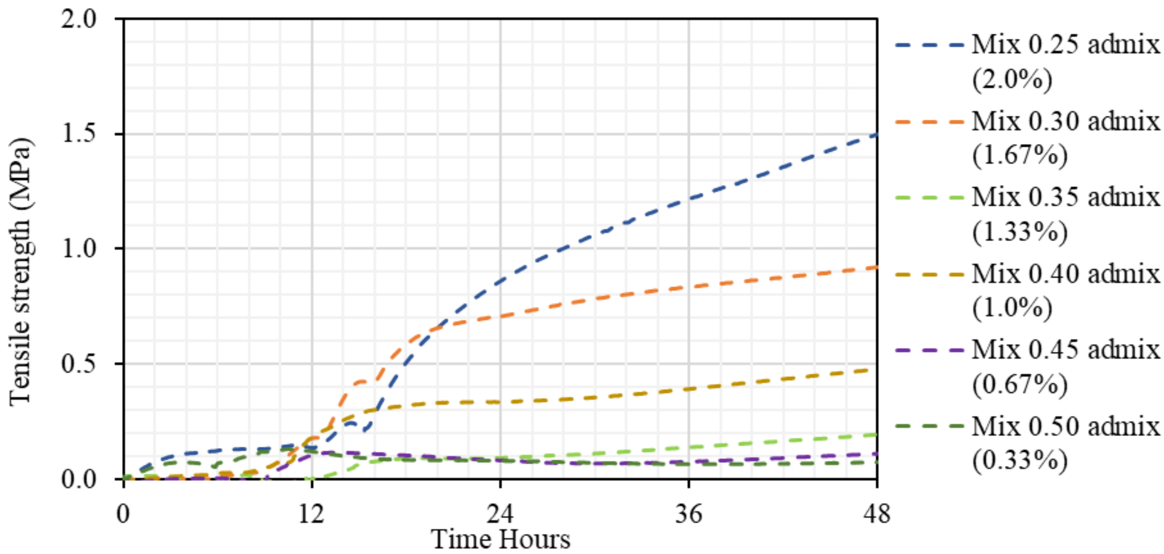

Figure 4-10: All mixes with HRWRA Ring-test results

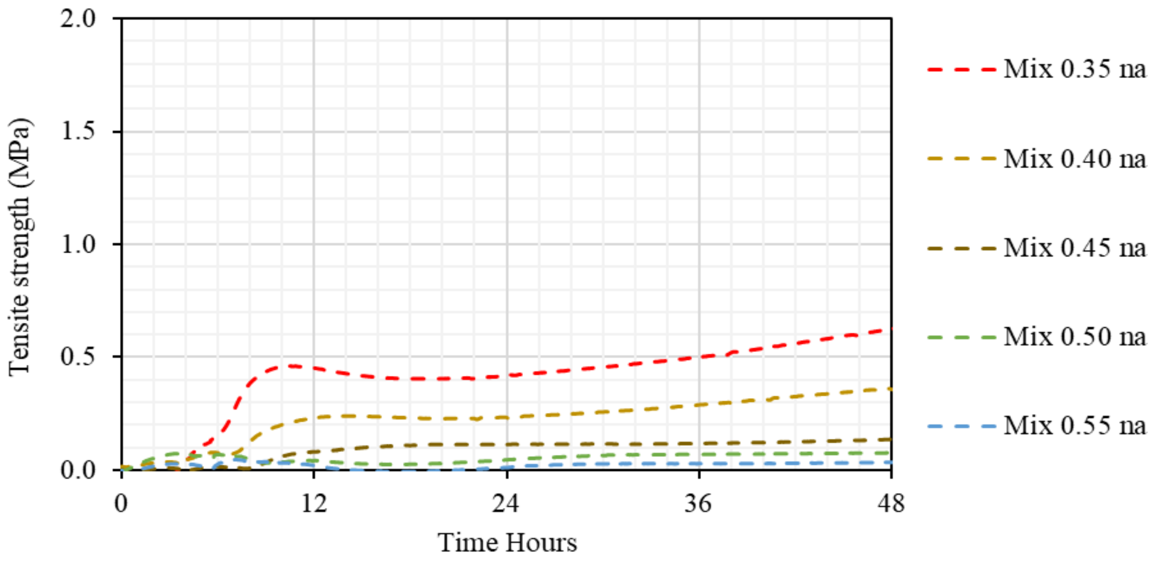

Figure 4-11: All mixes without HRWRA Ring-test results

The ring test was used in the research to see if the high strength concrete cracks when restrained shrinkage is measured. In Figure 4-12 the strain caused by the shrinkage of the concrete on the steel ring is measured by strain gauges on the inside of the steel ring. This strain is then used to calculate the stress on the outside of the steel ring asuming the E-value for steel is 200 GPa. That force is transferred directly to the inside of the concrete by using the dimentions of the steel and concrete specimens. Because the concrete did not crack in the first 48 hours the specimen was kept covered for 8 days, but even then no shrinkage cracking took place. The specimens were then opened and the strain immediately increased and the specimens cracked within 12 hours due to drying shrinkage. The stress at failure was to be 1.9 MPa when the concrete cracked more than 8 days after casting.

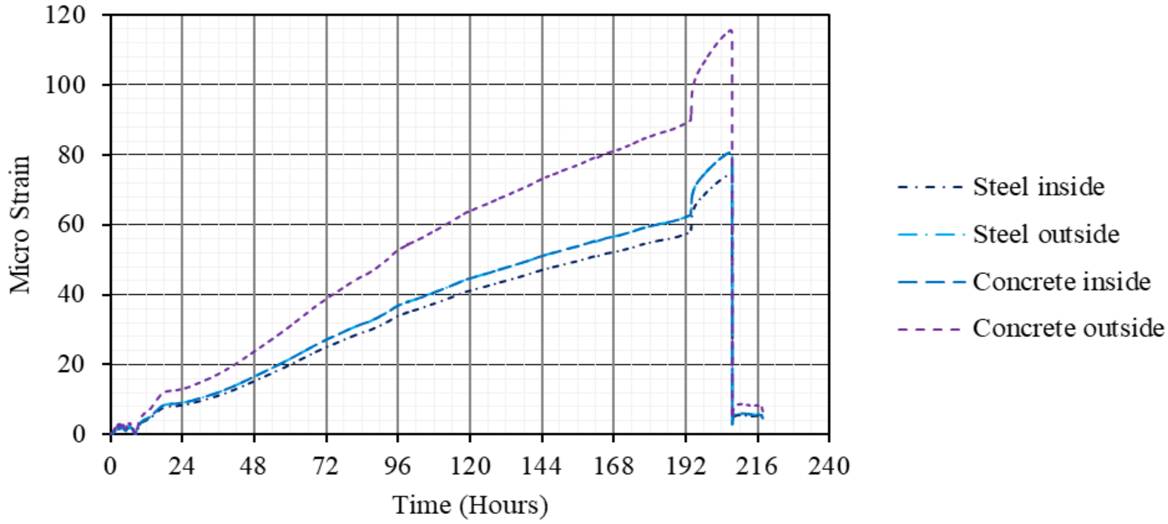

Figure 4-12: Strain measured in ring over time

4.6 SUCTION IN CONCRETE

Neither the measurement of suctions in concrete nor the use of the WP4 instrument is new (Holtz 2001, Slowik et al, 2013, and Tian et al, 2012). The technique of using the chilled mirror dewpoint is also not new for use in concrete (Schoeman, 2020). It is known that the technology works best for low water contents and high suctions. This is a statement made by the manufacturers of the apparatus and this caused the initial suction results for the concrete samples to be variable (WP4 users-manual). Repeated tests were conducted on the 0.25 mix and the results show that there is a clear repeatable pattern. The aim of the project was to find as many properties in the first 48 hours of concrete as possible to help with the determination of the point zero, from where the early age shrinkage can be measured. Where the apparatus seemed to be very helpful, was for the mixes with a w/c ratio below 0.4, where the early age shrinkage became significant. All results in Figure 4-13 are for the Mix 0.25 cast on different days with the sampling cups filled to slightly different volumes. These results show that for the first 12 to 14 hours the suction remained very constant, there after the suction increased at different rates up to about the 30-hour mark and then it flattened off to show the S-shape similar to the curves measured in clay-soils as seen in Figure 4-14. In this graph the turning point of the 6 different mixes that were tested shows a definite significant change in measured suction and the time of the change could be linked to a “knee-point” where the mix property changed or from where the real early age shrinkage could be measured.

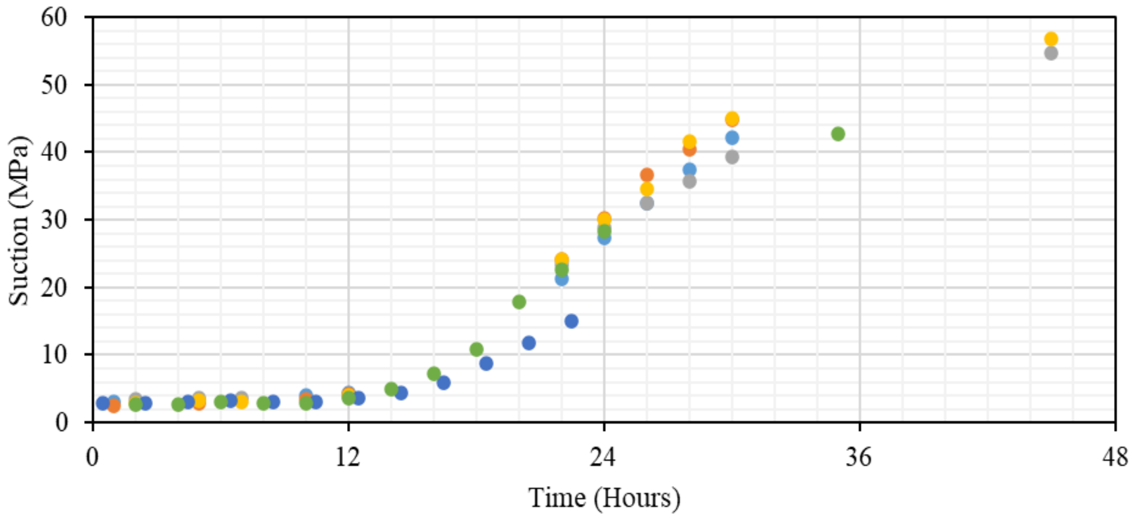

Figure 4-13: Suction for the various 0.25 w/c mixes over time

When the suction measurements on low water cement ratio mixes carried on beyond 48 hours, it showed a similar S-curve pattern than what geotechnical researchers find, linked to the moisture content in the soil (Al Haj & Standing, 2015). This S-shape pattern can be seen in Figure 4-14 where the black line shows the suction as the soil is losing moisture. The dotted line is when the soil is placed back in water, not applicable to this study.

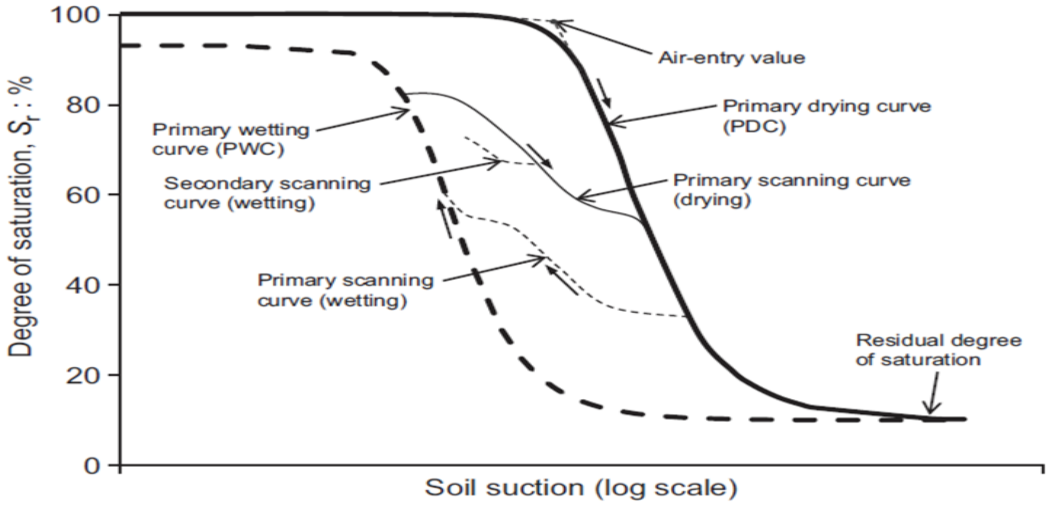

Figure 4-14: Example of a typical soil water retention envelope (Al Haj & Standing, 2015)

The graph in Figure 4-15 was produced using samples from the mix with a w/c ratio of 0.25 in the WP4 machine. In this result a S-shaped graph can be seen and the first turning point is at about 15 hours after casting. This point can also be seen as the air entry time or the point where the humidity in the concrete is less than 98%.

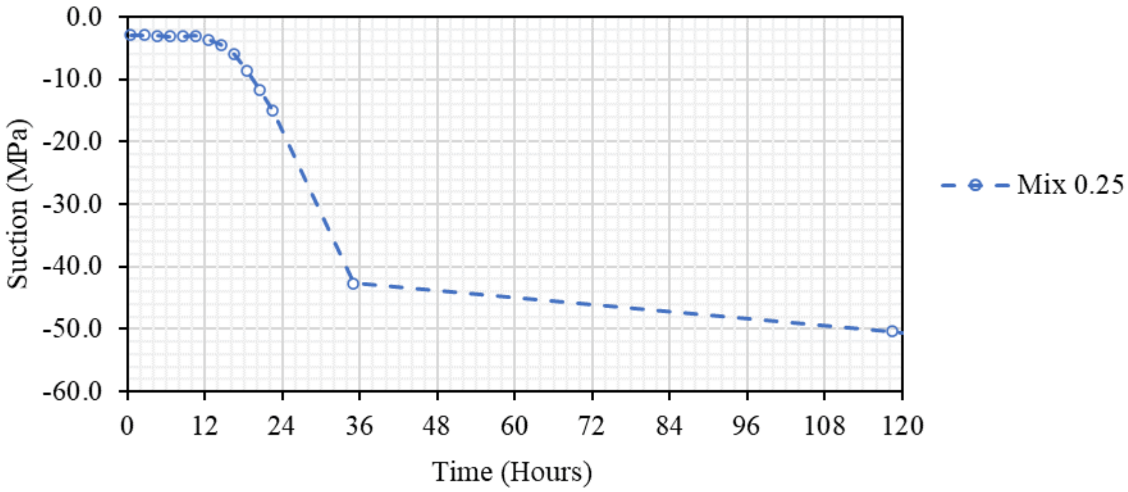

Figure 4-15: Suction for the mix 0.25 measured up to 120 hours

The suction shown in Figure 4-16 was measured for all the original mixes used to determine the workability curve. The early age influence of the different w/c ratio mixes show an interesting pattern. Al the mixes with a w/c ratio higher than 0.4 gave about the same suction at all time intervals. For w/c ratios of 0.4 and lower, the suction increased exponentially as the w/c is decreased. This indicates that the early age shrinkage can possibly be linked to suction measured with the WP4 apparatus. Further research is required to confirm this statement.

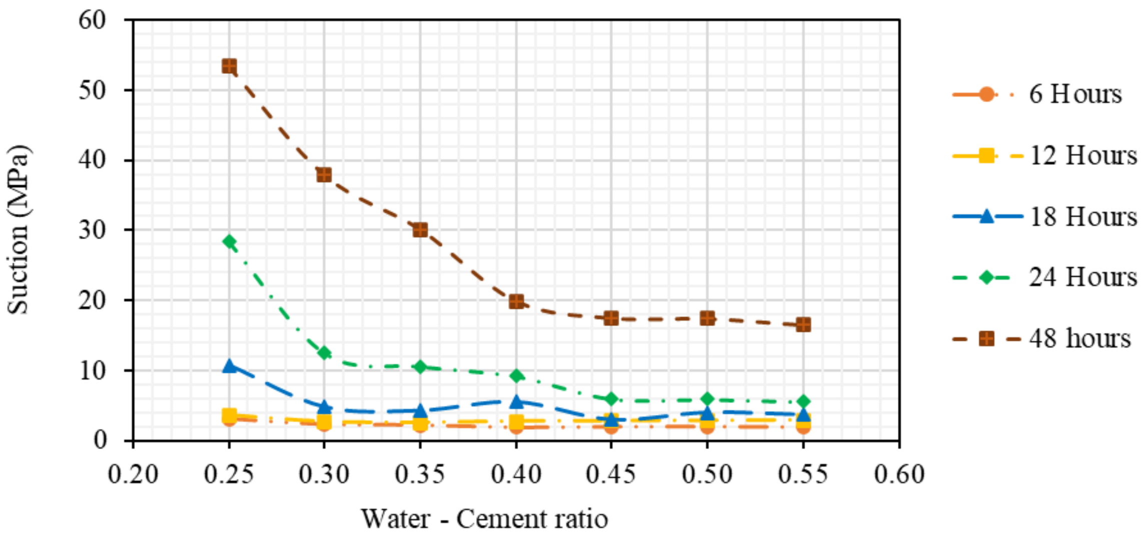

Figure 4-16: Suction comparison for mixes with admixture

For Mix 0.25 to Mix 0.45, with admixtures, it was possible to establish a point in time from where the suction changed from a horizontal line to a line with an incline. The estimated turning point (to the nearest half hour) as shown in Table 4-3 can be seen as a "knee-point". The average suction are the values at the beginning of the suction test by the WP4 machine up to the knee-point. The results indicate that the average suctions were higher for the lower w/c ratio mixes. The results also showed that a fixed suction value cannot be used, because the suction is higher at lower w/c ratios. This could be an indication that the capillary pores are smaller and in future it may be possible to use measured suction values to estimate the pore size.

Table 4-3: Estimated time of turning point for the mixes using the WP4 suction results

Mix no	Estimated time of turning point (hours)	Average suction at start (MPa)	Average suction directly after turning point (MPa)
Mix 0.25	14.7	3.03	5.00
Mix 0.30	11.5	2.29	2.76
Mix 0.35	10.5	2.16	2.64
Mix 0.40	10.0	2.06	2.77
Mix 0.45	8.0	1.78	2.15

4.7 NEEDLE TEST FOR SETTING TIME

Only the ASTM 403C-23 needle setting time test was used in the work because the aim was to get a definite setting time for the concrete when it changes from liquid to solid. The results as shown in Table 4-4 indicate the influence of the HRWRA For mixes containing HRWRA the difference between initial and final setting time is around 2 hours, but for the mixes without admixture the initial setting time is very early and the time span from initial to final setting time is noticeably higher at around 3 hours to 4 hours. The setting times for the mixes with admixture decreased with increased w/c ratio and for the mixes without admixture the trend is the opposite.

Table 4-4: ASTM 403-23 initial and final setting times using various diameter needles

Concrete mixes with admixture				Concrete mixes without admixture		
Mix no	Admixture as % of cement	Initial set (hours)	Final set (hours)	Mix no	Initial set (hours	Final set (hours)
Mix 0.25	2.00%	9.0	11.0	Mix 0.35 na	2.0	5.2
Mix 0.30	1.67%	7.9	10.3	Mix 0.40 na	2.5	6.3
Mix 0.35	1.33%	6.8	9.6	Mix 0.45 na	3.1	7.1
Mix 0.40	1.00%	6.6	8.9	Mix 0.50 na	3.7	7.5
Mix 0.45	0.67%	6.5	8.7	Mix 0.55 na	4.5	7.6
Mix 0.50	0.33%	6.4	7.8			

4.8 CONE PENETRATION TEST FOR DETERMINING A CHANGE IN SLOPE

Figure 4-17 indicates the shear resistance experienced by the cone with increasing penetration depth. The load recorded was divided by the cone surface contact area to calculate the shear resistance. The shear resistance increased with time, but not with penetration depth. At any given time after casting the shear resistance was fairly constant for penetration depths exceeding 25 mm and thus the shear strength was calculated as the average penetration resistance recorded between 25 mm and 40 mm.

In Figure 4-18 the cone penetration results are shown for mixes with w/c ratios varying from 0.25 to 0.55. The values used are the penetration force in kN divided by the area of the cone in contact with the concrete from the penetration depth of 25 mm to the penetration depth of 40 mm. This is the range of the penetration test where the result gives an almost constant shear force in MPa. Up to 20 mm penetration, the coarse aggregate in the mix had too much of an influence on the calculated strength and from 40 mm further the confinement of the mould started having an effect. The cone penetration test can also be stopped at 25 mm penetration as for the ASTM C403 test. Resistance can then be calculated at that point by dividing that force by the area of the cone in contact with the concrete, because the shear strength is constant at higher penetration depths.

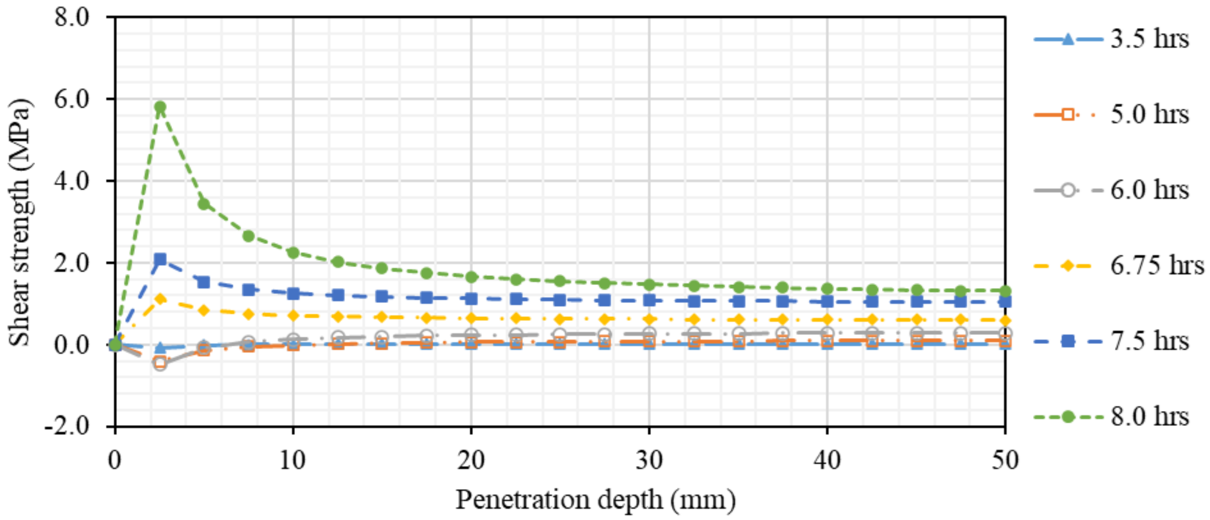

Figure 4-17: The results of the cone penetrating tests for mix 0.40 without HRWRA

These results clearly show a change in the slope, called a "knee-point" by some researchers. The "knee point" is where the linear slope, indicating the rate of strength development with time, suddenly increased significantly. This indicates rapid strength gain, which could be associated with the material changing from a Bingham flued to a solid. It is worth noting that this knee-point values varied significantly with w/c ratios and HRWRA dosage as can be seen in Figure 4-18.

The knee point moved on with time as the HRWRA dosage was increased in percentage to keep the workability of the mixes approximately constant. In Figure 4-18 the mix with the 0.55 w/c ratio show an immediate increase in shear strength from 5 hours onwards, indicating that the test started too late and the knee-point set was reached within 5 hours of casting. The mixes with w/c ratios of 0.45 and 0.35 show a flat line part, where after the knee-point set was reached At different times and the shear strength increased drastically but still at a constant rate. Although the mix with w/c ratio of 0.25 had the lowest w/c ratio, it contained 2% HRWRA and for the duration of the test period no change in slope was reached, indicating significant set retardation. The supplier of the HRWRA recommended a dosage of between 0.5% and 1.5% of the cement content, warning in their specification that using less would not make the admixture work at its full potential and using more could lead to set retardation.

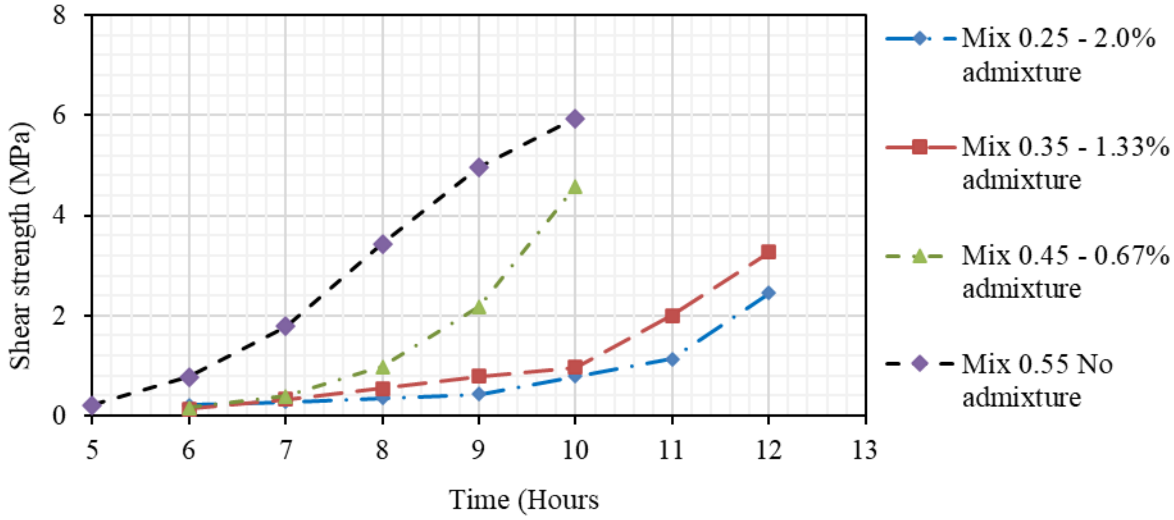

Figure 4-18: Cone penetration test results for mixes 0.25 – 0.55

In Figure 4-19 the results of a mix with a w/c ratio of 0.4 but without admixtures is shown, where the water content was increased from 225 to 295 litre per cubic meter, to give a mix with the same workability of 185 mm flow. With the comparison between the results of the two different test methods, the ASTM method gave a shear strength of about 12.5 MPa at the time when the cone shear strength showed the knee point and with this high-water content the shear strength of the mix was calculated from the cone penetration as 0.21 MPa.

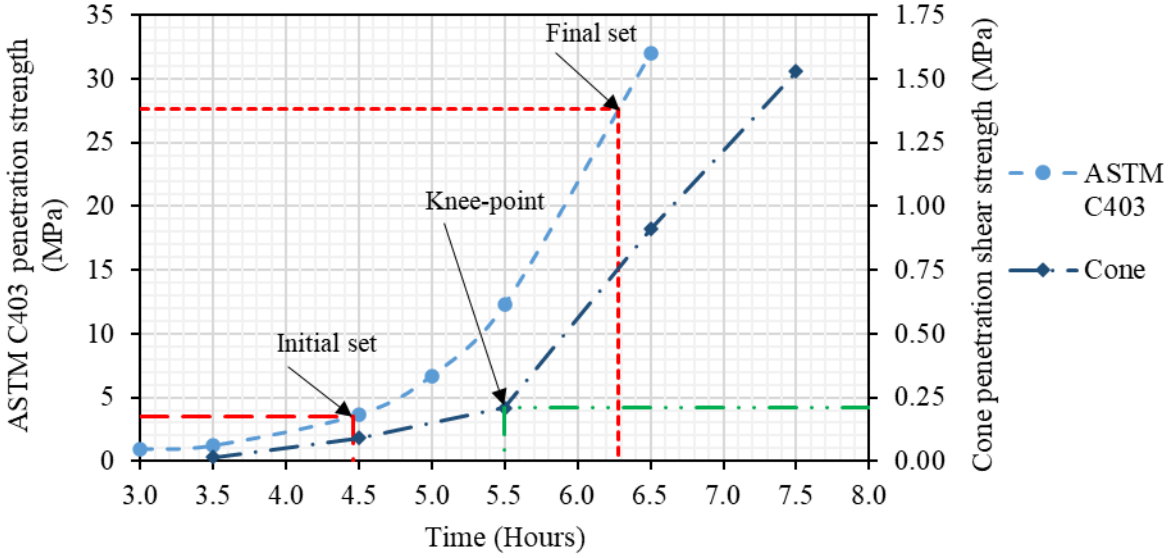

Figure 4-19: Results of setting time with cone and ASTM C403 needles.

The results in Table 4-5 are for the mix with a w/c ratio of 0.50 and no admixture using ASTM C403 and the cone penetration test as shown in Figure 4-19.

Table 4-5: Iinitial and final set times and knee-point

ASTM C403-16	Initial set	Final set
Time (hours)	4.46	6.28
Cone penatration	Cone penatration	
Time (hours)	5.50	

4.9 COMPRESSIVE STRENGTH

Designers makes use of concrete compressive strength and therefor it was important to conduct strength development testing over the first 48 hours to get a result chart for all the mixes used in this research. In Figure 4-20 the results of the mix with w/c ratio of 0.25 and 2% HRWR is shown. It is clear that there is a definite knee-point visible in the compressive strength. The strength tests were done every 2 hours and the results show that the knee point was in the region of 12 hours, with the compressive strength increasing to more than 60 MPa within 24 hours of casting.

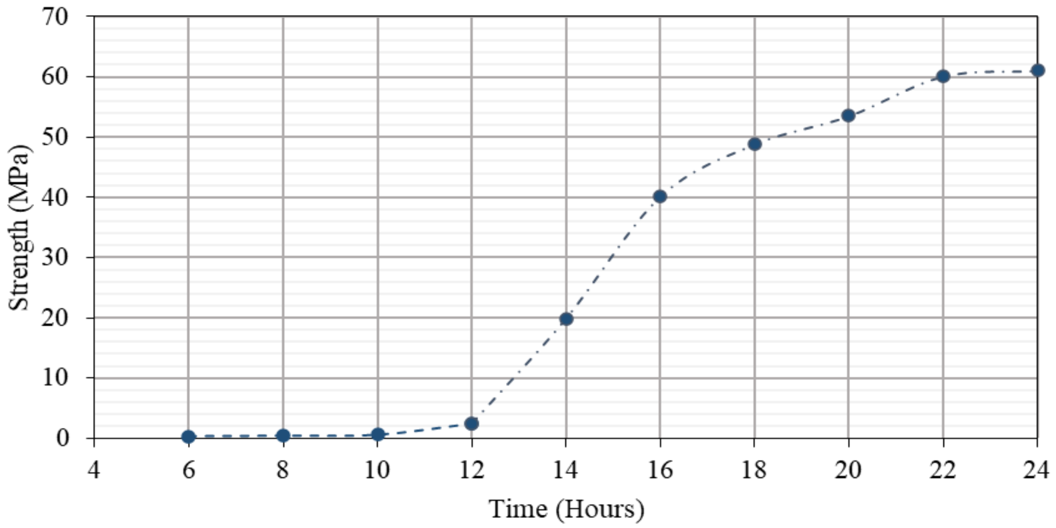

Figure 4-20: Cube results from mix with w/c ratio of 0.25

In Figure 4-21 the behaviour of the mixes with admixture up to mix 0.50 are shown. These results show the retarding effect of the HRWRA with the knee point of the mix with w/c ratio of 0.25 around the 12-hour mark with the mix with the 0.35 w/c ratio but less HRWRA at about 10 hours. As with the suction results, for the two mixes with a w/c ratio above 0.4 it is not 100% clear where the knee point is. In the graph the retardation effect was reversed at between 13 and 15 hours where the retarding effect of the HRWRA disappeared and the benefit of including HRWRA can be seen. After 18 hours all the strengths showed the performance expected for the different w/c ratios.

Strength gain of mixes without admixture can be seen in Figure 4-22. There is no cross-over of the strength gained in the first 48 hours. The 24-hour strength of the mixes are very similar for the two sets of mixes with the same w/c ratios. From 24 to 48 hours the mixes with the higher admixture dosage gain almost 10 MPa where the mixes without admixture gained very little strength.

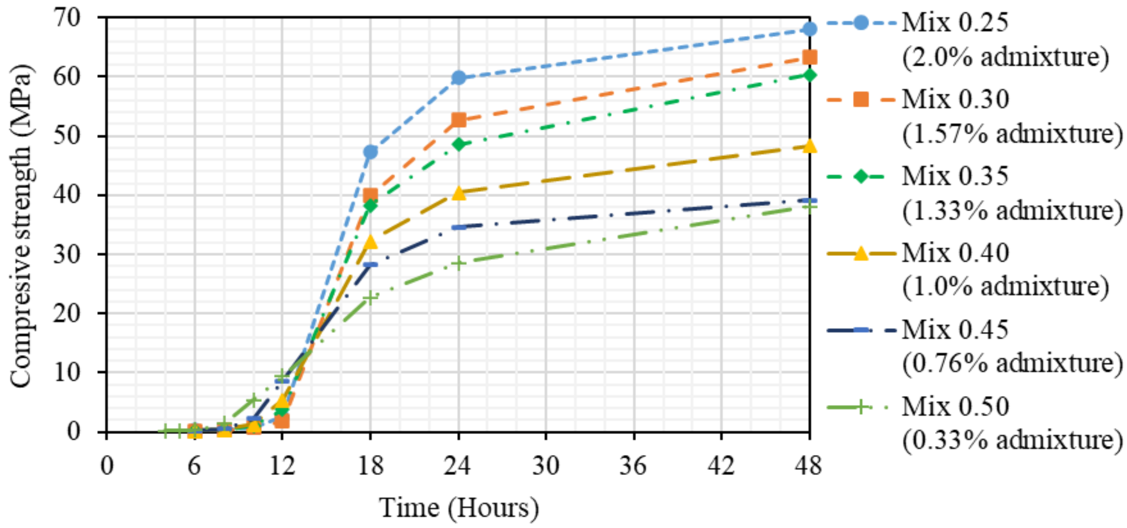

Figure 4-21: Concrete with admixture strength gain over the first 48 hours

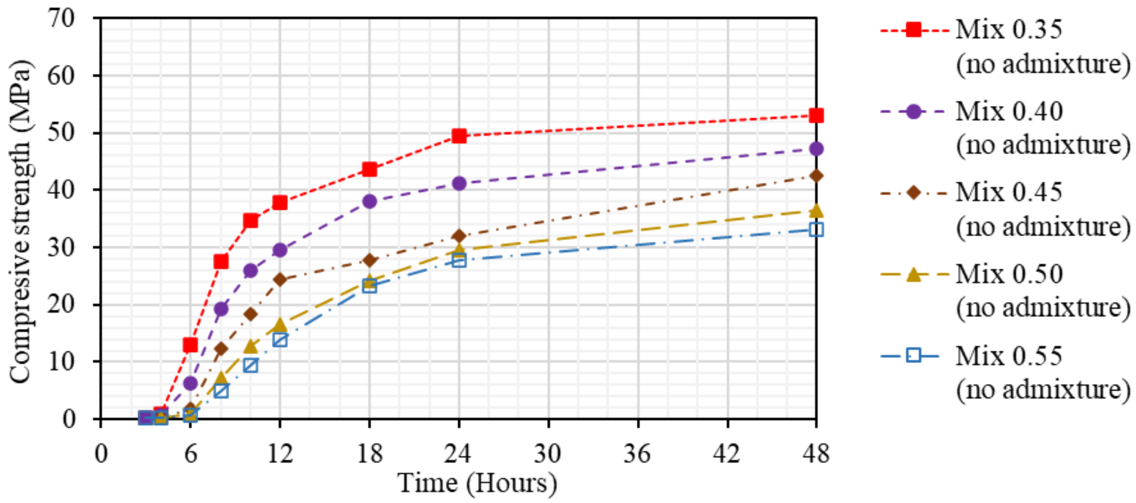

Figure 4-22: Concrete without admixture strength gain over the first 48 hours

Most of the early age tests are normally conducted 24 hours after casting and then the retardation effect causing the difference in early behaviour between the mixes with and without admixture would remain undetected. These difference in cube strength development for the various mixes with and without admixture clearly indicate that the use of a fixed time for time-zero is wrong.

The importance of mixing, casting and curing at different temperatures is shown in Figure 4-23. The results are from three mixes with the same free water content. The result shows the influence of the overnight storage of the material and the mixer drum, that differed by 5ºC on the early age strength development. After the mixing took place, the cubes were stored in the same room at 25ºC till the testing at the various times took place. This temperature difference changed the setting time by almost 2 hours. This was deliberately done to show the importance of keeping conditions the same. This graph also indicates that a concrete mix with the same w/c ratio, but without admixture, would gain strength noticeably earlier than when HRWRA is used. It is worth noting that the delay in strength development resulted in an additional 10% strength increase for the mixture containing HRWRA after 24 hours.

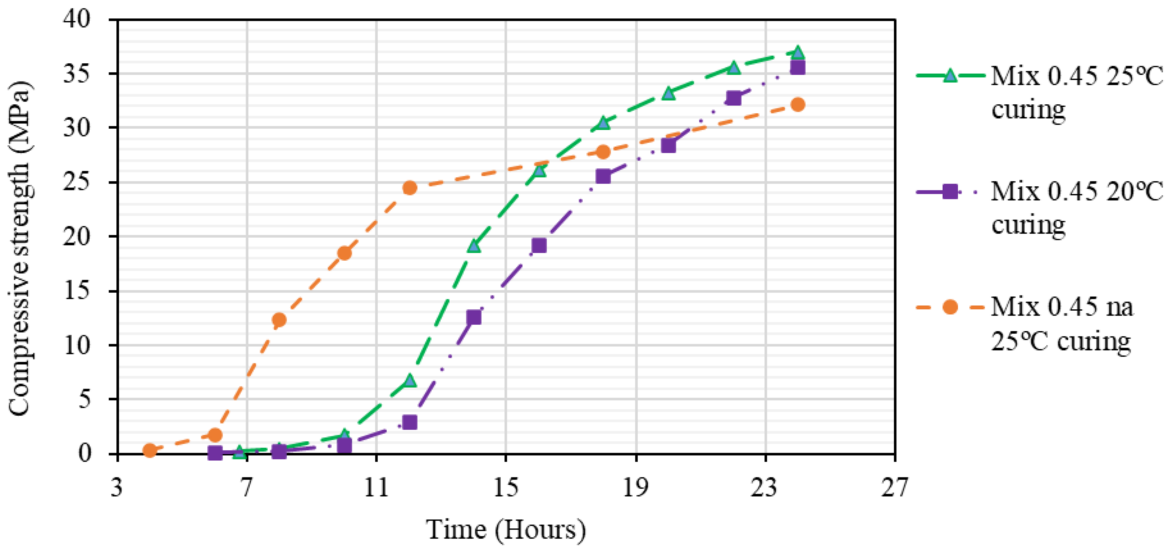

Figure 4-23: Mix 0.45 different mixing temperatures and the same mix without admixture

4.10 DIRECT AND INDIRECT TENSILE STRENGTH

There is no standard test method for direct strength testing of hardened concrete because of, not only the difficulty of gripping onto concrete and then pulling the concrete apart, but also the problem of ensuring that the specimen is axially loaded through the centre of the specimen.

There are a few methods suggested by casting concrete in specially shaped moulds. I beam, or dog-bone shaped specimen are used or cylinders are glued to metal heads that can be grabbed in a tensile testing machine. All these methods need skilled technicians to ensure useable results.

The tensile strength of concrete is mostly tested indirectly by two different methods. The split cylinder (SANS 6253:2006) and the Modules of Rupture (MoR) beam test (SANS 5864:2006) Both these tests have their own relationship to the compressive strength of the concrete. The split cylinder is close to 7% of the compressive strength and the MoR is close to 13% of the compressive strength (Osama et al. 2016, Alexander, 2021). Both these tests are usually only used at 28 days, giving no indication of early age properties of concrete. It is also not clear if it would be possible to do these tests at ages close to setting time.

During this research, split cylinder testing was also conducted at different early ages for the various mixes to determine the indirect tensile strength and compare that with the results from the direct tensile strength at early ages.

In Figure 4-23 the results of the original four mixes can be seen where the direct tensile tests were done as described in Chapter 3. The split cylinder strength results are shown in Figure 4-24.

The direct tensile strength for concrete is known to be about 5% to 10% less than the split cylinder or the MoR beams strengths (Wu et al., 2023). From the results in Figure 4-23 and Figure 4-24 it can be seen that at early age the direct tensile strength is higher than the split cylinder strength. It is only the 0.55 w/c ratio mix where the split cylinder strength is higher than the direct tensile strength after 8 hours and after 12 hours the mix 0.45 also gives a higher split strength. The relatively high direct tensile strength at early ages could be caused by suctions.

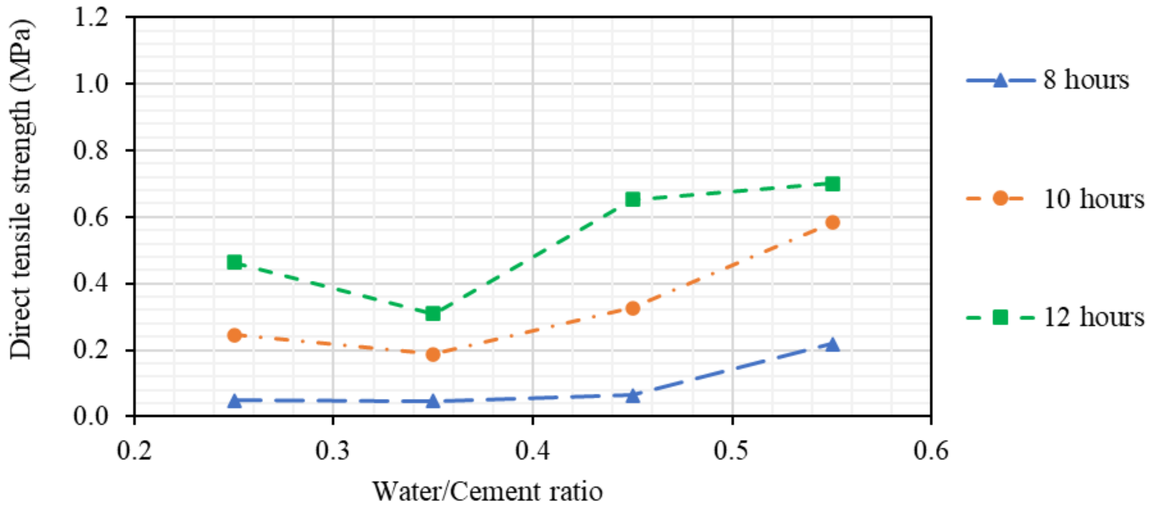

Figure 4-23: The direct tensile strength of mixes with HRWRA

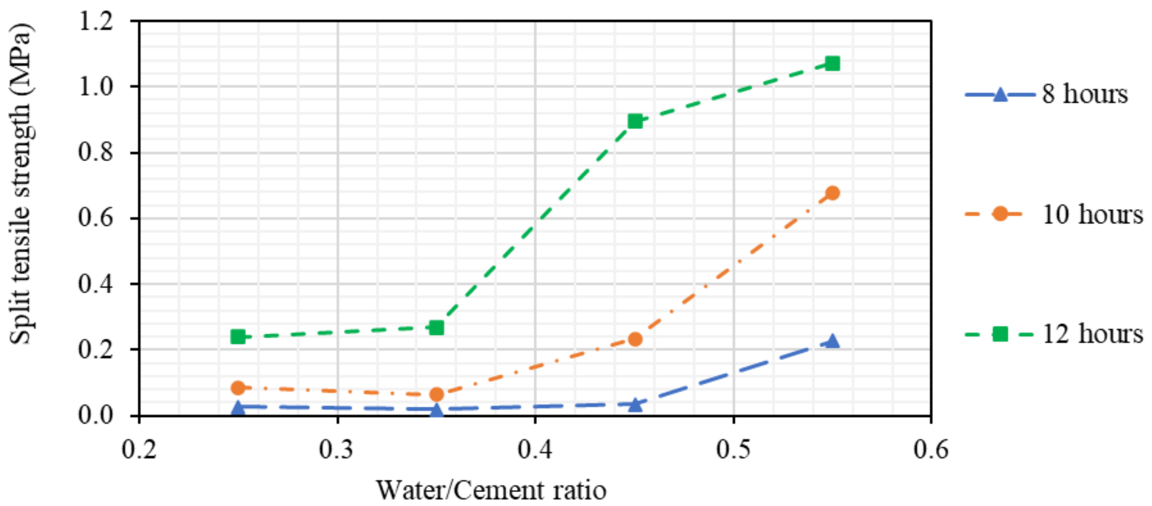

Figure 4-24: The split cylinder strength of mixes with HRWRA

In Figure 4-25 the split cylinder results for the mixes that were cast containing HRWRA are shown. A similar pattern as with the cube strengths show that the very early age strength of the low w/c ratio mixes were weaker than the higher w/c ratio mixes, but from about 18 hours to 30 hours the strengths revert back to what is expected, where the low w/c ratio mixes give the higher strengths.

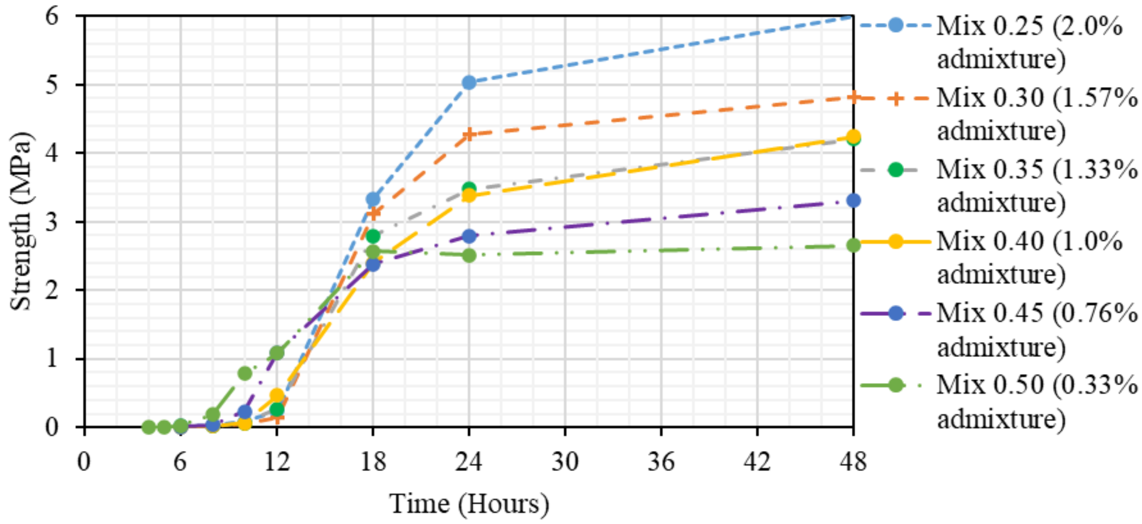

Figure 4-25: The split cylinder strengths at various time for the mixes cast with admixture

In Figure 4-26 the split cylinder results of the mixes without admixture are shown. The results show that the low w/c ratio mixes give a higher split cylinder strength from the start and it was higher than, that of the mixes with admixture shown in Figure 4-26. After 24 hours the results were however lower than for the same w/c ratio mixes containing HRWRA.

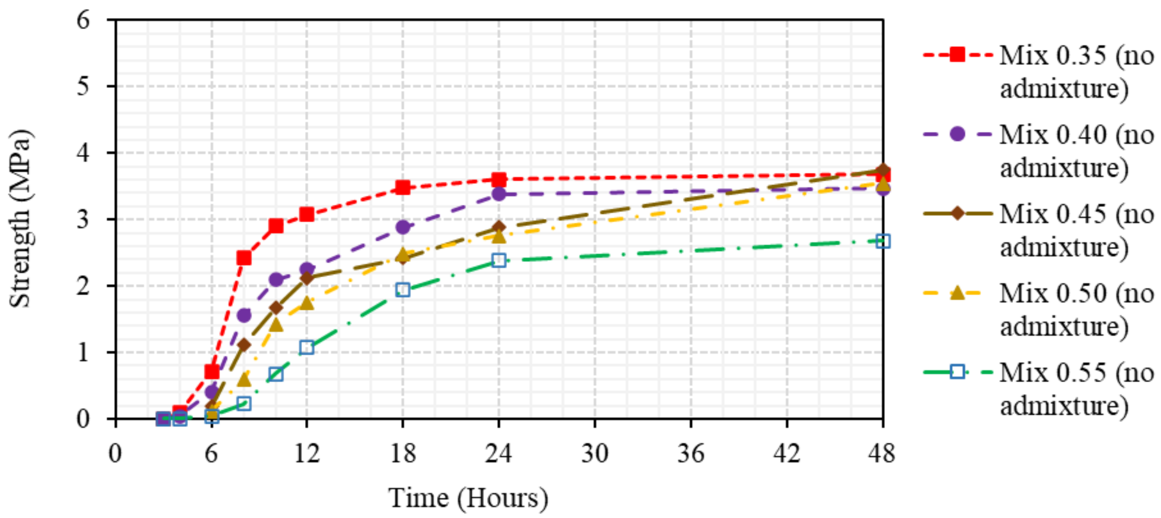

Figure 4-26: The split cylinder strengths at various times for the mixes cast without admixture

4.11 MODULUS OF ELASTICITY

The cylinders that were used to perform modulus of elasticity testing, were cast from the same mix that the strength gain cubes were cast from. The tests were done on different testing machines because of the low strength of the 10-hour and 12-hour cylinders. The cube strength at that time was used and the 40% strength was calculated by converting the cube strength to a cylinder strength (by multiplying strengths with 0.8). Three loading cycles were applied to each cylinder and the last cycle was used to calculate the E-value. The cylinder was then crushed to determine the strain at the maximum load reached at the different testing strains. For the 24-hour test the maximum load was reached just before the 0.004 strain which is very close to the 0.0035 that engineers use as the deformation limit in the design of structures. The 12-hour strain was at about 0.005 and the 10-hour test at about 0.011 strain at maximum stress. This pattern was followed for all the modulus of elasticity tests conducted for the different w/c ratio mixes. For the higher w/c ratio mixes it was also possible to do an 8-hour test as a result of the limited effect of the lower dosage of HRWRA time-zero has already occurred.

Figure 4-27 and Figure 4-28 shows the stress strain behaviour of mixtures containing HRWRA 12 hours and 24 hours after casting respectively. For Mix 0.55 no admixture was used, resulting in the highest stiffness at 12 hours and the lowest stiffness at 24 hours. Increasing HRWRA content resulted in reduced strength and stiffness 12 hours after casting, but the trend was reversed 24 hours after casting.

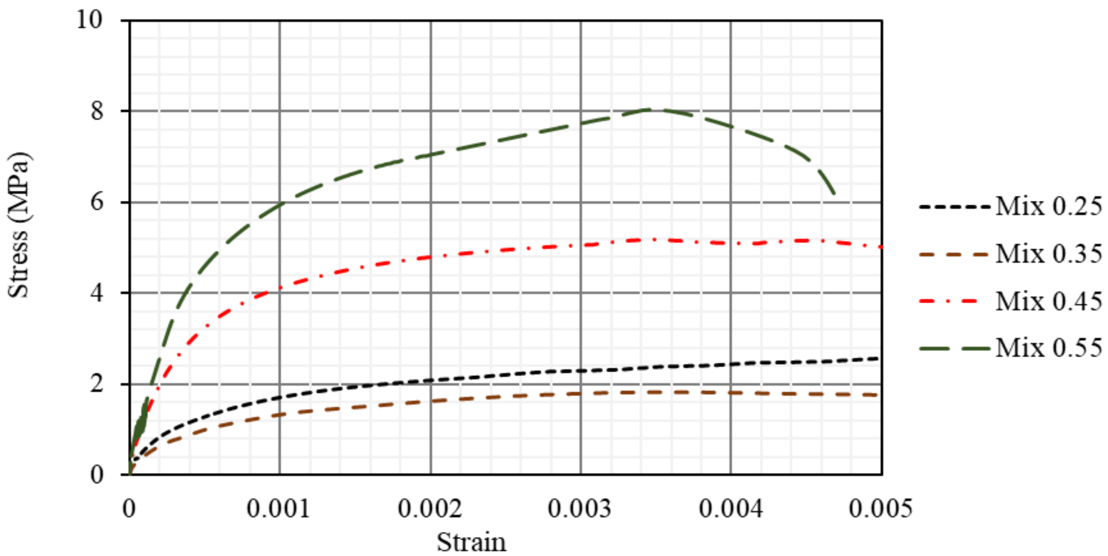

Figure 4-27: The 12-hour stiffness results

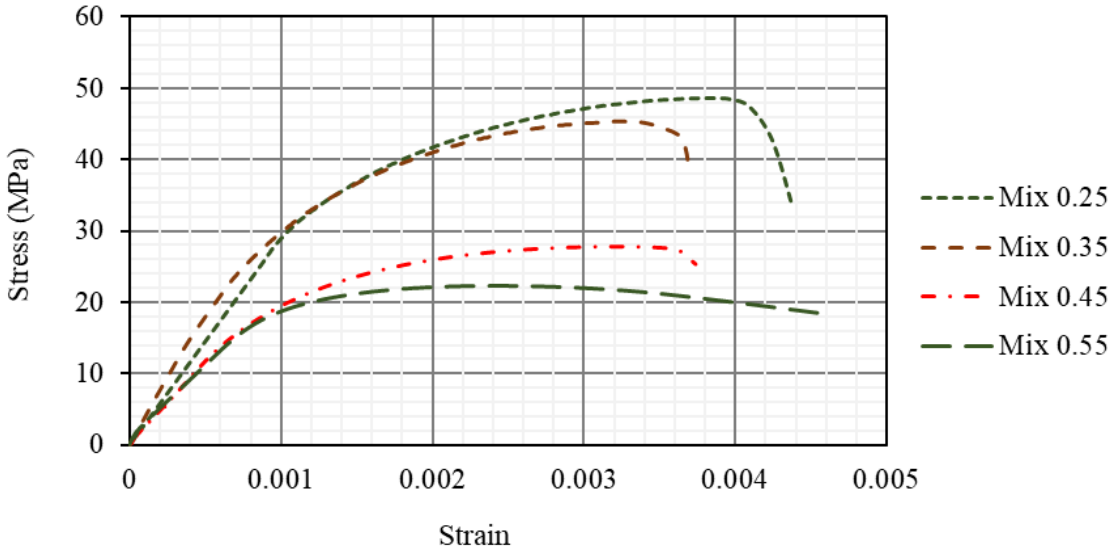

Figure 4-28: The 24-hour stiffness results

Table 4-5 shows results for the MoE determined from Figure 4-27 and Figure 4-28, as well as the strengths of the mixes from cubes and the cylinders used to do the stiffness tests. The 12-hour cylinder strengths are higher than the cube strengths at the low w/c ratio mixes, but as expected after 24 hours the pattern for all mixes is reversed. A similar tendency was observed between the direct and indirect tensile strength where the early age direct tension was higher than the indirect tensile strength for the lower w/c ratio mixes.

Table 4-5: Strength and E-values at 12 and 24 hours for various mixes

Time of test	Stiffness	Cube Strength	Cylinder strength
Hours	GPa	MPa	MPa
Mix 0.25			
12	2.33	2.42	2.70
24	34.4	58.9	48.7
Mix 0.35			
12	2.00	1.19	1.82
24	27.7	45.7	41.4
Mix 0.45			
12	8.40	6.78	5.18
24	25.5	35.6	33.9
Mix 0.55			
12	10.5	8.83	8.03
24	23.4	24.4	22.3

4.12 SUMMARY

To achieve the aim of the study the water content in all the mixes were kept the same at a water content that would be reasonable for a concrete mix with a maximum aggregate size of 5 mm. The intention was to have a similar workability for all mixes that could be compacted without having segregation or bleeding. When high strength concrete is designed it will never be without admixture. Therefore, the mix designs were started at the highest w/c ratioof 0.55 without admixture and worked down to the lowest w/c ratio of 0.25. The steps of increase in admixture were kept constant between the different w/c ratios to make sure that trends that develop should have a fixed pattern.

From the different types of tests conducted on the w/c ratio mixes it became clear that there is a different pattern between mixes with and without admixture for the same w/c ratio. The second observation was that the behaviour of the early age properties was different for mixes below a w/c ratio of 0.4 than for the mixes above 0.4 w/c ratio.

The volume of the specimen and the material type of the mould can also skew results for the heat of hydration and the setting time. Setting time is the time from where early age shrinkage should be measured to make results comparable all over the world.

The ring test also showed much lower stresses in the concrete than what the direct tensile strength was for the mixes that were tested for the lower w/c ratios. This indicates that early age shrinkage (chemical and autogenous) would not cause high strength concrete cracking.

A clear knee point was observed in the ring test, suction test, cone penetration test and the cube compressive test. The results obtained from the different early age tests conducted are compared in Chapter 5.

5 EARLY AGE PROPERTIES AND SETTING TIMES

5.1 INTRODUCTION

From Chapter 4 it became clear that until consensus is reached on what type of test to use to determine setting time, with a constant mould size and material type that is cast and cured under specific conditions, it will be impossible to compare results for deformation of concrete at early ages. The point in time from where the measurement of the deformation should start is, as shown in the literature review, still a point of debate.

From the literature it became clear that from mixing and placing there are four types of shrinkages that should be measured at early age. Plastic settlement shrinkage can only take place when the concrete is still a Bingham fluid and can only have an influence before final set. The second is chemical shrinkage that starts immediately after the cement gets in contact with the water. Chemical shrinkage is caused by the hydration process where the reaction product is smaller than the original products. There are test methods to determine chemical shrinkage, but the use of different materials and different volumes of material give different results. The third, autogenous shrinkage is part of the hydration reaction and starts when the free water is used up and the re-hydration of bound water causes suction in the capillary pores. Up to this point the chemical and autogenous shrinkage curves follow the same line and the point from where the two types of shrinkage take place at different rates is called time "Zero" (Sant et al., 2006). More than one test method is used, giving different results and different times from where the shrinkage measurements should be taken. The last deformation is cause by heat and the material expansion coefficient of each material and the heat of hydration plays a role in this deformation. Any temperature changes that take place in the concrete after setting will affect the strains measured and the effect of heat generated during hydration has to be taken into account in any early age measurements.

5.2 SETTING TIME RESULTS

The setting time test results were split into two-point and single point or knee point setting times. The results that were determined using the ASTM C403-23 provided initial and a final setting time. The fraction method calculating the first and second derivative from the heat of hydration results also giving two results, claiming to be initial and final set results. The use of the UPV also seems to give a result graph that could be seen as an initial and final set result but this test method was not part of this programme. The single point setting times were determined by measuring early age concrete properties at regular time intervals with the aim of finding a single turning point in time from where a change in properties occurred. In this chapter the trends observed for two-point and single point setting time tests are compared.

5.2.1 Two-point setting times

The standard setting time test used all over the world for concrete is the ASTM C403-16. The test results produce an initial and final setting time result for material smaller than 4.75 mm. In Figure 5-1 the initial and final set results from the various w/c ratio mixes with and without admixture are shown. The results confirm the fact that HRWRA retard the setting of concrete. For the research the admixture percentages as indicated in Table 4.2 was increased as the w/c ratio were reduced to give similar workability and that influenced the setting time. The mixes that could be cast without admixture showed that the setting time reduced as the w/c ratio reduced. From the results the admixture seems to have an influence on the difference between initial and final setting times.

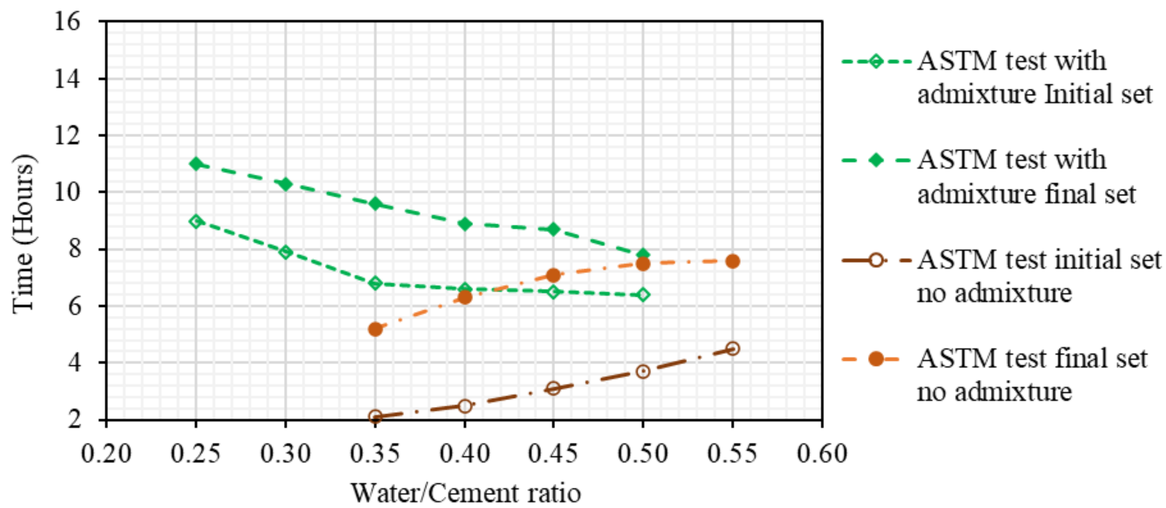

Figure 5-1: **Initial and final setting times using the ASTM test method**

Another two-point setting time method uses the Heat of Hydration (HoH) measurement, curing at 25 degrees and semi adiabatic conditions and then calculate the initial and final setting times as a percentage of the heat increase from the baseline which is defined as the casting temperature to the maximum temperature for each w/c ratio (Kopecskó and Baranyi, 2022). The method is called the fractions method. The results are shown in Figure 5-2. Researchers use the ASTM C191-19 method to determine the percentages required to calculate the initial and final setting time (Kopecskó and Baranyi, 2022). The results vary, but 21% for initial and 42% for final setting time were used for the calculation of the results in Figure 5-2.

Although the two different curing methods (constant ambient temperature and semi adiabatic) result in different maximum temperatures, the concrete had similar setting results for both the initial and final setting times. The results for w/c ratios of 0.25 and 0.50 differed by more than an hour, but literature indicates that the semi adiabatic measurements should be used to determine the setting times.

The results of Figure 5-1 and Figure 5-2 are supposed to be similar comparing the setting for mixes with admixture, but they differ significantly. The results differ less at the higher w/c ratios but there is still a noticeable difference. This significant difference in setting times between the ASTM C403-16 method and the fractions method at the low w/c ratios shows that using one or either of these methods to determine time zero will not result in accurate early age shrinkage values. It is known that the setting time test according to the ASTM method is an arbitrary result not linked to a specific change in the early age properties of the concrete. It is also known that the calculation of the setting times using the semi adiabatic and the HoH values are linked to the original ASTM and Vicat needle tests and will most probably be more accurate at higher w/c ratios.

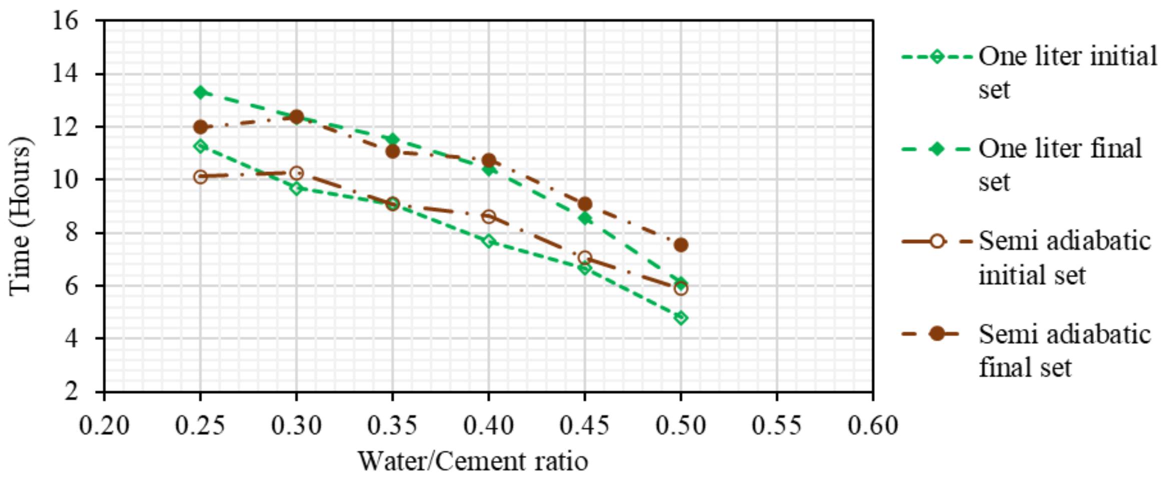

Figure 5-2: **Initial and final setting times using the fraction method**

The results in Figure 5-3 show trends when the first derivative of the semi adiabatic heat of hydration results (final set), the final set of the ASTM C403 method and the final set of the fraction method are compared. The results of the two methods that used the heat of hydration results are close, but the mixes from the ASTM method do not compare well. It is of concern that the ASTM final setting time is a needle penetration (load test) that measured at different time intervals until the interpolated strength value reached a strength of 27.6 MPa. This setting time can differ with up to two hours depending on the exact test method used. The initial setting time would also be another hour or two earlier, making the use of either of these methods to take as the time to start measuring early age shrinkage a risky decision.

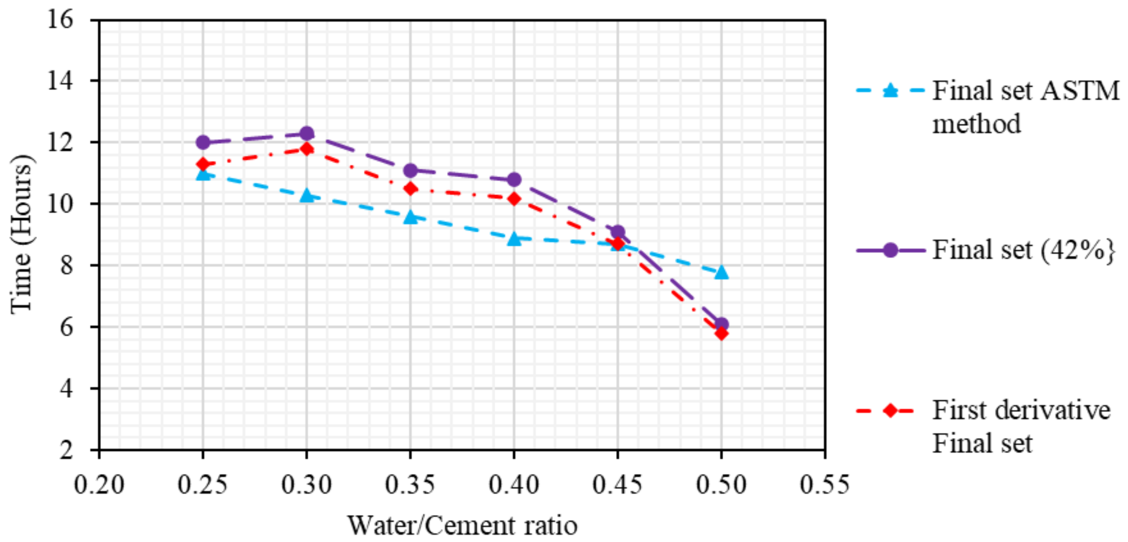

Figure 5-3: **Final setting times with and with admixture**

Figure 5-4 shows the difference in shrinkage when the time-zero for the different final setting times were used as determined using the three two-point test methods. The mix used to illustrate the difference was the mixture with a w/c ratio of 0.35 and with 1.33% HRWR. The figure shows the results for when the setting time determined using each of the methods was used as time-zero for the shrinkage graph. It is important to know that the shrinkage was measured using vertical movement over 290 mm. A thin plate, covered with oil, was used in this project, to prevent any evaporation. The measured deformation thus represents plastic settlement, chemical, autogenous and thermal deformation, defined as early age shrinkage for the remainder of this thesis. In the literature study it became clear that chemical and autogenous shrinkage are a major concern in high strength concrete, but looking at the setting time results and the influence of retardation, it is clear that plastic settlement shrinkage can cause a bigger problem when the data is presented using the final setting time of any of the two-point methods. The shrinkage results have the same slope from about 12 hours after the assumed setting time with about a 120 micro strain difference between all of the results excluding plastic settlement.

The high early age shrinkage recorded could be caused by the reduction in temperature directly after casting before the heat of hydration goes into the dormant period. It is shown in the literature study that water has a much higher shrinkage coefficient than the concrete and in this research the water content was 22.5% of the concrete volume (Zhutovsky,2015). Although this statement was not experimentally proven, it is a possible cause for high early age concrete shrinkage recorded shortly after casting.

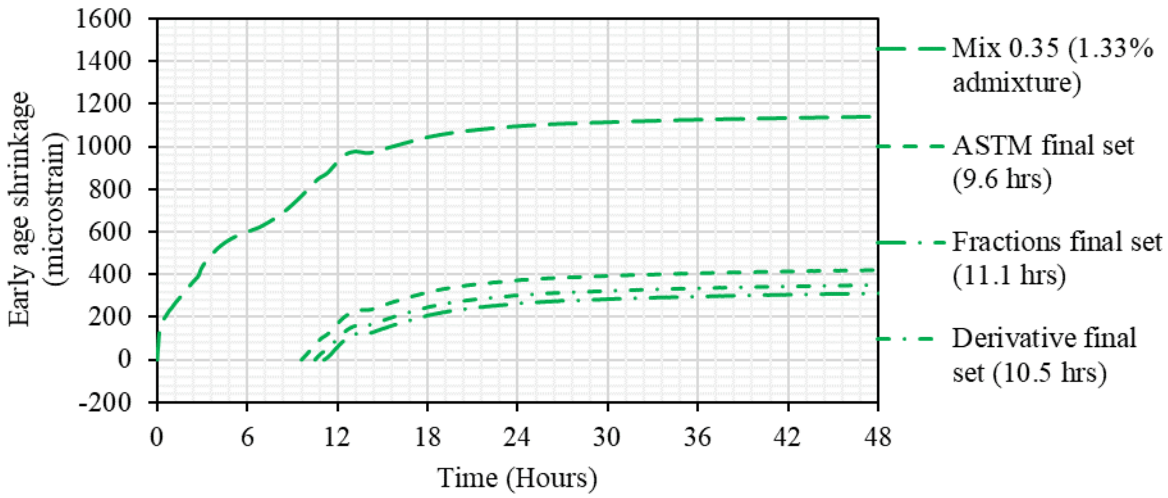

Figure 5-4: Shrinkage data comparison for Mix 0.35 with admixture

In Figure 5-5 the early age shrinkage results from the same mix (w/c= 0.35) but without HRWRA is shown. This mix had the lowest workability of all mixes but still the early age shrinkage was higher when measured as soon as possible after casting than the same mix with the HRWRA. When the shrinkage results are plotted from the final setting times of the three two-point setting time tests, they are very similar because the setting times are very close to each other.

The mixtures with admixture shrink more than the mixtures without admixture when time-zero from the different test methods is used. This observation confirms that the influence of HRWRA should be thoroughly investigated. More importantly the correct method for determining time-zero should be agreed upon among researchers.

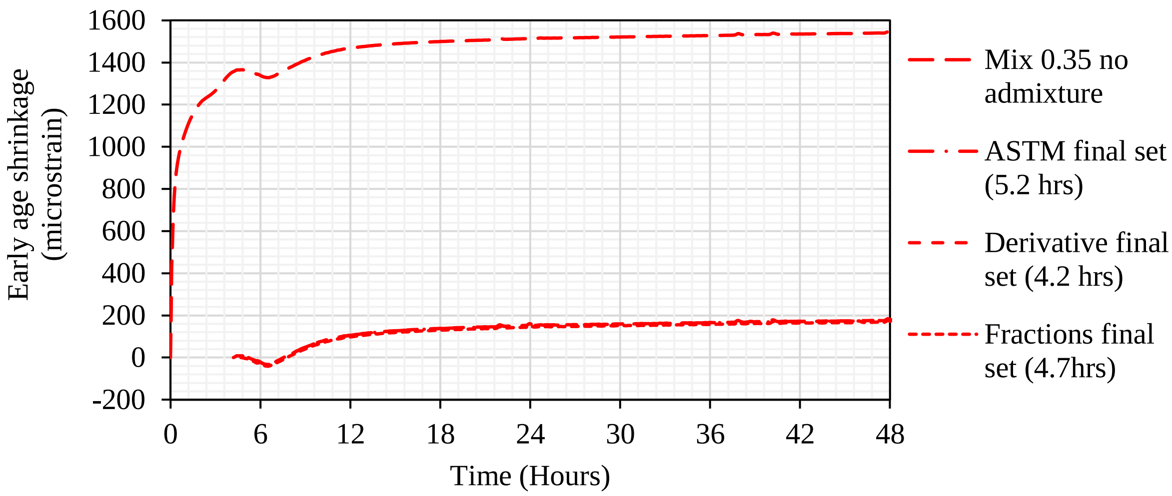

Figure 5-5: Shrinkage data comparison for Mix 0.35 without admixture

The setting time results shown in Figure 5-3 are from three setting time test methods where two setting times are determined for each method. Only the final setting time results for mixes with HRWRA are shown. In Figure 5-6 shrinkage results from Mix 0.45 was used to produce the graphs. The plastic settlement shrinkage of both Mix 0.45 with and without admixture is less than the shrinkage of Mix 0.35.

The two w/c ratios used in the determining of the results shown in Figures 5-4 to Figure 5-6 are on either side of the mix with a w/c ratio of 0.4 from where researcher believe concrete at early ages behave differently and the chemical and autogenous shrinkage can cause a problem (Bentz, 2008). These shrinkage results do not show this. The mix with a w/c ratio of 0.45 has a bigger variance between the results from the new time-zero determined using the final setting time of two of the methods used.

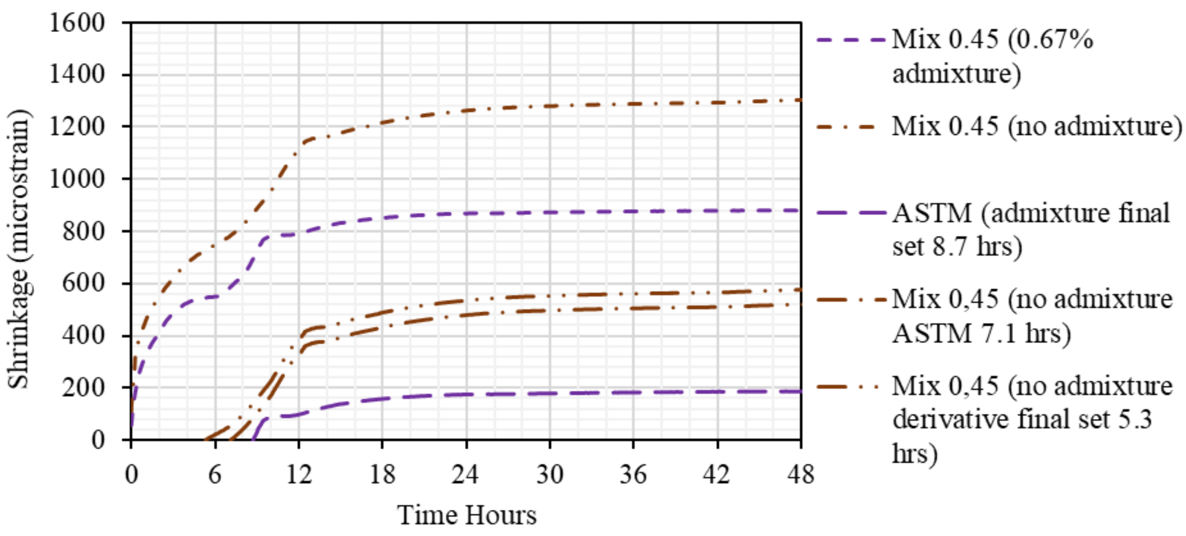

Figure 5-6: Shrinkage data for mix 0.45 with and without admixture

Because the current two-point test (ASTM C403-23) is a type of strength test and the two setting times are selected on a penetration strength, both these times can be manipulated by using the same strength mix that is connected to the same w/c ratio and the same water content. By adding 1% HRWR and reducing the water content the workability increased and so did the setting times. Mamirov (2019) and Benz et al. (2017) showed the different heat of hydration graphs for the same mix but with different aggregate types. If the two-point HoH method is used the difference between the mixes can be up to 4 hours at the 40% to 50% level of heat gain.

The results in Table 5-1 are the initial and final setting times as determined using ASTM C403-23 for each mixture. The results from the heat of hydration for the different mixes were obtained by using the fractions method where the time from casting at 21% for initial set and 42 % for final set of the temperature change between the casting and the maximum temperature reached in the semi adiabatic temperature measurements (Rolo, 2013, Kopecskó and Baranyi, 2021). It has to be mentioned that the percentages in the fraction method used came from researchers that used the ASTM C191-13 method (Vicat cement and mortar setting time test, Sant et al., 2009).

The values in the last column were determined by calculating the first derivative from the semi adiabatic heat of hydration results (rate of change in temperature). These values are all between the initial and final setting time as determined using the fraction method. The results from the derivative method are all higher than the final set time determined by the ASTM method. The initial setting time can be determined using the second derivative of the HoH results, but results from the calculations were too erratic and was not considered.

Table 5-1: Two-point setting time results (in hours)

Test type	**ASTM C403-23**		**Fraction of heat of hydration**		**Heat of hydration**
Mix no	Initial set (hrs)	Final set (hrs)	Initial set (hrs) (21%)	Final set (hrs) (42%)	First derivative Final set (hrs)
Mix 0.25	9.0	11.0	10,1	12,0	11,3
Mix 0.30	7.9	10.3	10,4	12,3	11,8
Mix 0.35	6.8	9.6	9,1	11,1	10,5
Mix 0.40	6.6	8.9	8,6	10,8	10,2
Mix 0.45	6.5	8.7	7,1	9,1	8,7
Mix 0.50	6.4	7.8	4,3	6,1	5,8
Mix 0.35 na	2.0	5.2	3,4	4,7	4,2
Mix 0.40 na	2.5	6.3	3,9	5,2	4,7
Mix 0.45 na	3.1	7.1	4,1	5,6	5,3
Mix 0.50 na	3.7	7.5	4,6	6,3	6,2
Mix 0.55 na	4.5	7.6	6,1	7,7	7,2

Figure 5-7 shows some of the two-point setting times results from summarized in Table 5-1. Based on literature reviewed and listed in Table 2-2 all of these values can be used as the point in time from where early age shrinkage should be measured. It obvious that a standardized method is needed to accurately determine the point in time when concrete starts acting as a solid material

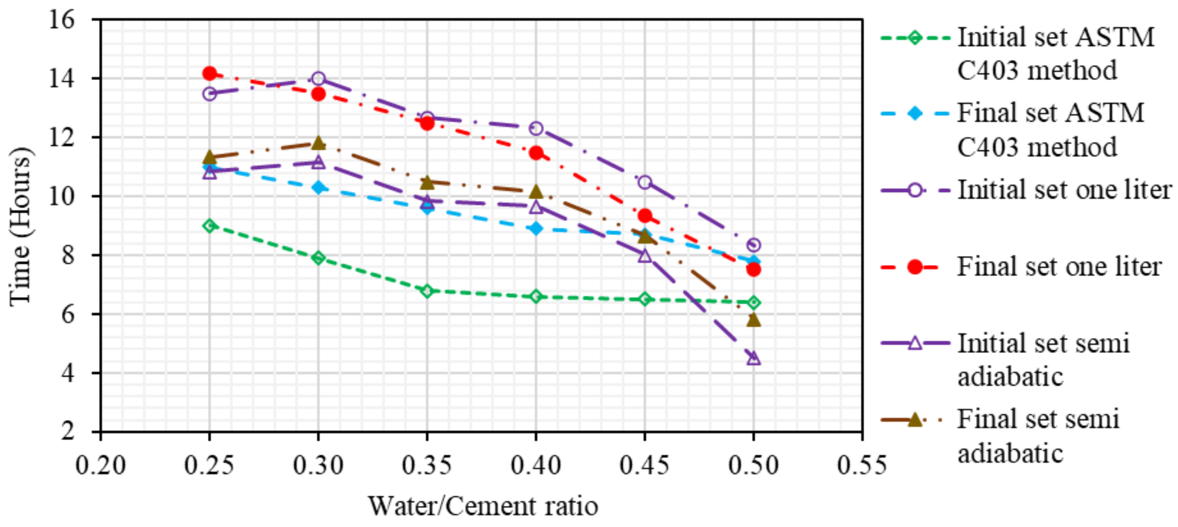

Figure 5-7: Results of all two-point setting times

5.2.2 Single point setting times

If considered the setting time results from the Figure 5-7 it is understandable that researchers would start looking for a better solution. Therefore, the research started to concentrate on a method to determine a single-point or a knee-point, sometimes referred to as time zero. The disadvantage was that they were mostly still guided by the Vicat or ASTM C403 method, instead of looking for the point in time from where early age concrete properties start developing. It was shown in the literature study that although promising research has been published, researchers still tend to try to use the initial and final setting times obtained from the needle tests. Pure cements or cement pastes are used and the temperatures are artificially kept constant instead of measuring the concrete property changes and search for a time-zero when concrete strength and stiffness start developing.

To avoid the confusion between using either the initial or the final setting time, single point setting times should be used as time-zero to start the measurement of the early age shrinkage. Single point setting time would normally be at a point where the early age property that is measured, starts changing and this is called a knee-point.

The point should represent a clear “knee-point” where the concrete changes from a Bingham fluid to a solid, resulting in a change in the rate of properties developing. To determine a point like that in concrete with a single test is a big challenge and requires an understanding of what influences concrete setting time or what is happening when plastic settlement no longer has any influence.

Load based tests

In Figure 5-9 results are shown for three types of tests where a load was applied and measured and a strength calculated in MPa. The tests are the ASTM C403-23 needle penetration test, a 20^o cone penetration test than needs to be researched for a potential addition to the ASTM test and the normal cube crushing strength test. The 20^o cone was penetrated at the same speed than the ASTM C403 penetration test but the results indicate a definite knee-point. In Figure 5-8 the difference between the penetration of the ASTM 403 needle test and the cone penetration test can be observed. The material used to form the different layers were all crushed aggregate that passed the 4.75 mm sieve with the same w/c ratio and water content. The photo shows that the penetration needle disturbed the layers and had a significant influence on the surrounding material whereas the cone penetration did not disturb the different layers that remained in an almost straight line, Although the needle only penetrated 25 mm and the cone to 50 mm, the cone caused less damage to the surrounding material. The needle test caused significant damage to the surrounding material, indicating that repeated tests on the same specimen could result in inaccurate results due to prior damage caused by earlier needle penetrations. The second advantage of the cone penetration test is that when calculating the shear stress using the cone surface area in contact with the concrete throughout the penetration, a constant shear strength in MPa is measured.

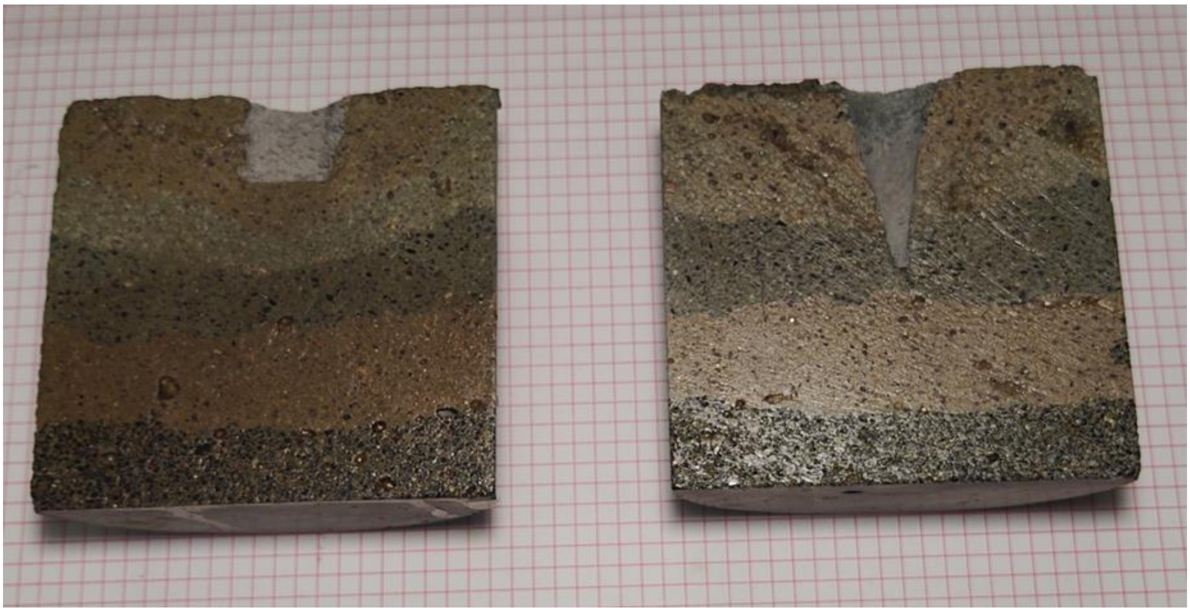

Figure 5-8: Influence of different penetration on concrete

The second strength test with a single point slope change is a normal cube strength test every two hours from a point where it is possible to demould the cubes. From all the tests done, this test shows the best knee-point for mixes with water to cement ratios of 0.4 and lower. The disadvantage with the cube test is the volume of concrete that must be mixed and the number of cubes required. In this study cubes were tested only every two hours, making the point of intersection not that accurate. The advantage is that it can be a very precise test for high strength concrete where the determination of the knee-point is required to measure the early age shrinkage from where a real change in the concrete strength took place.

The results of all the load type tests completed in this research from mixes with HRWRA, can be seen in Figure 5-9. In these results the admixture retarded the setting time as discussed before, but at higher w/c ratios, where the admixture was at a lower percentage the influence on the setting time reduced. This is expected from normal concrete technology principles when w/c ratios increase, resulting in weaker concrete strength with less cement. In this figure the two different strength type tests resulted in the cube strengths almost following the final setting time results and the cone penetration results the initial setting time result. The mixes with w/c ratios of 0.25 and 0.50 and admixture dosages of 2.0% and 0.33% gave setting time results that could be influenced by over dosing at 2.0% and not enough admixture at 0.33%.

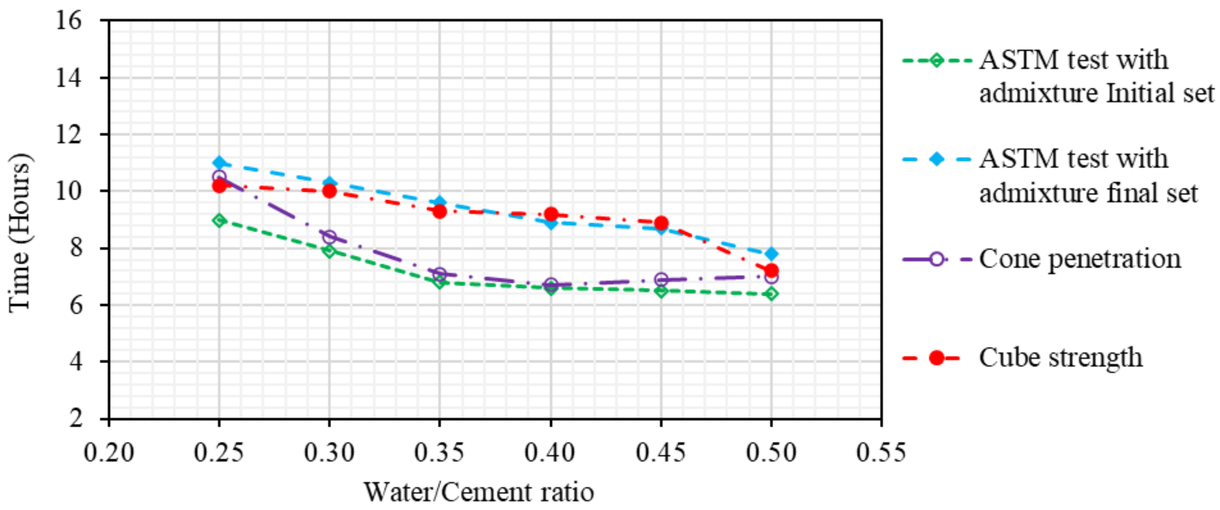

Figure 5-9: Knee-point load strength results from HRWR mixes compared to the ASTM method

Shrinkage and suction tests

In Figure 5-10 the effect of the w/c ratio on the rate of shrinkage can be seen. By determining the intersection between two lines indicating, a reduced shrinkage rate followed by an increased shrinkage rate, a knee point can be found as indicated with the vertical arrows. The time of slope change can be calculated by using the first derivative of the measured shrinkage. This knee-point is indicated in Figure 5-10 for mixes with HRWR. This method shows better results on low w/c ratio mixes, which is where early age shrinkage is a concern. The big difference between the shape of Mix 0.55 and the other three mixes was most probably caused by the inclusion of HRWR as all mixes with admixture showed a retardation in strength gain in the first 13 to 16 hours as mentioned before.

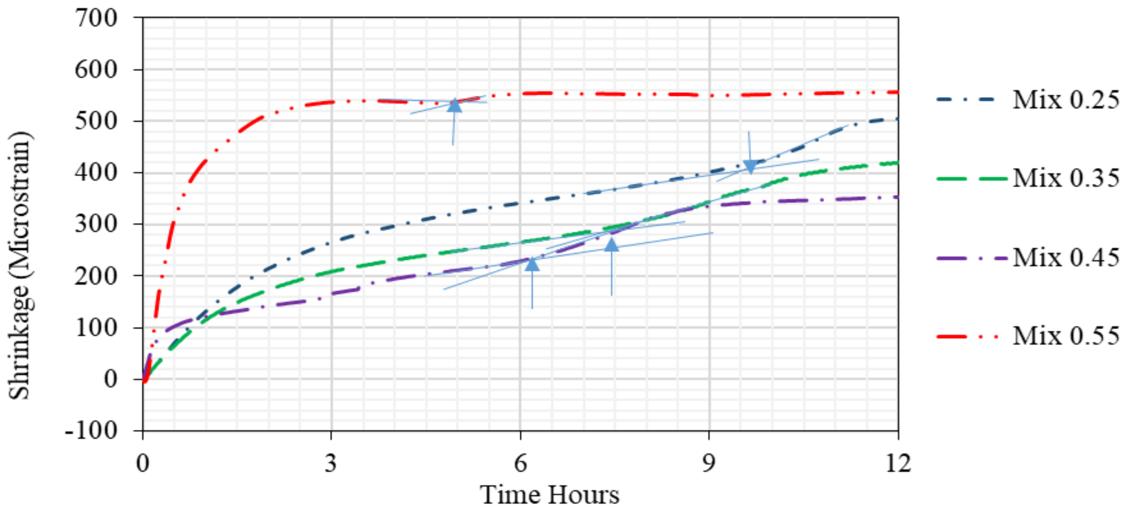

Figure 5-10: The intersections of the slope change line in the shrinkage results

The suction test results compare well with the shrinkage slope change and ring test results as indicated in Figure 5-11, where the similarities in shape between the tests are shown. Here again the setting time results for the mixes with a w/c ratio of 0.25 and 0.50 vary significantly, but at the other w/c ratio's the results are similar. The calculation of the derivative from the slope change from the early age shrinkage results shows almost the same setting time as that of the suction values measured with the WP4C apparatus for al mixes.

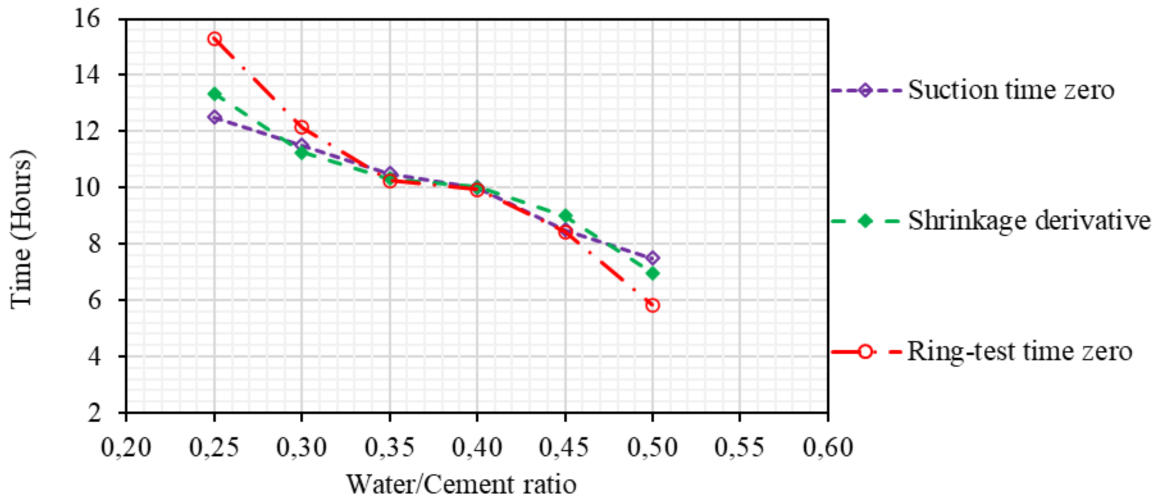

Figure 5-11: Setting times from time zero on early age tests

Figure 5-12 shows the comparison between the suction test done with the WP4C and the only pore pressure test conducted using a suction probe (normally used in soil testing). The probe was calibrated and saturated in distilled water, then placed into the centre of the concrete specimen before compacting the concrete. The disadvantage of using suction probes is that they are expensive, meaning that they cannot be left in the specimen until the concrete has hardened. The probe must be kept saturated and no air must be entrapped during this process. Therefore, only one comparative test was conducted. Using the WP4C is easier and faster, yielding good results. The probe results in Figure 5-12 show significant suction development up to 10.25 hours and the WP4C result for the same mix was 10.5 hours. The difference in estimating setting time of only 15 minutes. This small difference in Air entry level and the slope change in the suction results can be explained by the size of the castings and the difference in temperature between the specimens. The temperature of the probe was between 27 ºC and 30 ºC (normal heat of hydration curve) and the suction sample temperature was kept at 24 ºC.

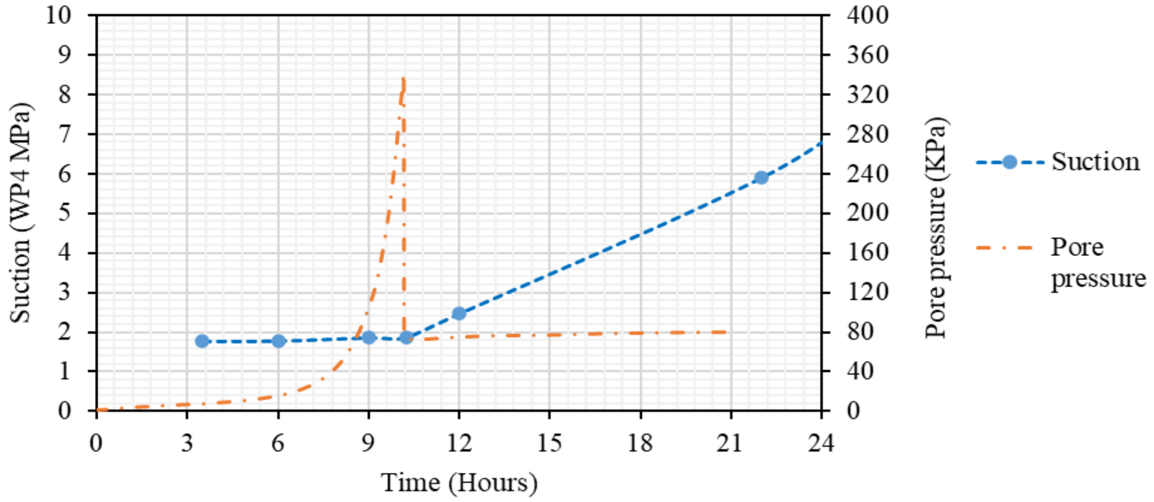

Figure 5-12: Pore pressure and the WP4C suction on mix 0.35

The restrained shrinkage ring test results are shown in Figure 5-13. The ring used was more sensitive and the concrete dimensions differed from the standard ASTM test method. The trend of the influence of the admixture is again clearly visible, showing that Mix 0.35 has no stress for about 10 to 11 hours after casting and then the measurable stress developed rapidly. Mix 0.50 on the other hand shows an increase in stress after 6 hours. The results also show that the stress at 12 hours is still very low for mixes with admixtures. The results also show that between 12 and 24 hours the lower w/c ratio mixes from 0.4 and below with admixture caused a tensile stress on the ring with an increased slope in comparison to the mixes with a w/c ratio higher than 0.40.

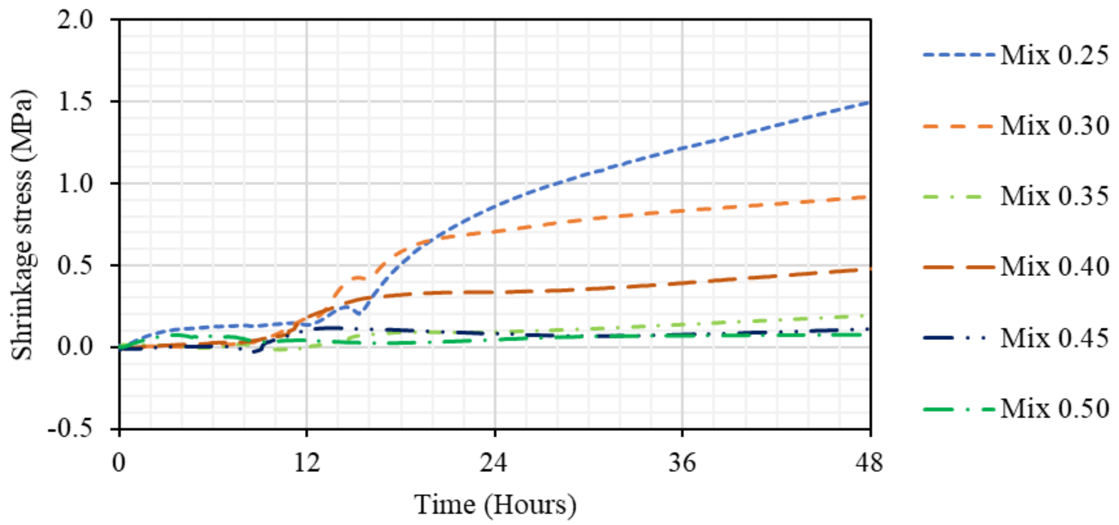

Figure 5-13: Ring test results from various mixes with HRWRA

In Figure 5-14 the results of the same mixes without HRWRA are shown and the trend shows a decrease in shrinkage as the water/cement ratio increases. The shrinkage stress development started earlier than for the same mixes with HRWRA. The mixes without the HRWRA show that the lower the w/c ratio in the mix the faster the tensile stress started developing after 48 hours the restrained tensile shrinkage stress was significantly lower than for the corresponding mix with HRWRA.

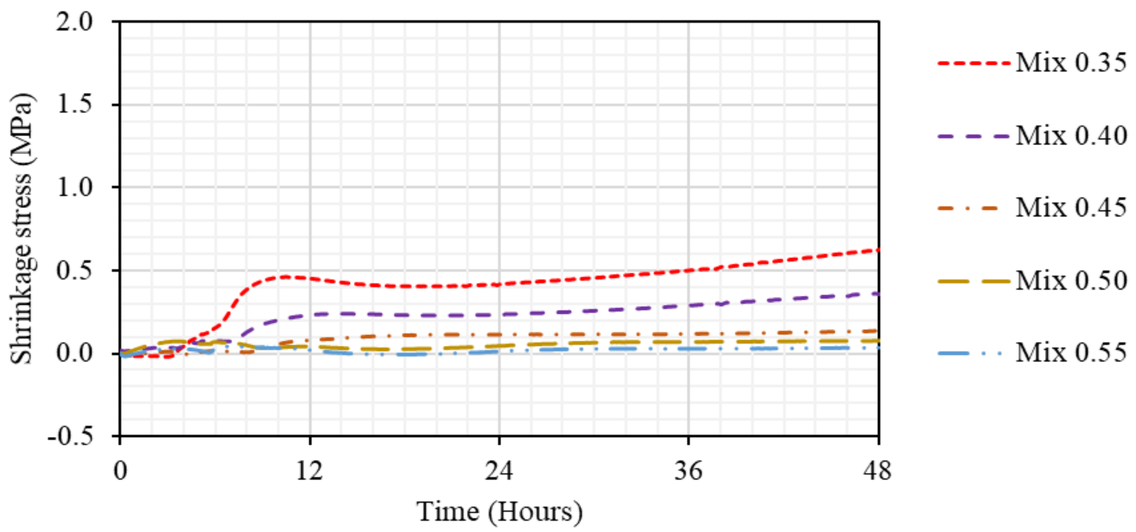

Figure 5-14: Ring test results from mixes without HRWRA

Figure 5-15 shows the point of increase in shrinkage stress applied by the concrete to the ring. This was used to determine the values that are shown in Table 5-2. The method of measuring is accurate, but the time from mixing to the time the test started must be added to have an accurate result. The effect of w/c ratio on the setting time when the concrete started developing internal stresses can clearly be seen in Figure 5-15.

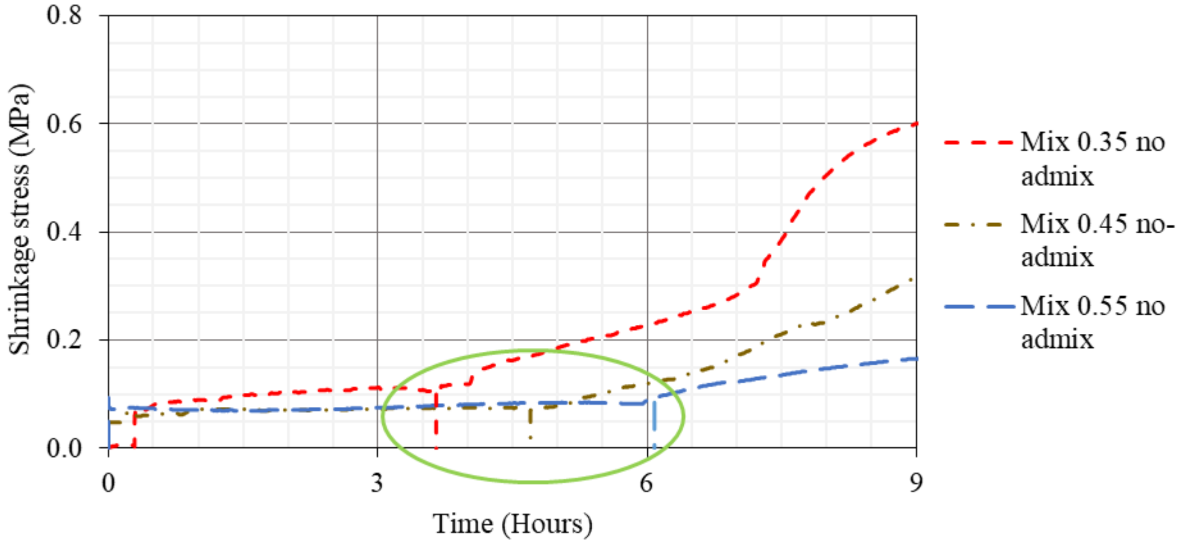

Figure 5-15: Ring test knee-point for three different mixes without admixture

Figure 5-16 shows the effect of admixture on the setting time. The same trend was experienced for all the mixes with and without admixture. The difference is however not constant. As the w/c ratio reduced, the admixture content as percentage of the cement content increased. This means that the difference between the two mixes at water/cement ratio 0.5 was minimal compared to the mix 0.35 where the difference in setting time, when the change in slope for the ring test is used as time zero, was almost 3 hours between the mix with HRWR and without the admixture.

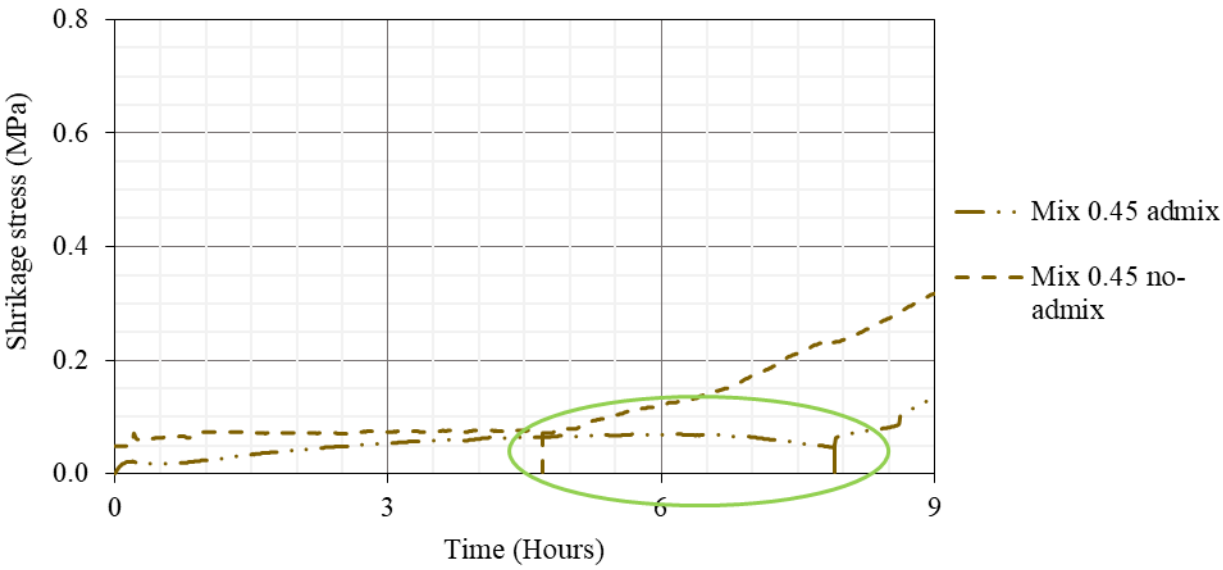

Figure 5-16: Ring test knee-point between same mixes but with and without admixture

Ultrasonic pulse velocity

Figure 5-17 shows the result of the single Ultrasonic Pulse Velocity (UPV) meter test done on mix 0.40 without admixture. The unit available to do the test did not read any value through the Bingham liquid, but as soon as the concrete became a solid that can transfer the pulse generated, it gave a reading. The apparatus gave an overflow reading up to 4.5 hours after casting. At 4,5 hours the first reading measured was calculated to be 1.66 kilometre/second. That reading rose to 3.42 kilometre/second at 7 hours. Between 7 and 8 hours after casting a steep rise to 4.29 kilometre/second occurred where after it then gradually rose to 4.4 kilometre/second after 48 hours. With this result as shown in Figure 5-17 there is a possible argument for an initial and final setting time. The cone penetration test that was done with the same mix gave a time zero of 4.6 hours which is close to the time when the first reading was measured. This test confirms that this method could also be a usable method to determine actual setting time. It was however not used in this project due to the lack of local availability of a suitable automated system that could be used to take UPV readings at a regular time interval. To avoid missing the first reading after overflow, readings for this dataset were taken manually every 15 minutes from 3 hours onwards giving the results as shown in Figure 5-17. With an automated system this test can give very useful early age concrete results. The test could indicate immediately when the concrete becomes a solid.

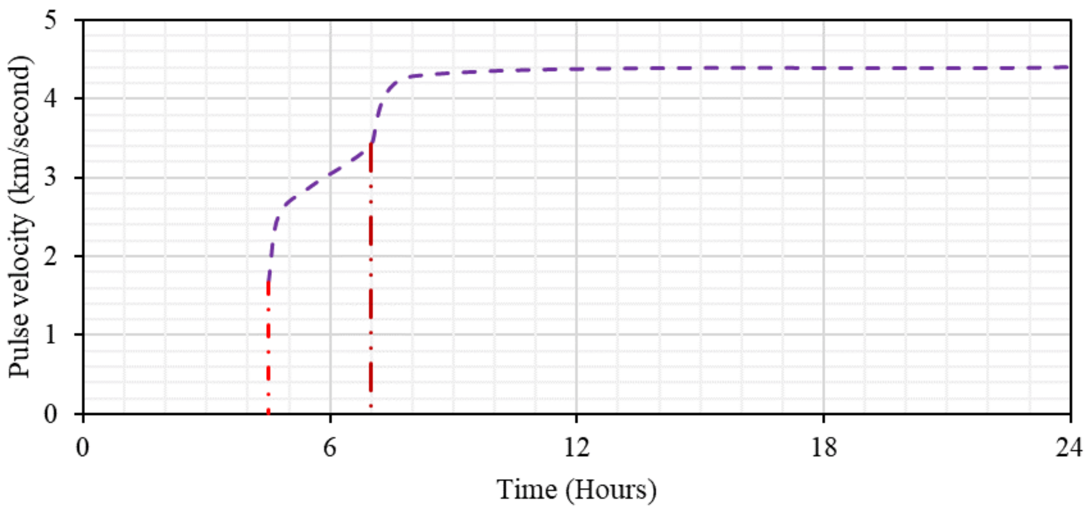

Figure 5-17: UPV measurement on fresh concrete

Summary of single point tests

Table 5-2 shows all the results from tests that concentrated on only one result giving the change from a fluid to a solid in concrete. These tests are the cone penetration, cube strength, shrinkage, restrained ring and the suction test. Two individual test results were added to the listed tests to allow a better understanding of some of the tests and these are the measured pore pressure in the wet concrete (using a suction probe) and the concrete temperature measured with a thermistor. An Ultrasonic Pulse Velocity Meter (UPVM) was also used to measure the increase in transmission speed through the concrete as it sets. These results were compared to the other results for the same mixes. The cone penetration setting times were all between the initial and final setting time results from the ASTM C403 test. The graph for the early age shrinkage had a definite slope change and without other testing it could be the point where the material changed to a solid. The restraining ring test was added to monitor the stresses that developed in the concrete at early ages and to monitor the possible cracking of the concrete.

Table 5-2: Single point tests to determine time zero (in hours)

Test type	**Cone penetration**	**Shrinkage test**	**Restrained ring**	**Cube strength**	**Suction WP4C apparatus**	
Mix no	Knee point (hrs)	Knee point (hrs)	Slope rise (hrs)	Cube strength knee point (hrs)	Knee point (hrs)	Pore pressure (hrs)
Mix 0.25	10.5	13.36	15.26	10.2	15.2	
Mix 0.30	8.4	11.26	12.62	10.0	11.5	
Mix 0.35	7.1	10.31	11.7	9.3	10.5	10.25
Mix 0.40	6.7	10.03	9.86	9.2	10.0	
Mix 0.45	6.9	8.99	8.42	8.9	8.5	
Mix 0.50	7.0	6.96	5.9	7.2	7.5	
Mix 0.35 na	4.0	3.49	3.14	4.0	Not measured	UPV (Pundit)
Mix 0.40 na	3.9	3.75	3.61	4.6		
Mix 0.45 na	4.8	4.75	4.03	5.4		4,5
Mix 0.50 na	5.3	5.64	4.65	6.0		
Mix 0.55 na	6.0	6.25	5.63	6.2	7.0	

What was then seen from the data was that all the mixes show very little stresses up to a certain point in time, from where the graph increases at a higher rate. The cube crushing strength is a known test and when the results were plotted on a graph it shows a very definite increase. The suction of the WP4C shows a definite change in slope for all mixes and is easy to use. The single pore suction was to confirm the data gathered from the suction data determined with the WP4C apparatus.

Figure 5-18 and Figure 5-19 are the results from Table 5-2 to show a better understanding of the different single point test results. Figure 5-18 shows a similar trend than for the two-point tests, confirming the influence the HRWRA had on the setting time.

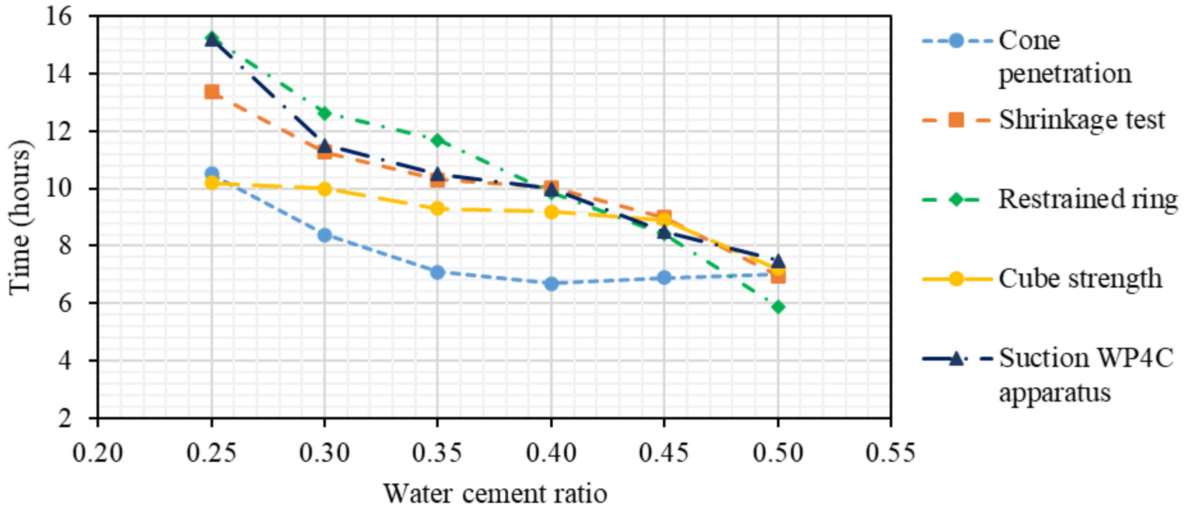

Figure 5-18: Single point results from Table 5-2 for mixes with HRWRA

Figure 5-19 shows the normal behavioural trend expected from the Duff Abrahams law for w/c ratio behaviour. The difference in setting time between the results for the mixes without admixture are also a lot smaller than those with the admixture.

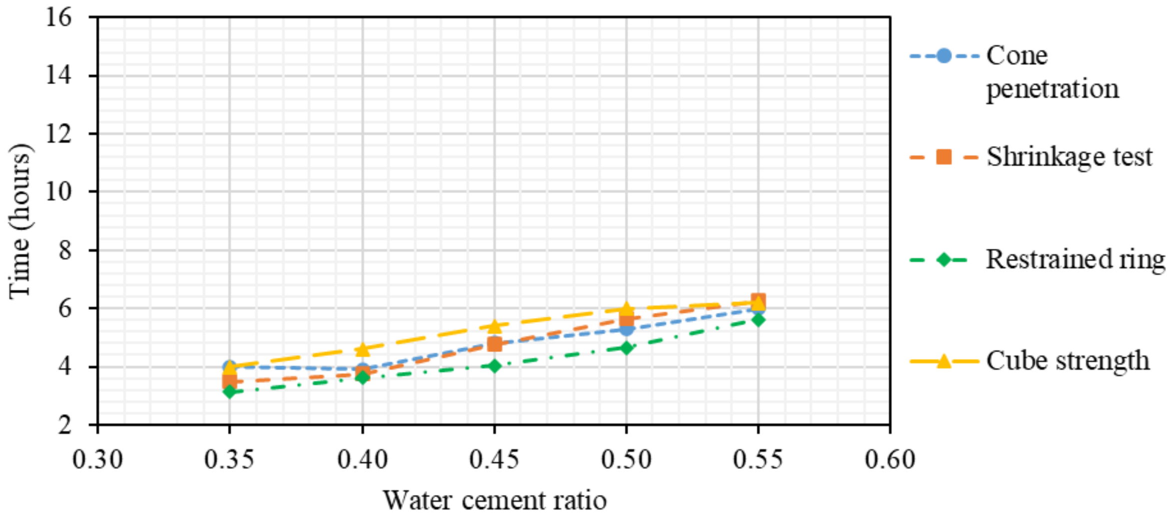

Figure 5-19: Single point results from Table 5-2 for mixes without HRWRA

5.3 TENSILE STRESS IN CONCRETE

The results in Table 5-3 are from the direct tensile tests done on the mixes containing admixtures (except mix 0.55 which was without admixture). The result of the ring test at the coresponding ages of 8, 10 and 12 hours was determined from the computer generated data. The ring test strain results were then used to compare the direct tensile strength to the real stress in the concrete at that time. The tensile stress that developed in the restrained ring test showed that shrinkage cracking was not possible during the test period as the tensile stress caused by shrinkage never exceded the tensile strength. The ring test for all mixes, did not show any drop in the stress during the first 48 hours after casting, indicating that no crack developed in the first 48 hours after casting.

Table 5-3: Results of direct tension and ring test

Direct tensile strength in kPa				
Time (hours)	Mix 0.25	Mix 0.35	Mix 0.45	Mix 0.55
8	48.5	46.5	20.0	219
10	247	188	327	584
12	463	309	653	701
Shrinkage ring stress in kPa				
Time (hours)	Mix 0.25	Mix 0.35	Mix 0.45	Mix 0.55
8	132	36.0	16.0	37.7
10	141	5.50	47.9	31.9
12	136	22.9	104	20.3
Setting times (hours)				
	Mix 0.25	Mix 0.35	Mix 0.45	Mix 0.55
Ring test	12.2	10.3	7.90	6.80
Strength and stress at setting times in kPa				
Tensile strength	470	190	20.0	219
Stress in concrete	140	6.00	16.0	37.0

5.4 SUMMARY

The results for the 9 different tests done on the 11 mixes, was summarized in Table 5-1 and Table 5-2. Researchers studying early age shrinkage could not decide on a fixed setting time as shown in Table 2-2 in the literature study. Neither could they decide on the type of test that should be used to measure the shrinkage. The most important aspect in this project was to keep the ambient temperature constant from 24 hour before mixing up to the final test for all materials, moulds and even the mixer pan. This made it possible to compare all test results with each other.

To compare the different trends observed, Figure 5-20 was created where all the setting time tests were divided into test groups where a load was applied and a force was measured for all the w/c ratio mixes with admixtures and a separate group without admixtures. Similarly, two groups were created where the setting times were determined by other methods and the average setting times for the different w/c ratios were plotted.

It was noticed that for all the tests where a force was measured and converted to a result, the shape of the data trend was similar. The comparison between the setting times calculated from load application and other setting time methods show that the admixture has an influence on the setting times. The mixes without admixtures gave similar setting times, when the average for the different tests were used whether calculated from load test data or from other setting time test methods. The use of admixtures resulted in differences between the different types of setting time measurements of almost 2 hours for w/c ratios below 0.45. This confirms that when load tests are used to determine setting time of mixes containing admixtures, the time when concrete change from a Bingham fluid to a solid can be wrongly estimated, causing the early age shrinkage to be measured from too early. This would result in an over-estimation of the cracking risk of concrete containing admixture.

These results show that the use of ASTM C403 could give a too early setting time for low w/c mixes and therefore using this result as "Time zero" can overestimate the early age shrinkage. This is most probably because the load values used in this test is arbitrary and came from an era where admixtures and high strength concrete was not yet developed.

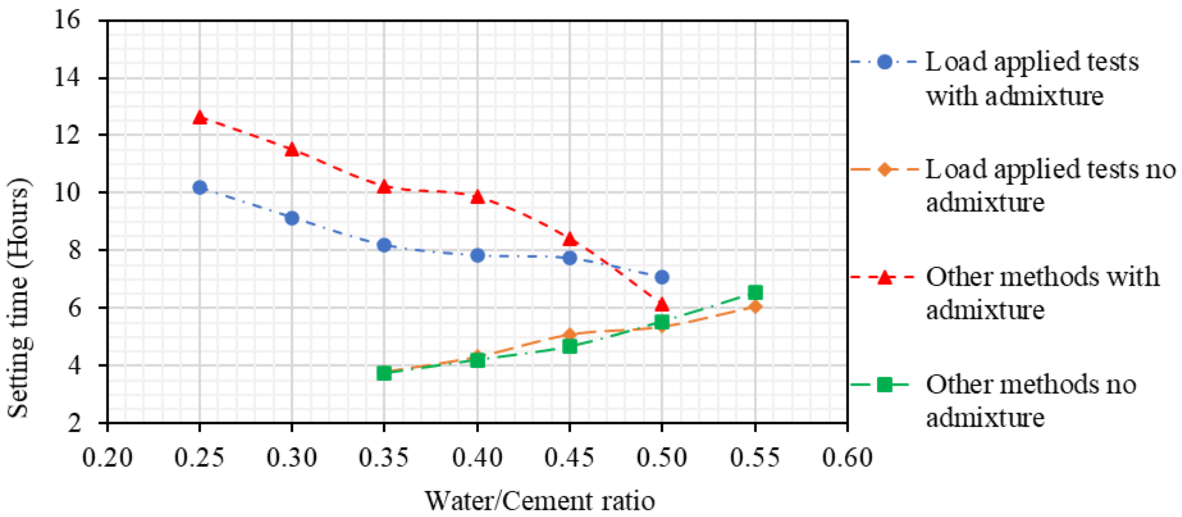

Figure 5-20: Comparison between setting time results

Based on the results of this study it is recommended that non-invasive tests should be consider as a measure to determine the change in slope representing "Time-zero" for high strength concretes with a water/cement ratio below 0.4. Three test methods gave very similar results, the ring test, the suction test done with the WP4C and the derivative calculated from slope change during the heat of hydration tests.

Although further research is required before a discission can be made regarding the use of a single set test, it is also important to test the concrete mixes with low w/c ratios when determining setting times and not only the cement or mortar pastes mixed to constant workability.

6 CONCLUSIONS AND RECOMMENDATIONS

6.1 INTRODUCTION

A study on early age behaviour of medium to low water/cement ratio concrete was conducted with the main aim to find a sudden change in the properties which could indicate the moment in time the concrete changes from a viscous liquid to a solid. The reason for the study was to develop a clearer picture from where early age shrinkage that can result in the development of internal stresses and cracks, should be measured. During the study, some standard tests were altered to fit in with some of the other tests that were conducted. Climate conditions, material types and ratios were kept as similar as possible. It is, however, not possible in a study of this magnitude, where almost two cubic meters of concrete was cast over a span of three years, that there would not be some differences. The cement came in two batches and although the mortar prism results were similar, chemically, the batches could differ slightly. That could affect results, such as the heat of hydration. The same applies to the water, admixture and aggregate that were not all from one batch. The tests were spread over a three-year period and repeated more than once and the values provided in the document are averages.

6.2 CONCLUSIONS

The main aim of the study was to determine if the point in time when concrete changes from a Bingham flued to a solid can be determined by one single test. This was done analysing the possible early age test methods that give only one time that can be called "Time-Zero" from where the early age shrinkage can be measured. From the literature study it became clear that the influence of w/c ratio, different types of cement, admixtures and the curing temperature all play a role in setting time. From this research, the early age properties gave different changing points in time with the different tests conducted.

The experimental results indicated that the time that it takes concrete to change from a viscous liquid to a solid is not a constant, but depends on w/c ratio, specimen size, temperature, inclusion of admixtures and the type of test used. The standard constant workability cement setting time tests can thus not be used to estimate the setting time of concrete, especially when admixtures are used.

Two-point setting time test methods such as the ASTM C403-16 and the calculation method using the heat of hydration graph do not give setting time values that represent the moment in time when the concrete starts acting as a solid material. The initial and final setting times determined from the heat of hydration results determined by the fraction and the derivative methods are based on the ASTM C403 method and it thus give similar behavioural trends, which cannot be linked to actual setting times.

It is possible to determine an actual point in time when concrete starts acting as a solid by using a single point set time indicator that takes all variables into account.

Testing early age cubes can provide the real time when the strength starts to increase. This is a safe method giving good results, but it involves a considerable amount of work. In this research, the cubes were only tested every two hours. Although it is claimed that the HRWRA do not retard, testing showed that at higher dosages they tend to retard significantly. However, after 24 hours there was no indication of that retardation. This test method is easy to use, showing a clear knee-point but, it is a lot of work and cannot be automated.

Throughout the project the early age properties from concrete made with a w/c ratio lower than 0.4 seem to indicate that the resulting concrete behave differently. The difference seems to be between tests where a load is applied and other types of tests where a property is measured like suction or restrained shrinkage. When load tests are used to determine setting time of mixes containing admixtures, the time when concrete change from a Bingham fluid to a solid can be wrongly estimated, causing the early age shrinkage to be measured from too early. This would result in an over-estimation of the cracking risk of concrete containing admixture.

Based on the results of this study it is recommended that non-invasive tests should be consider as a measure to determine the change in slope representing “Time-zero” for high strength concretes with a water/cement ratio below 0.4.

When shrinkage results were measured from the actual Time-Zero as determined for individual mixes, the shrinkage results showed clear trends that can be explained in terms of mix properties such as w/c-ratio.

6.3 RECOMMENDATIONS

Early age shrinkage results will form clear trends when the correct time zero is used as starting point for shrinkage measurement and researchers should consider their motivation for using specific values on this point in time. Different setting time test methods give different answers. For the industry to go forward using high strength concrete in particular further research is required:

- Different test methods should be used to determine setting times for not only concrete containing different cement replacement materials, but also more workable concrete mixes, including self-levelling and self-compacting concrete mixes.
- The moulds used when the time zero is being determined, should be made using specified material as mould material type can influence the result especially when the heat of hydration data is used to calculate the setting times.
- The possible influence of the volume of water, and the influence of the water temperature reduction in the initial stages of the heat of hydration on the immediate shrinkage of the concrete, should be considered.

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