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PROCESSING AND CHARACTERIZATION OF A LIGHTWEIGHT CONCRETE USING CENOSPHERES

FINAL RESEARCH REPORT

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16. Abstract

A study has been conducted in which a lightweight concrete was processed using ceramic microspheres, known as *cenospheres*, as a primary aggregate. The mechanical properties, including compressive strength, tensile strength, flexural strength and fracture toughness, were tested and cataloged. It was determined that the addition of high volumes of cenospheres significantly lowered the density of concrete but was also responsible for some strength loss. This strength loss was recovered by improving the interfacial strength between the cenospheres and the cement. The interfacial properties were quantified using interfacial fracture mechanics techniques. These techniques were also employed to find a suitable surface modifier with which to improve this interface. The admixture silica fume and the coupling agent Silane™ were found to be suitable candidates and both performed well in small-scale compression testing. Silica fume waseventually isolated as a prime candidate. The concrete produced with this admixture was tested and compared to a concrete with an equal volume fraction of cenospheres. The addition of silica fume improved the specific compressive strength of cenosphere concrete by 80%, specific tensile strength by 35%, specificflexural strength by 67% and specific fracture toughness by 73%. Therefore, the production of a high-performance lightweight concrete using cenospheres has been performed.

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PREFACE

This report presents the results of analytical and experimental work in which a lightweight concrete was fabricated and characterized and ultimately improved, creating a high-performance lightweight concrete. The concrete was produced using ceramic microballoons called *cenospheres* as a primary aggregate. These cenospheres initially showed a positive reduction in concrete density, which was a desired effect, but there was also some strength loss. This strength loss was attributed to the weak interface between the binder material (cement) and the cenospheres. This was shown quantitatively through interfacial fracture mechanics techniques. These techniques also facilitated the discovery of interface modifiers that were shown to improve the interfacial properties, thus improving the overall mechanical and fracture properties of this lightweight concrete. The main body of this report, which has been prepared in manuscript format, is broken into four sections.

The first section is the introduction, which provides relevant background information and motivation for this study. It is followed by the experimental procedures and results in the second section. This work focused on the characterization of this new type of lightweight concrete. This characterization included compressive, tensile and flexural strengths as well as fracture toughness. This section also isolated the weak interface between the cement binder and the cenospheres as a cause for strength loss.

The third section discusses interfacial strengths between the binder and the cenospheres. Bimaterial fracture techniques were employed to quantify the interfacial strength. These techniques were also used to find possible interfacial modifier candidates, which could be used to improve the materials properties.

The fourth section deals with the modified lightweight concrete. The most promising interfacial modifier was chosen and introduced into a similar concrete already tested. This concrete was characterized as in the second section and compared to previous data. The fifth and final section is the conclusion, which includes proposed future work in this area.

Following the main body are four appendices. Appendix A is a review of previous work. Appendix B is a detailed instruction on how to fabricate an aluminum silicate/cement bimaterial fracture specimen. Appendix C is all mix designs and relevant notes on the mixing of these concretes. Appendix D contains all the individual properties of the concrete component materials.

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INTRODUCTION

This study presents how a new type of lightweight concrete was developed using ceramic microspheres as a primary aggregate. These ceramic microspheres are a waste product, so they are relatively inexpensive and the use of them has the added benefit of decreasing the strain on the environment. This aggregate allowed the density to be reduced significantly but initially caused some strength loss. The strength was later regained with the use of interface modifiers. The result is a high-performance lightweight concrete.

Concrete is the number one structural material used in the world today. The demand to make this material lighter has been the subject of study that has challenged scientists and engineers alike. The challenge in making a lightweight concrete is decreasing the density while maintaining strength and without adversely affecting cost. Introducing new aggregates into the mix design is a common way to lower a concrete's density. Normal concrete contains four components, cement, stone, sand and water. The stone and sand are the components that are usually replaced with lightweight aggregates.

Many studies have been done with a wide range of fillers with the purpose of developing a lightweight concrete. Many of these studies used organic fillers in order to decrease the density. Aziz, et al. [1] studied the effects of cork granules. Slate used coconut fibers, all with little benefit. [2]

In recent years work has been carried out documenting the details of inorganic admixtures, such as flyash, and today flyash is widely used in the concrete industry. Flyash is inexpensive, has good pozzolanic properties (reacts with water to form cementitious materials), and can be half the density of cement. Naik, et al. has shown that flyash can not only decrease the cost and density, but also make the concrete stronger, more durable and more resistant to corrosion. [3]

Silica fume is another compound, which has been studied extensively and is used in concrete today. Tazawa, et al, have shown that silica fume can improve concrete strength, durability and corrosion resistance. [4]

An important by-product of flyash is *cenospheres*, relatively large (10-300 μm) thin-walled microspheres produced during flyash formation. Clayton and Back show that cenospheres are formed during the coal burning process by evolution of gas becoming trapped in a viscous molten glass matrix. [5] These cenospheres can be reclaimed from flyash readily and are relatively inexpensive as a bulk product. They are also considered a waste product, so any use of them decreases the strain on the environment. Wandell has suggested many uses for this material, including using them as fillers in polymers and concrete. [6]

A light micrograph of cenospheres is shown in Figure 1. These cenospheres have a low specific gravity, (approx. 0.67) which makes them ideal to be used as a predominant aggregate in a lightweight concrete.

The purpose of this study was to develop a lightweight concrete using cenospheres and characterize the mechanical and fracture properties of this new material. The ability to tailor the properties of this new material through the use of interface modifications was also investigated.

The first part of this study determined if cenospheres could be used as a replacement for fine aggregate. It was also determined if the cenospheres would segregate during the curing process due to their low specific gravity. This would cause the materials properties to vary throughout a specimen.

Three concrete mixes were made based on a control mix known as RIDOT Class XX AE. 50%, 75% and 100% of the fine aggregate were replaced in these mixes with cenospheres. All mechanical and fracture properties for these mixes were cataloged and all values reported are the result of at least five experiments. All results are reported with 95% confidence intervals.

Bimaterial fracture mechanics were employed to determine the interface properties of cenospheres and cement. These techniques have been used to characterize many materials, but no such work has been done in the area of infrastructure materials like concrete. These tests quantitatively showed that the inherent weakness in cenosphere concrete is the cenosphere/cement interface. Interface modifier candidates were also found using this technique. Certain desiccant, anti-leaching and admixture type modifiers were studied.

Small batches of concrete were mixed in order to further test interface modifier candidates. This was done to ensure that they would perform correctly under real world conditions. It was deemed necessary to test any potential interface modifier in a controlled and quick manner before investing the time and energy necessary to develop a large batch. These experiments showed that silica fume and a coupling agent called Silane™ had the best potential to improve the strength of the cenosphere concrete.

Silica fume was chosen as the best candidate to improve the performance of the cenosphere concrete due to its ease of use and low cost. A mix design was created incorporating this admixture and mixed on a large scale. All the mechanical and fracture properties of this material were investigated. These tests showed that all mechanical properties improved significantly with the addition of silica fume when compared to an equivalent concrete.

EXPERIMENTAL PROCEDURE AND RESULTS

Concrete Mix Design and Specimen Fabrication

All concretes used in this study were based on the Rhode Island Department of Transportation (RIDOT) Class XX AE. This mix design can be seen in Table 1.

The above recipe was our control batch (B1). The successive batches contained 13% (B2), 19.5% (B3) and 26% (B4) cenospheres of the unit volume. These numbers represented 50%, 75% and 100%, respectively, of the sand volume that was replaced with cenospheres. This method was chosen to maintain a constant consistency insofar as one fine aggregate (sand) was always replaced with another fine aggregate (cenospheres).

All batches of concrete were mixed at the Fiore Concrete Industries Inc. of South Kingston, RI except B3, which was mixed at Cardi Corp. of Cranston, RI. Batches were mixed according to ASTM C192-95. All batches were consolidated by rodding and allowed to harden for 24 hours and then placed in a wet bath and allowed to cure for 28 days according to ASTM C 192-95 before testing.

Individual component properties can be found in Appendix D. Batches B1 and B2 were mixed in during the 1999 season. All remaining batches were mixed during the 2000 season.

Viability of Using Cenospheres as a Primary Aggregate

A batch B4 was mixed and cast into a mold 305 mm (12") in height, 305 mm (12") wide, and 76 mm (3") deep. The specimen was allowed to air cure for two days and then cut along an edge from bottom to top.

With a flat internal surface exposed, it was possible to inspect the specimen under a microscope along the entire length. One of these photographs can be seen in Figure 2. From this one could determine the extent, if any, of cenosphere segregation.

To quantify these observations, the volume fraction was calculated from all the micrographs. This was achieved by drawing lines across the micrograph, as seen in Figure 2. An average number was calculated from fractional lengths of the lines taken up by cenospheres, and then taking an average of many lines. This gives an area fraction and this was assumed to be the volume fraction as well due to the spherical nature of the cenospheres.

It was determined that the cenospheres in this batch made up 28.75% ± 3.82% of the total volume. This agrees well with the value of 31% estimated using bulk density calculations. It can be seen in Figure 3 that although the volume fraction does not remain constant throughout the specimen, it varies randomly as one progresses towards the top of the specimen. It became evident from this simple experiment that cenospheres are a viable admixture to be used in concrete.

Density Analysis

The density was recorded by measuring the weight of the cylindrical specimens, which had a known volume.

The density measurements for the different concrete batches can be seen in Figure 4. As was expected, the density decrease was proportional to the amount of sand replaced with cenospheres to a low value of approximately 1800+/-34 kg/m^3 (112+/-2 lbs/ft^3), 22% lower than the control batch.

Compressive Strength

The compressive strength of the different concretes was measured according to ASTM C39-94. The specimen size used was 101.6 mm (4") in diameter and 203.2 mm (8") in height. These tests were performed on a Forney™ hydraulic testing system model FT-40.

The compressive strength for all batches of concrete can be seen in Figure 5. The control batch B1 showed a compressive strength of 44+/-2 MPa (6300+/-300 psi). The line shows what is an accepted benchmark for concrete strength, which is approximately 27.5 MPa (4000 psi). When cenospheres were initially added in B2, there was a 36% drop in compressive strength to 28+/-1 MPa (4060+/-150 psi). B3 showed a minimal rise to 31+/-1 MPa (4500+/-150 psi). B4 showed a compressive strength of 20+/-1 MPa (2900+/-150 psi), which represents a 55% drop in total compressive strength. Although this represents a significant drop in strength, the lower density must be considered. Figure 6 shows the specific compressive strength. This figure shows that the loss in strength is not so severe since the concrete is much less dense.

The mode of failure was also different for the cenosphere concrete. B1 showed a markedly shear type failure. The cenosphere concrete failed in a more violent way and almost always in a columnar fashion. The samples were usually completely destroyed. An example of both types of failure can be seen in Figure 7.

Tensile Strength

The tensile strength of all specimens was measured according to ASTM C 496-96. These tests were also performed on a Forney™ FT-40 using the alignment jig suggested in the ASTM document to ensure that the load was applied perfectly along the diametral line. The specimen size was exactly the same as for compression testing.

The tensile strength for all batches can be seen in Figure 8. B1 showed a tensile strength of 3.52+/-0.38 MPa (510+/-55 psi). The line shows an accepted benchmark for the tensile strength of concrete, which is approximately 2.00 MPa (300 psi). When cenospheres were added in B2, there was a 40% drop in the tensile strength to 2.07+/-0.14 MPa (300+/-20 psi). Again, there was a slight rise in strength for B3 to 2.29+/-0.25MPa (330+/-36 psi). B4 showed a tensile strength of 2.14+/-0.09 MPa (310+/-13 psi). Figure 9 shows the specific tensile strength. This figure shows that with further density decrease there was a gain in tensile strength.

Figure 10 shows a tensile specimen of cenosphere concrete after failure. This figure shows the "popping out" of the coarse aggregate. This is not beneficial to the concrete due to the fact that the coarse aggregate is what gives concrete its strength. One would prefer to see more cleaving of coarse aggregate.

Flexural Toughness

The flexural toughness was measured using ASTM C 293-94. These tests were performed on a MTS™ testing system Model 810. The specimen had a 152.4 mm (6") thickness and height and a 508 mm (20") length, which gave a 457.2 mm (18") test span.

The flexural toughness for all batches can be seen in Figure 11. B1 showed a flexural toughness of 4.34+/-0.35 MPa (630+/-51 psi). When cenospheres were added in B2, there was an 8% drop in toughness to 3.98+/-0.30 MPa (577+/-44 psi). B3 was unchanged within experimental error at 3.95+/-0.2 MPa (573+/-30 psi). B4 showed a significant drop in toughness to 2.72+/-0.02 MPa (394+/-30 psi). This represents a 37% drop in toughness when compared to the control batch.

Figure 12 shows the specific flexural toughness. This figure shows that there is a small rise in the flexural performance for B1 through B3. Then there is a sharp drop in B4 at the highest level of cenospheres.

Figure 13 shows a flexural specimen after failure. Again there is pop out of the coarse aggregate. This shows improper bonding within the matrix.

Fracture Toughness

There exists no standard test method for the determination of fracture toughness in concrete. ASTM 5045, a standard method for composite fracture toughness was employed instead. The following equation is used to relate load at failure to the critical stress intensity factor, K_{1c}.

$$K_{1c} = \frac{FS}{BW^{3/2}} * \frac{3\sqrt{x}\left(1.99 - x(1-x)\left[2.15 - 3.93x + 2.7x^2\right]\right)}{2(1+2x)(1-x)^{3/2}} \tag{1}$$

Where F is the load at failure, S is span, B is thickness, W is the specimen height, a is the crack length and x is a/W. This specimen had a length of 660.4 mm (26"), which gave a span, S, of 609.6 mm (24"). The specimen height, W, was 152.4 mm (6") and the thickness B was 81.3 mm (3.2"). The crack length was 76.2 mm (3"). The specimen geometry can be seen in Figure 14. The crack geometry can be seen in Figure 15. These tests were also performed on the MTS™ Model 810.

Figure 16 shows the fracture toughness for all batches. B1 showed a flexural toughness of 0.83+/-0.02 $MPa(m)^{1/2}$. This agrees well with previous experimental data that shows this type of concrete should have a K_{Ic} of around 1 $MPa(m)^{1/2}$. There is an extreme drop in the fracture toughness when cenospheres are added in B2 to 0.33+/-

0.02 $MPa(m)^{1/2}$. This represents a 60% drop in the fracture toughness. B3 had a fracture toughness of 0.25+/-0.05 $MPa(m)^{1/2}$. B4 was slightly lower at 0.21+/-0.03 $MPa(m)^{1/2}$.

Figure 17 shows the specific fracture toughness. This shows that the fracture toughness of the cenosphere concrete is an inherent weakness. Even normalized, the drop from B1 to B2, B3 and B4 is considerable.

Figure 18 shows a fracture specimen after failure. The pop out of coarse aggregate seen in the tensile and flexural specimens are also present in the fracture specimens.

Although all concretes tested close to or above industry benchmarks for certain applications, it would be beneficial to improve the overall strength as much as possible. The loss of strength seems to be due to the poor interface properties between the cenospheres and the cement. This can be seen clearly in the SEM micrograph Figure 19, which shows a cenosphere popped out of the cement matrix. Improving the interface properties of the cenospheres and cement is believed to be the key to performance increase.

SURFACE CUSTOMIZATION AND INTERFACE STRENGTH

Surface Customization

Four different surface customizations were attempted in order to improve the interfacial strength. These procedures are outlined below.

DESSICANT TYPE ADMIXTURE w/ ANTI-LEACHING AGENT

The desiccant used was calcium chloride ($CaCl_2$). This was mixed with the water used at a rate of 0.022 g per 1 g of water. The anti-leaching agent, sodium silicate, known also as water glass, was added at rate of 0.345 ml per 1 g of water. The water needed for the concrete was replaced with this solution.

SILANE™ (TYPE A174) TREATMENT

The cenospheres were coated with Silane™ before being mixed into the concrete. This was done in the following manner. A solution of 1080 cc of methanol, 120 cc of water and 4.3 g of Silane™ were mixed and poured into 428 g of agitated cenospheres. These were allowed to air dry for one day and then oven dried at 50°C for four hours. This recipe would be repeated until enough cenospheres were available.

SILICA FUME ADMIXTURE

Concrete batches were mixed similar to the previous batches, but 12% of the cement was replaced with silica fume by weight. The water was also increased slightly to adjust for the higher volume of cement and silica fume. A w/c ratio of 0.45 was normal for these types of concrete.

COMBINATION

It was decided to investigate the idea of a cumulative effect between these two interface modifiers. The concrete was exactly as the silica fume variant but using the Silane™ treated cenospheres.

Interfacial Strength

The interfacial strength of cenospheres and cement was tested using bimaterial fracture mechanics. Once the interfacial fracture toughness was established, similar bimaterial tests were performed to test the surface customizations.

Background

A bimaterial system is defined as two dissimilar; linearly elastic materials bonded or cast together. Figure 20 shows a bimaterial system with a central crack geometry. Material 1 is the more compliant of the two materials.

The bimaterial fracture experiments were done using a central crack geometry loaded in tension. The stress/strain field characterization is performed using what is known as the complex stress intensity factor. [7]

$$K = K_1 + iK_2 \tag{2}$$

This factor completely characterizes the stresses around the crack tip and shows a coupling of the opening mode and in-plane shear mode.

The material properties are accounted for with the mismatch parameter [7]:

$$\varepsilon = \frac{1}{2\pi}\ln\left(\frac{\dfrac{x_1}{\mu_1}+\dfrac{1}{\mu_2}}{\dfrac{x_2}{\mu_2}+\dfrac{1}{\mu_1}}\right) \tag{3}$$

where μ_i are the shear moduli and;

$$x_i = (3-\nu_i)/(1+\nu_i) \tag{4}$$

where ν_i are the Poisson ratios.

Finally, for a uniaxially stress state $(T = \sigma_{yy}^{\infty})$ in a central crack geometry, K_1 and K_2 can be expressed in terms of the remote loading [8]:

$$K_1 = \sigma_{yy}^{\infty}\sqrt{\pi a}\left[2\varepsilon\cos(\varepsilon\ln(2a)) + 2\varepsilon\sin(\varepsilon\ln(2a))\right] \tag{5}$$

$$K_2 = \sigma_{yy}^{\infty}\sqrt{\pi a}\left[2\varepsilon\cos(\varepsilon\ln(2a)) - \sin(\varepsilon\ln(2a))\right] \tag{6}$$

where $2a$ is the crack length.

Specimen Preparation

A description of specimen fabrication is provided in Appendix B. The cement used had a w/c ratio of 0.44, which is similar to the w/c ratio found in the concretes used throughout the investigation.

Test Setup

A photo of the test setup can be seen in Figure 21. An Instron™ Model 1125 testing machine was used. Prior to placing the specimen in the Instron™, the load cell was calibrated. The load cell was connected to the A/D software Labtech™ for automatic acquisition of the data.

Test Conduct

The specimen was then loaded at a rate of 8.62 kPa/s (1.25 psi/s) until failure, at which time the data from the software was checked for the maximum load at which failure occurred.

Results and Discussion

Following the experiment, the maximum load at failure was converted to stress and applied to equations (5) and (6) to determine the values of the critical complex stress intensity factor. After conducting multiple experiments an average value of K_{1c}=0.068±0.001 MPa-$m^{1/2}$ and K_{2c}=0.002±0.001 MPa-$m^{1/2}$ was found.

This is an extremely low value for a bimaterial interface. As a comparison, the bimaterial fracture toughness of an aluminum-polycarbonate interface is K_{1c}=1.00 MPa-$m^{1/2}$ and K_{2c}=0.5 MPa-$m^{1/2}$.

This quantitatively shows where the weakness in this material lies. To improve the strength of this material, one must improve the properties at the cement/cenosphere interface. The results of attempts to improve the interfacial properties are outlined in the following paragraphs.

The desiccant type admixture was a failure. The specimens were much weaker than any previously tried. The weakness was so prominent that most test specimens failed under their own weight even before they could be loaded into the testing apparatus. Therefore, this option was abandoned.

The silica fume showed impressive results. After conducting multiple experiments, an average K_{1c}=0.167+/-0.001 MPa-$m^{1/2}$ and K_{2c}=0.004+/-0.001 MPa-$m^{1/2}$ was found. This represents a 146% improvement in the interfacial fracture toughness.

The Silane™ was even more impressive showing an average K_{1c}=0.193+/-0.002 MPa-$m^{1/2}$ and K_{2c}=0.006 MPa-$m^{1/2}$. This represents a 184% improvement.

These tests have shown that the ability to improve the interfacial bond of cement and cenospheres can best be achieved using the surface treatment Silane™ and the admixture silica fume.

It is easy to understand why the silica fume works so well. Silica fume is 100 times smaller than cement, which gives it a microfiller effect. The silica fume particles are easily introduced between the cement grains. This is depicted in Figure 22. This effect reduces the space available for water and acts as a nucleation site for hydration products. There is also a pozzolanic effect. The particles are amorphous silica (+85% SiO_2) with an extremely high surface area. This reacts chemically with calcium hydroxide found in cement and forms calcium silicate hydrates or CSH. Increased CSH leads to higher strength.

It is not so easy to understand why Silane™ works. It is possible that Silane™ decreases surface wetting decreasing the formation of calcium hydroxide, which weakens the interface.

These materials needed to be tested under real working conditions. It was decided to do small batch compression tests before committing to large-scale batches.

Small Batch Compression Tests

All specimens were 50% cenosphere and 50% cement by unit volume, except when silica fume was added, and prepared according to ASTM C192-95. When silica fume was added it replaced 12% of the cement as was done in previous batches. The w/c was kept at 0.44 to be consistent. The results of these experiments can be seen in Figure 23. As was the case with previous observations, the addition of cenospheres decreased the compressive strength when compared to the control concrete from 21.14 MPa to 13.96 MPa. About 45% of this strength is regained with the addition of the Silane™ cenospheres. Even more encouraging is that more than 100% of the initial strength is regained with the addition of silica fume with an average compressive strength of 21.59 MPa. Although the combination of these two interface modifiers did not show cumulative results with an average strength of 15.47 MPa, which is lower than Silane™ alone.

These tests showed that although Silane™ shows promise as an interface modifier, more study would be needed to properly apply the procedure to real world situations. This along with a higher unit cost and difficulty in making large batches, it was determined not to attempt a large batch of Silane™ cenosphere concrete at this time.

On the other hand, the results of the silica fume in both the interface experiments and the small batch experiments along with the low cost and ease of mixing have led to the decision to pursue the making of a large batch of silica fume/cenosphere concrete and compare the results to previous concrete with the same amount of cenospheres.

PROPERTIES OF IMPROVED CONCRETE

The mix design of the final batch was similar to concrete B4, although 12% of the cement by weight was replaced with silica fume. The final mix design can be seen in Table 2. It was also necessary to increase the water content by 10 lbs. to allow for the higher volume of cementing materials. This batch was designated B4SF and was mixed and cured for twenty-eight days according to ASTM C192-95. This concrete was then compared to B4, which was similar to B4SF in all respects except for the addition of silica fume. This side-by-side comparison can be seen in Table 3.

- A density of 1840±48 kg/m^3 was very similar to the density of B4, which was 1810±34 kg/m^3.
- The compressive tests showed that B4SF had a specific compressive strength of 12.05±0.36 MPa/kg. This is an 80% improvement over B4.
- The tensile tests showed B4SF to have a specific tensile strength of 0.97±0.04 MPa/kg. This represents a 35% improvement over B4.
- The specific flexural toughness of B4SF was determined to be 0.20±0.02 MPa/kg. This represents a 67% improvement over B4.

- The specific fracture toughness of B4SF was determined to be 0.026±0.001 $MPa(m)^{1/2}/kg$. This was an 73% improvement over B4.

The cenosphere concrete even outperformed the control concrete in all respects except fracture toughness. This comparison can be seen in Table 4.

It was also interesting to note that not only were the properties of the cenosphere concrete improved by the addition of silica fume, but the modes of failure showed that improved interfacial properties were the key to these improvements as previously suggested. Figure 24 shows a compressive and tensile specimen after failure. The compressive specimen shows mostly shear failure and the tensile specimen shows very little pop out of the coarse aggregate. This reduction in aggregate pop out was also seen in both the flexural and fracture specimens after failure.

CONCLUSION

A study has been conducted in which a new lightweight concrete using ceramic microspheres, called cenospheres, was investigated. The viability of this type of concrete was determined, after which all the mechanical properties were characterized using various volume fractions of cenospheres. The properties of the concrete were then improved using interfacial modifiers.

After the first part of this study, in which this type of lightweight concrete was deemed viable, the mechanical properties were determined with different levels of success. The concrete exhibited acceptable levels of strength in all experiments for all volume fractions of cenospheres. However, there was a trend of decreasing strength with higher volume fractions of cenospheres. This loss of strength was determined to be due to the poor interfacial strength properties between the cenospheres and the binder material. It was decided that in order to improve the concrete's performance, this interfacial strength needed to be improved.

The interfacial strength was quantified using bimaterial fracture toughness techniques. This allowed the interfacial fracture toughness between the cenosphere and the cement to be quantified, after which interfacial modifiers could be tested. These tests led to two possible candidates. The coupling agent Silane™ and the admixture silica fume. Due to ease of manufacture, cost restrictions and performance, the admixture silica fume was chosen as the prime interface modifier.

The addition of silica fume to a concrete with a high volume fraction of cenospheres yielded impressive results. Although the lowered density remained virtually unchanged, there was an 80% improvement in compressive strength, 35% improvement in tensile strength, a 67% improvement in flexural toughness and a 73% improvement in fracture toughness. This concrete even outperformed the control concrete in all areas except fracture toughness.

The manufacture of a high-performance lightweight concrete using cenospheres is possible. Furthermore, the availability of the materials used in the development of this concrete are readily available and relatively cost effective. There are no special processes necessary and the concrete can be mixed at any existing concrete producing facility.

Proposed Future Work

One of the major obstacles encountered during this study was the inability to determine an exact w/c ratio during the mixing process. This was due to the fact that the adsorption properties of the cenospheres could not be accurately determined. It would simplify the manufacture of this type of concrete if the adsorption properties of cenospheres could be accurately calculated.

Furthermore, this study focused mainly on one type of mix design. The volume fraction of fine aggregate remained unchanged throughout the study and this was deliberate since it was a comparative study. However, it would be of great interest to perform a parametric study to determine optimal levels of all admixtures used in the concrete and also to investigate how low the density can be brought down without sacrificing strength.

The durability of the concrete is also of great interest. This can be determined using the rapid freeze/thaw test as well as ponding and rapid chloride permeability.

Finally, the coupling agent Silane™ showed great promise as a surface treatment for the cenospheres. This avenue should be investigated further to determine the exact mechanism by which this material improves the concrete's properties and to find an effective way to employ this method in an easy and cost effective manner.

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Table 1. Concrete mix design for control batch B1

CEMENT (PORTLAND TYPE II)	298 kg (658 lbs.)
FINE AGGREGATE (SAND)	521 kg (1148 lbs.)
COARSE AGGREGATE (19 MM)	645 kg (1423 lbs.)
COARSE AGGREGATE (3.2 MM)	160 kg (355 lbs.)
WATER (Represents a 0.44 water cement or w/c ratio)	132 kg (290 lbs.)

Table 2. Concrete mix design for final batch B4SF

CEMENT (PORTLAND TYPE II)	262 kg (577 lbs.)
SILICA FUME	36 kg (79 lbs.)
CENOSPHERES	119 kg (262 lbs.)
COARSE AGGREGATE (19 MM)	645 kg (1423 lbs.)
COARSE AGGREGATE (3.2 MM)	160 kg (355 lbs.)
WATER (Represents a 0.44 water cement or w/c ratio)	136 kg (300 lbs.)

Table 3. Comparison of cenosphere concrete with and without silica fume

PROPERTY	CENOSPHERES ALONE (B4)	CENOSPHERES W/ SILICA FUME (B4SF)	% CHANGE
DENSITY	1810±34 kg/m^3	1840±30 kg/m^3	+2%
SPECIFIC COMPRESSIVE STRENGTH	6.69±0.32 MPa/kg	12.05±0.36 MPa/kg	+80%
SPECIFIC TENSILE STRENGTH	0.72±0.03 MPa/kg	0.97±0.04 MPa/kg	+35%
SPECIFIC FLEXURAL STRENGTH	0.12±0.01 MPa/kg	0.20±0.01 MPa/kg	+67%
SPECIFIC FRACTURE TOUGHNESS	0.015±0.001 MPa(m)$^{1/2}$/kg	0.026±0.001 MPa(m)$^{1/2}$/kg	+73%

Table 4. Comparison of cenosphere concrete to the control concrete

PROPERTY	CONTROL BATCH (B1)	CENOSPHERES W/ SILICA FUME (B4SF)	% CHANGE
DENSITY	2307±32 kg/m^3	1840±30 kg/m^3	-20%
SPECIFIC COMPRESSIVE STRENGTH	11.6±0.33 MPa/kg	12.05±0.36 MPa/kg	+4%
SPECIFIC TENSILE STRENGTH	0.93±0.09 MPa/kg	0.97±0.04 MPa/kg	+4%
SPECIFIC FLEXURAL STRENGTH	0.16±0.01 MPa/kg	0.20±0.01 MPa/kg	+25%
SPECIFIC FRACTURE TOUGHNESS	0.045±0.002 MPa(m)$^{1/2}$/kg	0.026±0.001 MPa(m)$^{1/2}$/kg	-42%

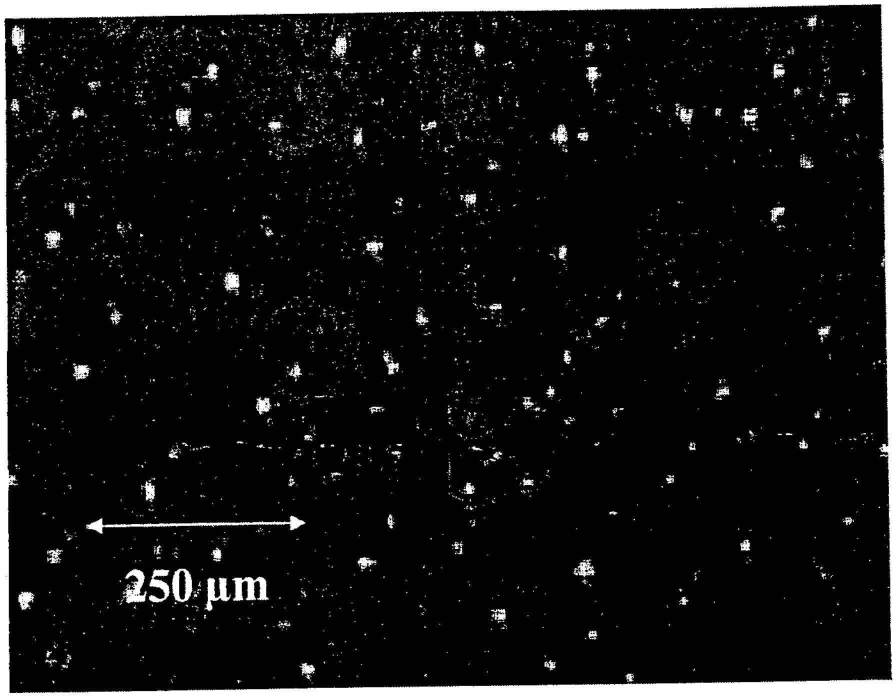


Figure 1. Light micrograph showing pure cenospheres (750x)

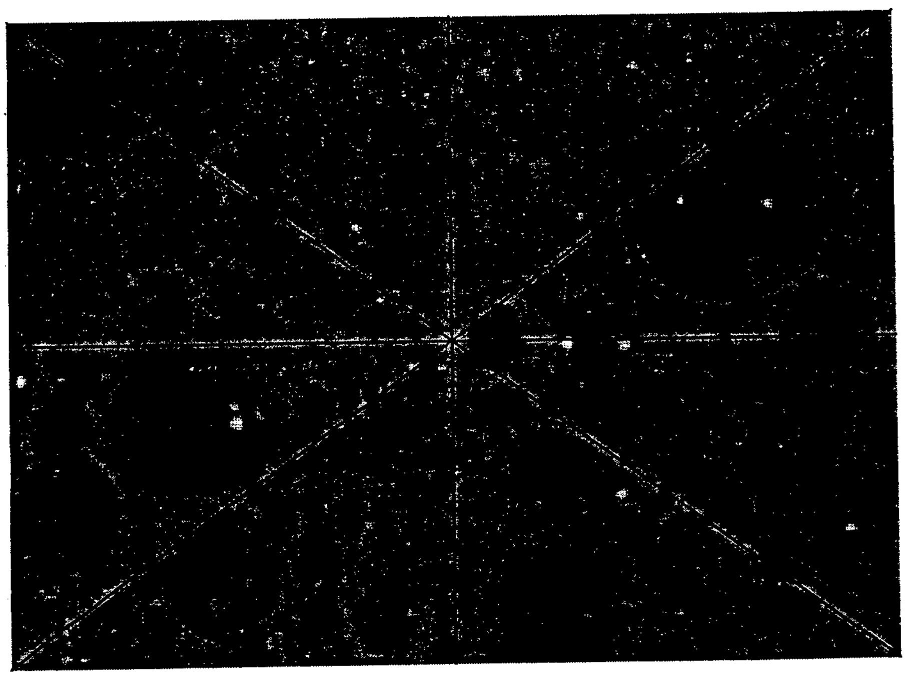


Figure 2. Light micrograph-showing cenospheres in concrete. The lines were used to determine the volume fraction of cenospheres at each point through the specimen.

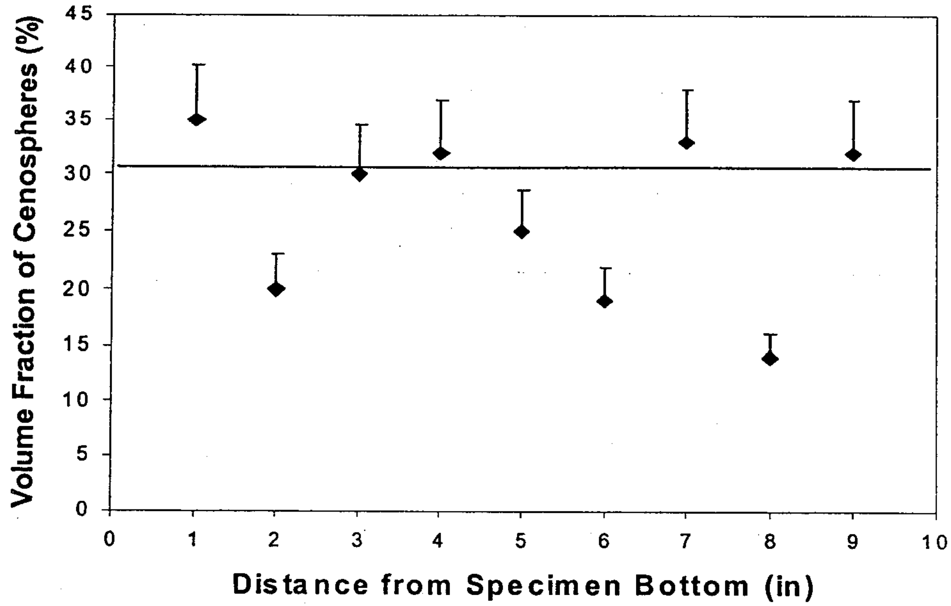


Figure 3. Volume fraction of cenospheres recorded at points throughout the concrete specimen. The line at 31% represents the volume fraction calculated using bulk densities.

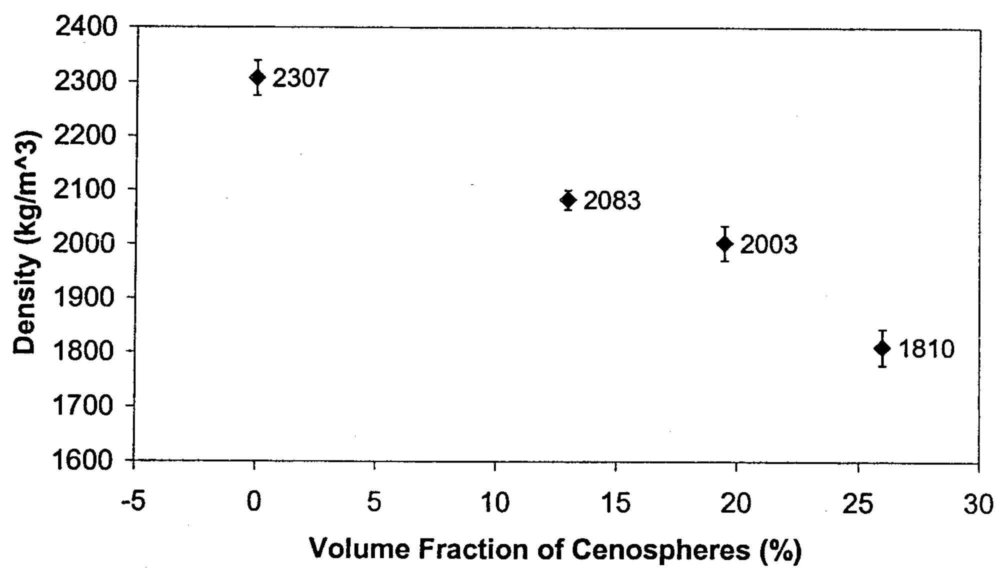


Figure 4. Density calculations for concretes with varying volume fractions of cenospheres.

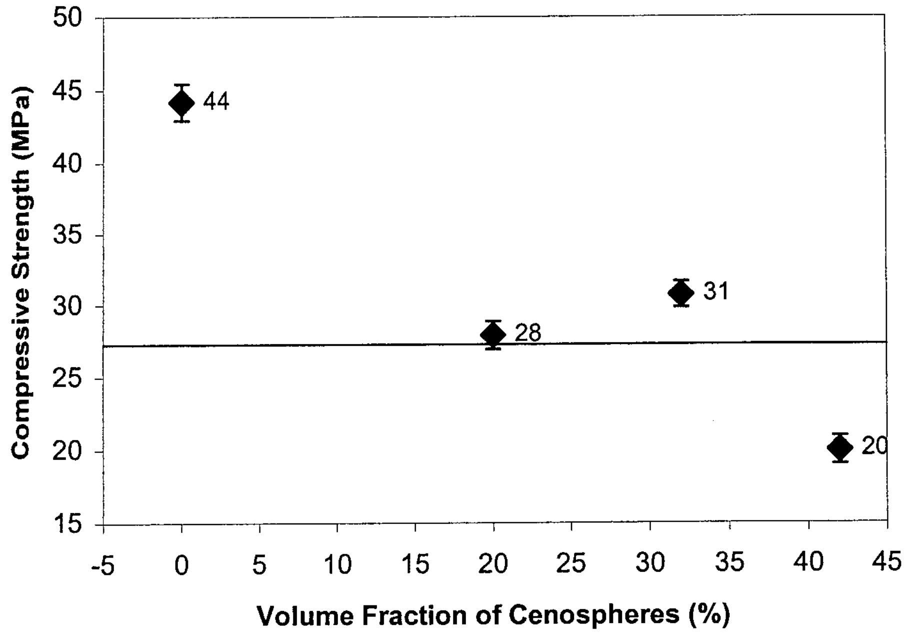


Figure 5. Compressive strength of concretes with varying volume fractions of cenospheres.

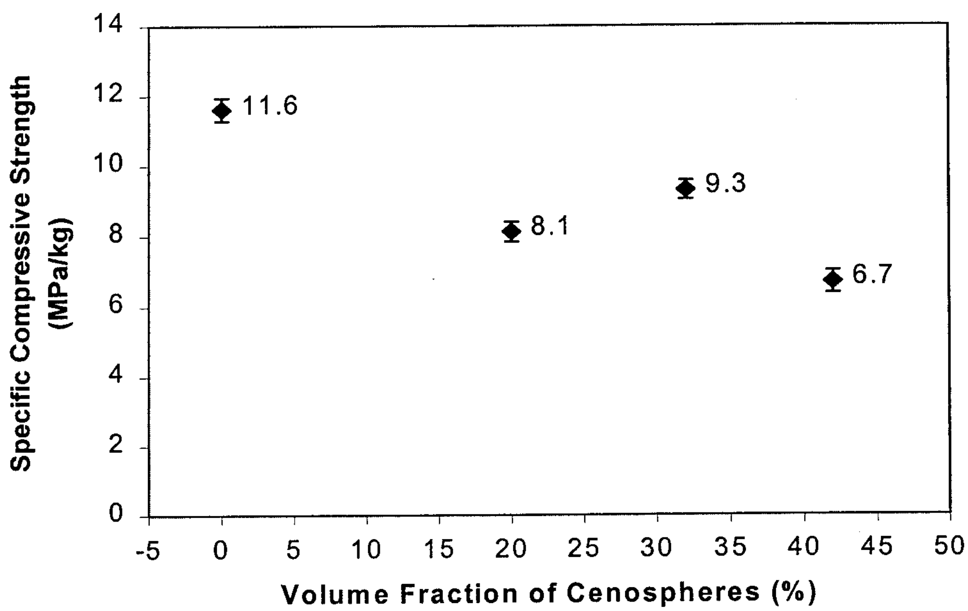


Figure 6. Specific compressive strength of cenosphere concretes.

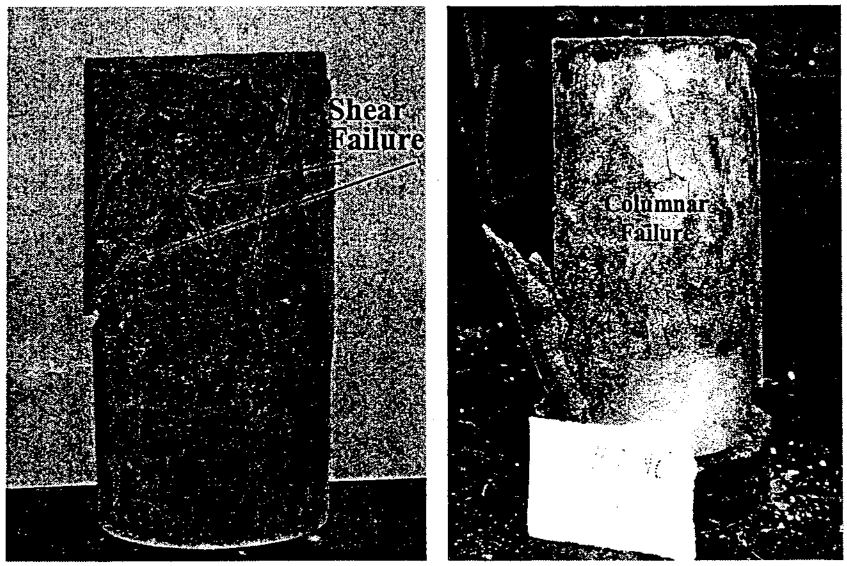


Figure 7. Normal failure mode for concrete (left) and failure mode of cenosphere concrete (right)

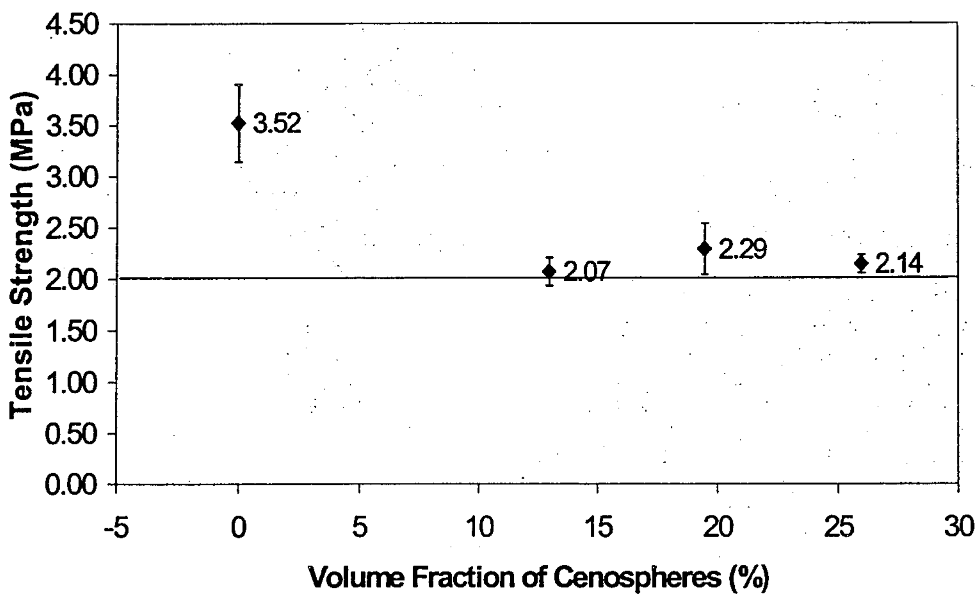


Figure 8. Tensile strength of concretes with varying volume fractions of cenospheres.

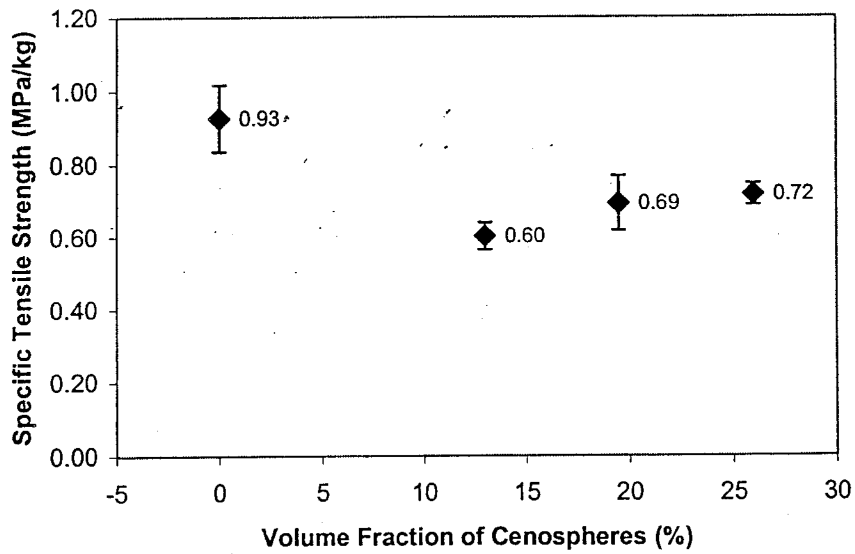


Figure 9. Specific tensile strength of cenosphere concretes.

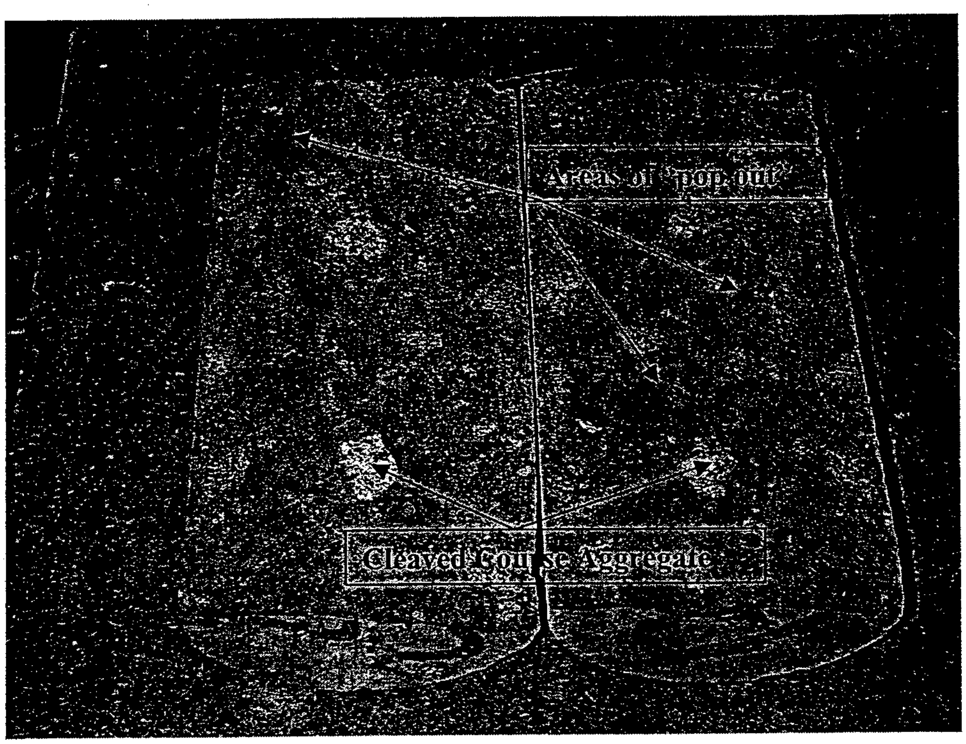


Figure 10. Tensile specimen showing both cleaved aggregate and "pop out".

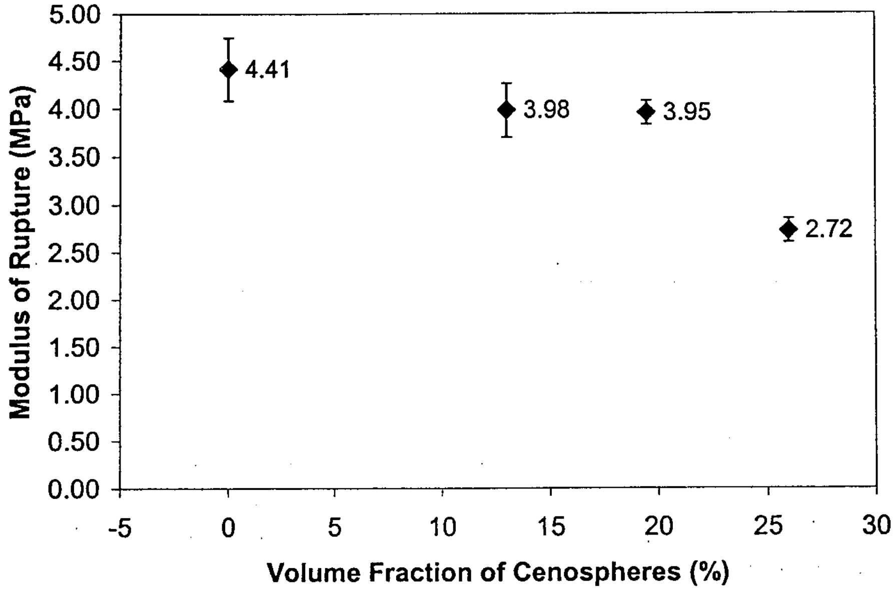


Figure 11. Flexural toughness of concretes with varying volume fractions of cenospheres.

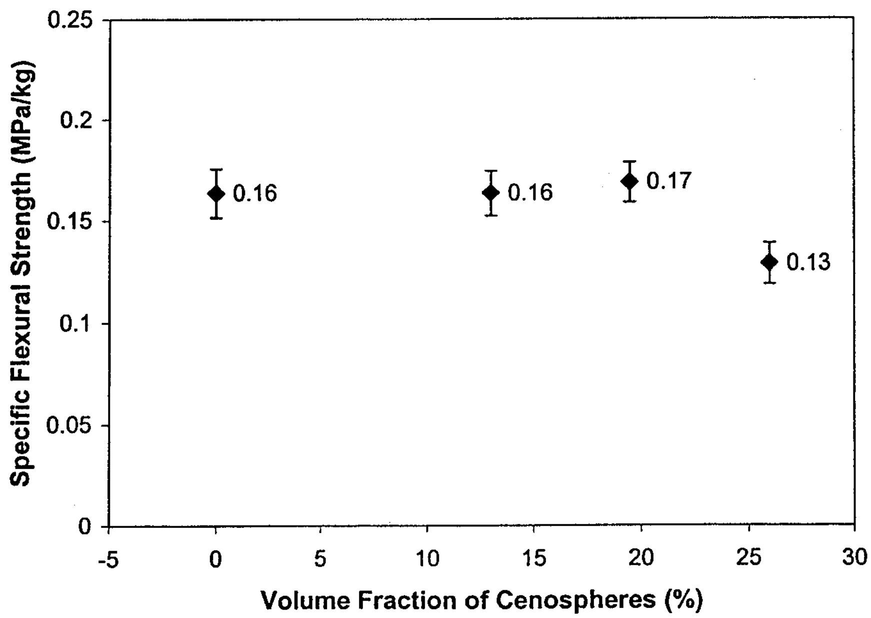


Figure 12. Specific flexural toughness of cenosphere concretes.

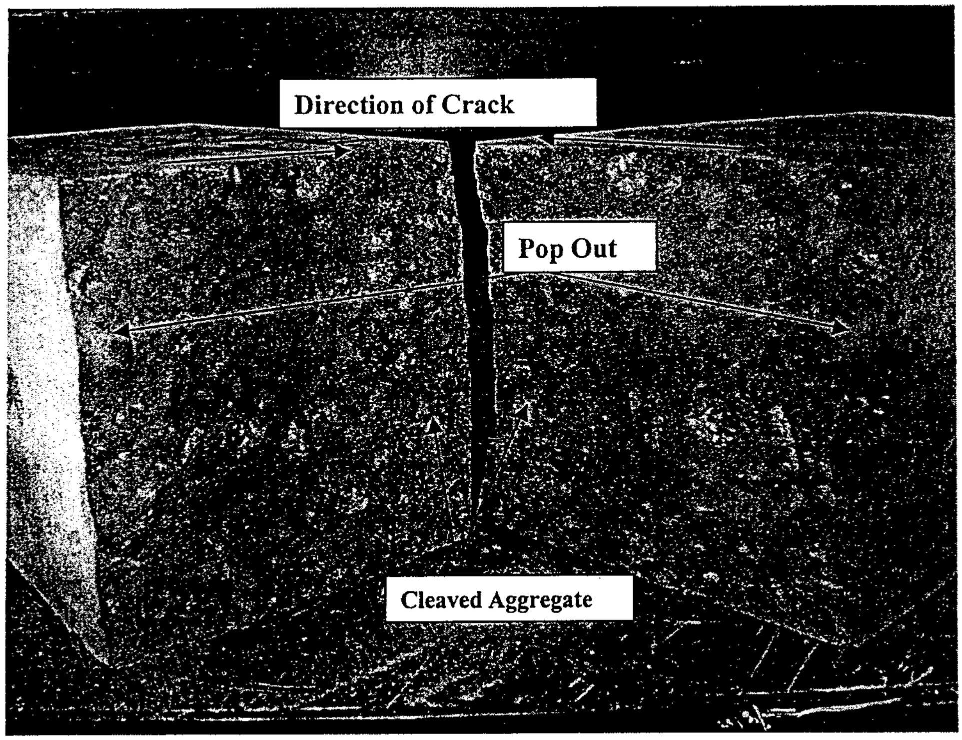


Figure 13. Flexural specimen showing cleaved aggregate and pop out.

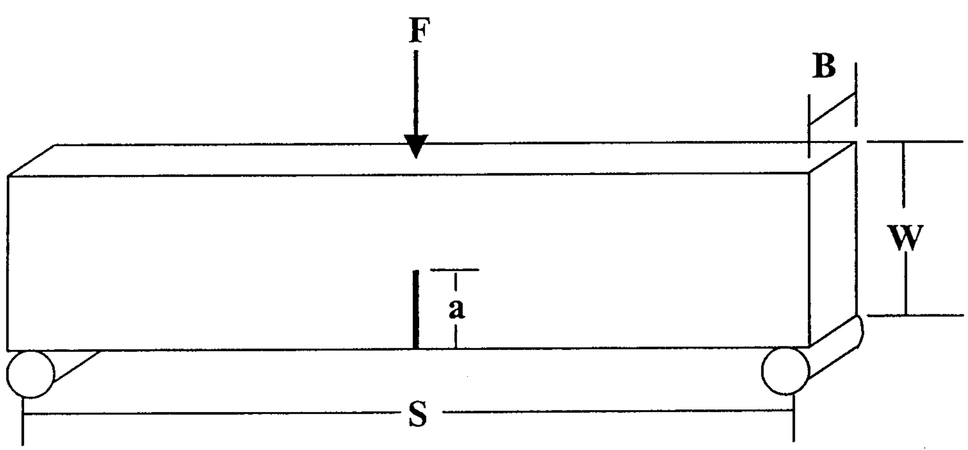


Figure 14. Fracture specimen geometry.

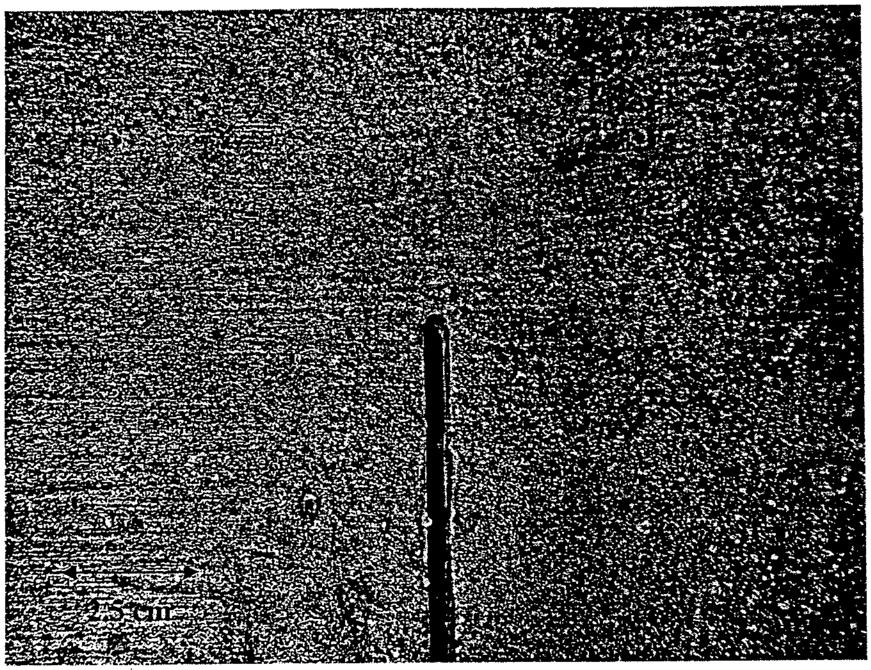


Figure 15. Geometry of crack used in fracture testing.

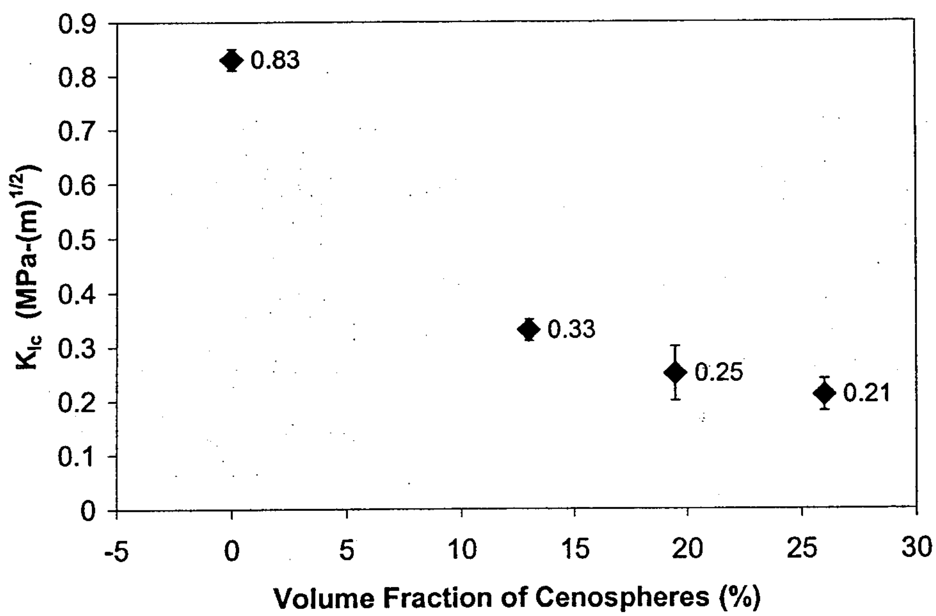


Figure 16. Fracture toughness of concretes using various volume fractions of cenospheres.

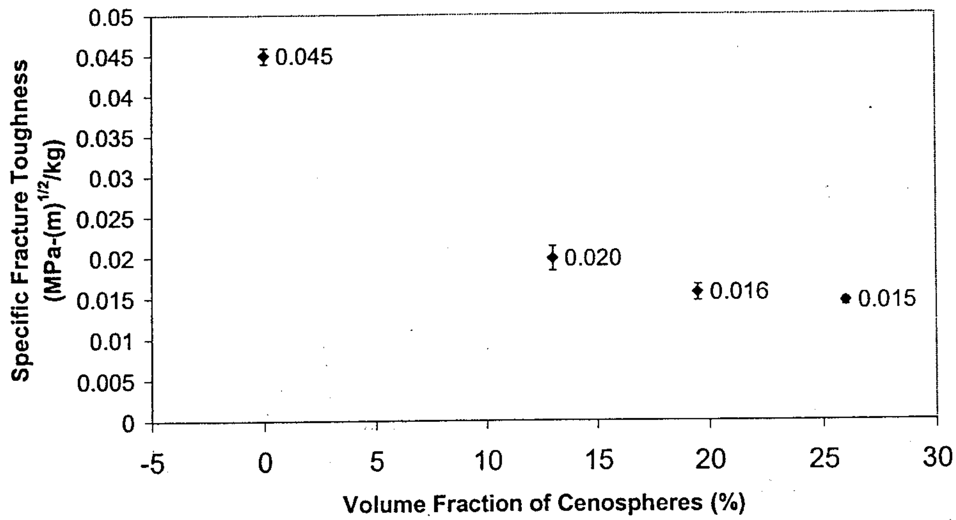


Figure 17. Specific fracture toughness of cenosphere concretes.

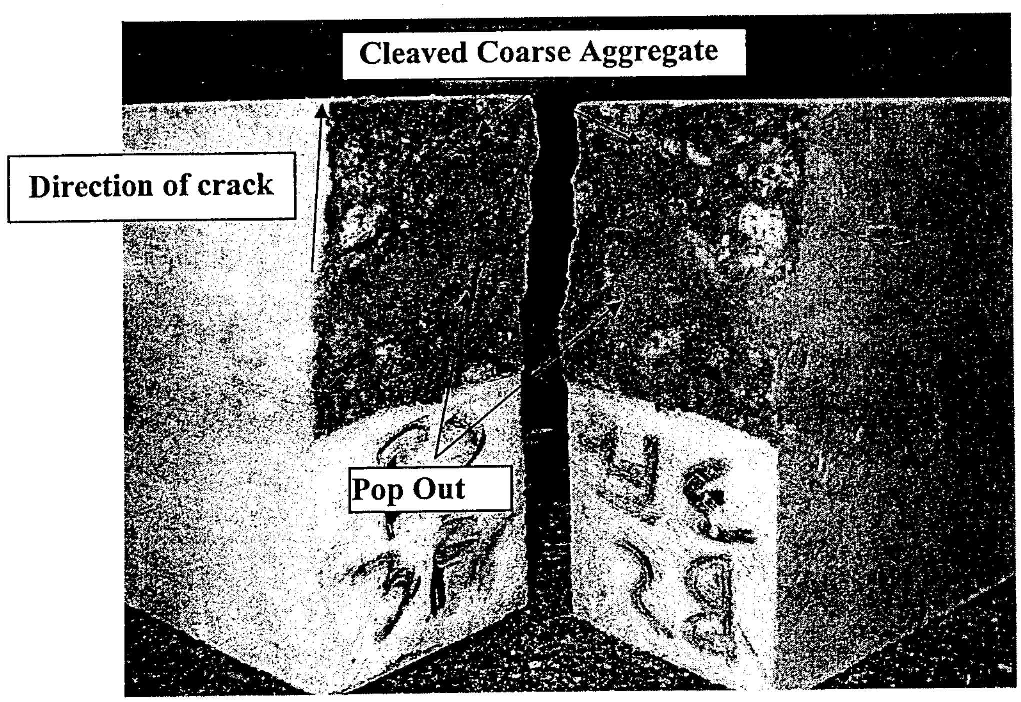


Figure 18. Fracture specimen showing cleaved aggregate and pop out.

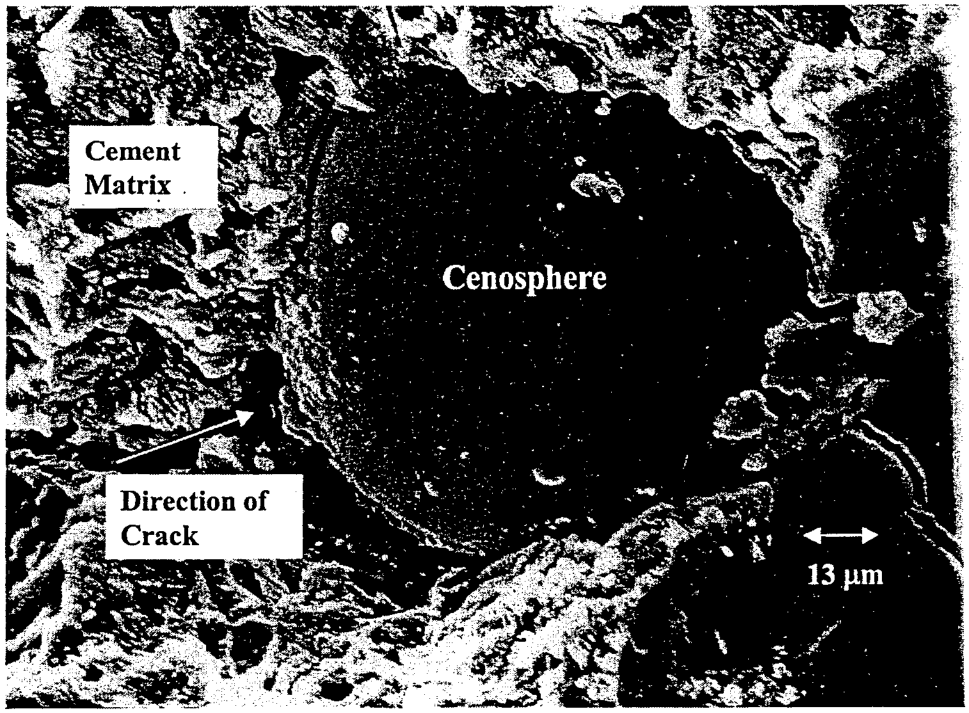


Figure 19. SEM micrograph of a cenosphere in the path of a crack.

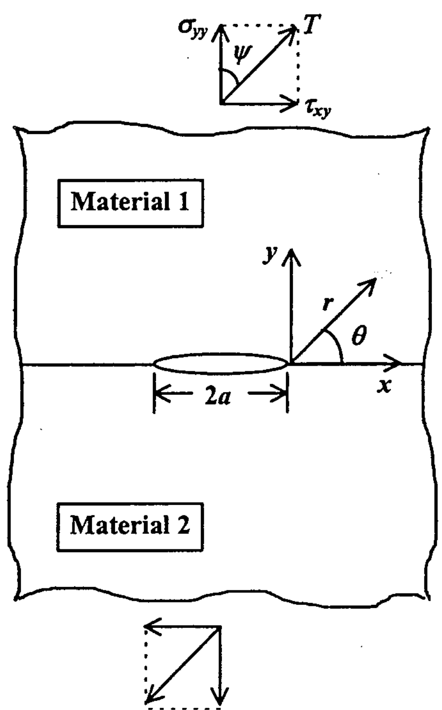


Figure 20. Geometry of a bimaterial fracture specimen.

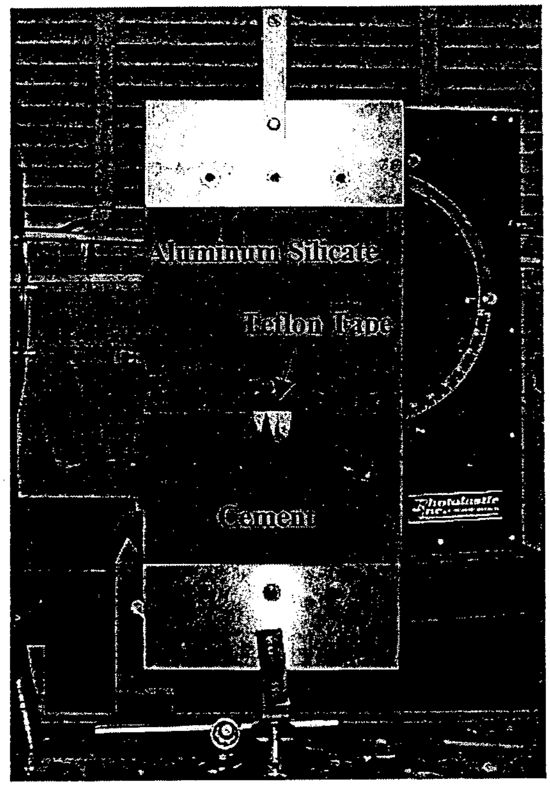
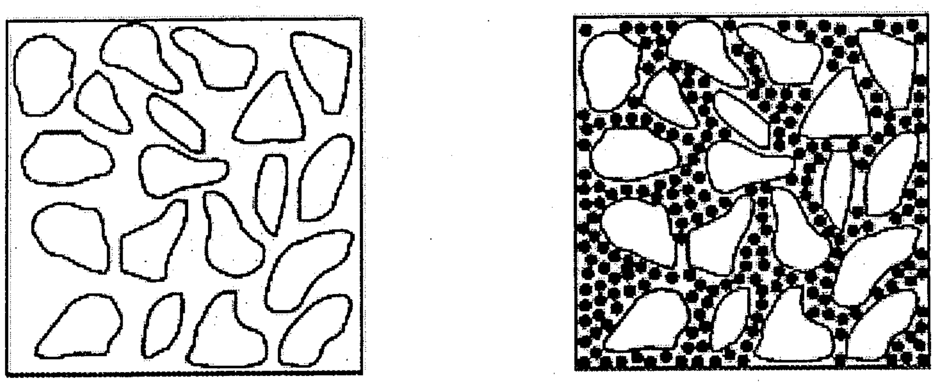


Figure 21. Experimental setup of a bimaterial fracture specimen.



i) Cement Only **ii) With Silica Fume**

Figure 22. Graphic showing the dense packing, which occurs with silica fume.

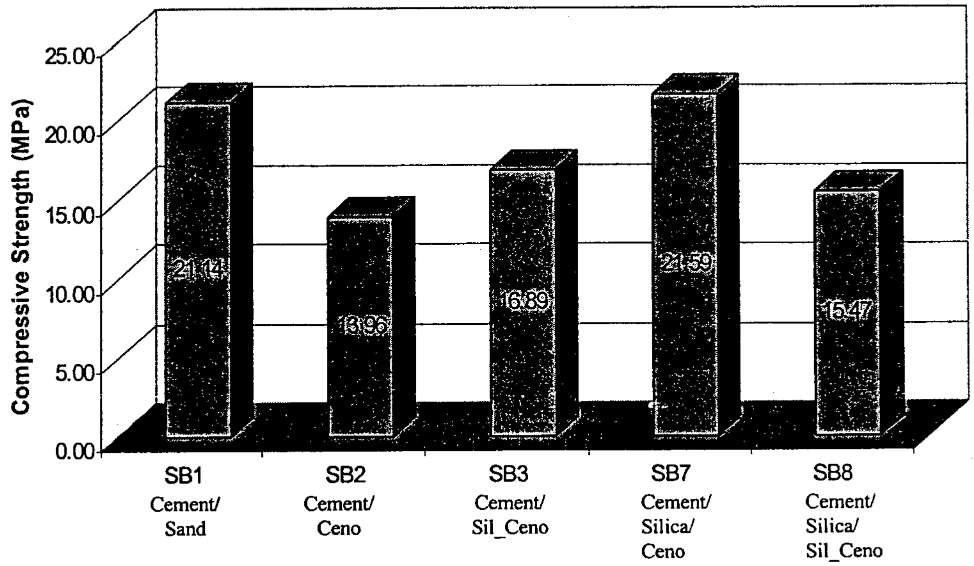


Figure 23. Results of small batch compression tests of various interface modifiers.

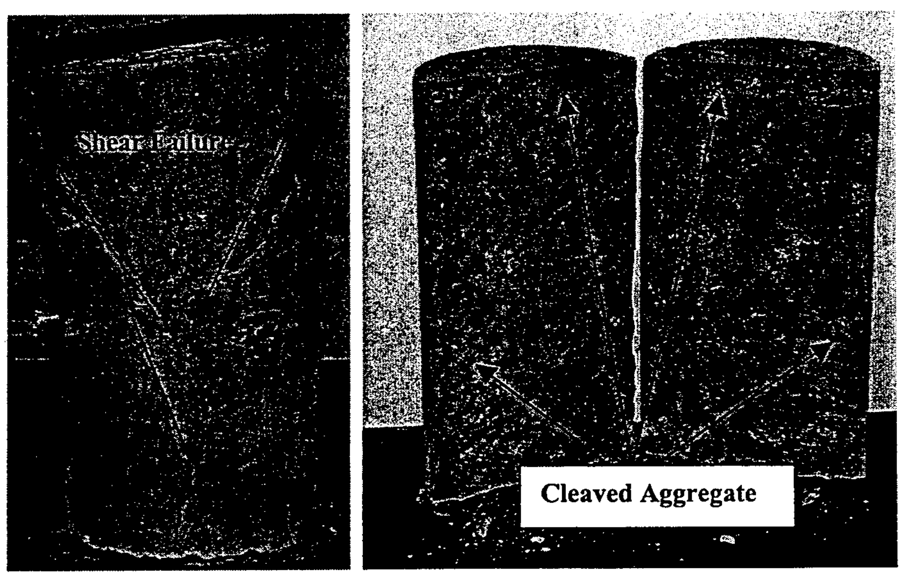


Figure 24. Failure modes of cenosphere concrete after the addition of silica fume. On the left, a compressive specimen showing shear failure, and a tensile specimen showing cleaved aggregate and no "pop out".

APPENDIX A

REVIEW OF PREVIOUS WORK

The field of lightweight concrete has been an intensive area of study for many years. It has always been of utmost importance to engineers to make the materials used in structures lighter and stronger, or at least to decrease weight without affecting strength, or increasing strength while maintaining a constant density. Naturally, it is also always a goal to decrease the cost of the material.

Concrete, which is normally a simple composite of cement, sand, stone, and water, has been modified using a wide array of aggregates, with the purpose of increasing performance. There have been many studies of concretes using organic fillers. Aziz, Murphy, and Ramaswamy (1979) performed an extensive study using cork granules in concrete. The results showed that the concrete was better suited as a filler or reinforcing material and did not perform well enough for structural applications. Slate (1976) performed a similar study using coconut fibers, and results were even less impressive. Other organic materials attempted have been rice-husk, sawdust, wood-chippings, and jute-stick particles, and, according to Aziz, Murphy and Ramaswamy, none performed as well as cork granules.

The next step is using non-organic materials to increase the performance of concrete. One of the most successful materials used to this end has been flyash. Vaillancourt (1999) shows that flyash is a by-product of the coal or heavy fuel oil burning process. It is waste a product, which makes it even more desirable as a concrete component. It is inexpensive, has good pozzalanic properties, and is less dense than cement. Naik, Singh, and Ramme (1998) have shown that high volumes of flyash in concrete not only decreases the cost and density while maintaining the performance specifications of concrete with no flyash, but can also improve mechanical properties, such as, compressive strength, tensile strength, flexural strength, and durability. Johnston and Malhotra (1987) came to similar results, but also determined specifications needed for the mixing and preparation of concretes containing high volumes of flyash, such as, water demand, amount of air-entraining admixture needed and the rate of slump loss in superplasticized concrete. Flyash has been studied extensively, and is used everyday in the concrete industry.

Silica fume is another compound, which has been studied at length and is used in concrete materials made today, although not as extensively as flyash. Tazawa, Mobuta and Ishii (1984) state that silica fume is a by-product of silicon metal and ferro-silicon manufacture. It consists of fine, spherical particles, has high silicon dioxide (SiO_2) content, and has shown superior pozzolanic properties. Tazawa, Mobuta and Ishii have also shown that concrete performance can increase as the silica fume content increases.

Another possible aggregate for concrete are cenospheres. Wandell (1996) shows that cenospheres are a lightweight by-product of flyash and are easily harvested from settling ponds due to the fact that cenospheres are less dense than water. Wandell also suggests, although no tests were performed, that cenospheres would be a good admixture for concrete since they are light and have shown good insulative properties as well as reduced shrinkage and warpage values.

Cenospheres alone have not been the subject of many studies. One of the more extensive was performed by Clayton and Back (1989), in which cenospheres were investigated in four areas. 1) Scanning electron microscopy of surface structure. 2) Photomicrography of particle cross section. 3) Measurements of porosity, surface area, and density. 4) Measurements of chemical composition. It was found that cenospheres range in size from 20-200 μm, are spheroidal and hollow and have a true density of 2.41 g/cm^3. Clayton and Back also give the complete chemical composition of the cenospheres studied.

Montgomery and Diamond (1984) investigated the influence of flyash cenospheres in cement pastes. The study showed that cracking in the cement generally went around the cenosphere with very little cleaving through the cenosphere. The conclusion made was that cenospheres act as an energy dissipating inclusions and do not necessarily weaken the system. Montgomery and Diamond also showed that there is very little bonding between the cenosphere and the cement paste, although some chemical etching was seen after a 50 day curing cycle.

Xuegan, Dongxu, Xiun and Minshu (1988) have stated that the bond between cement and aggregate is the weakest link in any concrete composite and most likely point of failure. This is especially true with cenospheres due to their low pozzolanic properties. Xuegan, Dongxu, Xiun and Minshu have suggested modifying this interfacial zone with reagents to form a reactive surface. $CaCl_2$ was used as the pretreatment in order to improve the bond strength between cement paste and aggregate through a combination of physical, chemical, and mechanical interlocking. This also decreased the formation of large portlandite crystals and the density in the interfacial region. Both of these conditions are favorable to increasing the mechanical performance of concrete. Compressive strength was improved by 12-24% and flexural strength was improved by 21-24%.

APPENDIX B

FABRICATION OF CEMENT/ALUMINUM SILICATE BIMATERIAL SPECIMENS

This section discusses the fabrication of bimaterial specimens in which one material was a solid (aluminum silicate) and one material, which was to be cast from a paste (cement). Though a simple process care must be taken to eliminate any boundary effects that can arise from the casting of a material.

Construction of the mold

In order to cast the cement paste onto the aluminum silicate block, it was necessary to construct a proper mold. The aluminum silicate block used was 257 mm (10.12") wide, 257 mm (10.12") high and 20.07 mm (0.79") wide. This material was obtained from the Maryland Lava Company and is 99.7% pure aluminum silicate. The cement block needed to be the same width and thickness, but it was decided to make it 50.8 mm (2") shorter in order to facilitate the casting.

The mold was simply four pieces of polycarbonate that would perfectly match the width and thickness and height of the bimaterial specimen. These pieces were cut out and machined to acceptable tolerances with the addition of three holes evenly spaced from the center in order to form boltholes in the cement casting. .

Preparation of the specimen

The aluminum silicate block had three holes drilled through, evenly spaced from the center, exactly as was done for the cement, in order to put the gripping bolts in place. The surface on which the cement was to be cast was then sanded with fine grit sandpaper to achieve a very smooth surface. This surface was then cleaned with methanol.

Teflon tape was applied to the exact center of the aluminum silicate block at a thickness of 38.1 mm (1.5") to represent a central crack. Packaging tape was applied along the edge, which would be removed after casting to make a pure interface. Then silicon was applied around the block to reduce water leakage.

The polycarbonate pieces were then arranged around the block to form the mold. These were all clamped firmly in place and then all joints were sealed with silicon to prevent leakage. The silicon was allowed to dry for one hour.

The cement paste was prepared according to the proper recipe and water content with all needed admixtures. This paste was poured into the mold from the top in three evenly spaced lifts. After each lift was placed into the mold banging the mold rigorously consolidated it. This also achieved the goal of removing all air from the paste.

After the final lift was introduced and initially consolidated, three 9.525 mm (3/8") bolts were placed into the holes. This required that the cement in the mold be consolidated further to ensure that the cement is uniform around the bolts. These bolts are to be removed after a few hours when the cement is firm but not completely hardened. If the bolts are removed after a complete cure, they can cause considerable cracking.

The top of the mold is then sealed to reduce water evaporation and the entire structure is allowed to cure for 48 hours. The polycarbonate pieces are then carefully removed leaving the completed specimen seen in Figure B1. The loading grips are then bolted in place and the specimen is loaded into the testing machine as seen in Figure B2.

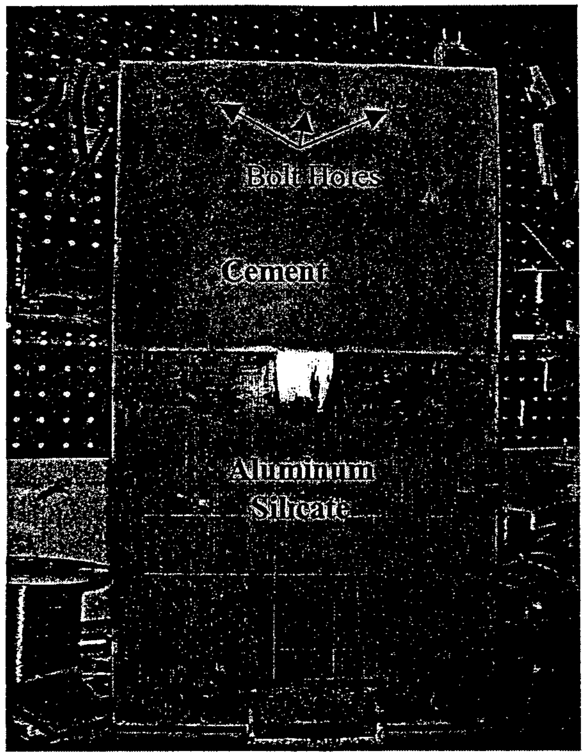


Figure B1. Bimaterial specimen after removal from mold.

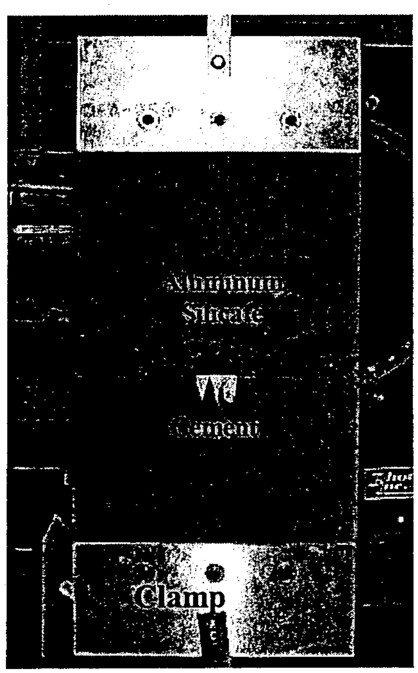


Figure B2. Bimaterial specimen ready for testing.

APPENDIX C

INDIVIDUAL MIX DESIGNS AND PLASTIC PROPERTIES

BATCH B1	
CEMENT (PORTLAND TYPE II)	299 kg (658 lbs.)
FINE AGGREGATE (SAND)	521 KG (1148 LBS.)
COARSE AGGREGATE (19 MM)	645 kg (1423 lbs.)
COARSE AGGREGATE (3.2 MM)	160 kg (355 lbs.)
WATER (Represents a 0.44 water cement or w/c ratio)	132 kg (290 lbs.)
SLUMP	157 mm (6.2 in.)
AIR CONTENT	5.80%
DENSITY	2307 kg/m^3 144 lb/ft^3

Notes: No water added

BATCH B2	
CEMENT (PORTLAND TYPE II)	299 kg (658 lbs.)
CENOSPHERES	60 kg (131 lbs.)
FINE AGGREGATE (SAND)	261 KG (574 LBS.)
COARSE AGGREGATE (19 MM)	645 kg (1423 lbs.)
COARSE AGGREGATE (3.2 MM)	160 kg (355 lbs.)
WATER (Represents a 0.44 water cement or w/c ratio)	132 kg (290 lbs.)
SLUMP	165 mm (6.5 in.)
AIR CONTENT	5.00%
DENSITY	2089 kg/m^3 130 lb/ft^3

Notes: 26 lbs. Water added to wet dry cenospheres

BATCH B3	
CEMENT (PORTLAND TYPE II)	299 kg (658 lbs.)
CENOSPHERES	89 kg (196 lbs.)
FINE AGGREGATE (SAND)	130 KG (287 LBS.)
COARSE AGGREGATE (19 MM)	645 kg (1423 lbs.)
COARSE AGGREGATE (3.2 MM)	160 kg (355 lbs.)
WATER (Represents a 0.44 water cement or w/c ratio)	132 kg (290 lbs.)
SLUMP	155 mm (6.1 in.)
AIR CONTENT	6.80%
DENSITY	2003 kg/m^3 125 lb/ft^3

Notes: 40 lbs. Water added to wet dry cenospheres

BATCH B4	
CEMENT (PORTLAND TYPE II)	299 kg (658 lbs.)
CENOSPHERES	119 kg (262 lbs.)
COARSE AGGREGATE (19 MM)	645 kg (1423 lbs.)
COARSE AGGREGATE (3.2 MM)	160 kg (355 lbs.)
WATER (Represents a 0.44 water cement or w/c ratio)	132 kg (290 lbs.)
SLUMP	163 mm (6.4 in.)
AIR CONTENT	5.00%
DENSITY	1810 kg/m^3 112 lb/ft^3

Notes: 60 lbs. Water added to wet dry cenospheres

BATCH B4SF	
CEMENT (PORTLAND TYPE II)	299 kg (658 lbs.)
CENOSPHERES	67 kg (148 lbs.)
SILICA FUME	36 KG (79 LBS.)
COARSE AGGREGATE (19 MM)	645 kg (1423 lbs.)
COARSE AGGREGATE (3.2 MM)	160 kg (355 lbs.)
WATER (Represents a 0.44 water cement or w/c ratio)	132 kg (290 lbs.)
SLUMP	147 mm (5.8 in.)
AIR CONTENT	4.80%
DENSITY	1840 kg/m^3 114 lb/ft^3

Notes: 60 lbs. Water added to wet dry cenospheres
10 lbs. Water added to allow for silica fume

APPENDIX D

CONCRETE COMPONENT PROPERTIES

FIORE CONCRETE 1999			
Aggregate Size	3/4	3/8	Fine
Bulk Specific Gravity	2.658	2.663	2.585
Apparent Specific Gravity	2.736	2.740	2.635
Absorption	1.08	1.05	0.72

FIORE CONCRETE 2000			
Aggregate Size	3/4	3/8	Fine
Bulk Specific Gravity	2.688	2.700	2.598
Apparent Specific Gravity	2.741	2.755	2.636
Absorption	0.71	0.74	0.56

CARDI CORP 2000			
Aggregate Size	3/4	3/8	Fine
Bulk Specific Gravity	2.653	2.667	2.589
Apparent Specific Gravity	2.700	2.735	2.624
Absorption	0.65	0.93	0.52

AWARDS AND PATENTS

This study was presented at the Society of Experimental Mechanics IX International Congress in Orlando. It was entered in the Student Paper Competition on June 6, 2000 by Shawn McBride and placed third among all papers presented.

The Board of Governors for Higher Education, State of Rhode Island and Providence Plantations has also decided to pursue two patents entitled "Lightweight Cenosphere Concrete Using Silica Fume" and "Lightweight Concrete Using Silane Treated Cenospheres".

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