An experimental study on the effects of casein protein in unreinforced lime mortar specimens

by

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Abstract

Casein, an organic milk protein, existed in masonry mortars from medieval times until the 18th century. It was understood to improve workability of the mortar. Recent mortar conservation projects have proposed its use, however, little information is existent on its effects on strength, a critical property in repair of masonry mortars. Adding 0.25% casein by mass was insufficient to improve flow properties of mortar. It was found that adding 0.5% casein by mass resulted in a flowable mortar, however, a 75% reduction in strength resulted, while bond strength increased, after 56 days. Reducing the water by 18% resulted in a 50% reduction in strength from that observed in the control sample, while maintaining flowable properties. Reducing the water more than 27% yielded a rapid-setting, non-flowable mortar. The addition of casein is a plausible alternative for repointing existing mortar joints. It has favourable flow properties, as well as improved bond strength.

Acknowledgements

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Chapter 1: Introduction

1.1 Background

Rehabilitation of historic masonry structures presents ongoing challenges concerning loading and stabilization. For most of the 20th century, existing unreinforced masonry buildings were reinforced and made stiffer. Much of this work occurred before the dynamic behaviour of structures and finite element analyses were well understood; it was found that stiffening the existing walls may lead to disastrous effects.

The *Standards and Guidelines for the Conservation of Historic Places in Canada* [1] includes a section dedicated to the maintenance and repair of lime mortars. Mortars shall be continuous, prevent water infiltration, and have a material strength compatible with that of the existing mortar and masonry units [1].

Casein is a protein naturally occurring in all types of milk; this research refers exclusively to micellar casein sourced from bovine milk. The casein protein is divided into three protein subgroups: α-casein, β-casein and κ-casein. Further subgroups of α-casein exist [2]. Bovine casein typically contains 48% α-casein, 34% β-casein and 15% κ-casein [2]. The chemical structure, comprising of a series of amino acids, is the same except for the number of phosphate groups present [3]. Solubility is pH-dependent: casein precipitates at a pH less than 4.6 [3], is partially insoluble near a neutral pH—κ-casein is soluble while the other types of casein are insoluble [4]—and dissolves completely at a pH greater than 10.0 [5]. Lime mortar, for which the main constituent is calcium hydroxide, has a pH of 12.6, equivalent to that of a saturated calcium hydroxide solution, therefore, casein dissolves in the mortar.

Casein is typically extracted from milk by means of acid precipitation and has been shown to increase the workability of the mortar and to improve adhesive properties [6]. In particular, the anionic phosphate component causes the protein to absorb calcium existent in masonry mortar, and thus behave as a superplasticiser additive [3]. Casein is also occasionally used to improve the workability of tile grout and in self-levelling grout specimens [7]. It has been used to increase the workability of mortars since medieval times in Europe [8] [9]. As far as could be determined, no major buildings have been constructed with casein mortar in Canada. It was used in glues and adhesives in the 19th century [10], and it is still used to the present day as an additive in certain paints.

This thesis seeks to investigate the structural properties of historic limestone mortar with a casein protein additive. First, a literature review of strengthening techniques that have used similar historic mortar additives is addressed. The report then devises a test procedure with the casein mortar to test its behaviour in tension, compression, flexure and shear, and in a bond capacity in both flexure and shear. Finally, a summary and analysis of all test results is presented.

The review of literature covers similar mortar-strengthening studies that shown improved resistance to lateral loading. These include reinforcing within the horizontal joint spaces during masonry repointing. The other study area was the addition of horsehair or similarly-sized tensile fibres to the mortar specimen. However, the structural effects of casein as a mortar additive have not yet been studied in detail by any other research group.

1.2 Statement of Motivation

Past experience has shown that in many cases, modern codes are inadequate to be applied as written on heritage masonry structures as they are intended for new construction [11] [12]. Due to the lack of research and available literature on alternative solutions, structural engineers continue to design inadequate and inappropriate structural upgrades to masonry buildings to the present day. Casein mortar had recently been proposed on a mortar conservation project in Canada for masonry wall repair, however, it was not used due to a lack of knowledge of the structural material [13].

The casein protein is a plausible candidate for improving the properties of mortar in structural rehabilitation applications. Most research on the protein additive investigates the protein itself from a chemical standpoint, with the chemical structure being fully researched. Its structural implications regarding mortar are not well known; these effects may be vital to understand the effects of casein on mortar that is existent in buildings. Little information has been published on the effects the addition of casein has on mechanical strength [14], and what little information does exist is incomplete.

It was predicted that the addition of casein, which often acts as a binding agent in glues, may substantially improve the bond strength while having little to no effect on the properties of a mortar cube by itself.

Furthermore, the cost of casein protein is minimal in comparison to the cost of other comparable structural enhancements, as it is used in only small quantities (less than 1% by mass) [13].

Chapter 2: Literature Reviews

2.1 Evidence of casein used in historic mortars

Little literature addresses the use of casein in masonry mortars. While each protein exhibits different chemical and structural properties, some tests have been conducted assessing proteins on a general basis.

Jasiczak and Zielinski [15] researched the structural effect of generic proteins on cement mortar specimens. The authors discussed the effects of protein specimen in general, including casein. Powdered protein was added to a standard cement paste. Two Portland cements were used for testing, the first, identified as CEM I, contained no fly ash, while the second, CEM II, contained fly ash [15]. The mortar prisms measured 40 millimetres in width and in height and 160 millimetres in length [15]. A wide range of testing was done: bending strength, compressive strength, shrinkage, volumetric density, absorption and exposure to freeze-thaw cycles. For the purposes of this thesis paper, the structural effects are those of interest.

Jasiczak and Zielinski [15] conducted two strength tests: compressive strength and bending strength. It was found that the additions of protein to cement mortar decreased the overall strength. The research group developed an empirical equation for the strength as a function of protein content. Air entrainment caused by the addition of a protein was identified as the primary reason for the decrease in strength, not the protein itself [15]. The only improvement noted by Jasiczak and Zielinski was the improvement in resistance to weathering [15].

Chandra and Aavik [14] tested a variety of proteins added to cement mortar, of which casein was one. Chandra and Aavik tested two casein specimens: one containing 0.075% casein by mass, the other containing 0.1% casein by mass. Percentages reflect the mass of protein added in relation to the mass of cement [14]. The research group found that the strength of casein-containing cement mortar was 42.5 MPa and 39.0 MPa for the 0.075% casein and 0.1% casein specimens respectively, compared to 43.0 MPa for standard cement mortar [14]. Flexural strength was 9.0 and 8.1 MPa for the 0.075% casein and 0.1% casein specimens respectively versus 7.0 MPa for standard cement mortar [14].

Chandra and Aavik indicated that the addition of casein causes an increase in air entrainment within the specimen [14], thereby reducing the compressive strength of the specimen. Air entrainment was reported to be 8.3% and 8.1% for casein-containing mortar versus 4.3% for standard cement mortar [14]. Chandra and Aavik noted that the addition of casein protein improves adhesion properties [14], despite increasing air entrainment.

Given the effects noted on casein in cement mortars, it was predicted that for the casein experiments in this study, the addition of casein into historic mortar would behave similarly, with the strength values being lower as a result of the type of mortar used.

2.2 Horsehair and other fibrous materials in mortar

Previous tests on the insertion of horsehair or similar fibrous materials into masonry mortar have been successfully tested [16] [17] [18]. It has been demonstrated that the addition of fibrous materials increased the tensile strength of the mortar specimen without any major effect on the mortar ductility. Evidence of the presence of horsehair in mortar dates to medieval times, similar to the known origins of the presence of casein in mortar.

Van Strydonck et al. [17] evaluated the constituents in historic mortar specimens by passing then through a sieve with apertures measuring 250 micrometres in diameter. As natural materials are organic materials, carbon dating was used as an estimation of the age of the specimens. The mortar was found to date between 1215 C.E. and 1297 C.E [17].

Ingham [16] outlines various means for the diagnosis of impurities in mortar from a durability and deterioration standpoint. The research report mentions the use of animal hair being contained in limestone-containing materials [16]. While much of the discussion focuses primarily on the existence of horse hair and other domesticated animal hair in plasters, mention of mortars was also made [16].

Chan and Bindiganaville [18] tested the addition of polypropylene fibres into lime mortar. Such fibres were mixed into a natural hydraulic lime (NHL) mortar at 0%, 0.25% and 0.5% composition by volume. Tests were conducted using NHL-2 based mortar, having a compressive strength of 2 MPa. The mortar was produced by mixing the natural hydraulic lime with masonry gold sand in a 1:3 ratio [18]. Chan and Bindiganaville concluded that propylene fibres increased the tensile and shear strengths while reducing the compressive strength. The first test, the compressive test, was conducted directly using a hydraulic press as shown in Figure 3-1. The strength was determined to be 1.3

MPa for the control sample and 0.39 MPa for both fibre concentrations [18]. A flexural test of the mortar was conducted by placing a hydraulic press in the middle of a specimen between two rollers acting as a simply-supported beam [18]. The rupture strength was found to increase marginally for the fibre-containing sample. Rupture strengths were reported as being 0.193 MPa for the control sample, 0.205 MPa for the 0.25% fibre sample, and 0.39 MPa for the 0.50% fibre sample [18]. The direct shear test was conducted in a similar manner to the flexural test, except with the supports and press surface flat, such that a shear plane failure occurred, as shown in Figure 2-1. The shear strength was found to be 0.480 MPa for the control sample, and 0.443 MPa for the 0.25% fibre sample and 0.590 MPa for the 0.50% fibre sample [18].

Figure 2-1: Direct Shear Test of Fibre-Reinforced Mortar Specimen in Chan and Bindiganaville [18]

Panesar et al. [19] assessed the use of cellulose nanofibers in lime mortar compounds [19]. It was reported that the flexural strength increased from 4.63 MPa for a lime mortar specimen with no fibre content to 7.53 MPa with the highest fibre content

[19]. Results of the research are presented below in Table 2-1. It was not reported how many samples were tested in order to produce this statistical data, nor the fraction of fibres to the total specimen. While the fibre content was specified, the units of the fibre content were never explicitly specified in the report. Panesar et. Al concluded that these fibres improved the tensile, flexural and shear strengths, while coincidentally reducing the compressive strength.

Table 2-1: Summary of fibre research by Panesar et al. [19]

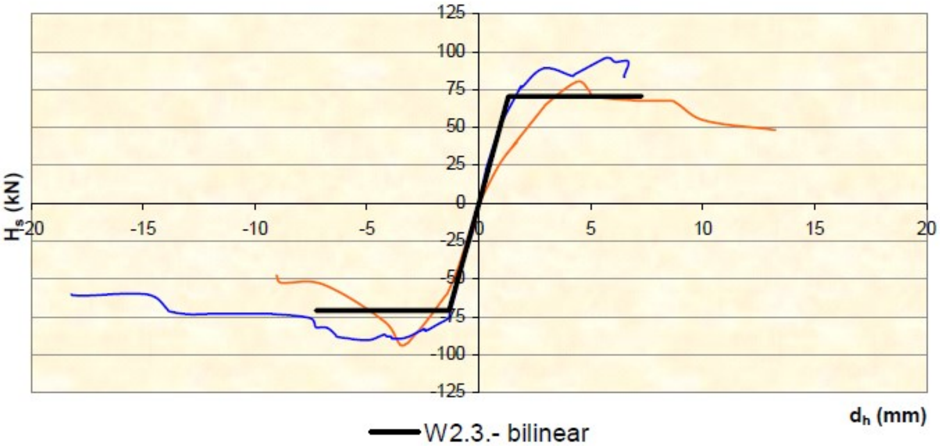
Fiber content	Control specimens (no fiber treatment)		Specimens containing treated fibers	
	Mean (MPa)	Standard deviation (MPa)	Mean (MPa)	Standard deviation (MPa)
0	4.63	0.55	–	–
0.05N	8.24	0.84	7.41	0.17
0.1N	7.81	0.71	6.08	0.38
0.2N	7.33	0.45	7.73	0.9
0.4N	7.53	0.39	7.69	0.44

Silva et al. [20] tested mortars reinforced with synthetic fibres. Two fibres were tested in detail: a fibre made of a polyethylene and poly propylene blend, and a fibre made of a polyacrylonitrile blend [20]. While only static testing was performed, the research presents interesting conclusions regarding the material properties. It was found that there was, in the best case, a 40% increase in a fibre-reinforced mortar containing a superplasticizer, exhibiting a compressive strength of 40.2 MPa and a flexural strength of 8.3 MPa, in comparison to a control sample without any fibre materials, which had a compressive strength of 29.1 MPa and a flexural strength of 5.9 MPa. Mortars without a superplasticizer were found to have substantially less improvement, observing a 8.6% decrease in compressive strength and up to a 7% increase in flexural strength [20]. Despite an improvement in strength, issues were raised with the use of polyacrylonitrile fibres; it was discovered that the workability of the mortar worsened with the addition of fibres [20].

2.3 Joint Reinforcement to improve strength

Reinforcement within the bed joints has been conducted in a masonry repair applications and has been proven to improve in-plane shear resistance of historic wall assemblies.

Studies conducted by Gouveia and Lourenço [21] tested walls reinforced in the joints on a shake table, creating a simulated seismic activity. A total of six walls were tested: three were confined by reinforcement around the perimeter, while three had horizontal bed joint reinforcement. One of the walls was a control wall with no reinforcement, while one wall had both confinement and joint reinforcement [21]. All walls were 143 mm in thickness [21]. Tests identified the lateral stiffness of the masonry wall, K, to be equal to the applied lateral load, H, divided by the horizontal displacement of the top of the wall, d [21]. The material used was concrete masonry units with dimensions of 200 mm × 96 mm × 143 mm [21]. An example is shown below in Figure 2-2.

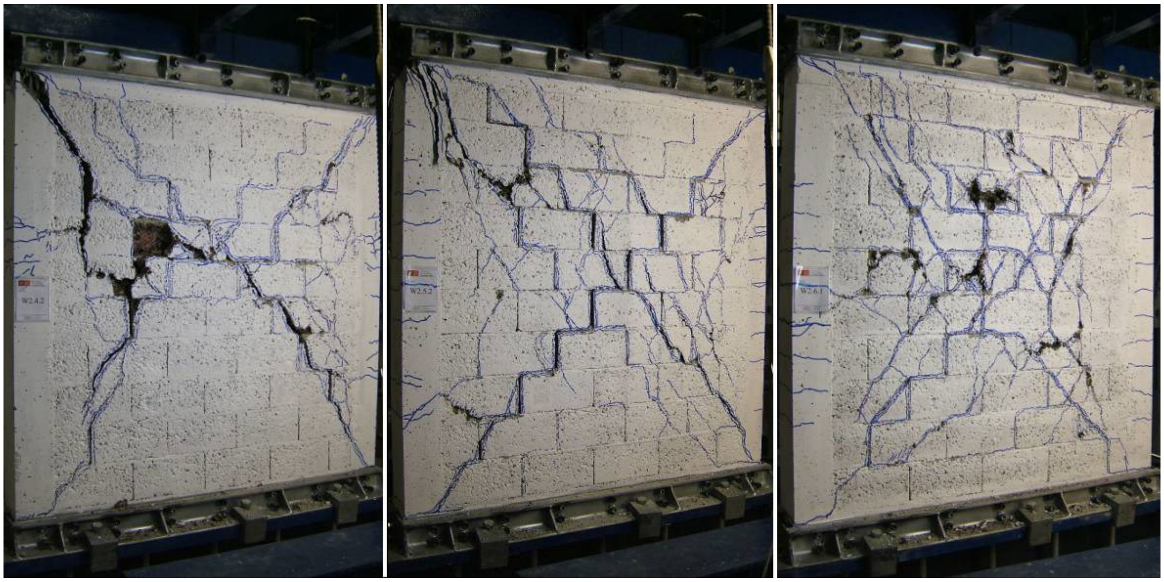


[21]

Figure 2-2: Load versus lateral displacement of unconstrained but horizontally reinforced wall sample [21]

(a) (b) (c)

Figure 2-3: Masonry walls not confined by reinforcement [21]



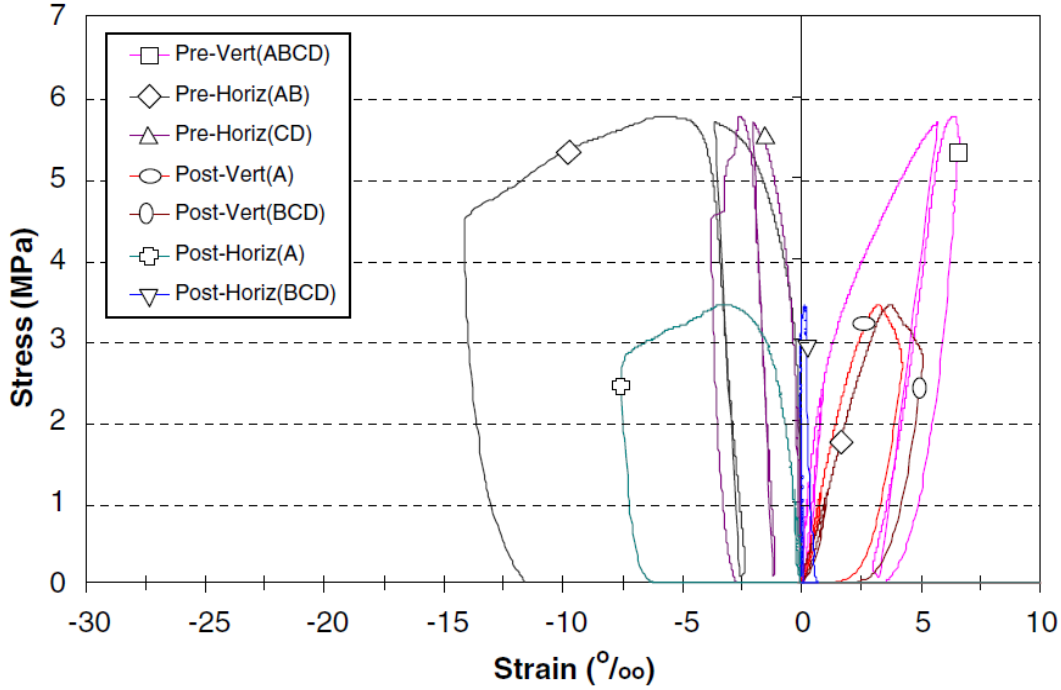
(a) (b) (c)

Figure 2-4: Masonry walls confined by reinforcement [21]

Studies conducted by Modena et al. [22] assessed various methods of strengthening historic masonry walls. The test experiments involved repointing the outermost 60 millimetres of the masonry walls and installing reinforcing bars that were 6 millimetres in diameter, 30 millimetres from the surface of the mortar joint [22]. Testing was undertaken by installing crack monitors wherever visual cracks were present. Installation of the reinforcing bar during repointing is shown in Figure 2-5.

Figure 2-5: Installation of reinforcement in existing masonry wall [23]

Unlike the load-displacement curves presented in the research of Gouveia and Lourenço, Modena et al. presented the results in a stress-strain curve as shown below in Figure 2-6 [22]. It was reported that the stresses were reduced by the addition of horizontal reinforcement within the structure.



[22]

Figure 2-6: Stress-strain curves for reinforcement retrofit. Note that all stresses (compressive and tensile) are shown to be positive [2].

Studies conducted by Tomazevic, Lutman and Petkovic [24] researched the seismic effects of reinforcement in unreinforced and reinforced masonry walls constructed from concrete masonry units. Concrete masonry units of 200 mm × 100 mm × 100 mm nominal dimension were used in the testing [24].

Tomazevic et al. [24] tested four different types of dynamic ground displacement in the masonry wall specimens used: monotonic (where the displacement increased in a linear fashion), cyclic displacements with an increasing amplitude over time, displacement amplitudes, repeated three times at each amplitude peak, with decreasing amplitudes between consecutive pulses, and a random motion pattern, simulating an earthquake [24].

In all cases, it was found that the stiffness decreased as the load increased, as shown below in Figure 2-7 [24].

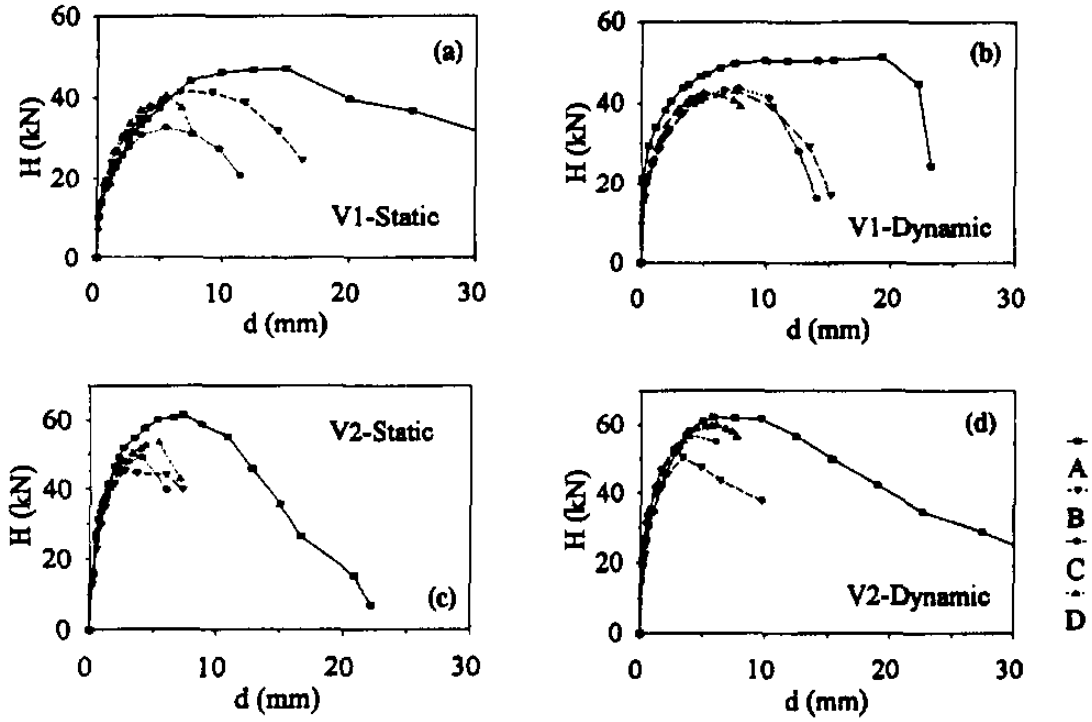


Figure 2-7: Load-displacement curves. [24]

By the equation established by Gouveia and Lourenço [21], the stiffness is the slope of the above curve in the elastic region, up to 40% of the peak load. The peak of the graph represents the ultimate point of failure of the shear wall.

Potential issues raised by the authors with these tests were that the masonry wall samples were constructed at half-size and at a half scale, however, full size masonry units were used in the construction of this same wall. While the interaction was similar, it may not be the same as a full-scale wall. At least one full-scale wall should have been built in order to compare any effects.

Issues raised with respect to reinforcement exist regarding heritage conservation. The confinement system used a series of vertical and horizontal reinforcing bars installed through the masonry. While this may increase the strength as presented in the report, this retrofit requires almost complete disassembly and reconstruction of the wall system, and therefore is not practical for most conservation applications.

2.4 Summary of Other Related Tests

Due to the lack of previous research focusing on the historical use of casein in masonry mortars, similar historic additives and retrofit techniques were reviewed in this chapter to supplement the little information available on casein and to determine how the testing would be undertaken. The one test that used casein showed that the addition of casein caused a marginal improvement in flexure. Other traditional additives were similarly used for strengthening. Based on this review, the preliminary hypothesis for this work was that the addition of casein would reduce the compressive strength [14] while improving the strength in tension and in flexure. This behaviour could be advantageous in a masonry wall system where repointing or filling a wall cavity is required, and could potentially serve as an alternative for undertaking extensive interventions necessary to retrofit reinforcement into an historic masonry wall.

Chapter 3: Review of Previous Tests and Standards

In order to devise an appropriate experimental procedure, a thorough review of previous tests on mortar additives was necessary, along with the equipment used to produce the results. Procedures were based on adaptations of established mortar testing procedures in a laboratory environment, adjusted when necessary to compensate for the alterations in material consistency observed by the addition of casein. Each method necessitated assessing the theoretical calculations required and the logistics of implementing the test in the laboratory setting. From this, an appropriate test method was selected. Various different researchers have used different test methods for arriving at the same ultimate conclusion.

The American Society for Testing Materials (ASTM) Standards [25] [26] [27] [28] [29] describe the dimensions and processes to test tensile and compressive mortar strengths, as well as brick-to-mortar bond strength and shear masonry bond strength. All of the ASTM Standards related to mortar testing specify that a minimum of three mortar prism samples shall be tested [26] [28] [27], and the results shall be within 10% of each other in order to make use of the test results in an engineering design [26]. Previous research testing mortar cubes and small masonry prisms was assessed for its procedure.

3.1 Compressive Strength Test Methods

Of the four tests in this study, this is the most straightforward test. This test is routinely undertaken following the procedure established in ASTM Standard C109 [26].

This procedure calls for the casting of a mortar cube measuring 50 millimetres in each Cartesian direction, and inserting into a testing machine with two smooth flat surfaces, such that a uniform compressive load dis applied across the face of the mortar cube. This method was used by Chan and Bindiganaville, as shown in Figure 3-1.

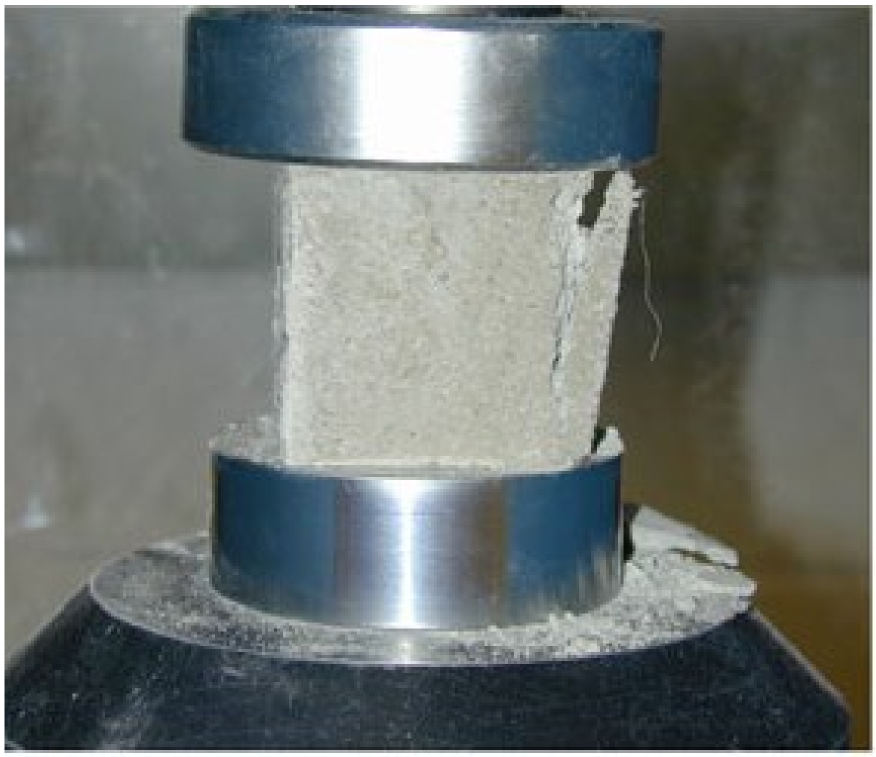


Figure 3-1: Compressive test conducted by Chan and Bindiganaville [18].

3.2 Tensile Strength Test Methods

The ASTM Standard C307 [28] requires the casting of oblique-shaped briquettes to enable splitting at the necked area on the specimen. The necked area of the specimen shall be a 25 mm × 25 mm cross section [28]. In this test, the failure stress is defined by the load divided by the area at the necking point. The ASTM Standard C307 for mortar tension tests is seldom used in practice due to complexities in the setup of the test bracket, and has not been referenced by any of the previous studies on lime mortar researched for this paper [28].

The split cylinder test defined in ASTM Standard C496 [27] is intended for use on concrete. It appears, however, that this standard has been used in other research to test masonry mortar in the past [16] [30] [31]. In the split cylinder test, the tensile stress is defined as the compressive load divided by the cross-sectional area of a diagonal cut through the cube, as shown in Figure 4-8 and Figure 4-9.

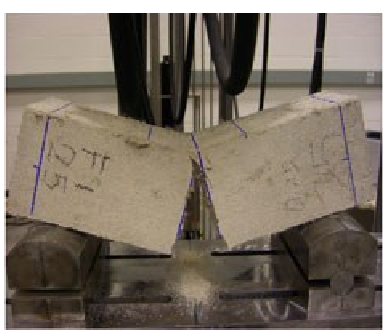
Some experimental work has been conducted by Ince [32] and Xu and Reinhardt [30], placing concrete and mortar cubes on the diagonal in order to determine the tensile strength. Lin and Wood [31] include references to indirect splitting testing conducted by Davies and Bose in 1968. The equations determining the tensile stress in this manner are similar to those used in the split cylinder test outlined in ASTM Standard C496 [27], where the diameter is defined as the area of a diagonal cut of a mortar cube between two opposite edges. This may be referred to alternatively as the Double-K method [31], named such by the cross section of the mortar cube support equipment. Similar to the double punch test, the only relationships found involve constructing a finite element model to determine the relationship between the transverse tensile stress and the applied compressive load, then working through the relationship results to determine the stress at failure through the compressive load at failure. This method requires knowing the entire stiffness curve first, before knowing the tensile point of failure.

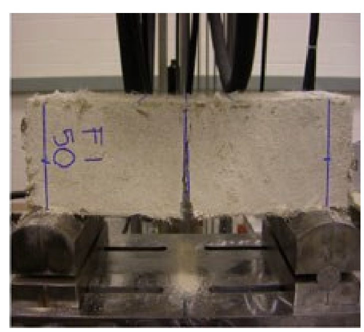
Conducting tensile tests by punching is a possible alternative to the direct tension test outlined in ASTM C307 [26]. This is typically undertaken by means of a double punch test, using two small metallic punches (or other material with a higher strength and stiffness). The relationship between load at failure and the tensile stress requires a three-dimensional finite element analysis with contact modelling [33]. Both the punch and the mortar specimen must be modelled in order for this method to be used. Due to the complexities in analyzing the results of a double-punch test, it was therefore decided that this method should not be used.

3.3 Flexural Strength Test Methods

3.3.1 Flexural Strength of Mortar

The method chosen for flexural testing experiments was to use a masonry block and to apply a central load in bending, despite not conforming to ASTM Standards. It was noted by the authors that this Standard is intended for fibre-reinforced concrete [34], yet it was used for masonry nonetheless. The method used herein is that described by Chan and Bindiganaville, referencing ASTM Standard C1609 [34]. Chan and Bindiganaville used rectangular prisms, measuring 100 millimetes in width and in height, and 400 millimetres in length [18]. The curved surfaces acted as pinned, point loads [18].





(a) (b) (c)

Figure 3-2: Flexural Testing undertaken by Chan and Bindiganaville [18]

The flexural test is based upon the principle that the stress distribution in a concrete-type beam is linear until the point of first crack on the tension side. By measuring the load applied, and given the distance between the point of load application and the support points, the bending moment at the point of load application can be determined. From a known bending moment and cross section, the tensile stress at the outermost surface can be determined.

3.3.2 Flexural Bond Strength

Flexural bond strength testing was conducted in accordance with ASTM Standard C1072 [25]. This involved using a minimum of two brick masonry units with a bending moment applied between the bricks [35]. The standard calculations are based upon the principle that a linear stress distribution exists across the masonry until the first crack occurs, at which point tensile failure has been reached. The properties of casein, which was historically used as a binder in glues, make the tensile bonding test particularly interesting.

3.4 Shear Strength Test Methods

3.4.1 Mortar Cube Tests in Shear

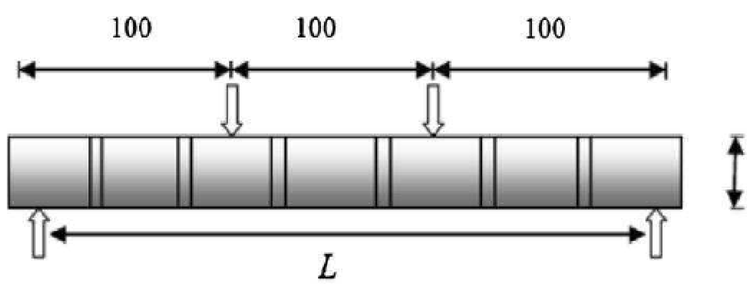
Currently, no ASTM standard is available for testing the shear strength of masonry mortar. This test was desired for purposes of completeness and to facilitate a comparison with shear bond tests. In practical modern applications, only the compressive strength is typically desired. One of the few tests found was that undertaken by Chan and Bindiganaville [18]. The research group undertook a direct shear test by using a shorter beam section than that used in the flexural test, such that a plane shear failure occurs as per short and deep beam theory. Equipment supports were flat, smooth surfaces offset from each other as opposed to the rounded surfaces used for the tension test [18]. A further advantage of the tests undertaken by Chan and Bindiganaville [18] was that most of the same equipment could be used for both the tensile strength test and the shear strength test. The only difference between the two tests was the shape of the supports and the distance between the supports.

3.4.2 Brick-to-Mortar Bond test in Shear

ASTM Standard E519 outlines the procedure to test the shear strength of a concrete block-to-mortar joint [36]. This is done by means of a diagonal compression test. Similar tests have been conducted by Ismail and Ingham [37], Alecci et al. [38], and by Borri et al. [39].

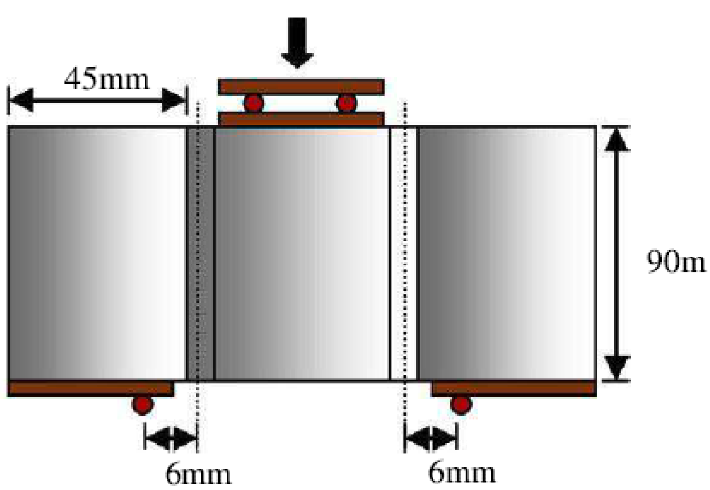
Potential concerns of this test are complexities in the setup. The test was designed and intended for testing concrete masonry units. This test has been undertaken by research groups on brick masonry in the past, with the following modifications: brick masonry units used instead of concrete masonry units, and the dimensions have been reduced from that specified in the ASTM Standard test.

Thamboo et al. [40] tested bond strength between units. Test apparati were similar to those used by Chan and Bindiganaville [18] for flexural and shear strength, except on a larger scale. Masonry units were arranged such that the mortar bed joints were aligned to the vertical axis. Rollers were used to initiate beam bending behaviour, and plates were used to simulate a shear failure [40]. While the tests were conducted on concrete masonry units, the report serves as a sample design regarding the construction of the masonry prism tests which are needed for this thesis paper.



[40]

Figure 3-3: Experimental flexural bond test setup by Thamboo et al. [40]



[40]

Figure 3-4: Experimental shear bond test setup by Thamboo et al. [40]

Thamboo et al. conducted tests after 14 days, 28 days and 56 days. Shear bond strength was determined to increase from 0.82 MPa to 0.93 MPa for wet-cured mortar specimens and increase from 0.89 MPa to 1.29 MPa for dry-cured mortar [40]. These strength comparisons were between 14 and 56 days [40].

While the strength comparison was the ultimate result, the mode of failure of the unreinforced lime mortar may be of interest. Costigan and Pavia [41] investigated the failure modes of lime mortar specimens. It was found that plastic deformation behaviour occurs with a lower-strength natural hydraulic lime mortar by itself (strengths of less than 2 MPa), while higher-strength mortars deform elastically to a brittle failure [41]. Given that the published strengths of the mortar that was used were less than 2 MPa for the short-term curing [42], it was expected that some plastic deformation would occur in the experiments that were to be conducted.

Chapter 4: Methodology

4.1 Methodology Background

The experimental tests involved the testing of natural hydraulic lime mortars with varying content of casein from 0% by mass to 1.5% by mass. Later tests would use a casein content not exceeding 0.5% by mass. A second set of tests was conducted, maintaining 0.5% casein by mass while varying the amount of water added to the mortar mixture. The amount of water added ranged between 3.5 litres and 5.5 litres for every 30 kilograms of mortar. While this is not the standard unit for specifying water content, this measurement unit was chosen as it reflects how mortar would be mixed by a masonry contractor in the field. Tests were conducted at 7 days, 28 days and 56 days to assess the setting process and the effects on this process caused by the addition of casein. Mortar cubes were casted for testing in compression and tension. Mortar prisms were casted to test bending and shear. Brick and mortar stack specimens were prepared to test cohesion, referred to hereafter as bond strength.

Workability in itself is a qualitative metric, and therefore, there is no one standard definition of workability in masonry mortars that is universally accepted. This report classifies workability as good or poor, based upon the worst case of the flowability, defined in Section 5.1.2, and strength hardening during the first two hours after mixing; a mortar will not be workable if it results in poor flow or hardens too rapidly. Wet mortar may be tested for flow rates and viscosity, the former of which was conducted in testing.

In a retrofit application, a higher workability that that needed for a new wall or wall rebuild is desired.

The primary intent of this experiment was to maintain consistent procedure between the different test specimens to facilitate a comparison of results. The specimens used a pre-mixed mortar, a packaged product, typically distributed in 30 kilogram bags in Canada. It contains a set ratio of lime, which may be a single type or a mixture of different types of lime, and masonry sand. The product used a 2:5 lime-to-sand ratio, that may not comply exactly with the lime-to-sand or cement-to-sand ratios specified in ASTM C270 [43]. Being a pre-mixed product, the ratio could be maintained constant throughout all experiments. Therefore, the ratio should bias the experimental results as little as possible. Nevertheless, natural tolerances in lime mortar and in the exact lime-to-sand mix were still existent.

Typical strength tests for the purposes of determining strength of a lime mortar product are conducted over a longer duration than cement mortar; this particular product reported test values after 7, 28 and 90 days instead of 7, 14 and 28 days. These were the durations specified by the manufacturer of the pre-mixed mortar. It was noted in the manufacturer's specifications that full strength is not reached until 365 days, however, testing at this duration was not conducted due to time constraints. The manufacturer's published strength at 90 days was 2.2 MPa, 75% of the 365-day published strength of 3.5 MPa [42].

4.2 Summary of Types of Tests Conducted

Compressive, tensile, shear and flexural tests were conducted. These tests were conducted after 7 days, 28 days and 56 days. Brick to mortar bond strengths were also conducted, employing two different means: a tensile bond test and a shear bond test.

Table 4-1: Summary of Relevant Standards for Mortar Specimen Tests

Time curing	7 days	28 days	56 days	ASTM Standard Reference
Mortar Prism Tests				
Compression	✔	✔	✔	C109
Tension (double-K)		✔	✔	C496
Tension (flexure)		✔	✔	C348
Shear		✔	✔	Chan and Bindiganaville [18]
Brick-to-Mortar Joint Interface Tests				
Tensile bond			✔	C1072
Shear bond			✔	Thamboo et al. [40]

Each casein-content and water-content test, represented as a single checkmark in Table 4-1, involved a minimum of 33 separate specimens for casting and testing. A total of over 320 specimens were tested. Surplus samples were cast to accommodate for accidental breakage. The number of repeats conducted for each case is shown below in Table 4-2.

Table 4-2: Summary of mortar cube tests conducted

Test Type	Time Days	0% Casein	0.1% Casein	0.25% Casein	0.5% Casein					1.0% Casein	1.5% Casein
		5.5 L / 30 kg	5.5 L / 30 kg	5.5 L / 30 kg	5.5 L / 30 kg	5.0 L / 30 kg	4.5 L / 30 kg	4.0 L / 30 kg	3.5 L / 30 kg	5.5 L / 30 kg	5.5 L / 30 kg
Compres-sion	7	3	3	5	3	3	3			3	3
	28	5	4	4	6	3	3	3	3		5
	56	4	4	4	4	3	3	3	3		3
Tension	28	4	4	4	5	3	3	3	3		5
	56	4	5	5	4	3	3	3	3		3
Shear	28	8	6	5	12	6	6	6	6		12
	56	8	9	8	10	6	6	6	6		6
Bending	28	4	4	5	4	3	3	3	3		
	56	3	4	3	3	3	3	3	3		2
Shear Bond	56	2	2	3	3						
Flexural Bond	56	5	4	4	7		2	1			

4.3 Lime Mortar Specification

The mortar used for all mortar samples was a pre-mixed natural hydraulic lime mortar named HLM-500 [42], produced by King Masonry Products. The product is a 2:1:5 ratio by volume of Natural Hydraulic Lime to Hydrated Lime to Masonry Sand. The published compressive strengths from the manufacturer are 1.1 MPa after 7 days, 2.2 MPa after 28 days, 4.3 MPa after 90 days and 5.5 MPa after 365 days [42].

The manufacturer's specifications indicate that 5.5 litres of water should be added to each 30-kilogram bag of mortar. There was no means to measure the water contained within the hydrated lime powder; this was treated as a constant given the similarity in environmental conditions between all tests.

Initial test mortar cubes were cast before the test wall samples were constructed in order to define the test matrix. It has been noted in previous testing by the National Research Council of Canada [13] that the addition of too much casein in the mortar will prevent the mortar from properly hardening.

4.4 Casein Specification

The casein sample used was a pure Micellar Casein, produced by NKD Nutrition. It was produced by means of milk extract, with no further chemicals added.

4.5 Specimen Preparation Procedure

4.5.1 Mortar-curing environment

All specimens were kept in a high-humidity environment, either by using a humidification cabinet as shown in Figure 4-1 or by completely covering the mortar specimens with wet towels. Inside the high-humidity chamber, the mortar cubes were kept in a saturated lime water solution, and the brick and mortar stacks were covered in moisture-proof bags as shown in Figure 4-2.

Figure 4-1: Humidification Chamber used to maintain greater than 90% humidity level

Figure 4-2: Stack bond samples curing in humidification chamber before testing

4.5.2 Determining quantities of constituents in the mortar mixture

The amount of casein was measured using a centigram balance, and the water was measured using either a graduated cylinder or a balance, based on a density of 1000 kilograms per cubic metre. The water content was deemed to be precise to within 5 millilitres.

4.5.3 Mixing Procedure

The casein protein was added to the water first as shown in Figure 4-3 and mixed thoroughly by hand, such that the final casein/water mix was as shown in Figure 4-4. This casein-and-water mix was then added to the mortar, which was measured before the addition of the water and casein mix in a bucket using a decigram balance, deemed to be precise within 0.1 grams.

Figure 4-3: Casein and water before mixing

Figure 4-4: Casein and water thoroughly mixed

Figure 4-5: Dry mortar mix, with lime and sand pre-portioned

A paddle mixer was used for those mixes involving more than 5 kilograms of mortar. For preliminary test mixes where only a small quantity of mortar, less than 2 kilograms, was needed, mixing was conducted by hand. Mixing was continued until the mortar was visually a near-homogeneous consistency throughout. If it was discovered during pouring that some mortar was not mixed, all poured mortar was returned to the mixing container and re-mixed until a visually homogeneous consistency was obtained.

4.5.4 Casting forms

A combination of pre-manufactured brass cube forms and wooden cube forms that had been purposely constructed for this experimental testing, were used. Interior dimensions were constructed to within a tolerance of one millimetre. Compression and tension tests used a combination of brass cube forms—capable of casting a maximum of 12 mortar cubes—and wooden cube forms for the remainder of the mortar cubes, due to the need to cast a large batch at once. Flexural prism specimens used exclusively wooden forms due to the unavailability of pre-manufactured forms to cast the necessary prism form size. In order to prevent moisture absorption and minimize adhesion of fresh mortar to the formwork, the wood forms were immersed in an oil-based form-release agent for at least 24 hours during their construction. This was essential, particularly for the specimens containing a higher amount of casein. The humidity chamber in which the forms were retained during casting maintained close to 100% relative humidity and prevented the wooden forms from drying during the curing process. Petroleum jelly was used as an additional form-release agent for both the wooden forms and the brass forms; this minimized the number of mortar prism specimens that broke prematurely, prior to testing. It also prevented any discernable water absorption from occurring. By taking these measures, no discernable difference was evident on the surface after removing the mortar cubes from the casting forms.

Mortar Casts with 0.25% casein or less frequently had rough edges after casting, as shown below in Figure 4-6, even after tamping. This is a result of casting mortar into a small space. Mortar with less than four litres of water per 30 kilograms of mortar also exhibited rough edges as seen here. This was deemed to be the result of poor workability, as defined in Section 4.1.

Figure 4-6: Mortar cube casted from sample with poor workability

4.6 Mortar Prism Tests

ASTM C109 calls for a loading rate of 1.5 millimetres per minute [26]. The loading rate was not explicitly specified in ASTM C496 or ASTM C348. In order to maintain consistent test setup, all tests used a common loading rate of 1.5 millimetres per minute, which was found to be a sufficiently slow rate to observe fully static specimen behaviour, as shown by the load-displacement curves in Appendix A and Appendix B . The point of zero force and zero displacement were reset manually for every specimen test. Before the load cell made full contact, the zero displacement point was set manually at the point that the first minuscule non-zero load was detected by the load cell. See Figure 4-7, Figure 4-8, Figure 4-10 and Figure 4-12 for experimental setup.

4.6.1 Mortar Prism Testing in Compression

Compressive Strength testing is the most common test conducted on masonry mortar and is the most critical property in assessing masonry walls against modern masonry codes [11]. These tests were conducted in accordance with ASTM Standard C109, as shown in Figure 4-7. This Standard prescribed cubes of 50 millimetres in each Cartesian coordinate direction to be tested by means of a direct compression test [26]. A minimum of three cubes were to be tested in compression for each duration and for each different casein content [26]. The mortar cubes were inserted in a testing machine, the machine itself conforming to ASTM Standard C270. The load-displacement curve was also recorded; see Appendix A.1 for the load-displacement curves.

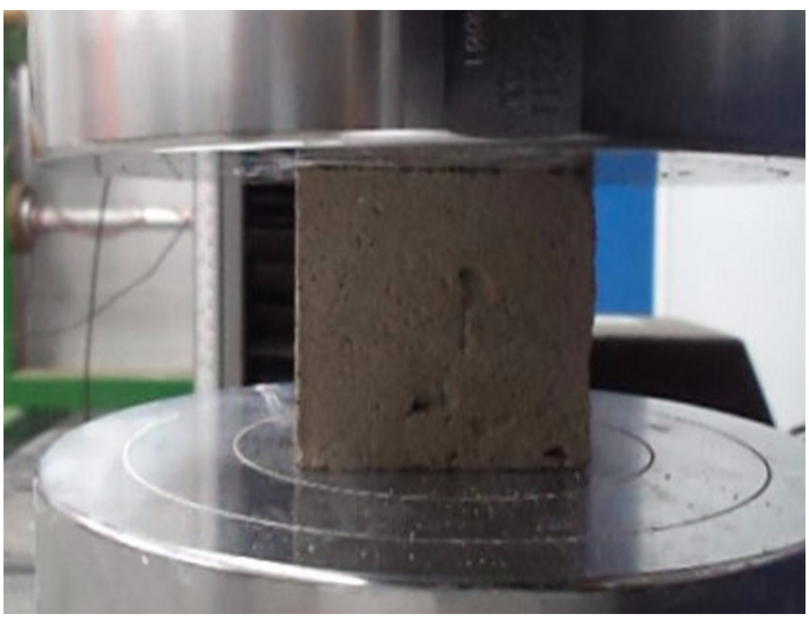
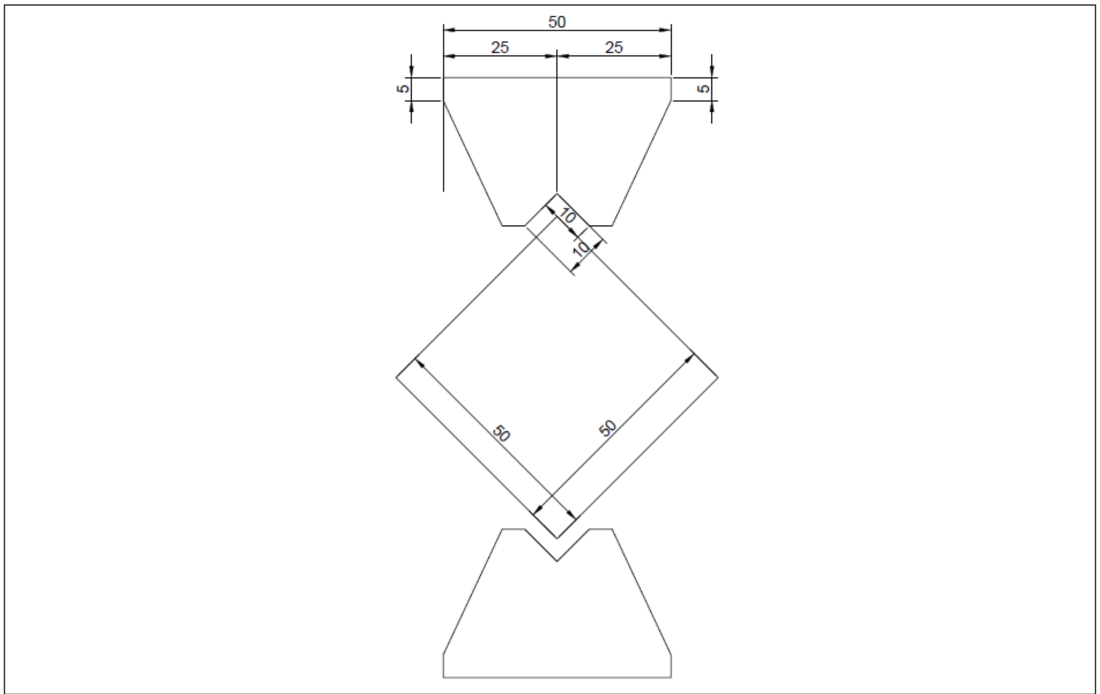


Figure 4-7: Compressive Test as conducted

4.6.2 Mortar Prism Testing in Tension

Tensile tests were conducted using the Double-K method outlined by Ince [32]. As with the compression test, mortar cubes 50 mm dimensions in each Cartesian coordinate direction were cast. While no dimensions were specified, 50 millimetres was chosen to maintain the same casting procedure and formwork as used for the compression samples. A minimum of three cubes were to be tested in tension for each curing duration and for each different casein content [26]. The mortar cubes were inserted in a testing machine, the machine itself conforming to ASTM Standard C270 [43]. The difference between this and the compression tests was the construction of an angled support bracket, applying pressure no more than 10 millimetres from the corner of the mortar cube along opposite edges, initiating a horizontal transverse tensile stress across the centre of the mortar cube. The support system typically uses two brackets comprising of a plate and small angle welded to each other, resembling the letter K, hence the name “Double-K Method”. Due to the construction tools available, it was easier to construct an equivalent support bracket out of a solid steel block, maintaining the 10 mm angle dimensions and requiring no welding. See Figure 4-8 for the experimental setup as constructed.



CONCEPTUAL TEST SETUP
NOT TO A STANDARD SCALE
MASONRY CUBE IS 50 mm × 50 mm × 50 mm

Figure 4-8: Double-K tension test as constructed

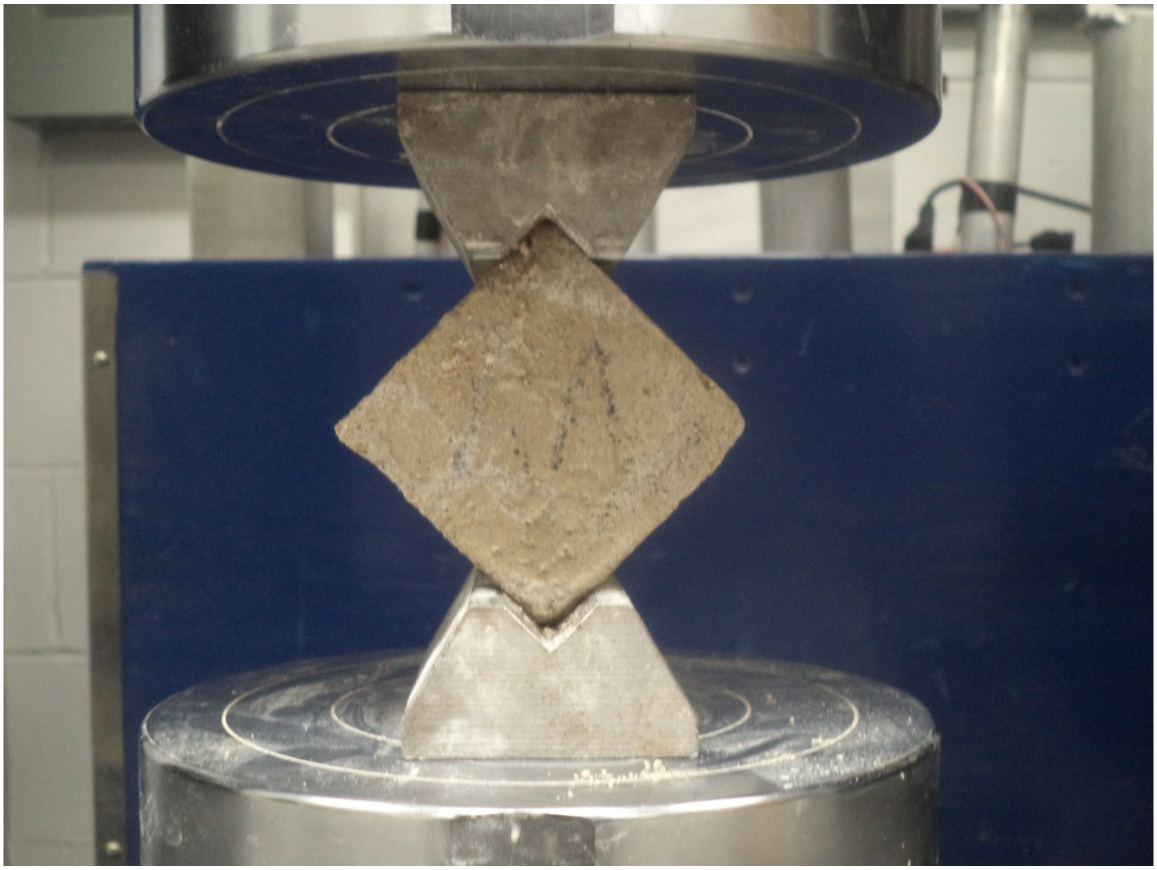


Figure 4-9: Tensile Test using Double-K Method as implemented

4.6.3 Mortar Prism Testing in Flexure

Flexural testing is used to determine the flexural strength, otherwise known as the modulus of rupture. Flexural mortar tests were conducted using the method outlined in ASTM Standard C348 [29], as described in Section 3.3. A 1:1:4 prism ratio was used to ensure that bearing stresses through the specimen were not a concern. The dimensions were 160 millimetres in length, 40 millimetres in width and 40 millimetres in height. The span distance was 130 millimetres between supports, with the load exerted 65 millimetres from each support, as drawn in Figure 4-10 and as shown in Figure 4-11. The arrow in Figure 4-10 represents the load application by the testing machine.

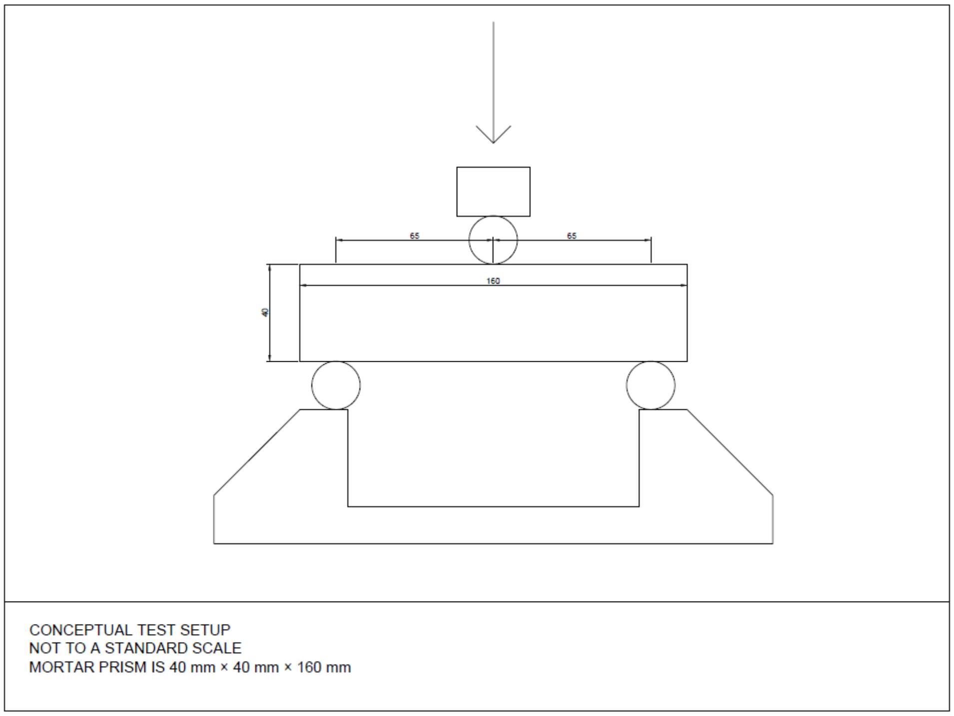


Figure 4-10: Drawing of flexural test setup

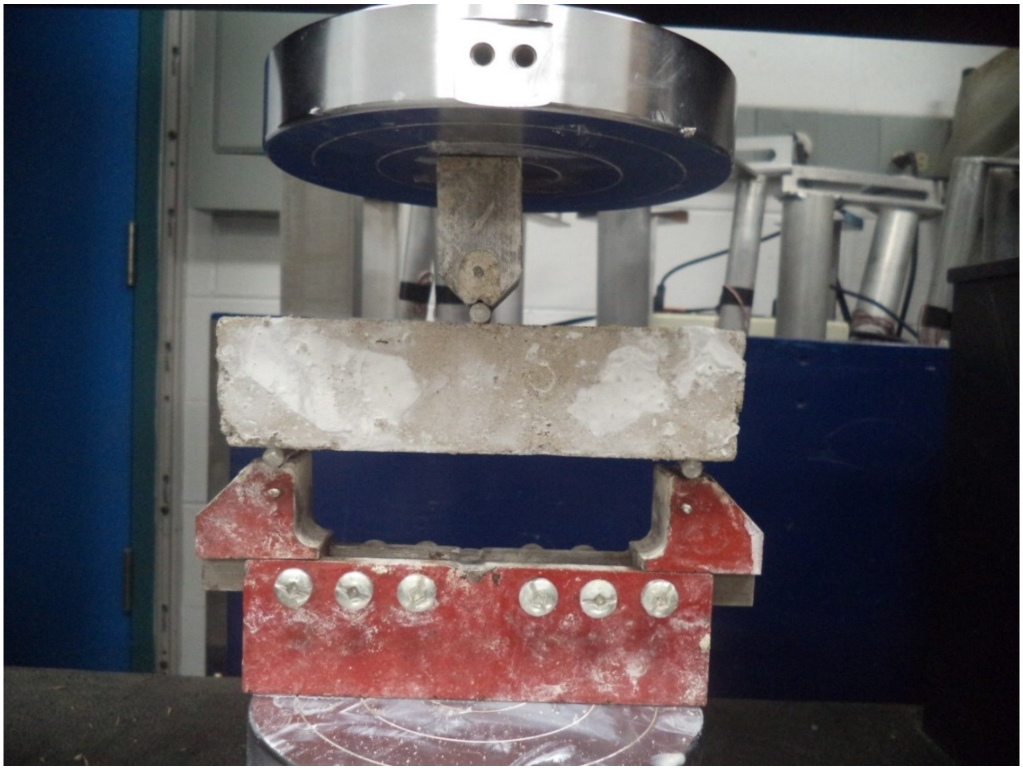


Figure 4-11: Flexural Test apparatus of mortar prisms as conducted

4.6.4 Mortar Prism Testing in Shear

Shear tests were conducted by means of a direct shear test as outlined by Chan and Bindiganaville in Section 3.4, as drawn in Figure 4-12 and as shown in Figure 4-13.The supports were flat surfaces, with a minimal distance—less than 5 millimetres—between facing supports. For the purposes of calculations, exact dimensions in the horizontal plane are not critical. The arrow in Figure 4-12 represents the application of load by the testing machine.

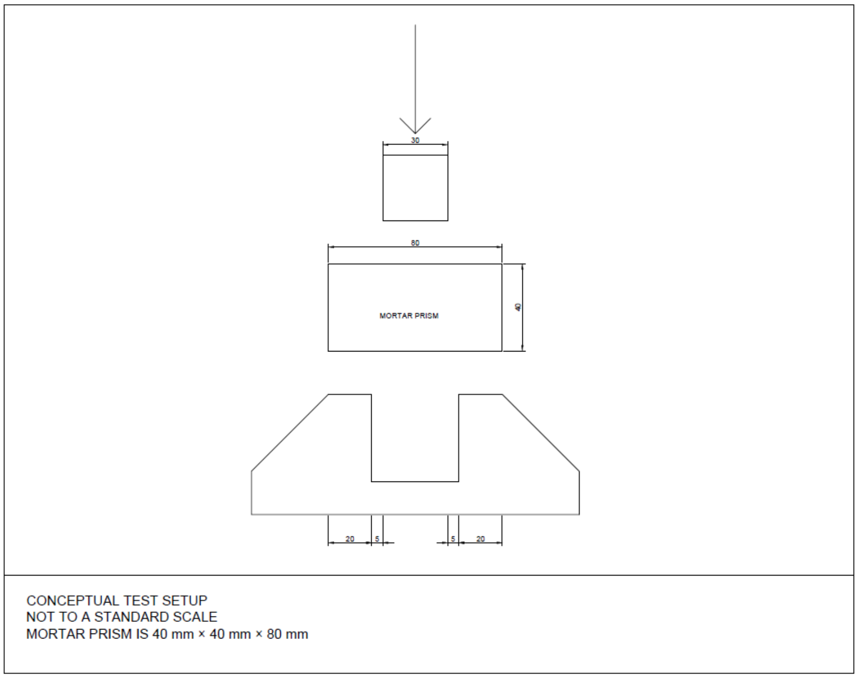


Figure 4-12: Sketch of shear strength test by direct shear

Note that the dimension of 80 millimetres is half the length of the flexural specimens at 160 millimetres. The fractured specimens from the 3-point bending test could be subsequently used to conduct shear tests as shown in Figure 4-13; each half was used for a shear test. The shear stress concentrations were across the midline of the specimen, while the flexural stress concentrations were localised around the point of failure on the underside of the specimen at midspan, which became the extreme corners in the shear test specimens.

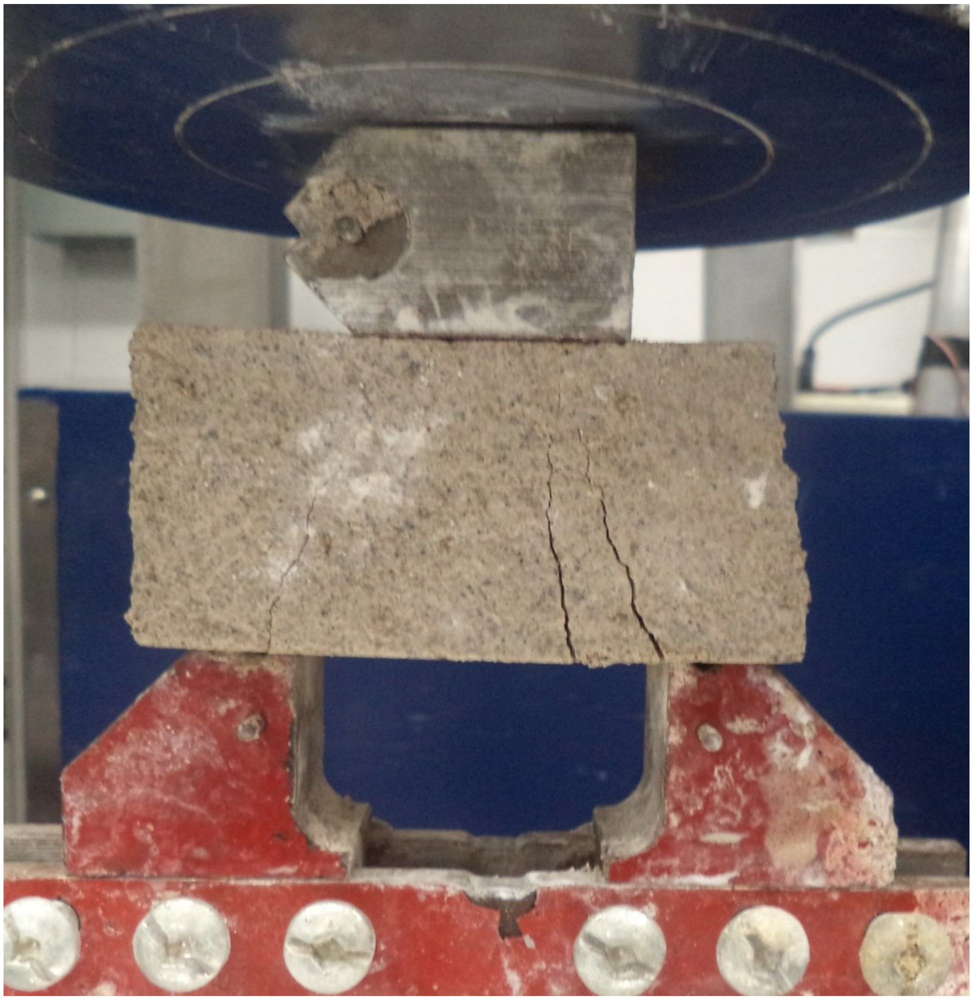


Figure 4-13: Direct Shear Test as Conducted

4.7 Brick-to-Mortar Bond Test

Bricks used were clay bricks intended for repair of masonry walls on heritage buildings, manufactured by the Watsontown Brick Company. These bricks are intended to be used in conjunction with lime mortars, as was used in testing. The structural properties of the brick unit in itself were outside of the scope of this research and were not tested.

4.7.1 Flexure Bond Test Setup

Flexure bond testing was conducted by means of a mechanical bond wrench tool in accordance with ASTM Standard C1072 [25].The primary variation was that instead of a free-standing apparatus, an apparatus attached to a fixed steel column was used, given the conditions available in the structural testing laboratory. A photograph of the bond wrench apparatus in use is shown below in Figure 4-16.

The lever arm acts as a clamp supported by the topmost brick. The load from the applied brick is transferred to the top brick. The clamping action induces a downward force on the front of the brick column while simultaneously inducing an upward force on the rear of the brick column.

The ASTM Standard C1072 also establishes the procedure for constructing stacks of bricks for testing [25]. It requires a form the size of the brick to be laid atop the previous brick, to be filled with mortar, as shown in , and to be removed with the following brick immediately placed after removal of the form [25]. As the bricks measured 102 mm × 210 mm, the length and width had to be increased by 10 millimetres in order for the form to fit atop the brick. Due to the increased flowability that the ASTM Standard C1072 was not intended to accomodate, the 0.5% casein specimens required that the form surrounding the mortar joint remain in place for several hours after filling in order to produce a proper mortar joint.

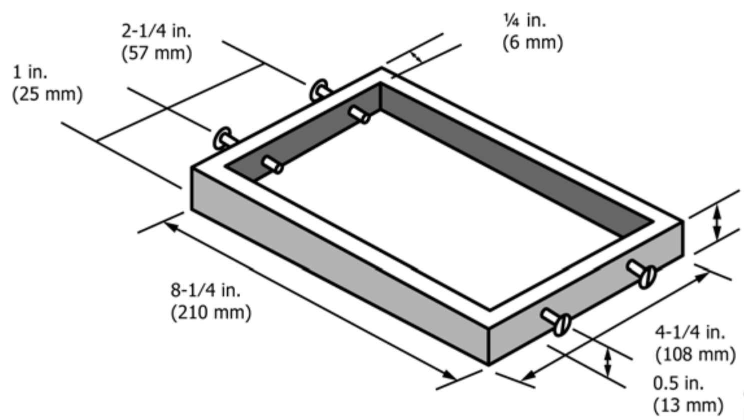


Figure 4-14: Form for stacking bricks as specified in ASTM C1072

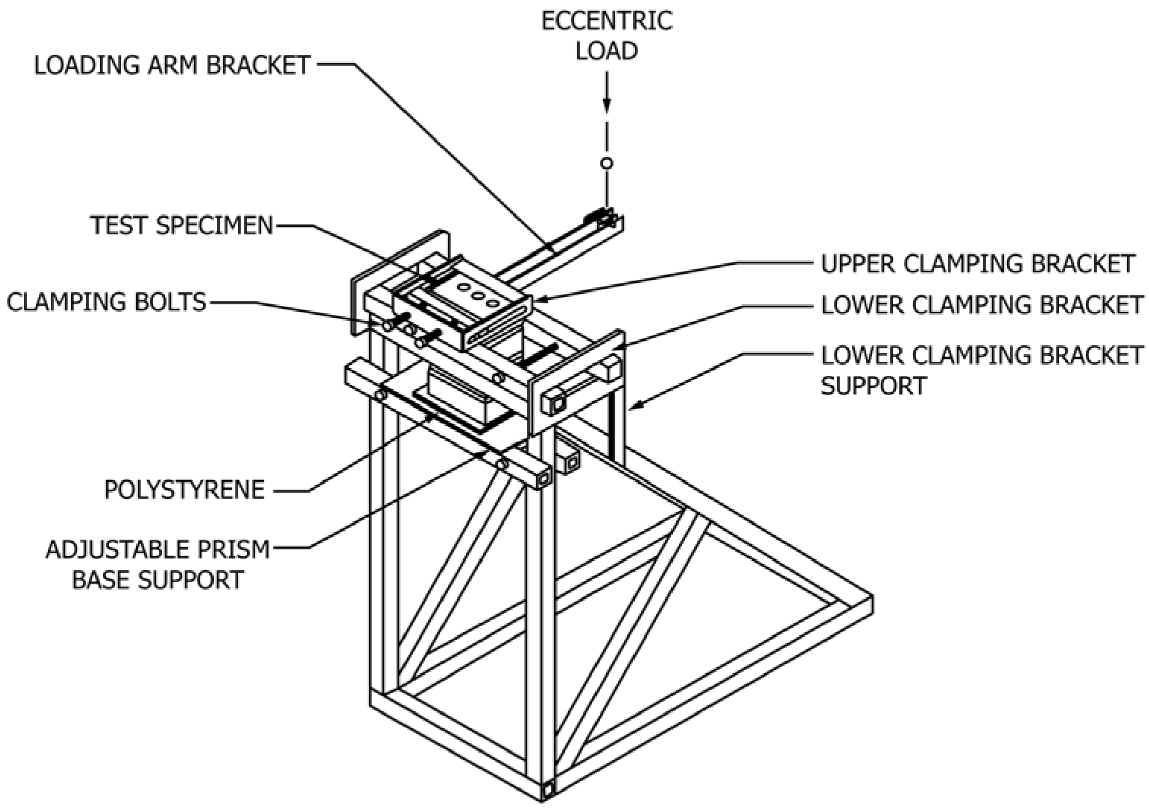


Figure 4-15: Bond wrench apparatus as illustrated in ASTM C1072 [25]

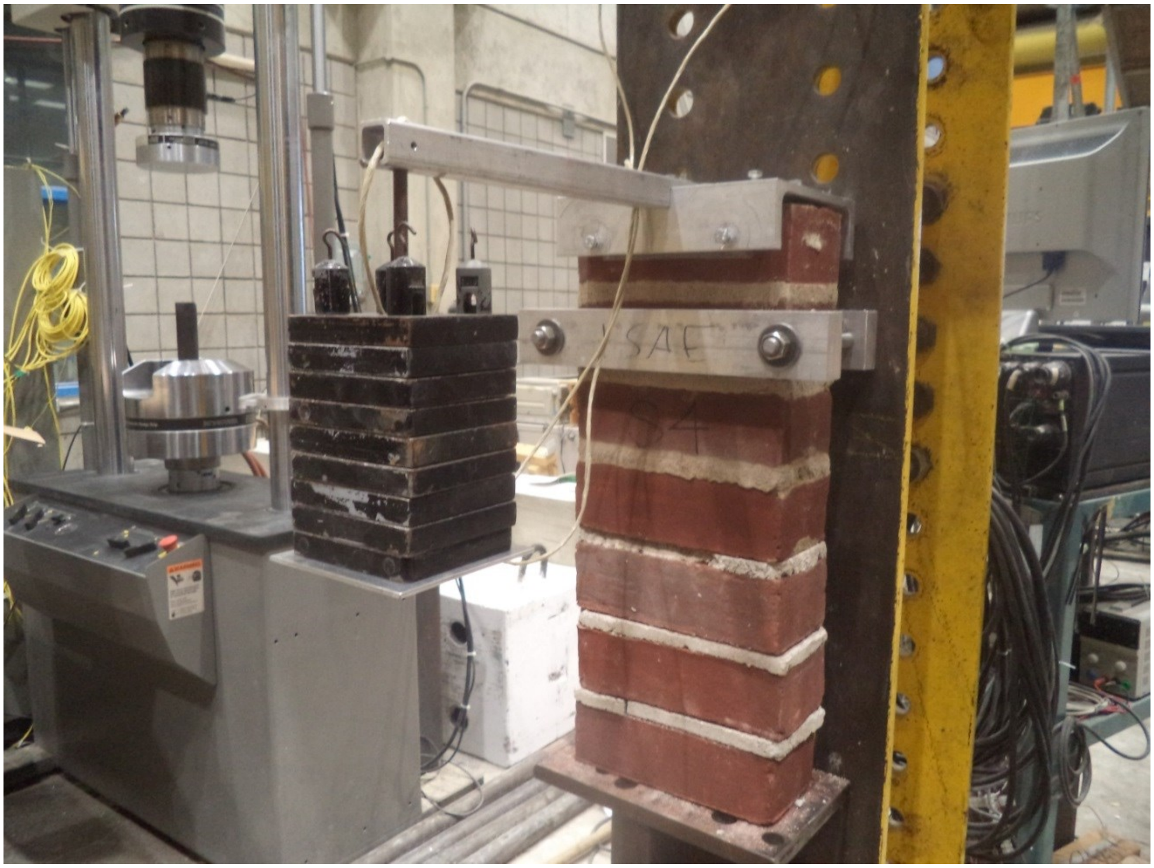


Figure 4-16: Bond wrench testing apparatus as constructed

The test apparatus had a mass of 1860.8 grams. The centre of mass was located a distance of 74 millimetres from the centre line of the brick assemblage. The eccentricity of the applied load was measured to be 339 millimitres. Assuming an acceleration due to gravity of 9.81 metres per second squared, it was possible to determine the forces exerted on the lever arm and consequently the bending moment on the mortar joint.

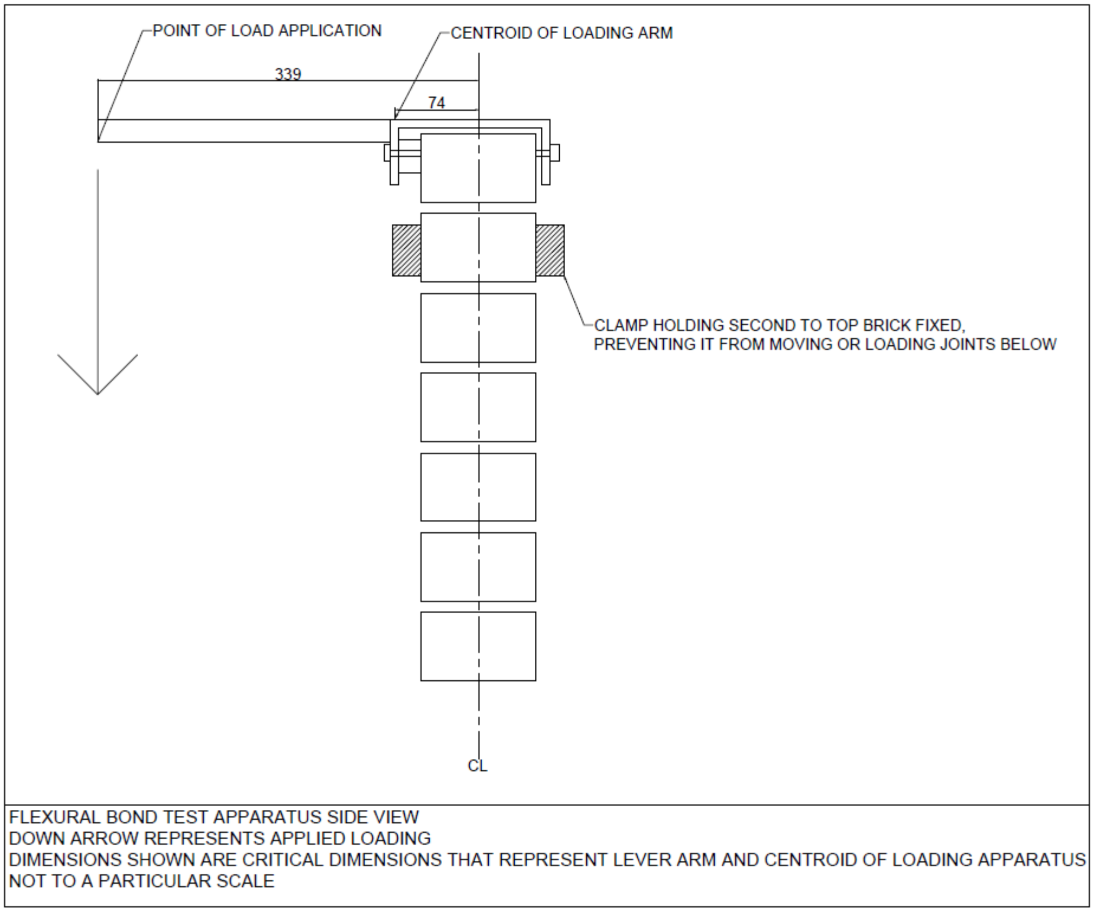


Figure 4-17: Bond wrench with critical dimensions shown

4.7.2 Shear Bond Test Setup

Shear bond testing was conducted by means of a direct shear test. While the ASTM standards do not cover shear strength as it is not a test used in modern design codes [11], pertinent specimen preparation methods outlined in the ASTM flexural test were followed as closely as possible [25]. A stack of three bricks was used, with mortar joints being made flush with the face of the bricks as much as possible. The ASTM C1531 shear test of masonry in situ [44] uses this method to determine shear bond strength. The stack of bricks were tested on the short edge, following the shear bond testing method used by Thamboo et al. [40]. The load was applied to a small point on top the middle brick in the stack as shown in Figure 4-18, while the two outer bricks were supported from below. The size of the load application and supports was not critically important for this test; the critical testing case was to maintain a small space between the load application and the mortar joint. Distances could not be maintained identical due to the minor variations in the sizes of the clay bricks themselves.

Construction of the 3-high stack of bricks was conducted in accordance with the procedure for constructing stacks of bricks set forth in ASTM Standard C1072 [25]. As with the flexural stack, the 0.5% casein specimens required that the form surrounding the mortar joint remain in place for several hours after filling in order to produce a proper mortar joint.

Figure 4-18: Shear bond test as conducted in the laboratory setting

4.8 Flow Table Test

For each batch of mortar, for all casein and water concentrations, a flow table test was conducted. The standard flow table as depicted in ASTM Standard C109 was used [26]. The Standard required the flow table to be struck 25 times over a time duration of 15 seconds, at a constant frequency of 1.67 Hz [26]. The standard cone, as shown in Figure 4-19 measured 50 millimetres in height and 99 millimetres in internal diameter [26].

Figure 4-19: ASTM Standard flow table

For each flow test, the final diameter, as shown in Figure 4-20, was recorded in millimetres as the specially calibrated caliper was not available. Measuring the diameter using a ruler is a permissible alternative to the special caliper as per the ASTM Standards [26]. In the case of a discrepancy between diameter measurements across a minimum of four cross-sections, the mean value of the four measurements was used.

Figure 4-20: Mortar after 25 oscillations of table

For the batches containing 5.5 litres of water per 30 kilograms of mortar, the cones were filled to a height of 25 millimetres instead of the full cone height of 50 milllimetres. This was done to prevent the specimens containing 5.5 litres of water per 30 kilograms of mortar and 0.5% casein or more from overflowing the standard test table. The water ratio was maintained constant for comparison purposes; only the amount of casein was varied in these set of tests.

Chapter 5: Experimental Test Results

5.1 Standard Water Content Specimens

The generic comparison charts of the entire set of data, reduced to four graphs, are shown in Figure 5-1 through Figure 5-4. Note that 'cas' in the graph legends represents the percent casein by mass. The four plots show the ultimate strengths in compression, tension, flexure and shear respectively. The lines represent a linear interpolation of the mean strengths at each prescribed casein content value. The smaller dots represent the results of the individual repeats. The same data is plotted in Figure 5-5 through Figure 5-8, except in terms of the casein content as opposed to the curing time. These plots represent of load-displacement curves for each, a sample of which is shown in Figure 5-9. See Appendix A for the full series of load-displacement curves for all test combinations and stress-strain curves for the compression tests.

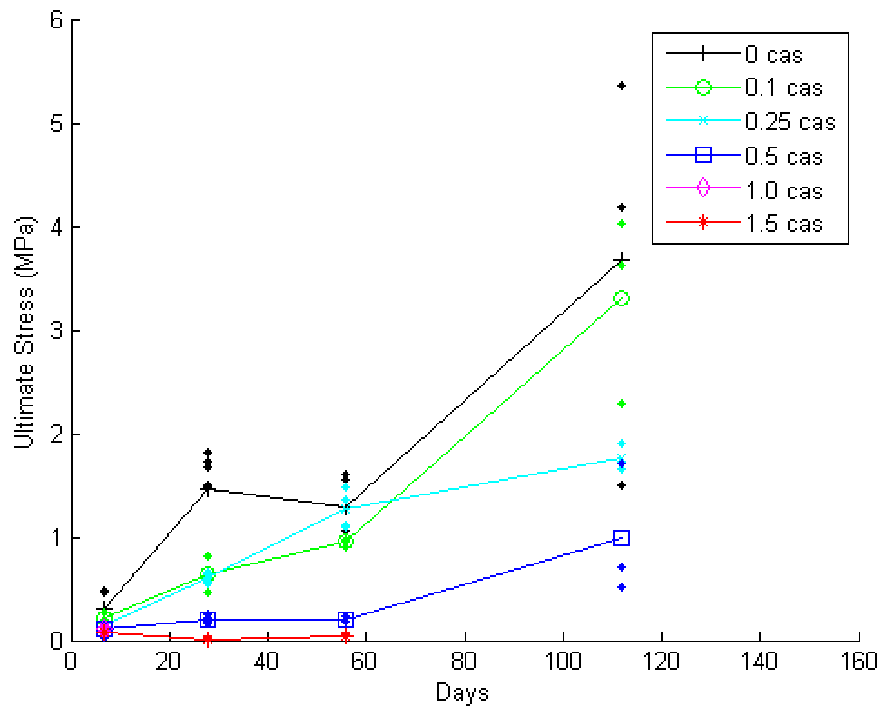


Figure 5-1: Ultimate Compressive Strength versus Curing Time

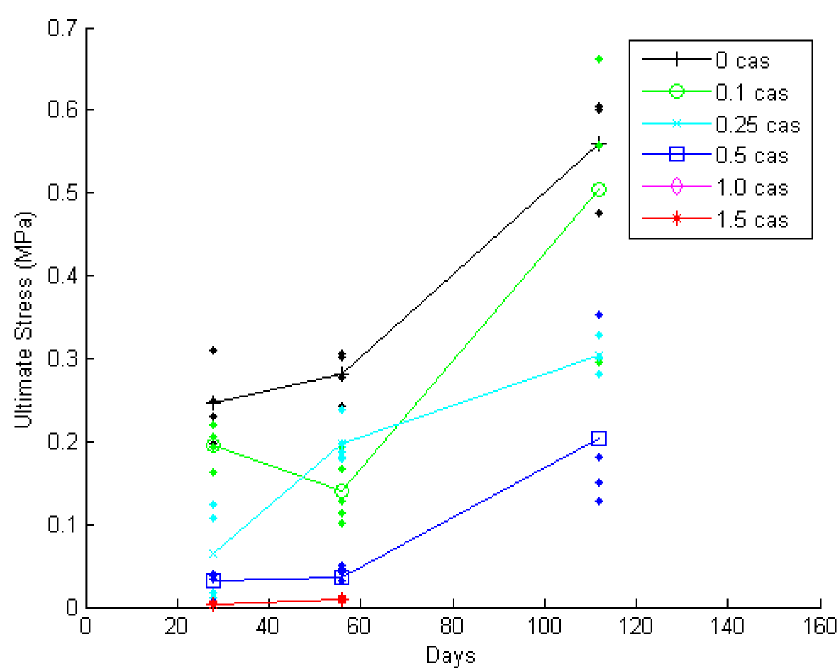


Figure 5-2: Ultimate Tensile Strength versus Curing Time

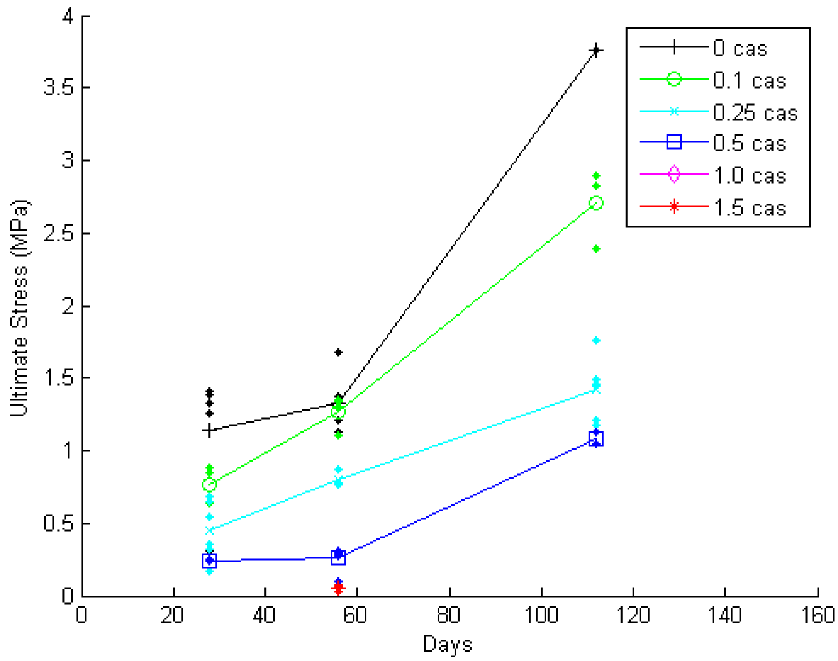


Figure 5-3: Ultimate Flexural Strengths versus Curing Time

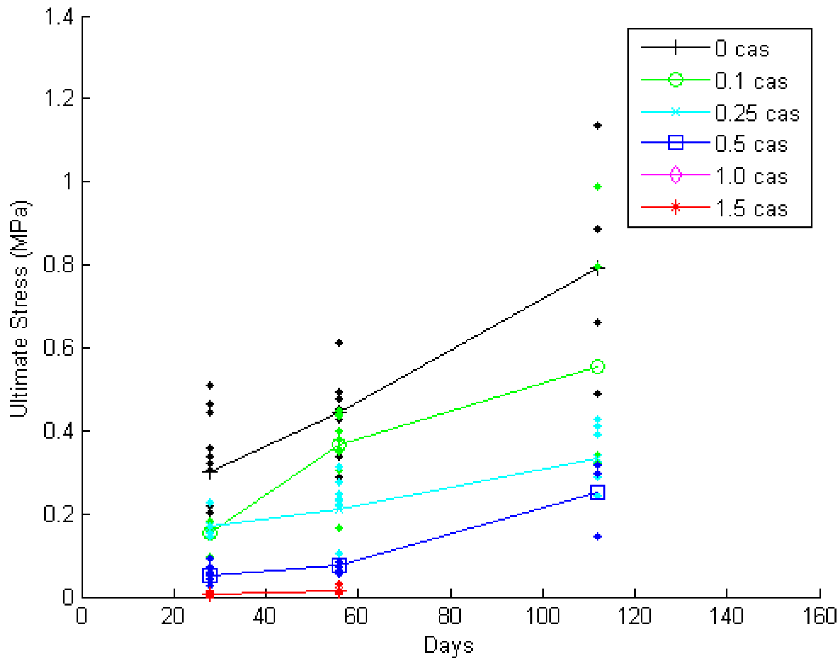


Figure 5-4: Ultimate Shear Strengths versus Curing Time

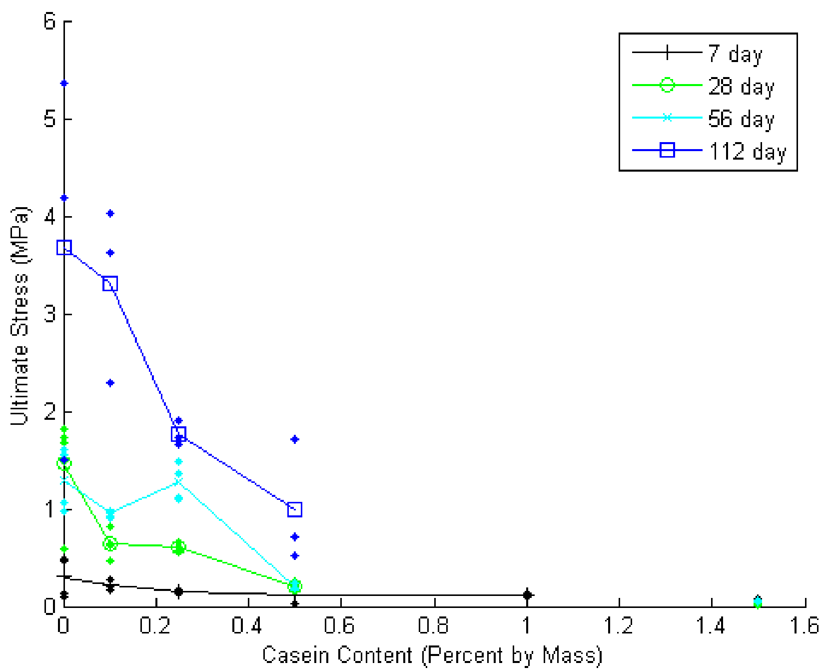


Figure 5-5: Ultimate Compressive Strengths versus Casein Content

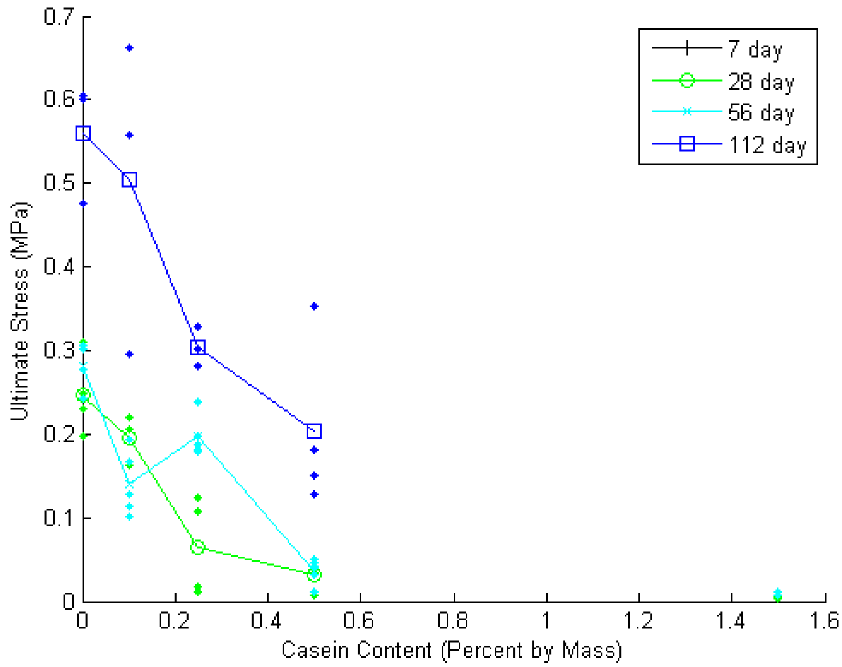


Figure 5-6: Ultimate Tensile Strengths versus Casein Content

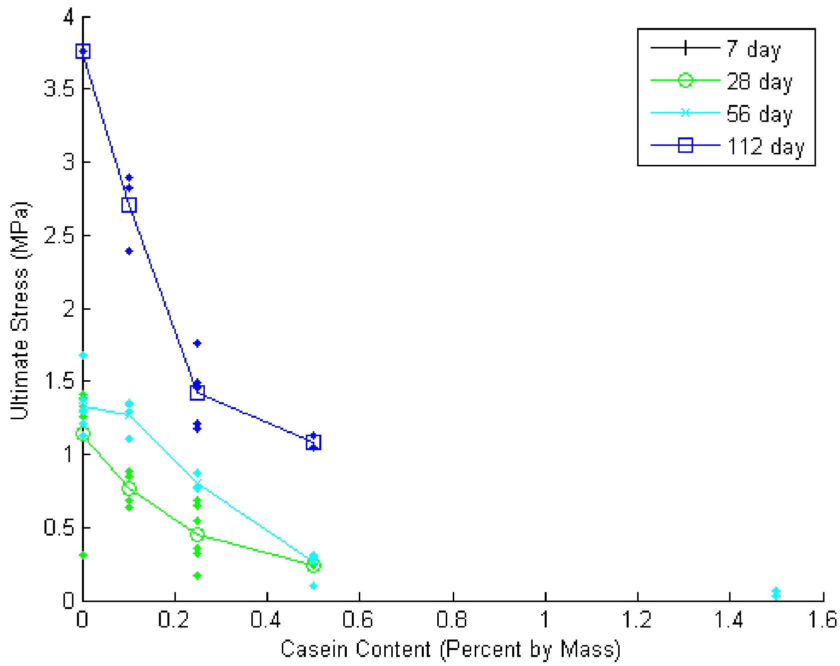


Figure 5-7: Ultimate Flexural Strengths versus Casein Content

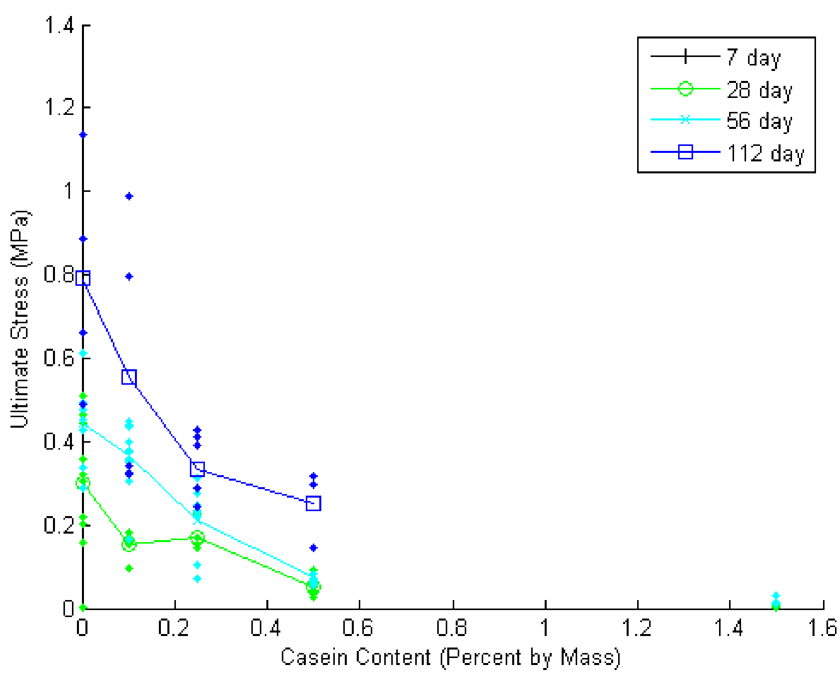


Figure 5-8: Ultimate Shear Strengths versus Casein Content

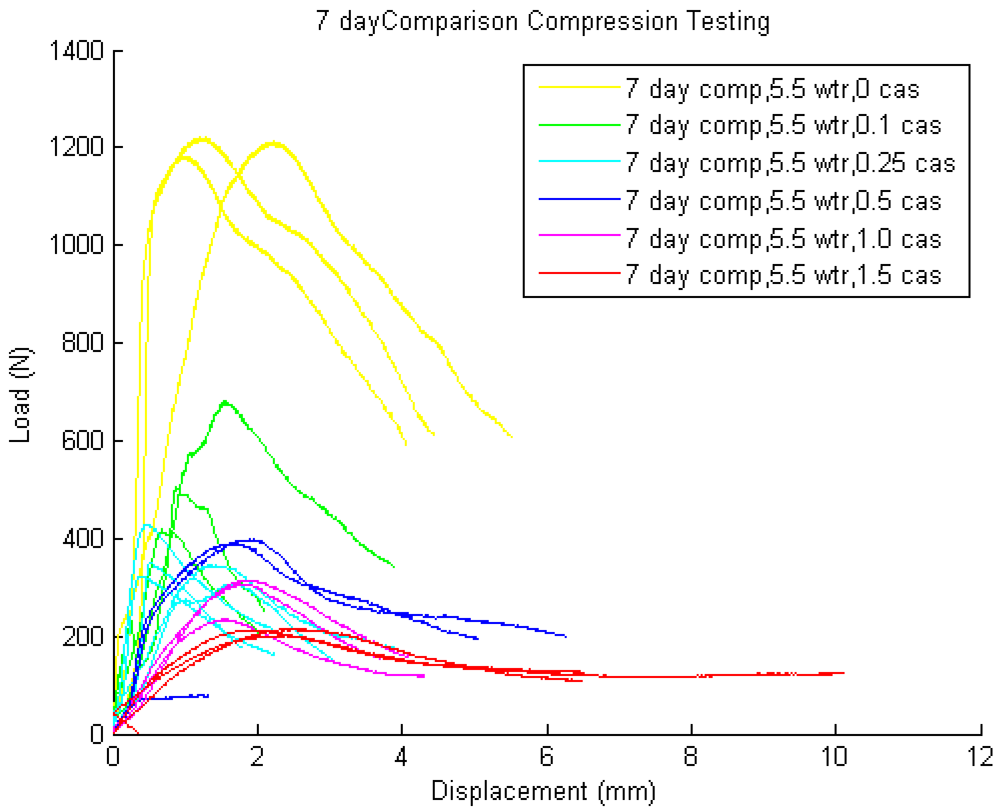


Figure 5-9: Sample Load-Displacement curve for 7 day testing in Compression and normal water content

Below in Table 5-1 and Table 5-2 are the mean and standard deviation results for the reduced water specimens, organized first by the type of test conducted. While there were not enough repeats of each specimen to have confidence in the Standard Deviation, the deviation values are shown for purposes of completeness.

Table 5-1: Means and Standard Deviations for each trial, organized by casein content

Water Content (L / 30 kg mortar)	Test Type	Casein Content (% by mass)	Duration Of Curing	Mean Ultimate Strength (MPa)	Standard Deviation (MPa)	Number of Repeats
5.5	comp	0	7 day	0.298	0.202	6
5.5	comp	0	28 day	1.466	0.445	6
5.5	comp	0	56 day	1.298	0.331	4
5.5	comp	0	112 day	3.682	1.983	3
5.5	comp	0.1	7 day	0.214	0.053	3
5.5	comp	0.1	28 day	0.633	0.142	4
5.5	comp	0.1	56 day	0.954	0.031	4
5.5	comp	0.1	112 day	3.308	0.910	3
5.5	comp	0.25	7 day	0.141	0.019	5
5.5	comp	0.25	28 day	0.598	0.042	4
5.5	comp	0.25	56 day	1.265	0.186	4
5.5	comp	0.25	112 day	1.764	0.124	3
5.5	comp	0.5	7 day	0.116	0.073	3
5.5	comp	0.5	28 day	0.209	0.026	6
5.5	comp	0.5	56 day	0.198	0.022	4
5.5	comp	0.5	112 day	0.984	0.641	3
5.5	comp	1	7 day	0.114	0.018	3
5.5	comp	1.5	7 day	0.084	0.003	3
5.5	comp	1.5	28 day	0.015	0.006	5
5.5	comp	1.5	56 day	0.049	0.013	3
5.5	tens	0	28 day	0.246	0.047	4
5.5	tens	0	56 day	0.282	0.029	4
5.5	tens	0	112 day	0.561	0.073	3
5.5	tens	0.1	28 day	0.196	0.024	4
5.5	tens	0.1	56 day	0.141	0.038	5
5.5	tens	0.1	112 day	0.505	0.189	3
5.5	tens	0.25	28 day	0.065	0.059	4
5.5	tens	0.25	56 day	0.197	0.024	5
5.5	tens	0.25	112 day	0.304	0.023	3
5.5	tens	0.5	28 day	0.033	0.013	6
5.5	tens	0.5	56 day	0.036	0.016	5

5.5	tens	0.5	112 day	0.203	0.102	4
5.5	tens	1.5	28 day	0.004	0.001	5
5.5	tens	1.5	56 day	0.010	0.002	3
5.5	flex	0	28 day	1.136	0.466	5
5.5	flex	0	56 day	1.333	0.190	6
5.5	flex	0	112 day	3.757	0.000	1
5.5	flex	0.1	28 day	0.764	0.124	4
5.5	flex	0.1	56 day	1.272	0.116	4
5.5	flex	0.1	112 day	2.703	0.277	3
5.5	flex	0.25	28 day	0.454	0.206	6
5.5	flex	0.25	56 day	0.806	0.053	3
5.5	flex	0.25	112 day	1.418	0.240	5
5.5	flex	0.5	28 day	0.243	0.007	4
5.5	flex	0.5	56 day	0.263	0.078	6
5.5	flex	0.5	112 day	1.088	0.060	2
5.5	flex	1.5	56 day	0.052	0.025	2
5.5	shear	0	28 day	0.302	0.149	11
5.5	shear	0	56 day	0.445	0.099	8
5.5	shear	0	112 day	0.793	0.279	4
5.5	shear	0.1	28 day	0.154	0.030	6
5.5	shear	0.1	56 day	0.365	0.089	9
5.5	shear	0.1	112 day	0.555	0.315	5
5.5	shear	0.25	28 day	0.170	0.034	5
5.5	shear	0.25	56 day	0.213	0.083	8
5.5	shear	0.25	112 day	0.335	0.085	6
5.5	shear	0.5	28 day	0.051	0.017	12
5.5	shear	0.5	56 day	0.075	0.013	10
5.5	shear	0.5	112 day	0.254	0.096	3
5.5	shear	1.5	28 day	0.005	0.003	12
5.5	shear	1.5	56 day	0.016	0.009	5

Table 5-2: Means and Standard Deviations for each trial, organized by curing time

Water Content (L / 30 kg mortar)	Test Type	Casein Content (% by mass)	Duration Of Curing	Mean Ultimate Strength (MPa)	Standard Deviation (MPa)	Number of Repeats

5.5	comp	0	7 day	0.2983	0.20182	6
5.5	comp	0.1	7 day	0.21357	0.053483	3
5.5	comp	0.25	7 day	0.14078	0.018823	5
5.5	comp	0.5	7 day	0.11563	0.0728	3
5.5	comp	1	7 day	0.11441	0.017841	3
5.5	comp	0	28 day	1.4657	0.4453	6
5.5	comp	0.1	28 day	0.63349	0.1419	4
5.5	comp	0.25	28 day	0.59773	0.042323	4
5.5	comp	0.5	28 day	0.20875	0.026346	6
5.5	comp	0	56 day	1.2977	0.33062	4
5.5	comp	0.1	56 day	0.95435	0.031165	4
5.5	comp	0.25	56 day	1.2652	0.18648	4
5.5	comp	0.5	56 day	0.19788	0.021791	4
5.5	comp	0	112 day	3.6816	1.9829	3
5.5	comp	0.1	112 day	3.3083	0.90975	3
5.5	comp	0.25	112 day	1.7639	0.12374	3
5.5	comp	0.5	112 day	0.98429	0.64095	3
5.5	tens	0	28 day	0.24624	0.047244	4
5.5	tens	0.1	28 day	0.19599	0.024011	4
5.5	tens	0.25	28 day	0.06455	0.058859	4
5.5	tens	0.5	28 day	0.032526	0.012557	6
5.5	tens	0	56 day	0.28181	0.029153	4
5.5	tens	0.1	56 day	0.14068	0.037931	5
5.5	tens	0.25	56 day	0.19689	0.024011	5
5.5	tens	0.5	56 day	0.036074	0.015796	5
5.5	tens	0	112 day	0.56073	0.072968	3
5.5	tens	0.1	112 day	0.50511	0.18859	3
5.5	tens	0.25	112 day	0.30405	0.022821	3
5.5	tens	0.5	112 day	0.20304	0.10223	4
5.5	flex	0	28 day	1.1362	0.46638	5
5.5	flex	0.1	28 day	0.76389	0.12356	4
5.5	flex	0.25	28 day	0.45444	0.206	6
5.5	flex	0.5	28 day	0.24314	0.006808	4
5.5	flex	0	56 day	1.3332	0.19042	6
5.5	flex	0.1	56 day	1.2721	0.11565	4
5.5	flex	0.25	56 day	0.80581	0.053327	3
5.5	flex	0.5	56 day	0.26265	0.077964	6
5.5	flex	0	112 day	3.7567	0	1
5.5	flex	0.1	112 day	2.7032	0.27677	3

5.5	flex	0.25	112 day	1.4184	0.24033	5
5.5	flex	0.5	112 day	1.0877	0.0604	2
5.5	shear	0	28 day	0.302	0.14895	11
5.5	shear	0.1	28 day	0.15422	0.029997	6
5.5	shear	0.25	28 day	0.16953	0.03384	5
5.5	shear	0.5	28 day	0.051423	0.016908	12
5.5	shear	0	56 day	0.44515	0.098566	8
5.5	shear	0.1	56 day	0.36472	0.088589	9
5.5	shear	0.25	56 day	0.21256	0.083166	8
5.5	shear	0.5	56 day	0.07528	0.012514	10
5.5	shear	0	112 day	0.7932	0.27924	4
5.5	shear	0.1	112 day	0.55523	0.31518	5
5.5	shear	0.25	112 day	0.33467	0.085421	6
5.5	shear	0.5	112 day	0.25369	0.095717	3

5.1.1 Discussion on Standard Water Content Results

Adding 0.5% casein by mass lowered the observed strengths by 70% - 80% in comparison to the control sample. The strength reduction was approximately the same percentage in all tests, regardless of whether a compression, tension, flexure or shear test was conducted. Increasing the concentration of casein lowered the strength further, however, the reduction was found to be non-linear. The 1.5% casein mix caused a 90% reduction in strength compared to the baseline control after 7 days; this became more pronounced as the curing time increased. The bending specimens at a casein content of 1.5% casein by mass were of insufficient strength to withstand insertion in the test machine; fracture occurred before the test could be conducted. The 0.1% casein mix caused a 40% reduction in strength compared to the baseline control. The 0.25% casein mix caused a strength reduction of about 60% compared to the baseline control specimen.

It was noted that after 56 days of curing, the specimens containing 0.1% casein approached the strength seen in the control sample. The specimens containing 0.25% casein also increased in strength, with results closer to, but still less than, the control specimens. However, the specimens containing 0.5% casein and 1.5% casein exhibited no improvement in strength above that seen at 28 days, in any of the failure modes. Furthermore, the 1.5% casein mix exhibited no strength improvement beyond the initial curing, with similar compressive strength results between the 7 day and 56 day tests at 10% of the 7 day control specimen.

5.1.2 Flow Table Testing of the mortar specimens

Flow recordings for each of the casein-containing specimens were determined using the flow test method in ASTM Standard C109. As the 1.0% casein and 1.5% casein specimens would exceed the diameter of the flow table if the standard cone was filled to the specified standard height of 50 millimetres [26], a reduced height of 25 millimetres was used for this test as a comparative study. The reduced height, which was measured to the nearest millimetre, was recorded and noted in Table 5-3 beside the "Initial Height" heading. Results for the final flow diameter and the percent increase in diameter are shown below in Table 5-3. As the flow table was a manually-operated apparatus, the frequency of oscillation of the table did not perfectly match that specified in the Standard [26].

Table 5-3: Flow Recordings of Mortar Mixes for Standard Water Content Specimens

Mortar Mix	Diameter Increase	Classification (By observation)
No Casein	58%*	Not Flowable
0.1% Casein	11%*	Not Flowable
0.25% Casein	31%*	Not Flowable
0.5% Casein	110%*	Flowable
1.0% Casein	67%*	Flowable
1.5% Casein	67%*	Flowable

The flowability upon casting was found not to directly correspond to a large variation in mortar strength. For the purposes of this research, a mortar specimen was deemed to be flowable if it could be poured into and completely fill the prism forms without requiring the use of any tamping. A mortar specimen was deemed to be marginally flowable if a spatula was required to spread it evenly through the cube forms but still filled the forms completely without any tamping. A mortar specimen was deemed to be not flowable if tamping was required. By a general trend, flowable mortar specimens exhibited greater than a 60% increase in diameter versus non-flowable mortar specimens.

Flow results, for the purposes of real-world mixing, could be classified into two groups: 'highly flowable' and 'not flowable'. The high-flowable category comprises the samples that double in diameter, while the low-flowable samples exhibit less than a 50% increase in diameter. The high-flowable category comprises of those samples that exhibited liquid-like properties: the mortar could easily be poured into the moulds. A sudden transition occurred from a low-flowable mortar to a high-flowable mortar with a small change in the casein content or the water content; no specimens could be considered as moderately flowable.

From the results of the experiments using the default water concentration of 5.5 litres per 30 kilograms of mortar [42], it could be deduced that no further testing with a casein content greater than 0.5% by mass could be of any further benefit. Furthermore, as a mortar mix containing 0.5% casein by mass produced a ‘highly flowable’ mortar, increasing the casein content further would result in few practical installation advantages, given that flow properties were the primary historical reason for adding the casein protein.

5.2 Reduced Water Content Specimens

However, given the substantial difference in flow table results between the 0.25% and 0.5% casein specimens, a further sample retaining 0.5% casein by mass but reducing the relative water content was conducted. This will produce similar mortar-to-water ratios to that provided in the historic casein mortar mix as described in *The City and Countrey Purchaser* [9].

It was decided to reduce the relative water content in nominal half litre increments per 30 kilograms of mortar, resulting in 9%, 18% 27% and 36% water reductions respectively. The reasoning for this approach is that masonry mortars in Canada are typically packaged in 30-kilogram bags, and these percentage reductions correspond with half-litre reductions per bag of mortar.

Below are the strength comparison charts of the entire set of data, reduced to four graphs as shown in Figure 5-10 through Figure 5-13. Note that 'wtr' in the graph legends. represents litres of water added per 30 kilograms of mortar. The following four plots show the ultimate strengths in compression, tension, flexure and shear respectively. The same data is plotted in Figure 5-14 through Figure 5-17, except in terms of the casein content as opposed to the curing time. See Appendix B for the full series of load-displacement curves for all test combinations and stress-strain curves for the compression tests.

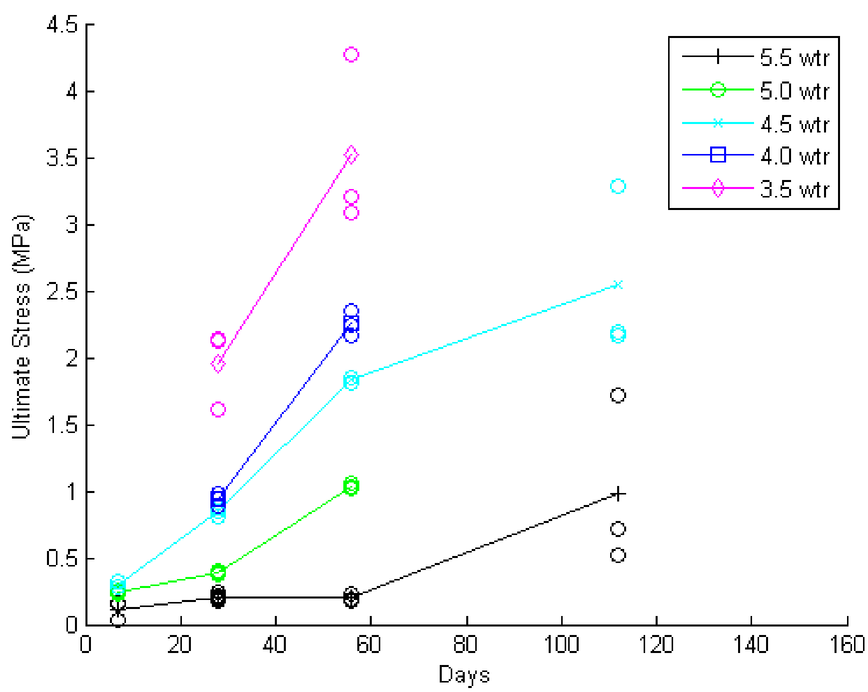


Figure 5-10: Ultimate Strength vs Curing Time in Compression, Colour represents water content

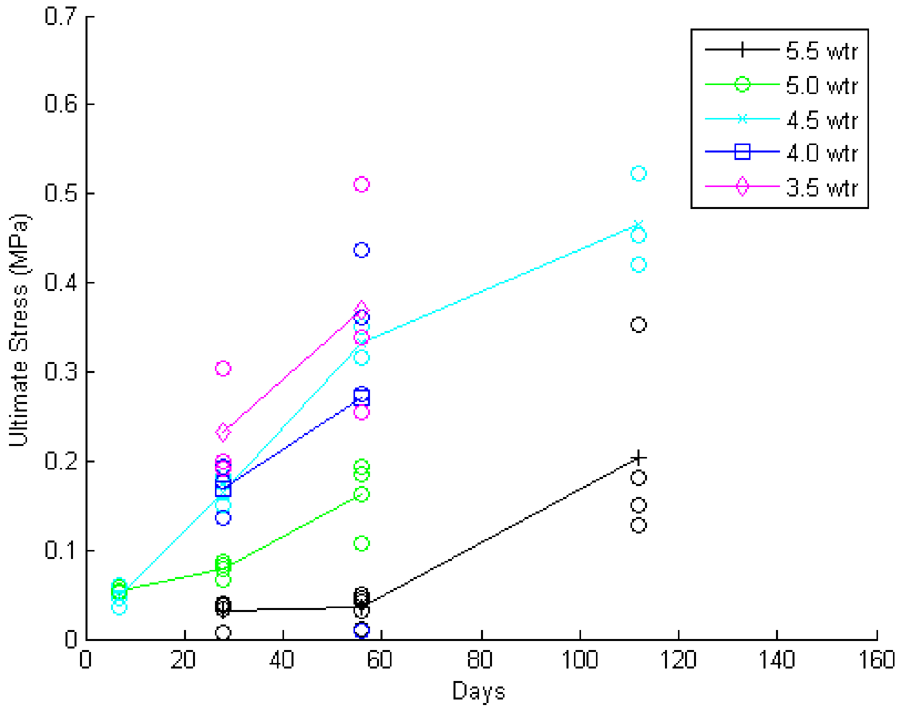


Figure 5-11: Ultimate Strength vs Curing Time in Tension, Colour represents water content

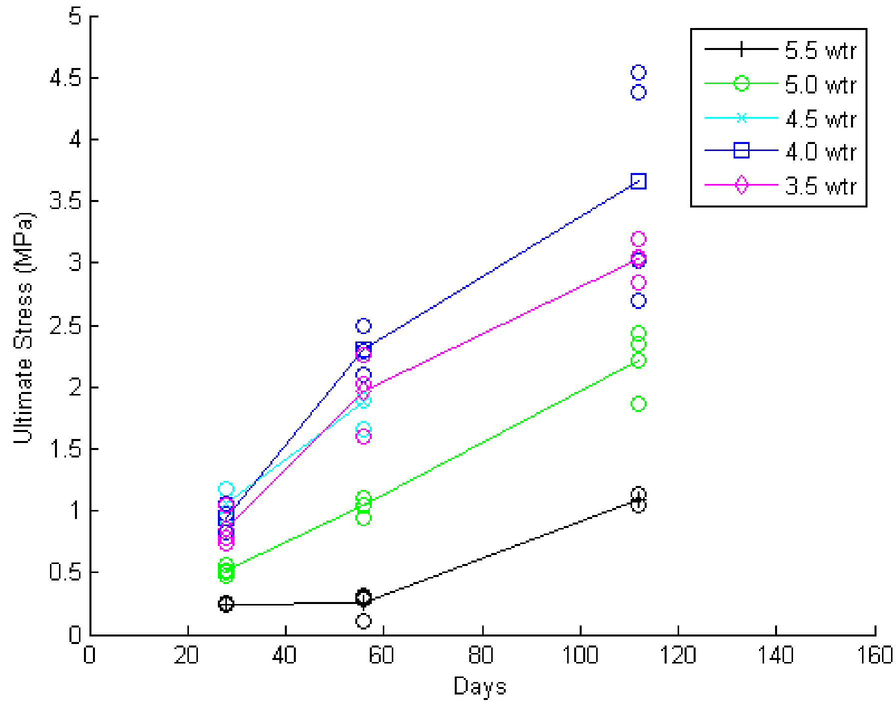


Figure 5-12: Ultimate Strength vs Curing Time in Flexure, Colour represents water content

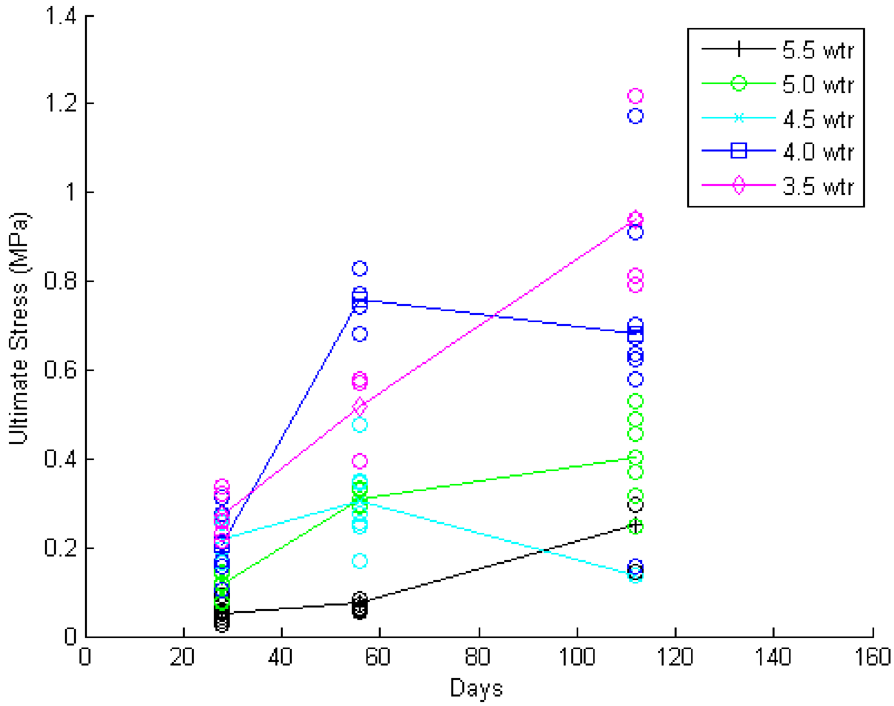


Figure 5-13: Ultimate Strength vs Curing Time in Shear, Colour represents water content

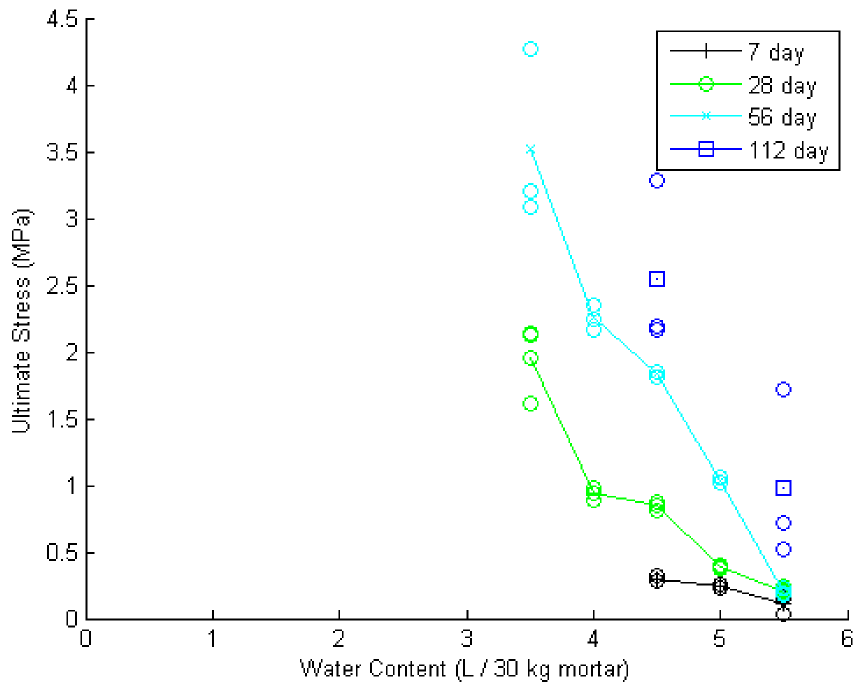


Figure 5-14: Ultimate Strength vs. Water Content in Compression, Colour represents Curing Time

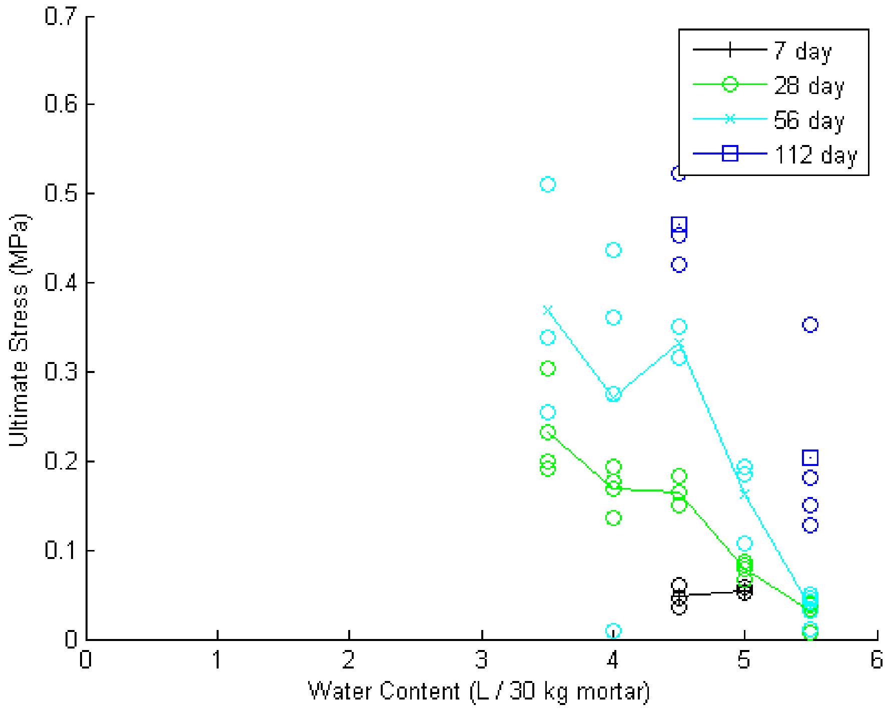


Figure 5-15: Ultimate Strength vs. Water Content in Tension, Colour represents Curing Time

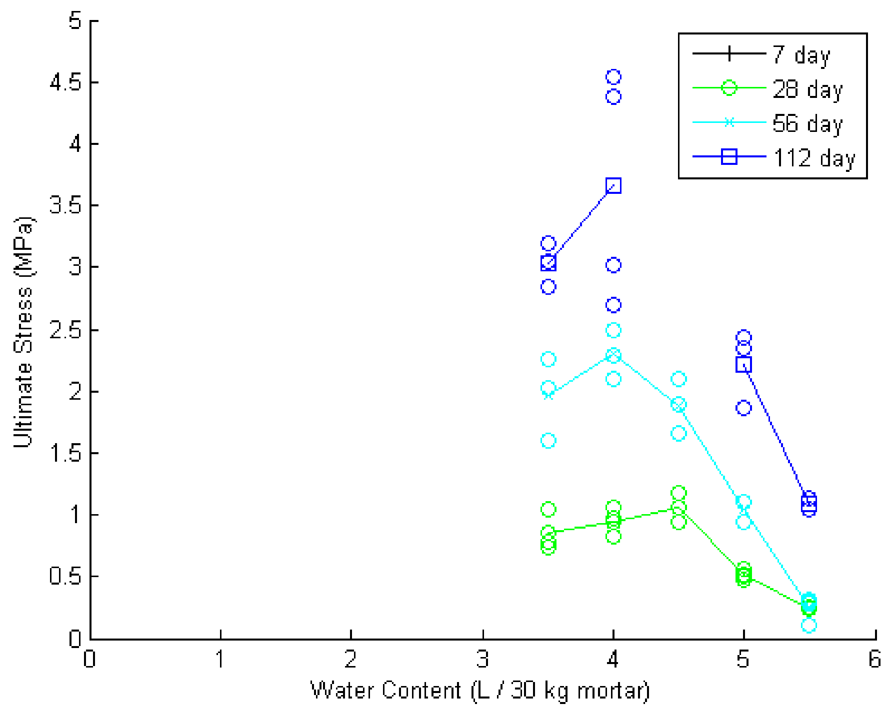


Figure 5-16: Ultimate Strength vs. Water Content in Flexure, Colour represents Curing Time

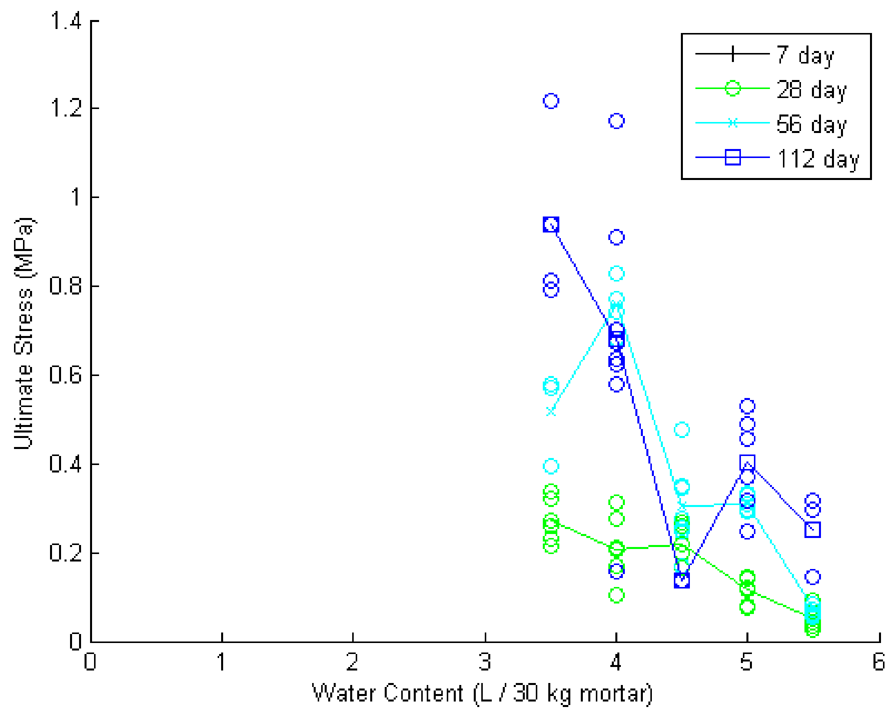


Figure 5-17: Ultimate Strength vs. Water Content in Shear, Colour represents Curing Time

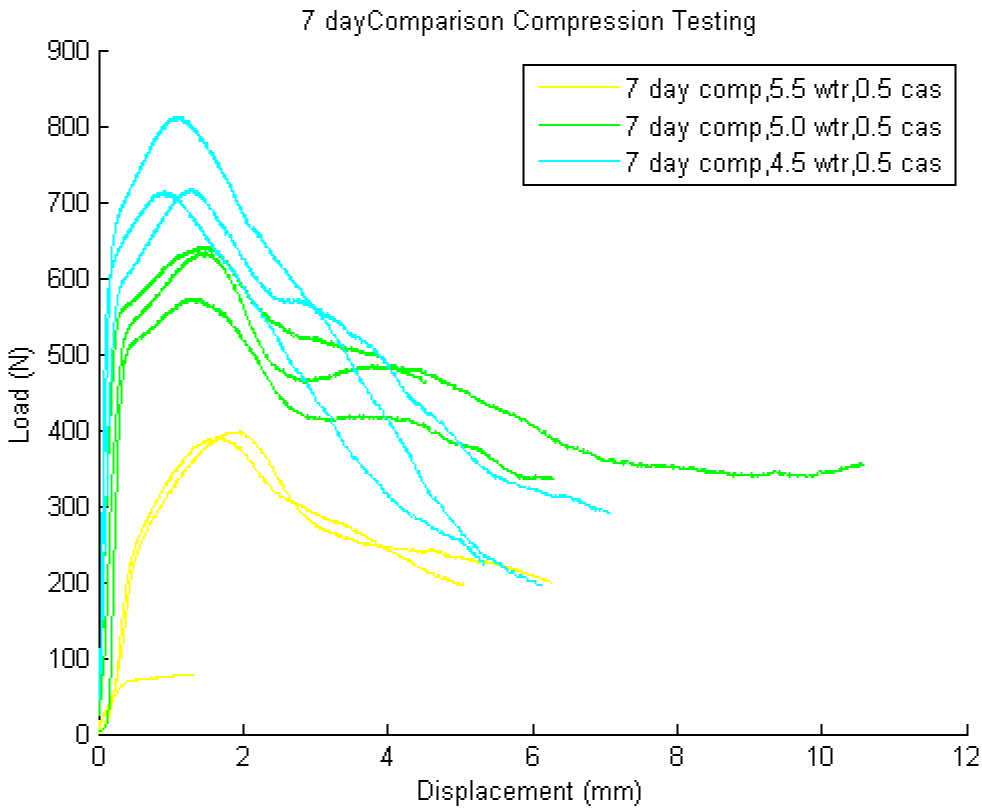


Figure 5-18: Sample Load-Displacement curve for 7 day compression samples with reduced water content

Below in Table 5-4 and Table 5-5 are the mean and standard deviation results for the reduced water specimens, organized first by the type of test conducted. While there were not enough repeats of each specimen to have confidence in the Standard Deviation, the deviation values are shown for purposes of completeness.

Table 5-4: Mean and Standard Deviation results for each trial, organized by curing duration

Casein Content (% by mass)	Test Type	Water Content (L / 30 kg mortar)	Duration Of Curing	Mean Ultimate Strength (MPa)	Standard Deviation (MPa)	Number of Repeats
0.5	comp	5.5	7 day	0.116	0.073	3
0.5	comp	5.0	7 day	0.247	0.015	3
0.5	comp	4.5	7 day	0.299	0.022	3
0.5	comp	5.5	28 day	0.209	0.026	6
0.5	comp	5.0	28 day	0.391	0.015	3
0.5	comp	4.5	28 day	0.843	0.032	3
0.5	comp	4.0	28 day	0.936	0.041	3
0.5	comp	3.5	28 day	1.956	0.298	3
0.5	comp	5.5	56 day	0.198	0.022	4
0.5	comp	5.0	56 day	1.038	0.031	2
0.5	comp	4.5	56 day	1.842	0.023	3
0.5	comp	4.0	56 day	2.253	0.095	3
0.5	comp	3.5	56 day	3.519	0.657	3
0.5	comp	5.5	112 day	0.984	0.641	3
0.5	comp	4.5	112 day	2.544	0.643	3
0.5	tens	5.0	7 day	0.055	0.004	3
0.5	tens	4.5	7 day	0.047	0.012	3
0.5	tens	5.5	28 day	0.033	0.013	6
0.5	tens	5.0	28 day	0.079	0.011	3
0.5	tens	4.5	28 day	0.166	0.016	3
0.5	tens	4.0	28 day	0.169	0.030	3
0.5	tens	3.5	28 day	0.232	0.063	3
0.5	tens	5.5	56 day	0.036	0.016	5
0.5	tens	5.0	56 day	0.162	0.048	3
0.5	tens	4.5	56 day	0.333	0.025	2
0.5	tens	4.0	56 day	0.271	0.186	4
0.5	tens	3.5	56 day	0.368	0.130	3
0.5	tens	5.5	112 day	0.203	0.102	4
0.5	tens	4.5	112 day	0.466	0.052	3
0.5	flex	5.5	28 day	0.243	0.007	4
0.5	flex	5.0	28 day	0.513	0.050	3
0.5	flex	4.5	28 day	1.063	0.113	3
0.5	flex	4.0	28 day	0.949	0.121	3

0.5	flex	3.5	28 day	0.855	0.163	3
0.5	flex	5.5	56 day	0.263	0.078	6
0.5	flex	5.0	56 day	1.047	0.096	3
0.5	flex	4.5	56 day	1.883	0.218	3
0.5	flex	4.0	56 day	2.298	0.195	3
0.5	flex	3.5	56 day	1.966	0.335	3
0.5	flex	5.5	112 day	1.088	0.060	2
0.5	flex	5.0	112 day	2.216	0.312	3
0.5	flex	4.0	112 day	3.658	0.937	4
0.5	flex	3.5	112 day	3.033	0.177	3
0.5	shear	5.5	28 day	0.051	0.017	12
0.5	shear	5.0	28 day	0.118	0.032	6
0.5	shear	4.5	28 day	0.221	0.047	6
0.5	shear	4.0	28 day	0.206	0.078	6
0.5	shear	3.5	28 day	0.273	0.054	5
0.5	shear	5.5	56 day	0.075	0.013	10
0.5	shear	5.0	56 day	0.310	0.018	6
0.5	shear	4.5	56 day	0.303	0.098	7
0.5	shear	4.0	56 day	0.760	0.055	5
0.5	shear	3.5	56 day	0.516	0.104	3
0.5	shear	5.5	112 day	0.254	0.096	3
0.5	shear	5.0	112 day	0.402	0.109	6
0.5	shear	4.5	112 day	0.138	0.000	1
0.5	shear	4.0	112 day	0.682	0.290	8
0.5	shear	3.5	112 day	0.940	0.195	4

Table 5-5: Mean and Standard Deviation results for each trial, organized by water content

Casein Content (% by mass)	Test Type	Water Content (L / 30 kg mortar)	Duration Of Curing	Mean Ultimate Strength (MPa)	Standard Deviation (MPa)	Number of Repeats
0.5	comp	5.5	7 day	0.116	0.073	3
0.5	comp	5.5	28 day	0.209	0.026	6
0.5	comp	5.5	56 day	0.198	0.022	4
0.5	comp	5.5	112 day	0.984	0.641	3
0.5	comp	5.0	7 day	0.247	0.015	3

0.5	comp	5.0	28 day	0.391	0.015	3
0.5	comp	5.0	56 day	1.038	0.031	2
0.5	comp	4.5	7 day	0.299	0.022	3
0.5	comp	4.5	28 day	0.843	0.032	3
0.5	comp	4.5	56 day	1.842	0.023	3
0.5	comp	4.5	112 day	2.544	0.643	3
0.5	comp	4.0	28 day	0.936	0.041	3
0.5	comp	4.0	56 day	2.253	0.095	3
0.5	comp	3.5	28 day	1.956	0.298	3
0.5	comp	3.5	56 day	3.519	0.657	3
0.5	tens	5.5	28 day	0.033	0.013	6
0.5	tens	5.5	56 day	0.036	0.016	5
0.5	tens	5.5	112 day	0.203	0.102	4
0.5	tens	5.0	7 day	0.055	0.004	3
0.5	tens	5.0	28 day	0.079	0.011	3
0.5	tens	5.0	56 day	0.162	0.048	3
0.5	tens	4.5	7 day	0.047	0.012	3
0.5	tens	4.5	28 day	0.166	0.016	3
0.5	tens	4.5	56 day	0.333	0.025	2
0.5	tens	4.5	112 day	0.466	0.052	3
0.5	tens	4.0	28 day	0.169	0.030	3
0.5	tens	4.0	56 day	0.271	0.186	4
0.5	tens	3.5	28 day	0.232	0.063	3
0.5	tens	3.5	56 day	0.368	0.130	3
0.5	flex	5.5	28 day	0.243	0.007	4
0.5	flex	5.5	56 day	0.263	0.078	6
0.5	flex	5.5	112 day	1.088	0.060	2
0.5	flex	5.0	28 day	0.513	0.050	3
0.5	flex	5.0	56 day	1.047	0.096	3
0.5	flex	5.0	112 day	2.216	0.312	3
0.5	flex	4.5	28 day	1.063	0.113	3
0.5	flex	4.5	56 day	1.883	0.218	3
0.5	flex	4.0	28 day	0.949	0.121	3
0.5	flex	4.0	56 day	2.298	0.195	3
0.5	flex	4.0	112 day	3.658	0.937	4
0.5	flex	3.5	28 day	0.855	0.163	3
0.5	flex	3.5	56 day	1.966	0.335	3
0.5	flex	3.5	112 day	3.033	0.177	3
0.5	shear	5.5	28 day	0.051	0.017	12

0.5	shear	5.5	56 day	0.075	0.013	10
0.5	shear	5.5	112 day	0.254	0.096	3
0.5	shear	5.0	28 day	0.118	0.032	6
0.5	shear	5.0	56 day	0.310	0.018	6
0.5	shear	5.0	112 day	0.402	0.109	6
0.5	shear	4.5	28 day	0.221	0.047	6
0.5	shear	4.5	56 day	0.303	0.098	7
0.5	shear	4.5	112 day	0.138	0.000	1
0.5	shear	4.0	28 day	0.206	0.078	6
0.5	shear	4.0	56 day	0.760	0.055	5
0.5	shear	4.0	112 day	0.682	0.290	8
0.5	shear	3.5	28 day	0.273	0.054	5
0.5	shear	3.5	56 day	0.516	0.104	3
0.5	shear	3.5	112 day	0.940	0.195	4

Flow Results for the reduced water specimens is shown below in Table 5-6. As the 1.0% and 1.5% casein specimens were not conducted in this case, it was possible to perform the flow test to the standard, filling the cone to a full height of 50 millimetres.

Table 5-6: Flow Test results for Reduced Water Content specimens

Mortar Mix	Diameter Increase	Qualitative Classification (By observation)
5.5 L / 30 kg	58%*	Flowable
5.0 L / 30 kg	14%	Marginally Flowable
4.5 L / 30 kg	100%	Flowable
4.0 L / 30 kg	60%	Marginally Flowable
3.5 L / 30 kg	12%	Not Flowable

Again, it was possible to categorize the specimens into ‘highly flowable’ and ‘not flowable’ groups, as listed in Table 5-6 and described in Section 5.1. The flow properties of the mortar did not exhibit a direct correlation to the mortar strength.

5.2.1 Reduced Water Test Summary

It was found that reducing the water content increased the strength while maintaining similar flowable properties of the 0.5% casein mortar mix with the normal water ratio. Compared to the baseline control specimen (no casein, 5.5L water per 30 kilograms mortar), a strength reduction of 65% in comparison to the control was observed for the 9% water reduction specimen, and a strength reduction of 50% was observed for the 18% water reduction specimen after 7 days. The 0.5% casein specimen with the normal water content exhibited a 75%-80% strength reduction in comparison to the control specimen.

It was found that a ratio of 4.5 litres of water per 30 kilograms of mortar resulted in the best balance between increased workability and strength, an 18% water reduction from the manufacturer's specification of 5.5 litres of water per 30 kilograms of mortar. A ratio of 5.0 litres of water per 30 kilograms of mortar resulted in similar flow results, however, a lower mortar strength resulted. It was noted by visual, qualitative observations during mixing that 3.5 litres of water per 30 kilograms of mortar was insufficient, as unmixed mortar often remained in the bottom of the mixing container. Flow results were not desirable for this specimen series. Using 4.0 litres of water per 30 kilograms of mortar was sufficient to ensure full, complete mixing, however, the resultant mortar mixture was not flowable, as seen in the flow results in .

The samples using 4 litres and 3.5 litres of water per 30 kilograms of mortar, 27% and 36% reductions on water respectively, caused a very rapid initial setting of the mortar. While a strength greater than the baseline control was observed for the specimen containing 3.5 litres of water per 30 kilograms of mortar, the addition of casein provided no benefit in workability. This aligns with the historic casein mortar mix described in *The City and Countrey Purchaser*, indicating that this mortar must be worked quickly [9].

Note that all samples from batch V3 do not follow the expected linear correlation regarding material densities while the replicate samples from batch Y5 do follow the expected linear correlation. It is from this data that it was deduced that a mistake was made with the preparation of this batch. In a reduced water specimen, this could likely be attributed to the mixing process. It was decided to re-cast a new batch of cubes and prisms with 4.5 litres of water per 30 kilograms of mortar, Batch V7, in order to conduct new tests on the data.

5.3 Brick-to-Mortar Bond Test Specimens

Given the historical use of the casein protein in glues and adhesives, it was deemed important to test the brick-to-mortar bond strength of the mortar specimen. An increase in bond strength would be a further benefit in addition to workability. As these tests were more expensive to conduct, and the desired result was to observe the effect of adding casein on bond strength as opposed to material strength, it was decided only to conduct these tests after 56 days of curing. It would be expected that the bond strength would increase overtime in a similar fashion to the tensile and compressive strength.

The rapid setting of the mortar presented challenges when building the stack bond specimens, resulting in potentially inconsistent bond properties between the top of the stack and the bottom of the stack.

A significant proportion, approximately 40%, of the samples were affected by breakage that occurred before the loading and testing could occur. These samples, for which no useable data could be extracted, are denoted as ‘BBL’ in Table 5-7 through Table 5-9. The brock-to-mortar bond specimens could break accidentally at any stage throughout the specimen preparation and testing process: mortar hardening at a rapid rate that prevented initial formation of a bond, transporting the samples from the casting location to the humidified storage room, transporting the samples from the humidified storage room to the testing machine, and undesired impulse loads exerted on lower specimens during testing of specimens higher in the stack.

Table 5-7: Mass applied to end of armature required to break brick-to-mortar bond tests

	No Casein	0.1% Casein	0.25% Casein	0.5% Casein 5.5 L water per 30 kg mortar	0.5% Casein 4.0 L water per 30 kg mortar	0.5% Casein 4.5 L water per 30 kg mortar
Repeat #	Mass in grams	Mass in grams	Mass in grams	Mass in grams		Mass in grams
1	4000	BBL*	BBL*	8000	BBL*	BBL*
2	500	1000	2750	5500	BBL*	BBL*
3	1000	3900	2200	19800	BBL*	3000
4	1000	2600	BBL*	4000	4400	29000
5	BBL*	BBL*	BBL*	13400		
6	2200	11000	10000	13000		
7	BBL*	BBL*	8000	33000		

* BBL = Broke before Loading

Note that there was a wide range in results, similar to what was found in the sudden failure modes. Both the bricks and mortar may cause variability in this test, as clay bricks intended for historic repair applications were used.

The stack bond specimen with a water ratio of 4.0 litres of water per 30 kilograms of mortar was unable to bond properly. It was noted that all but one of the mortar joints in the stack de-bonded after 56 days of curing, before inserting into the testing machine.

Table 5-8: Bending Moment (in N·m) required to break brick-to-mortar bond tests

	No Casein	0.1% Casein	0.25% Casein	0.5% Casein	0.5% Casein 4.0 L water per 30 kg mortar	0.5% Casein 4.5 L water per 30 kg mortar
	BBL*	BBL*	BBL*	27.94586	BBL*	BBL*
	3.0125797	4.674798	10.49256	19.63477	BBL*	BBL*
	4.6747984	14.31567	8.664123	67.17422	BBL*	BBL*
	4.6747984	9.993898	BBL*	14.64811	BBL*	11.32367
	BBL*	BBL*	BBL*	45.89782	15.97789	97.75905
	8.6641233	37.91917	34.59473	44.56805		
			27.94586	111.0568		

* BBL = Broke Before Loading

The final value desired was the tensile stress at the outermost surface, where failure of the joint first occurred. This was calculated using the mean brick and mortar joint dimensions of 102 mm × 214 mm.

Table 5-9: Tensile stress (in MPa) at outermost surface of mortar joint

	No Casein	0.1% Casein	0.25% Casein	0.5% Casein	0.5% Casein 4.0 L water per 30 kg mortar	0.5% Casein 4.5 L water per 30 kg mortar
	BBL*	BBL*	BBL*	0.076745	BBL*	BBL*
	0.008273	0.012838	0.028815	0.053921	BBL*	BBL*
	0.012838	0.039314	0.023793	0.184474	BBL*	0.031097032
	0.012838	0.027445	BBL*	0.040227	0.043878	0.268466
	BBL*	BBL*	BBL*	0.126044		
	0.023793	0.104133	0.095004	0.122393		
	BBL*	BBL*		0.304984		

* BBL = Broke Before Loading

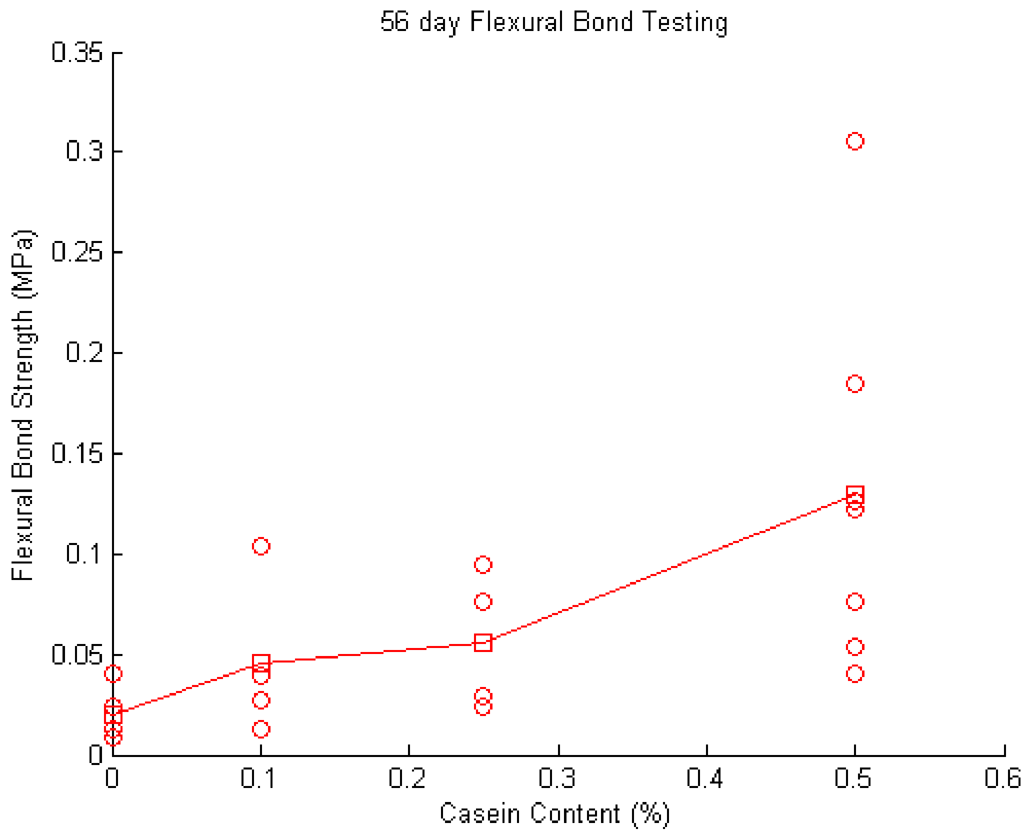


Figure 5-19: Flexural Bond Test Results. The large green dots represent the mean strength for each discrete casein value tested.

It was observed that the flexural bond strength increased with the addition of casein. In this case, the bond strength would increase until reaching the same value as the material tensile strength, at which point the tensile strength would govern. It was found that the samples containing 0.5% casein did not break cleanly on the face of the brick as shown below in Figure 5-21, indicating that the bond strength was similar to the tensile strength of the material at 0.5% casein. The reported bond strengths of 0.18 MPa and 0.30 MPa reflects this, given that after 56 days for a 0.5% casein sample with the baseline water content, the tensile strength was 0.05 MPa and the flexural strength was less than 0.15 MPa.

Figure 5-20: Mortar Cleanly Broken from brick surface; failure path followed interface

Figure 5-21: Mortar not cleanly broken; failure path through the mortar itself

In most brick-to mortar bonds, the masonry units absorb some of the water in the fresh mortar upon immediate construction. The amount of water absorbed by each brick varied by an unknown amount due to natural variability of the bricks used. As a consequence of this, the mortar in the mortar joint may have had a lower effective water content than the mortar casted by itself in the prism forms. As a result, the measured bond strength in this experimental setup was greater than both the measured tensile and flexural strengths.

Reducing the water content to 4.5 litres of water per 30 kilograms of mortar maintained adhesion properties resulting from the added casein, however, the bond strength was no longer larger than the tensile and flexural strengths. For the few specimens that were tested successfully, the recorded bond strengths of 0.03 MPa and 0.26 MPa were marginally less than the interpolated 56 day flexural strength that would be expected to range between 0.2 MPa and 0.3 MPa. Given the wide range in results, there is not sufficient data for this latter relationship to be definitively conclusive. In all of these tests, the mortar broke clean from the face of the brick along the shortest joint line, as shown in Figure 5-20.

The results would also conclude that the bond tests were governed by the measured flexural strength, not the measured tensile strength. A further explanation of the difference in results between the tensile and flexural strengths is made in 5.5. Considering that the bond test is a flexural test, this result could be expected.

5.3.1 Shear Bond Test Results

The shear bond tests were conducted by means of a test setup as shown in Figure 4-18. Results for the shear bond tests are shown below in Figure 5-22. Due to the difference in procedure, impulse loads were not a concern in these tests. As with the flexural tests, issues of premature bond failure were encountered, which occurred during the casting and curing stages. Some data points are missing in Figure 5-22 for the 0% casein and 0.1% casein specimens as a result of this.

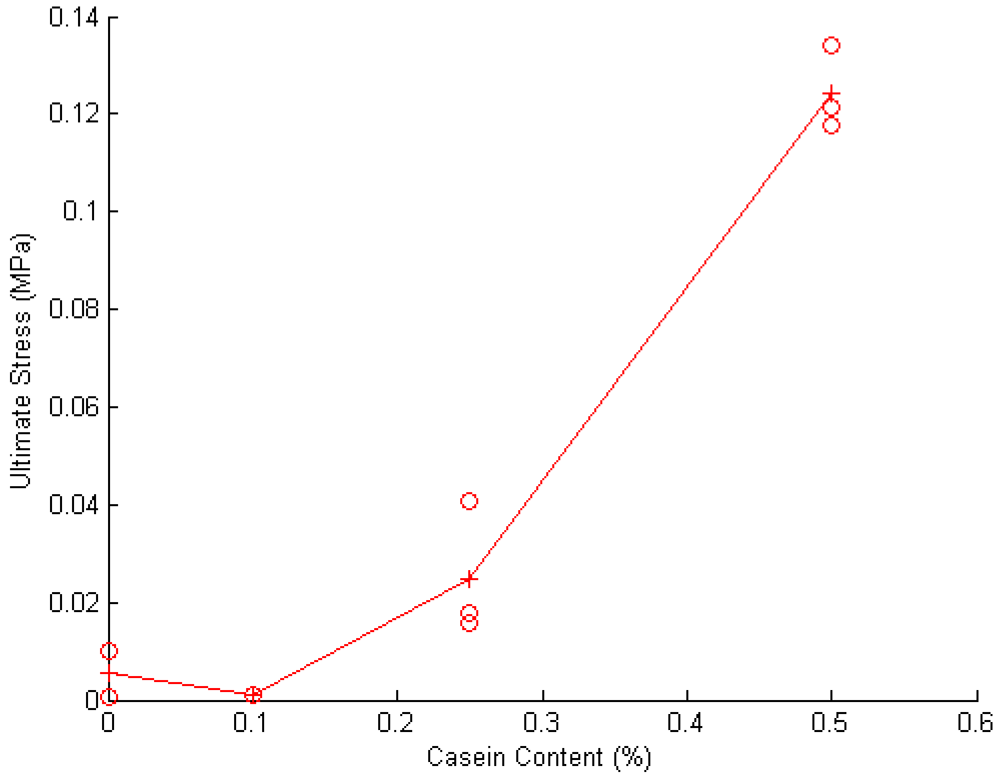


Figure 5-22: Ultimate Strength of Brick-to-Mortar Bond Tests

The shear bond tests, shown in Figure 5-22, followed the same pattern observed with the flexural bond tests, exhibiting a 56 day strength improvement by a factor of 8 with respect to the control specimen. Due to a limited quantity of bricks that could be acquired, shear bond tests were not conducted with the reduced water content specimens.

The 0.5% casein stacks of bricks did not fracture clean from the face of the brick. This indicated that the bond strength was greater than the shear strength for the mortar specimen. Brick-to-mortar bond strengths in shear, using a mix containing 0.5% casein and 5.5 L of water per 30 kg of mortar, were calculated to be 0.14 MPa as shown in Figure 5-22, while the 56 day mortar prism shear strength using the same casein and water ratios was calculated to be less than 0.1 MPa as shown in Figure 5-8. The shear bond tests containing 0.5% casein by mass fractured as shown in Figure 5-21, while all other specimens fractured along the brick-to-mortar interface as shown in Figure 5-20.

5.4 General qualitative observations

Some plastic deformation was observed in the load-displacement curves, particularly in the compressive tests and the shear tests as the specimens approached the point of failure. See Appendix A and Appendix B for the complete series of load-displacement curves.

The shear tests exhibited a partial flexural failure first, cracking partially at the mid-span of the specimen, before assuming further loading, which would increase to a complete failure in shear, with slightly angled failure lines. This would indicate that the shear failure pattern contained a tensile failure component, and thus was not a pure shear failure.

Behaviour of the mortar specimens aligned with the research found regarding proteins. The ratios used were substantially greater than those tested by Chandra and Aavik and Jasiczak and Zielinski,

5.5 Observations Comparing Flexural Tests Versus Shear Tests

Throughout all of the experimental results, the modulus of rupture, or the flexural strength, is approximately three times the value of the tensile strength. As this was a comparative study, the flexural strength was consistently higher than the tensile strength by a factor of about three in all specimens, thereby not affecting the comparison of strength from one sample to another. While the flexural strength is normally greater than the tensile strength, the two strengths are theoretically related and do not normally differ by this great of an amount. The first plausible explanation for this result is that the rectangular prism specimens are transmitting a large portion of the strength in bearing.

Research has shown that the split cylinder test will indicate a tensile strength to be lower than the actual strength of the specimen. For a split cylinder, the observed tensile strength using a split cylinder test ranges between 80% and 90% of the true tensile strength [31] when tested with concrete. The 10-millimetre clearance of the angle as established in the experimental procedure may reduce this diameter further, below what was assumed by the calculations in determining the split cylinder tensile strength. This would be consistent with the observed crack pattern propagating to one side of the angle bracket instead of passing through the corner of the mortar cube. This effect was observed to be consistent with all mortar cube tests, regardless of casein content or water content. Furthermore, it has been previously established that the modulus of rupture or flexural strength will be observed as being higher in an experimental test scenario than a direct tensile test [45], despite theoretically representing the same quantity.

The combination of the error factors on both test methods described above leads to the second plausible explanation regarding the increased recorded strength in flexure. In reality, it may be a combination of both bearing stresses and recording methods.

Chapter 6: Conclusions and Recommendations

6.1 Concluding Remarks

Mortar prisms were tested with the casein content being the only altered variable in order to determine the effects of casein alone on an historic lime mortar mixture. Brick-to-mortar bond tests were also conducted. Bond tests are similar to mortar tests conducted in situ, which typically yields a higher material strength than mortar cubes tested individually. It was found that at least 0.5% casein by mass was necessary to improve workability, however, this resulted in a mortar strength reduction of 80% with respect to the standard lime mortar mix with no casein. Following the results of this casein analysis, 0.5% casein was maintained while reducing the water content in half litre increments per bag of mortar to determine if casein affected the appropriate water ratio. It was found that reducing the water content by 18% yielded the most favourable strength to workability balance across all of the tests when 0.5% casein was added.

This is the first study to comprehensively investigate casein in particular as a mortar additive to determine its behaviour in all forms of static loading: compression, tension, flexure, shear and bond strengths. The ultimate objective of this thesis paper was to conduct a comparative study between specimen types, varying the casein and water content of the mortar. Determining the exact material strength was not the intended result.

Based upon the results of the experimental tests, it was deduced that casein may serve as a useful historic lime mortar additive in certain circumstances. It would likely be best suited to a retrofit application where the existing lime mortar has partially deteriorated and consolidation of a masonry wall is required. In particular, improvement in flow properties and brick-to-mortar bond strength lead to the use of a casein mortar as a favourable retrofit application.

6.1.1 Limit in bond strength improvement

The point at which the tensile strength is equal to the flexural bond strength establishes the effective maximum casein content. At a casein content less than the effective maximum amount, the bond strength governs, and decreases as the casein content decreases. At a casein content exceeding the effective maximum amount, the mortar strength governs, and decreases as the casein content increases. It is therefore necessary to know both the flexural bond strength and the flexural mortar strength for the casein content being used, the lower strength would need to be used in design calculations.

The effective maximum casein content was found to vary based on the amount of water in the mortar; the flexural strength was greater in the reduced water specimens, while the flexural bond strength, while not definitively conclusive, changed very little by a reduction in water content.

6.1.2 Limitations pertaining to data collection and analysis

Flow Table test results were indicative of, but not perfectly correlated to, workability. While they are provided here for completeness, the quantitative results were highly variable, and are inconclusive by themselves.

Variability of the material was inevitably existent, despite following the same procedure as closely as possible for every test specimen. In many circumstances, the material strengths were not within the 10% tolerance criteria established in ASTM Standard C109 [26], despite casting the required number of test specimens outlined in the standard. Further testing of a large number of specimens would be required in order to determine a specified standard strength to account for the variability of both the mortar mix and the casein.

6.2 Recommendations

6.2.1 Procedural Recommendations

A refined casting procedure that remains in compliance with ASTM Standard C1072 [25] or casting of surplus stack bond tests should be conducted to account for the accidental breakage of masonry joints that occurred within the first 24 hours after initial casting and immediately before testing. Reproducibility would enable the experimental test results presented in Chapter 5: to be confirmed and hence a definitive conclusion would result.

While substantially more costly and more complicated to design and set up as an experiment, conducting the flexural brick-to-mortar bond test using a testing machine inducing a fixed load on the end of the lever arm may be preferable. The ASTM Standard C1072 [25] specifies an eccentric load, yet does not specify any details as to what type of load this shall be. A loading plate for using fixed quantity large weights was implemented to provide a load. Using a testing machine or controlled jack would eliminate undesired impulse loads that are inherent with loading by traditional weights that may lead to premature specimen failure, as it would be able to exert a controlled loading of gradually increasing magnitude.

6.2.2 Longer Term Testing

In several circumstances, the strength of the casein-containing mortar specimens approached the baseline control specimen after 56 days. In order to properly test the required specimens to determine if the strength reduction in comparison to the baseline control specimen is lower still, longer term testing of the mortar cubes is needed. It is outside of the scope of this study due to time constraints, however, further testing at 112 days and 365 days has been planned, using the mortar casting and testing procedures described in Chapter 4.

6.2.3 Further Experiments

It may also be beneficial to test the effect of casein in different lime mortar mixes. A Natural Hydraulic Lime based mix was used as it is the standard currently recommended and used most often in the heritage conservation field. Throughout all tests, only a single binder to aggregate ratio was used.

Varying the type of lime used may be of interest. The addition of casein in a hot lime mix may be affected in a slightly different manner, considering that degradation was noted at high temperatures in previous literature. Mixing ordinary hydrated lime and sand will produce a useable mortar; this was also used historically [9]. Based upon the few pieces of literature available, it would be expected that the addition of casein would exhibit a similar behaviour on other types of mortar. In certain historic conservation projects, a mixture of lime and cement is used as the binder in the mortar.

Varying the lime-to-sand ratio may be of interest. In order to maintain consistency, a pre-mixed mortar with a constant lime-to-sand ratio of 3:5 [42] was used throughout all experimental tests. The mortar mix outlined in *The City and Countrey Purchaser* specified a 1:1 lime to sand ratio [9]. Reducing the amount of sand will increase material strength; the effects of casein might vary slightly while exhibiting similar trends.

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Appendices

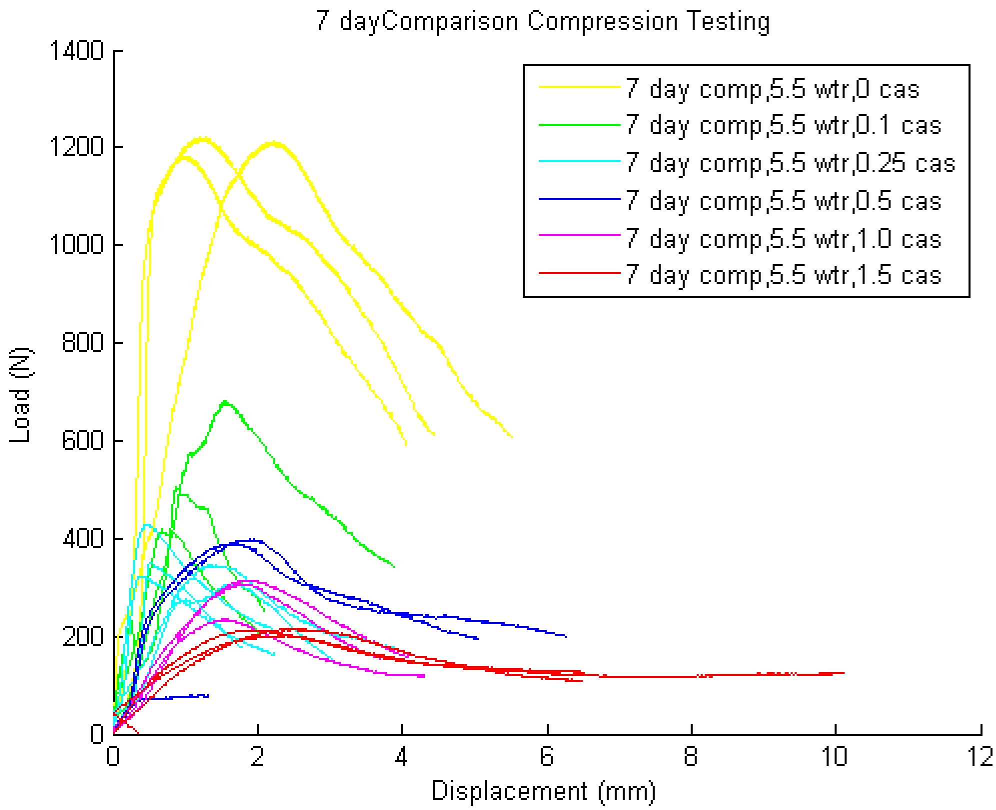
Appendix A - Load-Displacement Curves of Standard Water Concentration Specimens

The following abbreviations are used in the legends of the load-displacement curves throughout this appendix.

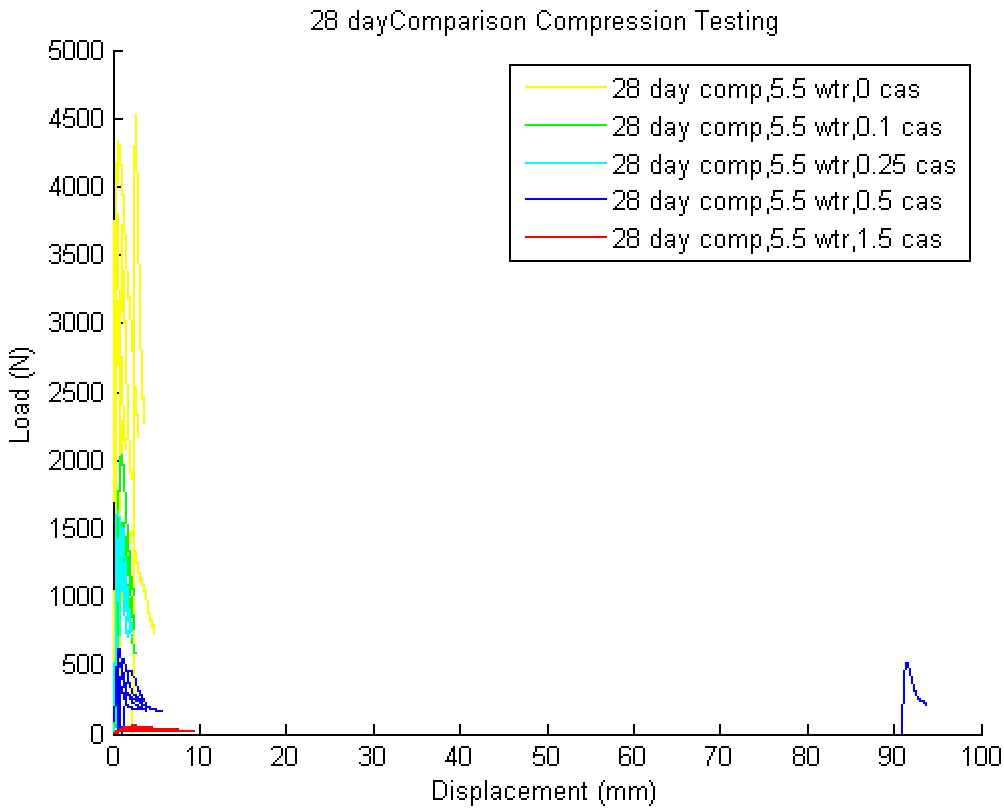
- wtr – Litres of water per 30 kilograms of mortar
- cas – Percent casein by mass
- comp – Compression
- tens – Tension
- flex – Flexure

A.1 Compression Tests

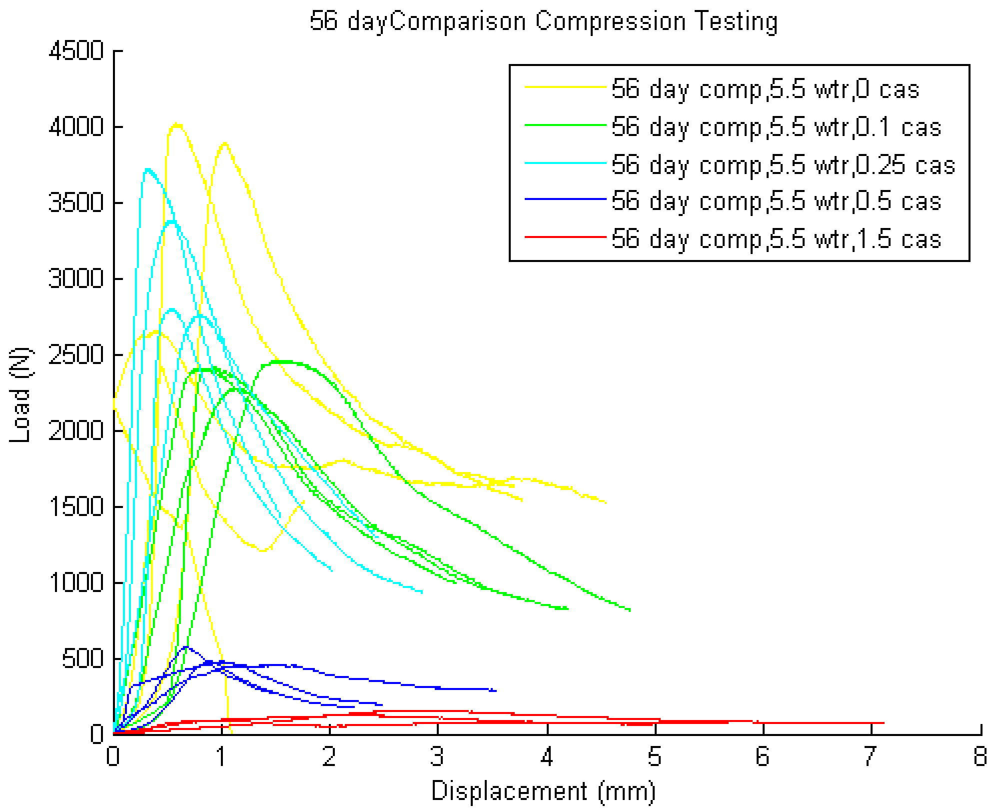
Load-displacement curves as recorded by the Instron 5582 test machine for the reduced water specimen tests in compression after 7, 28 and 56 days curing time are shown below in Appendix Figure A-1 through Appendix Figure A-3 inclusive.



Appendix Figure A-1: 7 day compression load-displacement curve



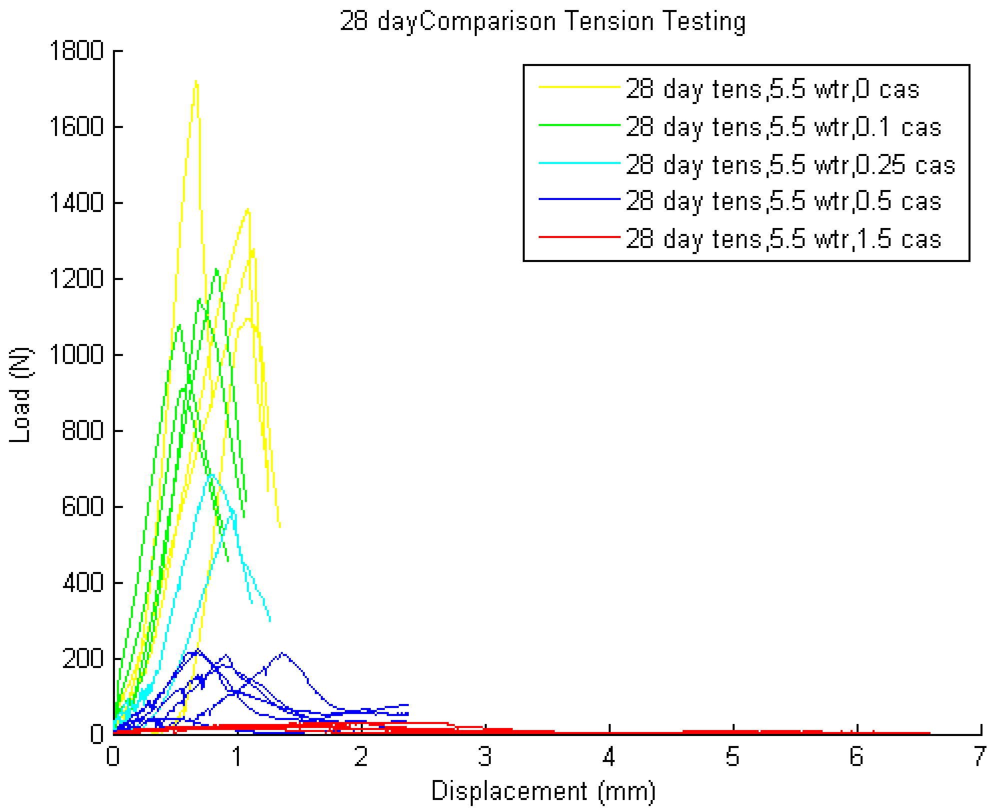
Appendix Figure A-2: 28 day compression load-displacement curve



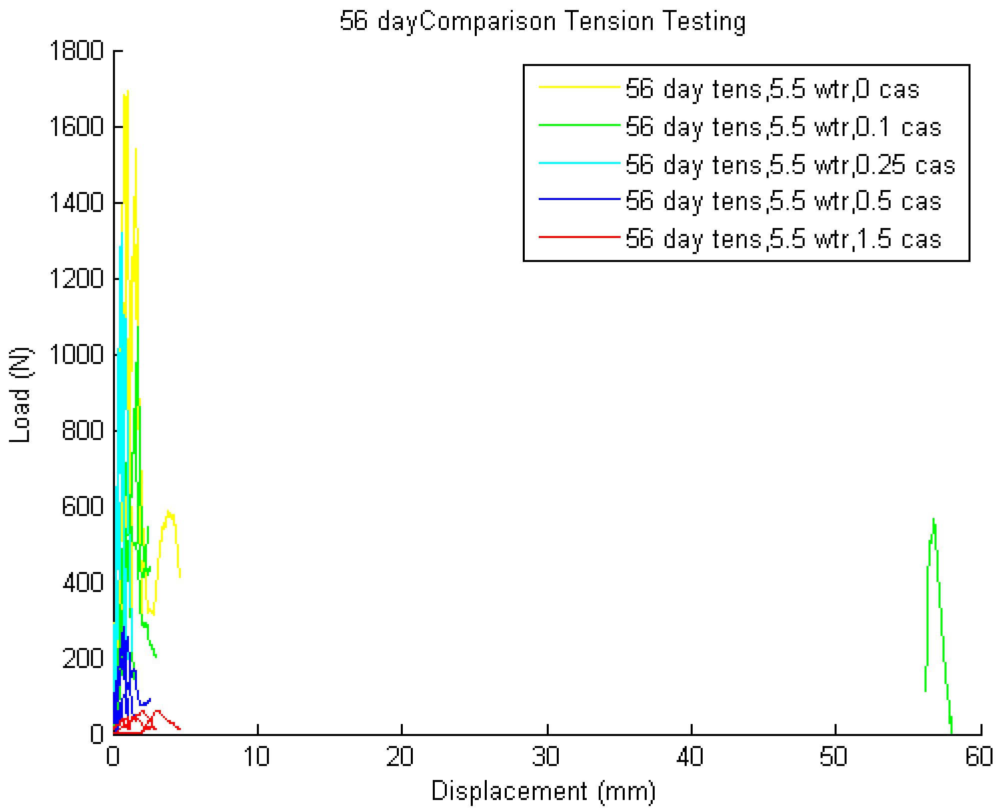
Appendix Figure A-3: 56 day compression load-displacement curve

A.2 Tension Tests

Load-displacement curves as recorded by the Instron 5582 test machine for the reduced water specimen tests in tension after 28 and 56 days curing time are shown below in Appendix Figure A-4 and Appendix Figure A-5. Note that the testing machine operated in compression as shown in Figure 4-9, therefore, the load and displacement values shown are those that were imposed on the point of load application above the specimen.



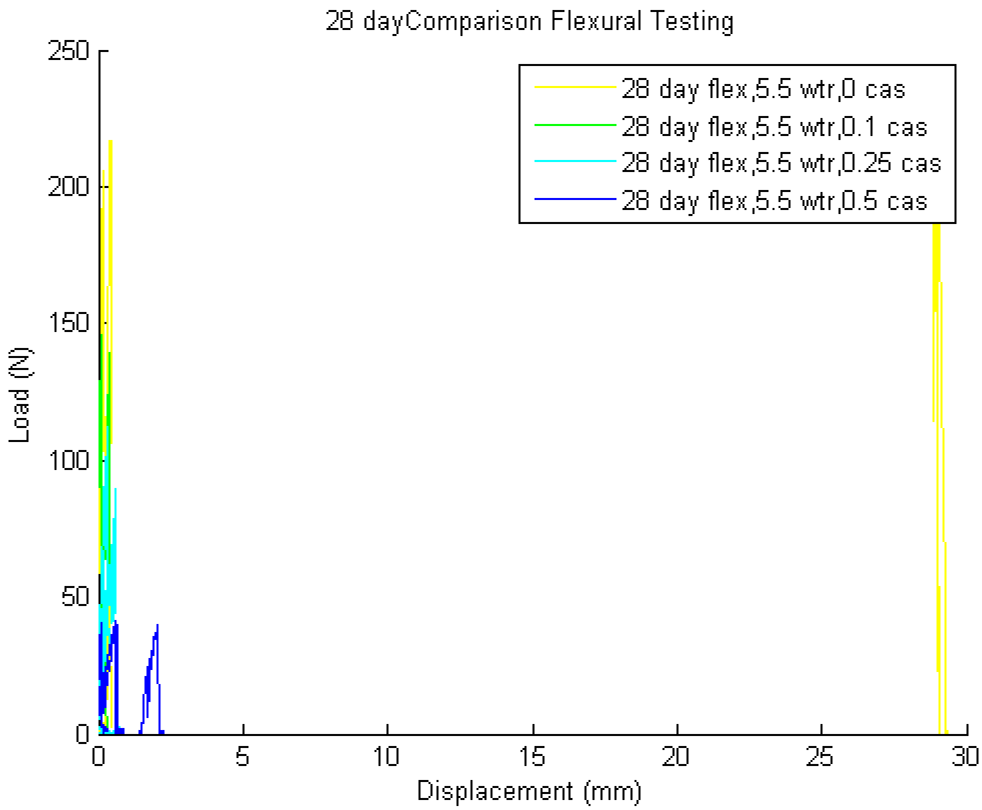
Appendix Figure A-4: 28 day tension load-displacement curve



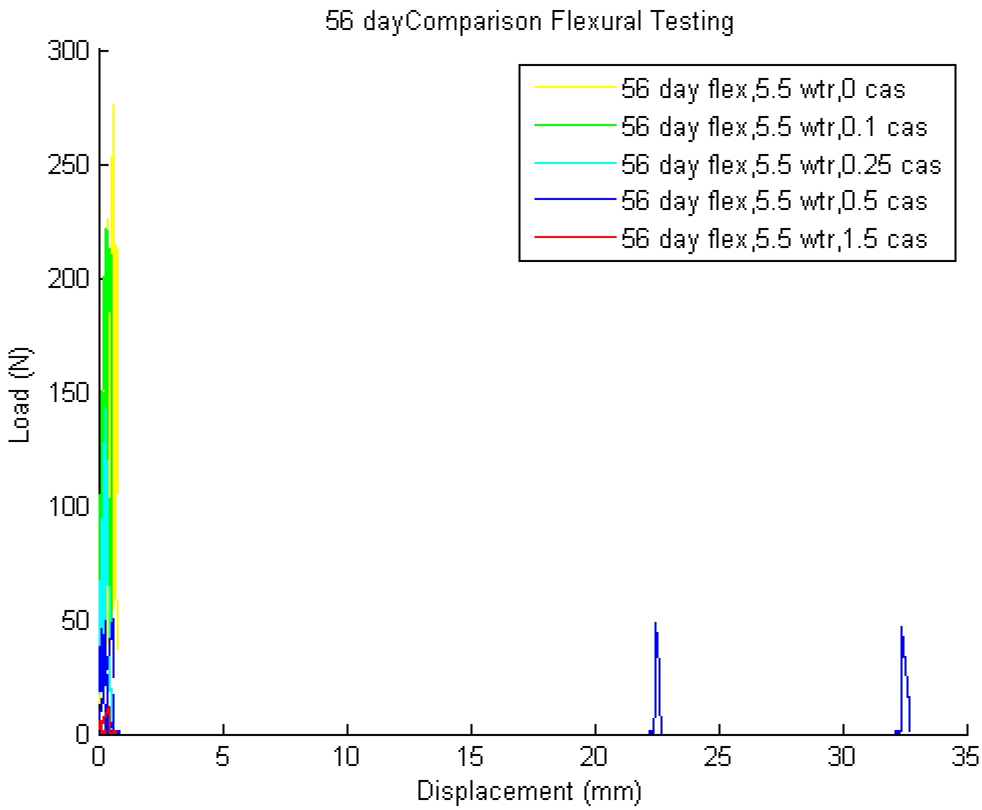
Appendix Figure A-5: 56 day tension load-displacement curve

A.3 Flexure Tests

Load-displacement curves as recorded by the Instron 5582 test machine for the reduced water specimen tests in flexure after 28 and 56 days curing time are shown below in Appendix Figure A-6 and Appendix Figure A-7. Note that the testing machine operated in compression as shown in Figure 4-11, therefore, the load and displacement values shown are those that were imposed on the point of load application above the specimen.



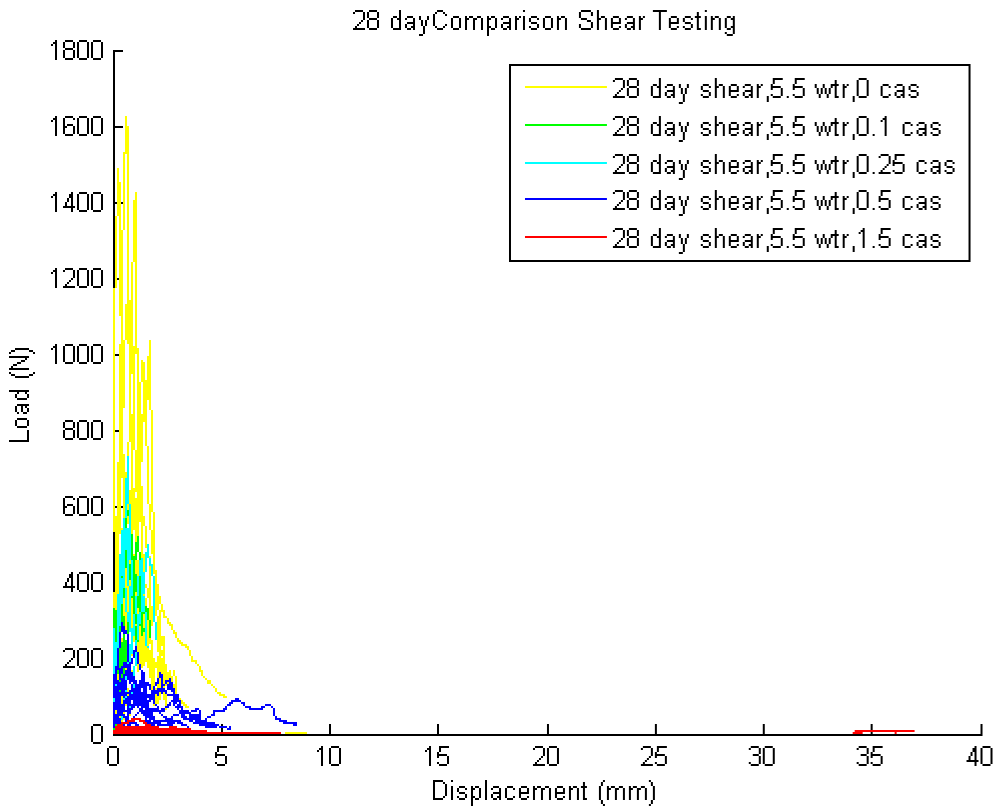
Appendix Figure A-6: 28 day flexure load-displacement curve



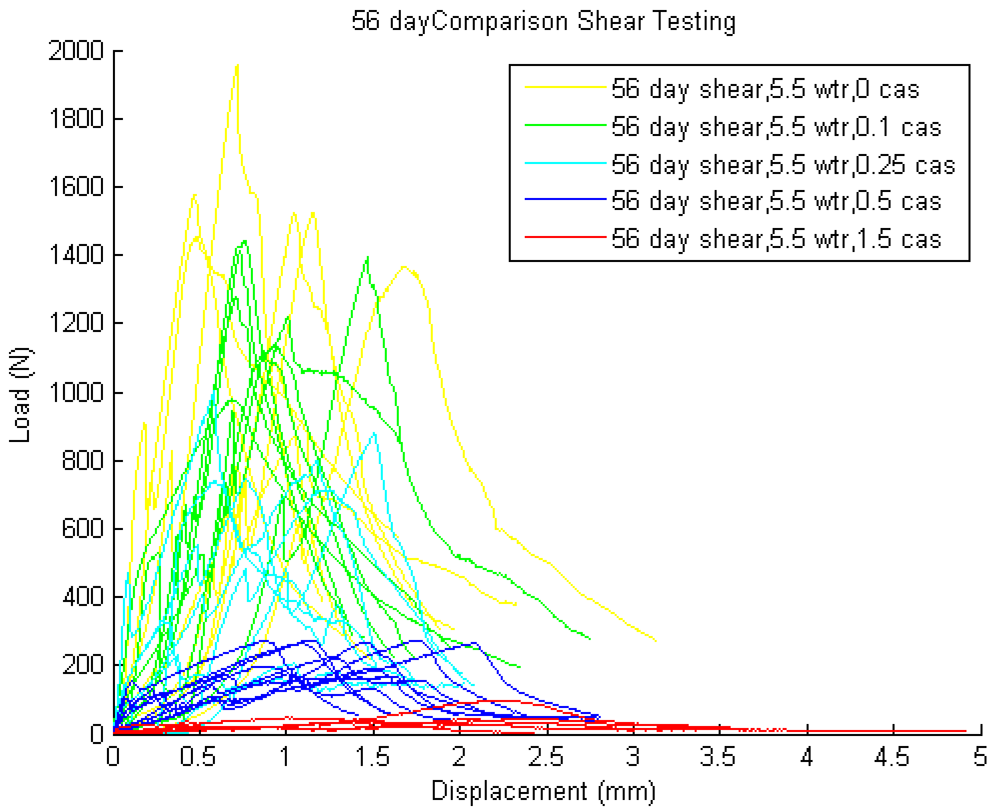
Appendix Figure A-7: 56 day flexure load-displacement curve

A.4 Shear Tests

Load-displacement curves as recorded by the Instron 5582 test machine for the reduced water specimen tests in flexure after 28 and 56 days curing time are shown below in Appendix Figure A-8 and Appendix Figure A-9. Note that the testing machine operated in compression as shown in Figure 4-13, therefore, the load and displacement values shown are those that were imposed on the point of load application above the specimen.



Appendix Figure A-8: 28 day shear load-displacement curve



Appendix Figure A-9: 56 day shear load-displacement curve

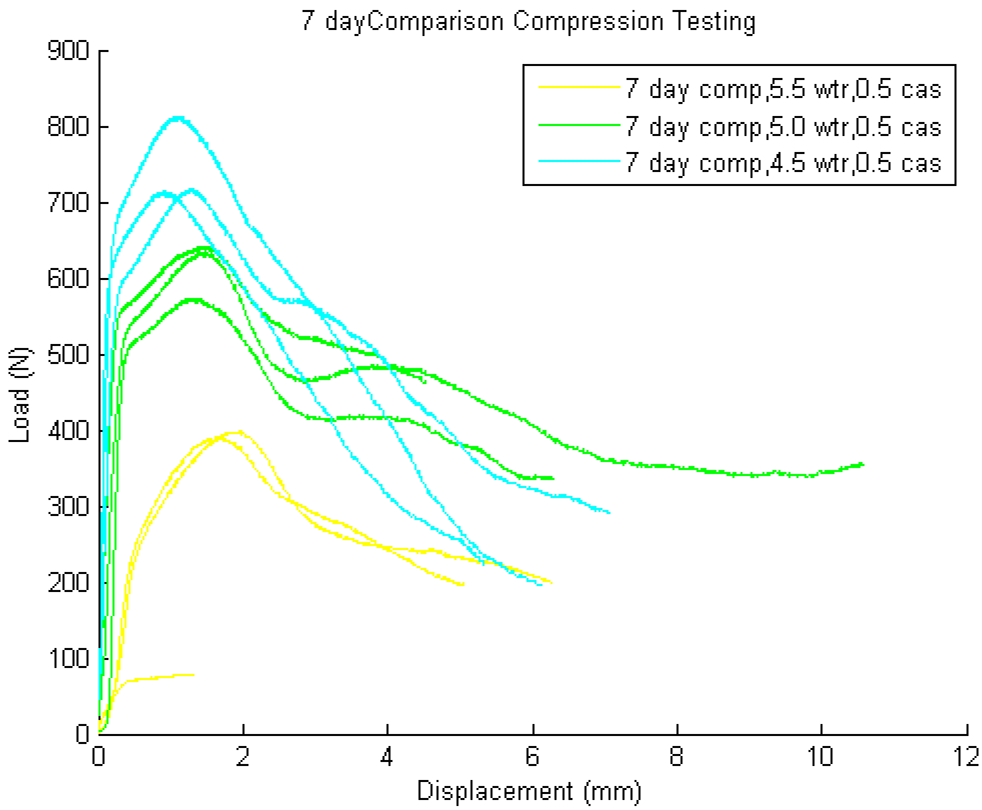
Appendix B - Load-Displacement Curves of Reduced Water Concentration Specimens

The following abbreviations are used in the legends of the load-displacement curves throughout this appendix.

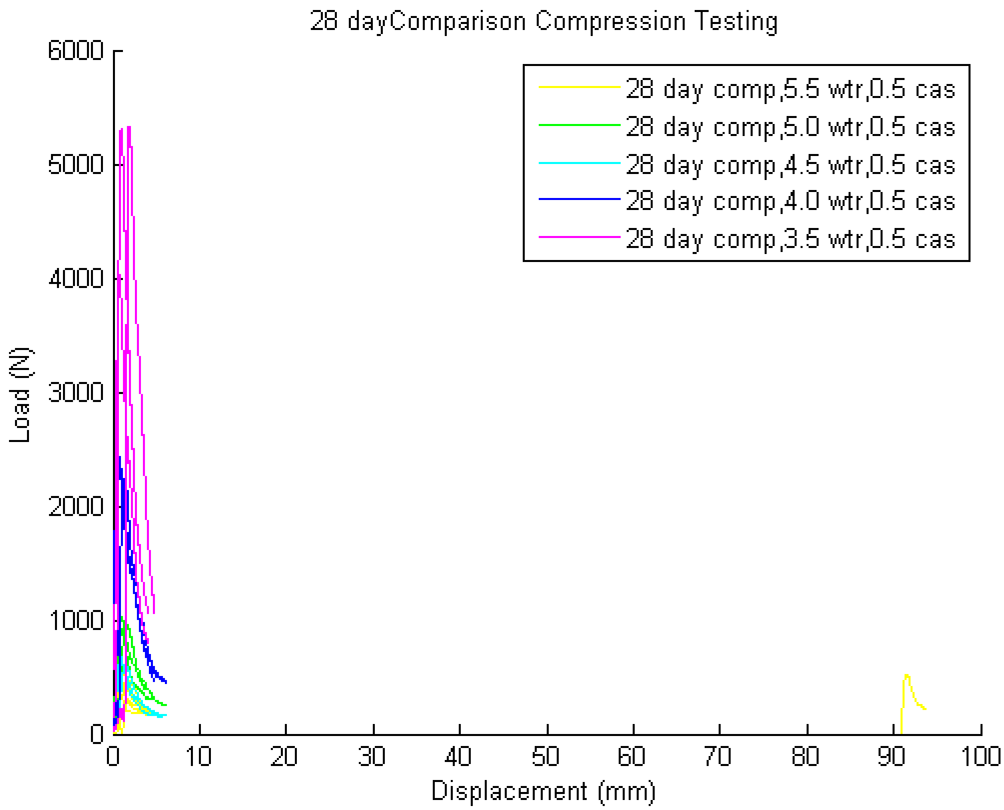
- wtr – Litres of water per 30 kilograms of mortar
- cas – Percent casein by mass
- comp – Compression
- tens – Tension
- flex – Flexure

B.1 Compression Tests

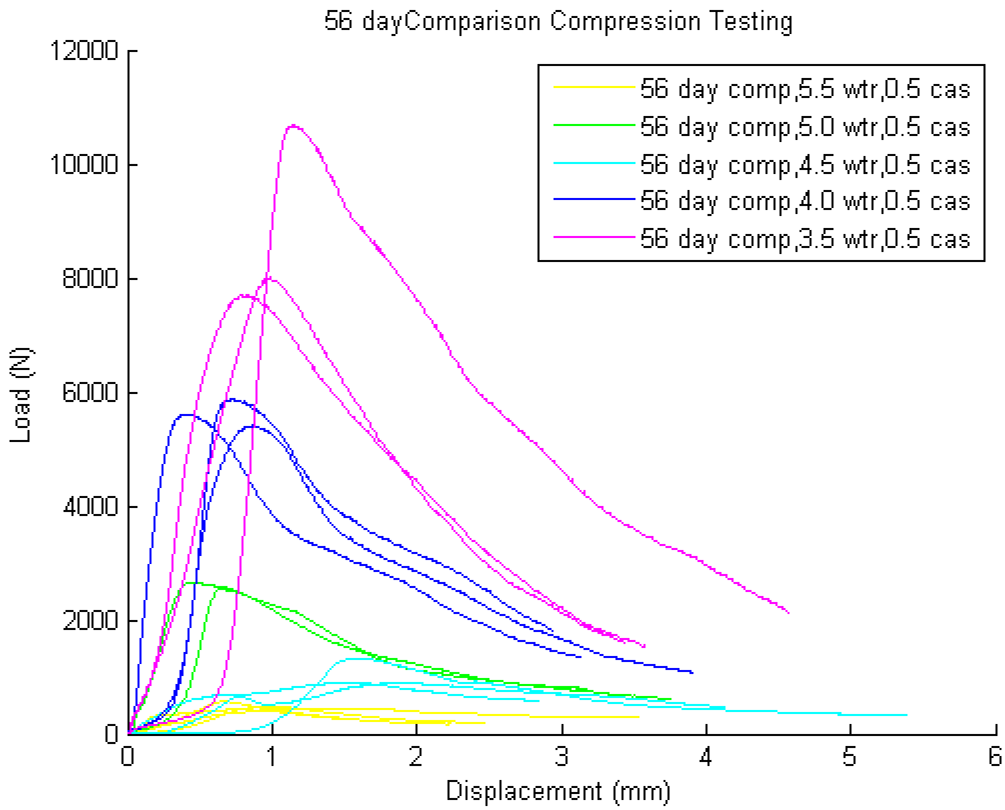
Load-displacement curves as recorded by the Instron 5582 test machine for the reduced water specimen tests in compression after 7, 28 and 56 days curing time are shown below in Appendix Figure B-1 through Appendix Figure B-3 inclusive.



Appendix Figure B-1: 7 day compression test load-displacement curve



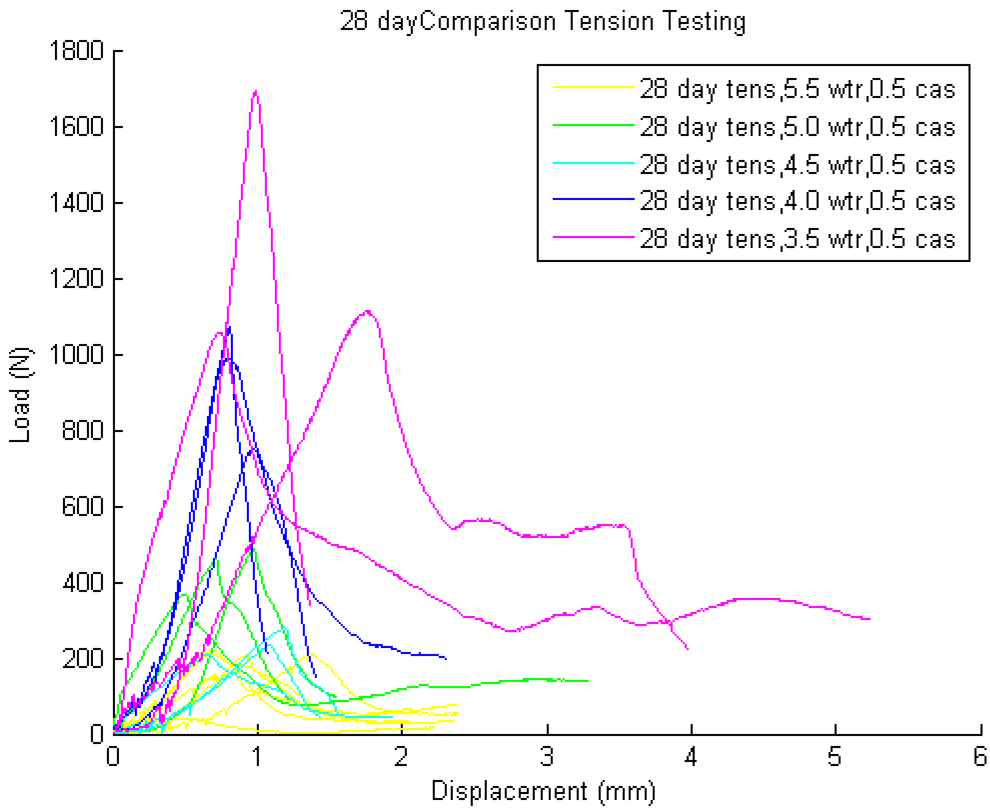
Appendix Figure B-2: 28 day compression load-displacement curve



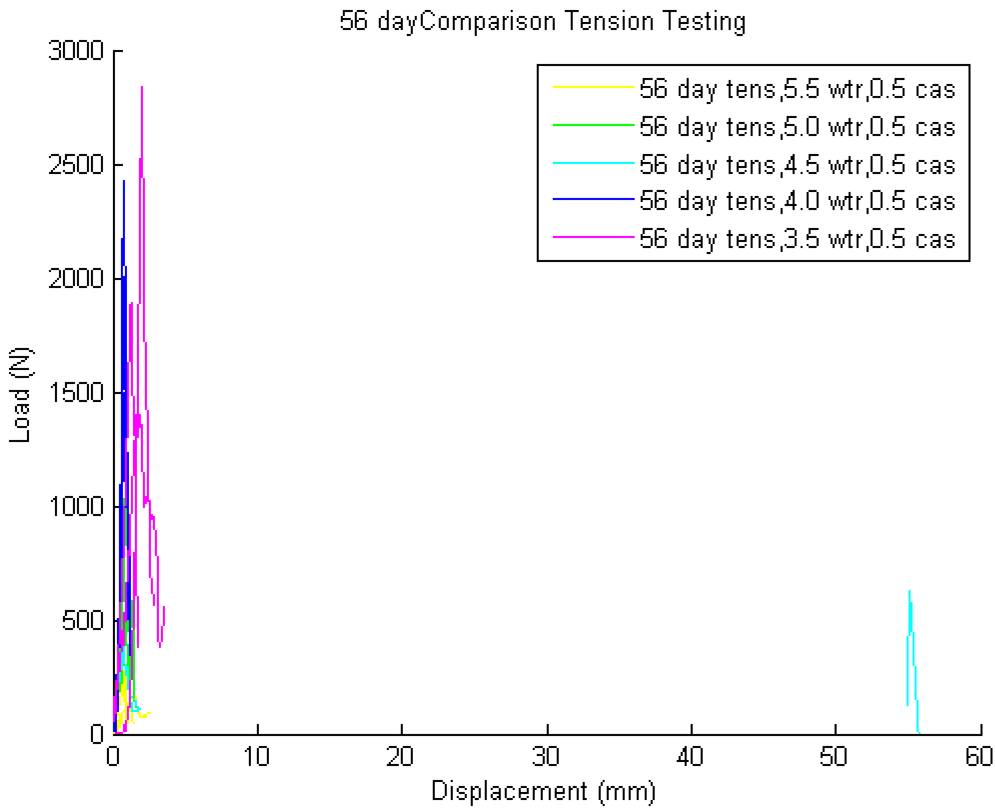
Appendix Figure B-3: 56 day compression load-displacement curve

B.2 Tension Tests

Load-displacement curves as recorded by the Instron 5582 test machine for the reduced water specimen tests in tension after 28 and 56 days curing time are shown below in Appendix Figure B-4 and Appendix Figure B-5. Note that the testing machine operated in compression as shown in Figure 4-9, therefore, the load and displacement values shown are those that were imposed on the point of load application above the specimen.



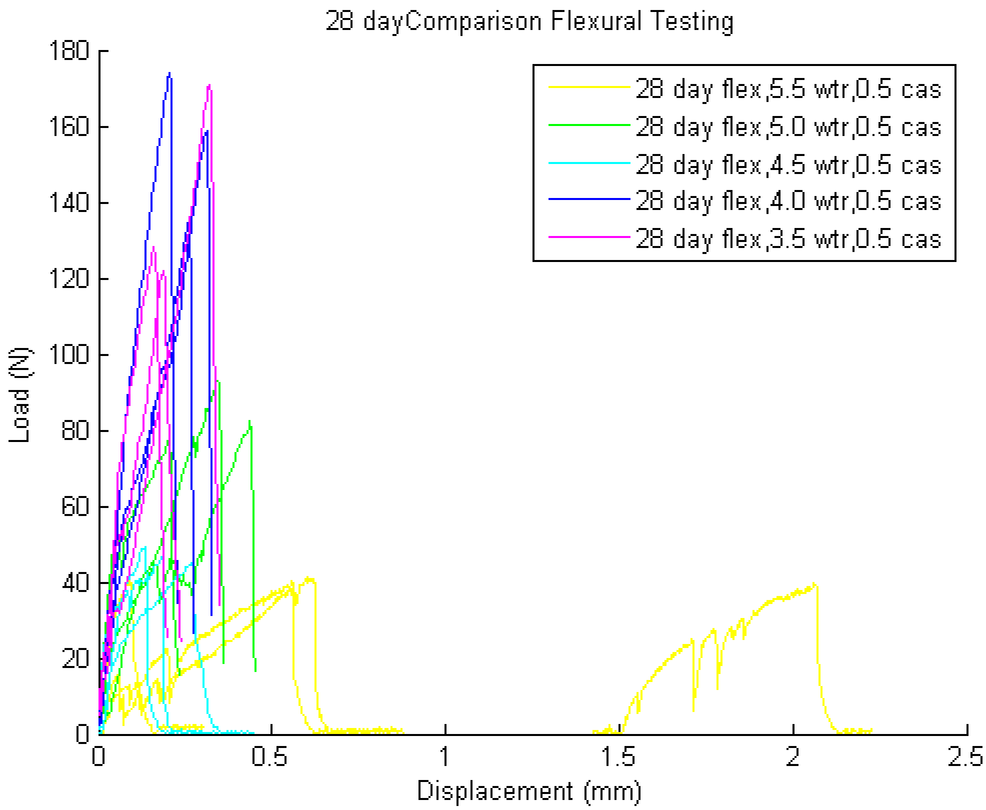
Appendix Figure B-4: 28 day tension load-displacement curve



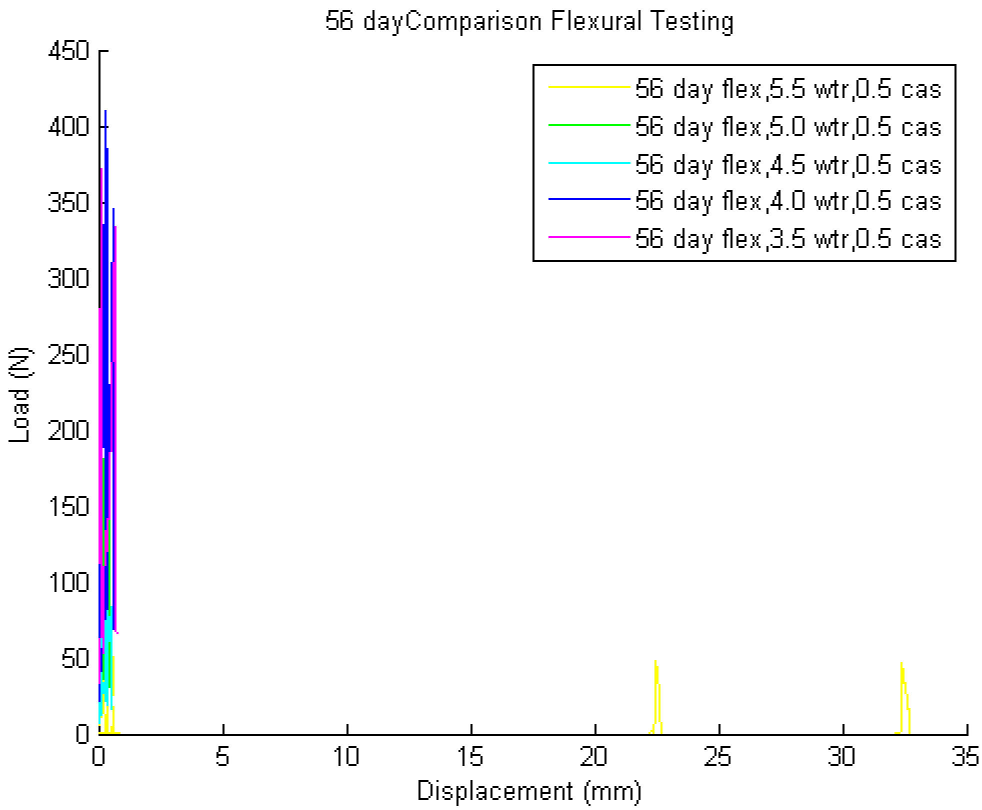
Appendix Figure B-5: 56 day tension load-displacement curve

B.3 Flexural Tests

Load-displacement curves as recorded by the Instron 5582 test machine for the reduced water specimen tests in flexure after 28 and 56 days curing time are shown below in Appendix Figure B-6 and Appendix Figure B-7. Note that the testing machine operated in compression as shown in Figure 4-11, therefore, the load and displacement values shown are those that were imposed on the point of load application above the specimen.



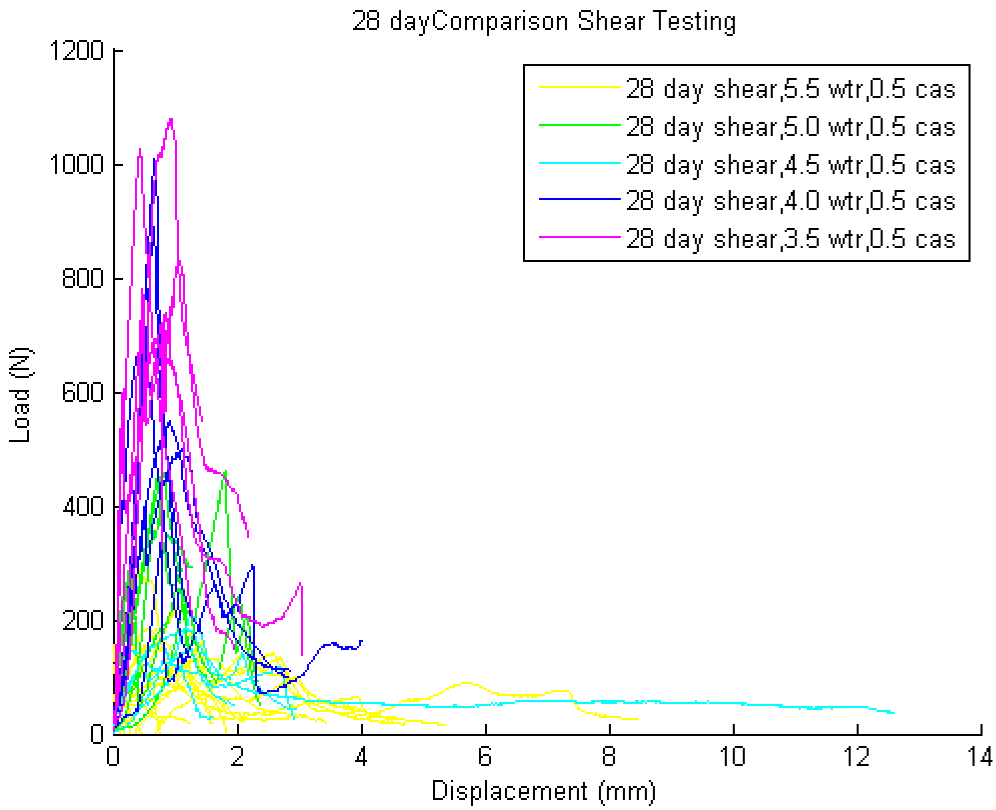
Appendix Figure B-6: 28 day flexure load-displacement curve



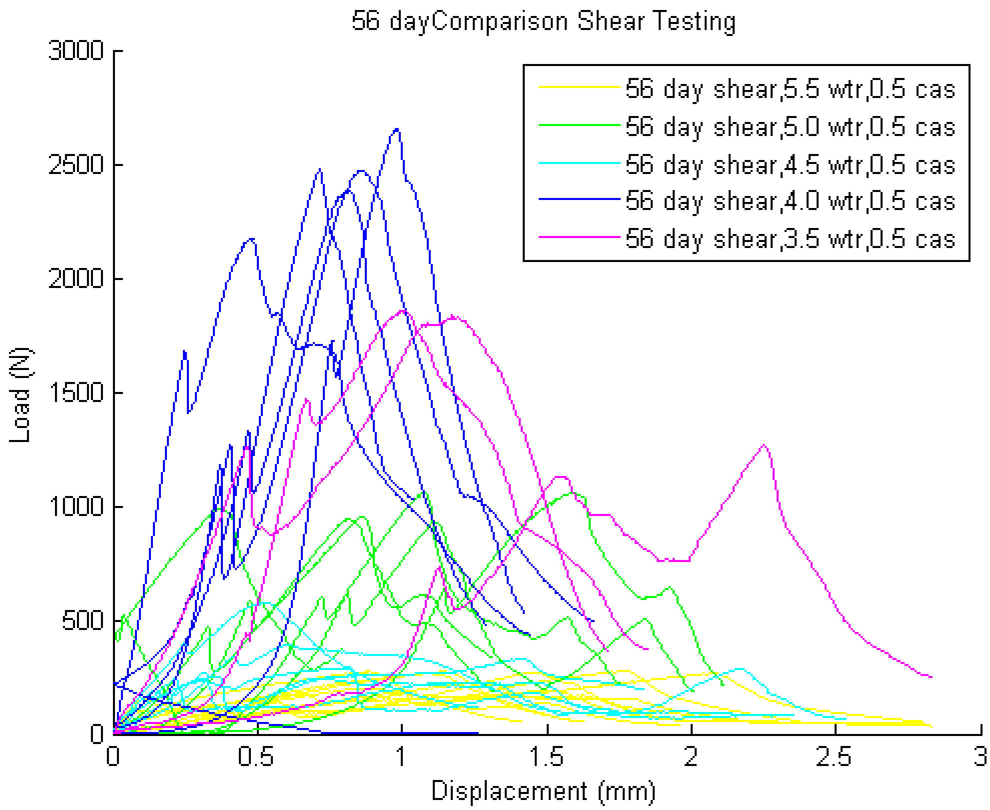
Appendix Figure B-7: 56 day flexure load-displacement curve

B.4 Shear Tests

Load-displacement curves as recorded by the Instron 5582 test machine for the reduced water specimen tests in flexure after 28 and 56 days curing time are shown below in Appendix Figure B-8 and Appendix Figure B-9. Note that the testing machine operated in compression as shown in Figure 4-13, therefore, the load and displacement values shown are those that were imposed on the point of load application above the specimen.



Appendix Figure B-8: 28 day shear load-displacement curve

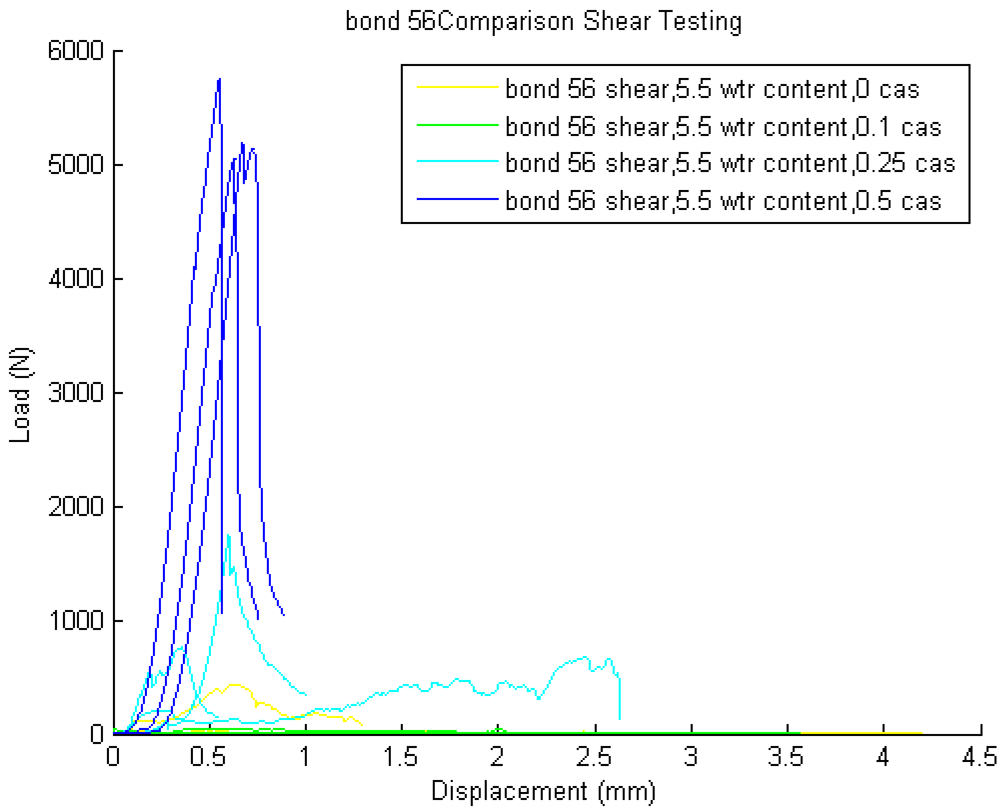


Appendix Figure B-9: 56 day shear load-displacement curve

Appendix C - Load-Displacement Curve of Shear Bond Tests

Below is the load-displacement data curve as recorded by the Instron 5582 test machine for the brick-to-mortar shear bond tests after 56 days. The following abbreviations are used in the legends of the load-displacement curve in this appendix.

- wtr – Litres of water per 30 kilograms of mortar
- cas – Percent casein by mass
- bond 56 – 56 day bond test



Appendix Figure C-1: 56 day shear bond test load-displacement curve