Using Cenospheres to Develop New Asphalt and Cement Based Concrete Materials

by

Dr. Arun Shukla
Dynamic Photomechanics Laboratory
Department of Mechanical Engineering and Applied Mechanics

Dr. Arijit Bose
Department of Chemical Engineering

And

Vikrant Tiwari and Shawn Patrick McBride
Dynamic Photomechanics Laboratory
Department of Mechanical Engineering and Applied Mechanics

University of Rhode Island
Kingston, RI 02881, USA

August 2001

URI-TC Project No. 536110

Prepared for

University Of Rhode Island
Transportation Research Center

1. Report No URITC FY99-10	2. Government Accession No. N/A	3. Recipient's Catalog No. N/A
4. Title and Subtitle Using Cenospheres to Develop New Asphalt and Cement Based Concrete Materials		5. Report Date November 2001 6. Performing Organization Code
7. Authors(s) Dr. Arun Shukla, Dr. Arijit Bose, Vikrant Tiwari, Shawn Patric McBride		8. Performing Organization Report No.
9. Performing Organization Name and Address University of Rhode Island Mechanical Engineering & Applied Mechanics Department 92 Upper College Road Kingston, RI 02881		10. Work Unit No. (TRAIS) N/A 11. Contract or Grant No. 536110 13. Type of Report and Period Covered Final
12. Sponsoring Agency Name and Address University of Rhode Island Transportation Research Center Kingston, RI 02881		14. Sponsoring Agency Code URITC 99-10 A study conducted in cooperation with the USDOT

15. Supplementary Notes

16. Abstract

This report describes first year's research activities of a three year proposed project dealing with the use of cenospheres to develop new asphalt and cement based concrete materials. The project's primary aim is to study and experimentally determine the acoustic and mechanical properties of the newly developed lightweight concrete and asphalt materials. The acoustic behavior of different grades of cenospheres rich concrete is being studied and investigated experimentally. Properties such as absorption coefficient, reflection coefficient and acoustic impedance and their dependence on frequency are being measured. The effect of cenospheres on the acoustic properties of the above mentioned materials is under observation and work is in progress to bring out the complete acoustic frequency response characteristics of cenospheres rich concrete. A study has also been conducted in which a lightweight concrete was processed using ceramic microballoons (cenospheres) as a primary aggregate. The mechanical properties, including compressive strength, tensile strength, flexural strength and fracture toughness, were tested. It was determined that the addition of high volumes of cenospheres significantly lowered the density of concrete and was also responsible for some strength loss. This strength loss was recovered by improving the interfacial strength between the cenospheres and the cement producing a high-performance lightweight concrete. Tests were also conducted on cenosphere rich asphalt concrete to study the effect of cenospheres on the strength of the asphalt concrete. Cenospheres were also coated by two different polymers namely Styrene-Acrylonitrile and Styrene-Butadiene and their effect on the strength of cenosphere rich Asphalt concrete was studied.A large part of first year effort was focused on setting up equipments and establishing the testing procedure. This will help in research efforts in the second and third year of the project.

17. Key Words Lightweight concrete, Asphalt, Fracture toughness, Flexural strength, Cenospheres, Sound Absorption, Absorption coefficient, Reflection Coefficient, Acoustic Impedance		18. Distribution Statement No restrictions. This document is available to the public through the University of Rhode Island, Transportation center, 85 Bnar Lane, Kingston, RI 02881.	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 47	22. Price N/A

Form DOT F 1700.7 (8-72) Reproduction of completed page authorized (art. 5/94)

TABLE OF CONTENTS

LIST OF FIGURES.

ABSTRACT

This report describes first year's research activities of a three year proposed project dealing with the use of cenospheres to develop new asphalt and cement based concrete materials. The project's primary aim is to study and experimentally determine the acoustic and mechanical properties of the newly developed lightweight concrete and asphalt materials.

The acoustic behavior of different grades of cenospheres rich concrete is being studied and investigated experimentally. Properties such as absorption coefficient, reflection coefficient and acoustic impedance and their dependence on frequency are being measured. The effect of cenospheres on the acoustic properties of the above mentioned materials is under observation and work is in progress to bring out the complete acoustic frequency response characteristics of cenospheres rich concrete.

A study has also been conducted in which a lightweight concrete was processed using ceramic microballoons (cenospheres) as a primary aggregate. The mechanical properties, including compressive strength, tensile strength, flexural strength and fracture toughness, were tested. It was determined that the addition of high volumes of cenospheres significantly lowered the density of concrete and was also responsible for some strength loss. This strength loss was recovered by improving the interfacial strength between the cenospheres and the cement producing a high-performance lightweight concrete.

Tests were also conducted on cenosphere rich asphalt concrete to study the effect of cenospheres on the strength of the asphalt concrete. Cenospheres were also coated by

two different polymers namely Styrene-Acrylonitrile and Styrene-Butadiene and their effect on the strength of cenosphere rich asphalt concrete was studied.

A large part of first year effort was focused on setting up equipments and establishing the testing procedure. This will help in research efforts in the second and third year of the project.

CHAPTER 1

ACOUSTIC PROPERTIES OF PARTICLE REINFORCED CEMENT, CONCRETE AND ASPHALT

1.1 ABSTRACT

The acoustic behavior of different grades of cenosphere-rich concrete has been investigated experimentally. Properties such as absorption coefficient, reflection coefficient and acoustic impedance, and their dependence on frequency have been measured. The effect of cenospheres on the acoustic properties is under observation and work is in progress to bring out the complete acoustic frequency response characteristic of cenosphere-rich concrete.

1.2 INTRODUCTION

Absorption characteristics of different materials have been studied for a long time. Cenosphere (ceramic microballoons)-rich concrete has been recently tested in the Mechanical Engineering Department of University of Rhode Island. It has shown promising results, from a mechanical perspective. Here we study acoustic properties of this cenosphere filled material to determine if it can be used as sound barrier near highways and other roadways. Its effect on sound absorption properties of asphalt is also of interest.

As it is standard for acoustic characterization, properties such as absorption coefficient, reflection coefficient and acoustic impedance of the samples are determined and their variation with frequency is studied.

1.3 REVIEW OF PREVIOUS WORK

A number of techniques have been employed to study the acoustic characteristics of different materials. They include the standing wave method, transfer function method [1], one microphone technique and the protruding tube method [2]. These techniques are compared with each other [3] and found to give matching results. Related work on sound absorption mechanisms of porous asphalt pavement was done by Michiyuki Yamaguchi, Takuya [4], Ishida, M., Meiarshi, [5] and Watts [6][7]. Michiyuki Yamaguchi and Takuya studied absorption properties of asphalt as a function of density, pore size and thickness for different frequencies. Ishida and Meiarshi analyzed the relationship between noise reduction and the thickness and aggregate size of the drainage asphalt road surface. Watt studied porous asphalt surface and noise barriers. He used the Boundary Element Method (BEM) approach to determine the extent to which the noise reducing benefits of asphalt could be added to the screening effects of noise barriers in order to obtain overall reduction in noise levels.

1.4 THEORETICAL CONSIDERATIONS

The sound absorbing performance of a material is defined by its sound absorption coefficient α, the ratio of the unreflected sound intensity at the surface to the incident sound intensity. Sound absorption coefficient varies with frequency and is also a function of material thickness, density and pore size. The unit of sound absorption is Sabins (**A**), one Sabine is one square foot of total absorption by the specimen. Other acoustic properties include the sound reflection coefficient which is a ratio of the amount of total reflected sound intensity to the total incident sound intensity, and acoustic impedance which is defined as ratio of sound pressure acting on the surface of the sample to the associated particle velocity normal to the surface.

Theory behind acoustical measurement can be explained as follows. Consider an acoustic plane wave in a standing wave tube. At a particular position in the tube, the sound pressure due to the incident wave at a particular instant of time is given by

$$p_i = A \cos 2\pi f t \qquad (1)$$

and the sound pressure due to the reflected wave at the same point at the same instant of time is given by

$$p_r = B \cos 2\pi f \left(t - \frac{2y}{c} \right) \qquad (2)$$

where

p_i , p_r are sound pressure of the incident and reflected wave in Pa

f is the frequency of excitation in Hz

y is distance of point from sample in m

c is the velocity of sound in the tube in m s^{-1}

t is time in s

A, B are amplitudes at p_i and p_r respectively.

The total sound pressure at this point, p_y will therefore be:

$$p_y = p_i + p_r = A \cos 2\pi f t + B \cos 2\pi f \left(t - \frac{2y}{c} \right) \qquad (3)$$

By applying the addition theorem i.e.

$$\text{Cos}\,(\theta - \phi) = \text{Cos}\,\theta\ \ \text{Cos}\,\phi + \text{Sin}\,\theta\ \ \text{Sin}\,\phi \qquad (4)$$

it can be seen that the sound pressure will have a maximum value of (A+B)Cos2πft when y=λ/2 and a minimum value when y=λ/4 where λ =wavelength = c/f. A microphone situated at a distance λ/2 from the sample will receive an alternating sound pressure of frequency f and amplitude (A+B).

The absorption coefficient (α) of the sample will be then

$$a = 1 - \left(\frac{B}{A}\right)^2 \quad . \qquad (5)$$

This equation can be written as

$$\alpha = 1 - r^2 \qquad (6)$$

Using the standing wave apparatus, we can measure the ratio, n, of the maximum to minimum sound pressure in the tube, that is the standing wave ratio:

$$n = \frac{P_{MAX}}{P_{MIN}} \qquad (8)$$

Therefore $$n = \frac{A+B}{A-B} \qquad (9)$$

An analogy can be drawn between acoustic standing wave ratio and the standing wave ratio measured in electromagnetic wave guides. Hence

$$\frac{B}{A} = \frac{n-1}{n+1} \qquad (10)$$

Therefore absorption coefficient can be expressed in terms of the standing wave ratio by substituting eq(10) in eq(5) yielding:

$$a = 1 - \left(\frac{n-1}{n+1} \right)^2 \qquad (11)$$

1.5 EXPERIMENTAL PROCEDURE

The experimental setup for doing the acoustic studies has been installed as shown in Figure 1.1. It consists of a B&K standing wave apparatus (Type 4002) with crystal type microphone (25 mv/Pa, 2nF), frequency generator (Bendix advance technology center 343), four channel phosphor oscilloscope (Tektronix TDS 3014) and low noise preamplifier with band pass filter (Model SR560, Stanford research systems). The experimental setup is calibrated by conducting experiments on standard test samples. Comparison of experimental and theoretical values for an aluminum sample can be seen in Figure1.2 . Small difference can be accounted by sound energy dissipation as sound travels along the tube length and the acoustic impedance of air.

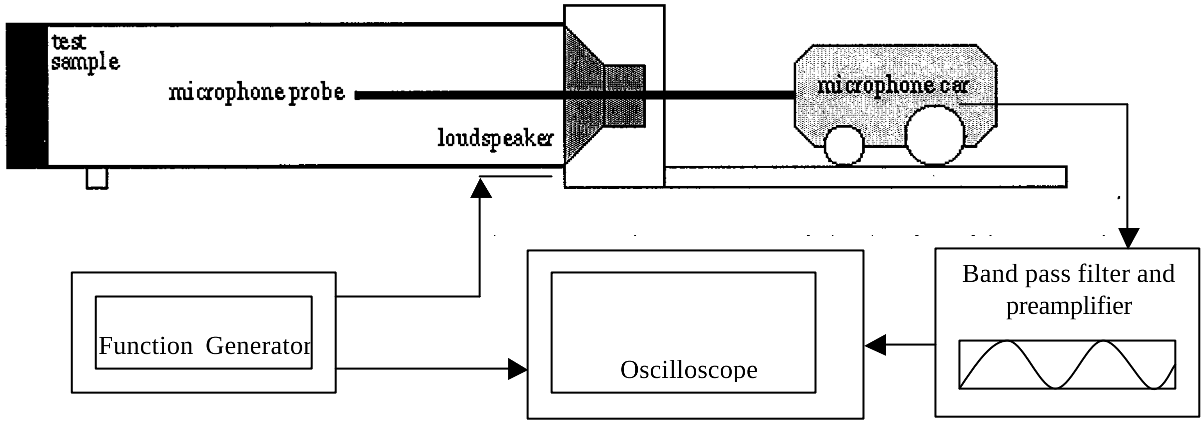

Figure 1.1 Experimental setup for acoustic studies.

In the experimental procedure the test specimen is placed in a specimen holder, fitted at one end of the impedance tube. A loudspeaker capable of generating the desired frequency is placed at the other end of the tube. A microphone is positioned in such a way that it can be moved longitudinally along the tube but remains coaxial with the tube. When the test specimen is placed in the holder the sound levels at the point of maximum and minimum sound intensity can be measured and the standing wave ratio can be calculated.

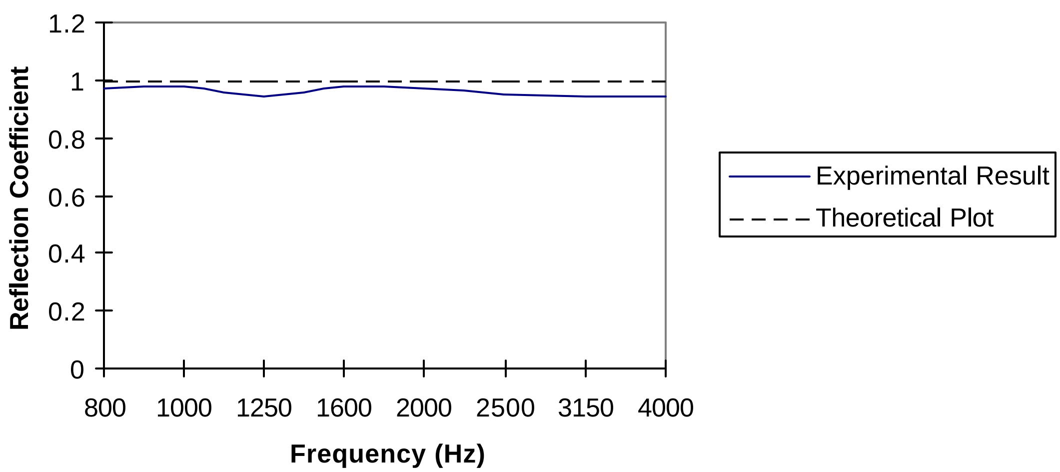

Figure 1.2 Comparing experimental results with theoretical values of Aluminum sample for calibration.

Amplifier and filters are used to clear the noise and record test signals. Using ASTM C384 [8] standards, the absorption coefficient, reflection coefficient and acoustic impedance at any frequency can be determined for the specimen. Specimens of different thickness and size have been examined for the frequency range between 120Hz to 4000Hz, range where human ear is most sensitive to sound.

1.6 SPECIMEN PREPARATION

Specimens for conducting experiments have been made in the Dynamic Photomechanics Laboratory in the Mechanical Engineering Department and the Transportation lab in the Civil Engineering Department. Care has been taken to follow predetermined standards. About 50 specimens of cenosphere-rich cement with different percentage of cenospheres (from 0 to 70% by volume) and different sizes have been made. For each volume fraction of cenosphere six samples of different (1, 2, and 3 inch long) lengths and different (3.9 and 1.13 inch) diameters were made. Some of them can be seen in Figure1.3 . Water to cement ratio was maintained at 0.4 for all of the specimens. At higher percentage of cenospheres, wet cenospheres were used to ensure the proper workability of the resulting mixture. Apart from the specimens for standing wave apparatus, sheets of one square foot dimension with different cenosphere percentage have been made and will be used for measuring the longitudinal wave velocity for the purpose of calculating acoustic impedance (ρv).

The weight of all the samples was carefully measured to calculate their mass density and it was found that mass density decreases from 1900 kg/m^3 for zero percentage cenospheres to 1110 kg/m^3 for seventy percent cenospheres as seen in Figure1.4 .

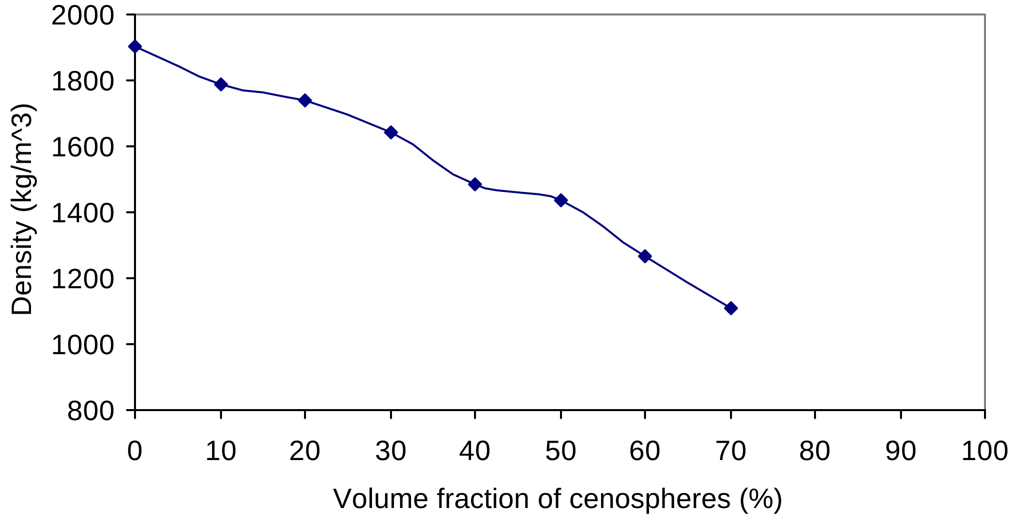

Figure 1.4 Change in mass density with percentage increase of cenospheres in cement samples.

Asphalt specimens having different cenospheres content have also been made for conducting acoustic experiments. For making the specimens surface course design was used for mixing the fine and coarse aggregate with 6% asphalt by weight. The design for the specimens came from a CAMA software program owned by the Civil Engineering Department in URI. The mineral aggregates were obtained from Cardi in four different sizes. These include both the coarse aggregate; which include the ¾", ½", and the 3/8" size denominations; and the fine aggregate, which was denominated as sand. These materials were subjected to sieve analysis, see American Society for Testing Material (ASTM) test C136 [9] or American Association of State Highways and Transportation Officials (AASHTO) test T27. Figure1.5 and Figure1.6 shows the results of sieve analysis for both fine and coarse aggregates respectively. Mix design for these samples is discussed in detail in chapter 3 of this report.

Aggregates were then subjected to specific gravity tests, see ASTM C127 [10] and C128 [11] and AASHTO T84 and T85. A comparison of the densities of the coarse and fine aggregates was thus obtained, and as expected they are all almost identical.

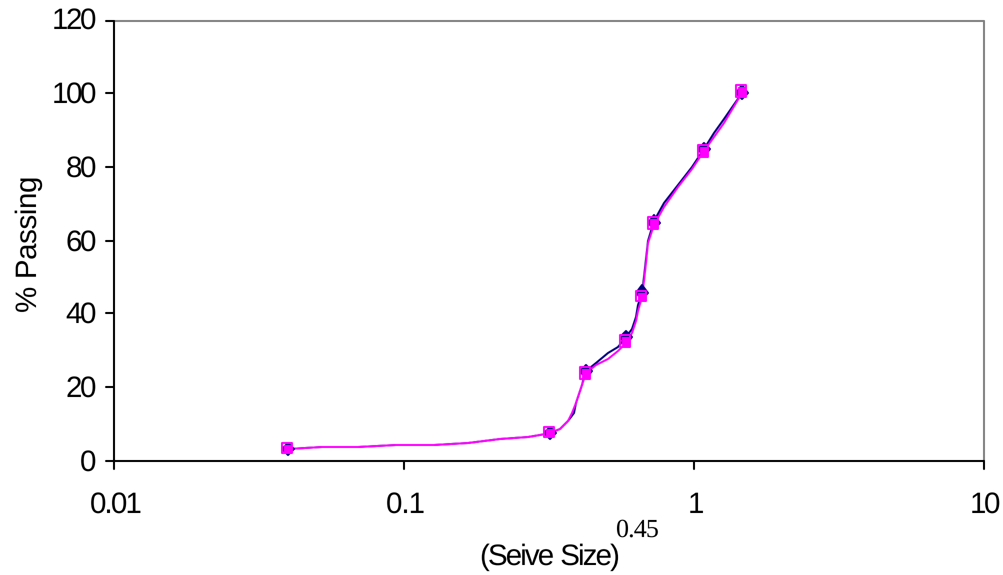

Figure 1.5 Gradation chart for Sand.

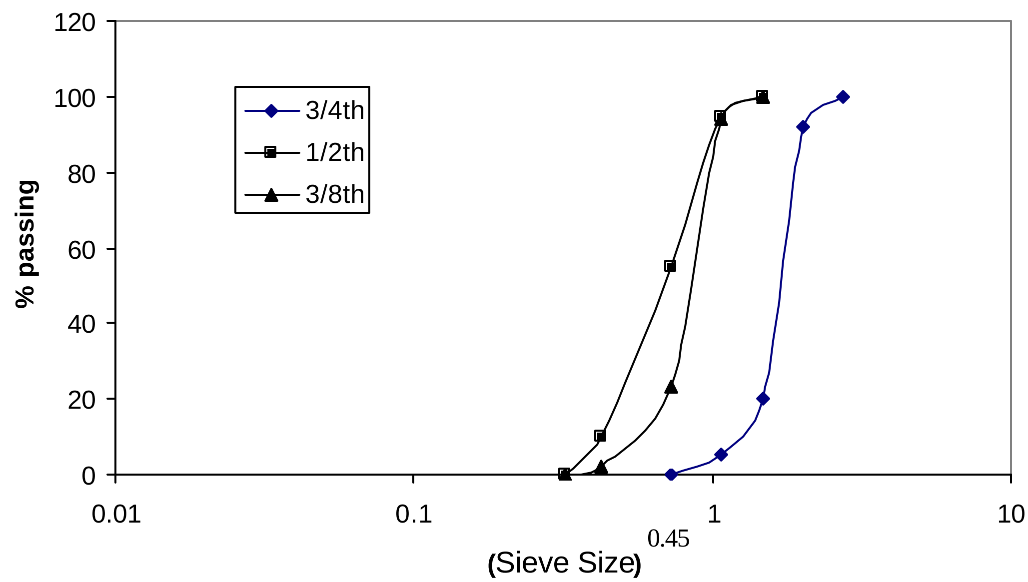

Figure 1.6 Gradation chart for Coarse aggregate.

Before making the samples aggregates were placed in an oven overnight along with molds and hammer, and the oven was set to 325°F. Approximately two hours before mixing asphalt was also added to oven. Both the aggregates and asphalt were mixed together thoroughly in metal bowl before being placed in the compacting mold. Mixture was compacted by the 50 blows on both side of the Marshall specimen. After compaction was over each sample was allowed to cool in the mold before being taken out. Weight of each sample was carefully measured to calculate the mass density of the specimens as shown in Figure1.8 .

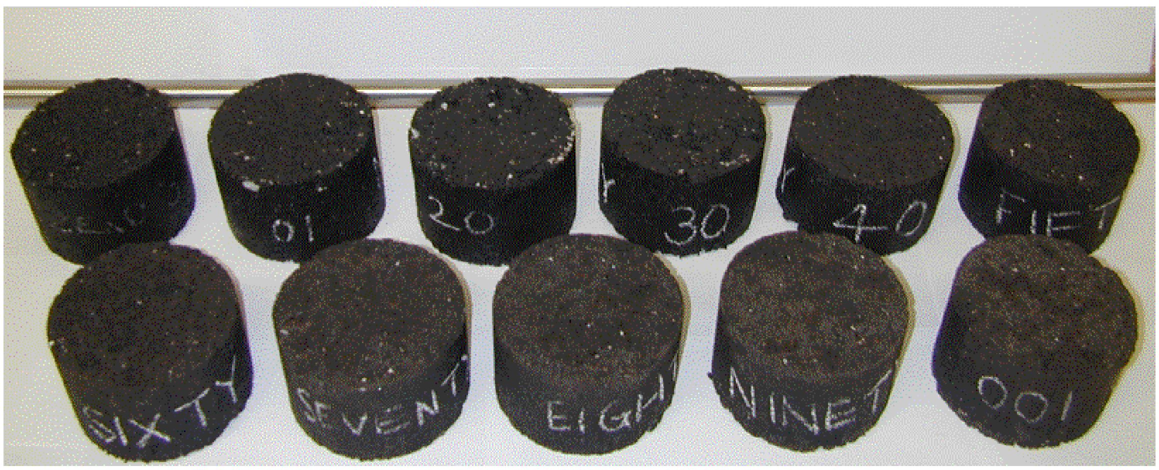

Figure 1.7 Asphalt Samples with different percentage of cenospheres.

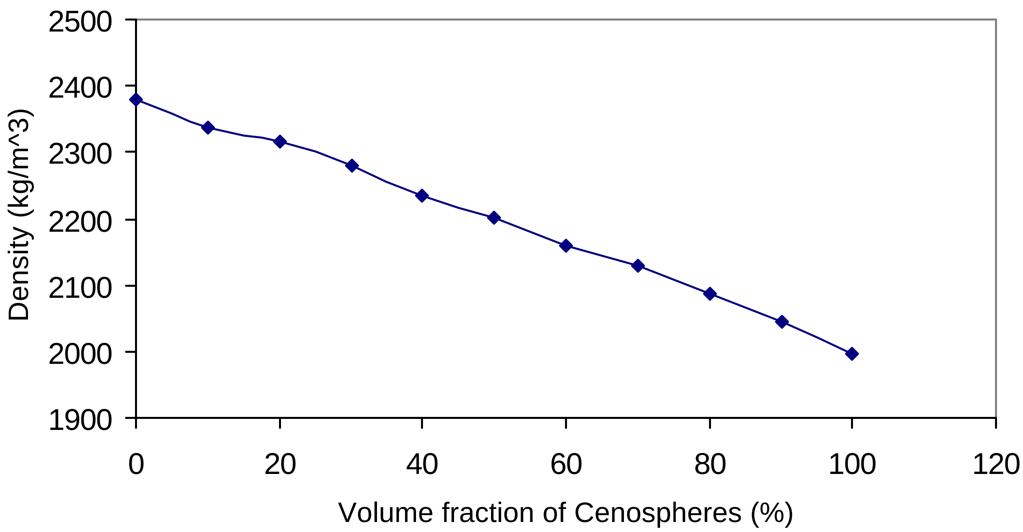

Figure 1.8 Change in mass density with percentage increase of cenospheres in Asphalt samples.

Bulk specific gravity test was performed on all the samples by measuring the saturated weights of the samples in air and water. Results (Figure1.9) show that there is not much variation in values of bulk specific gravity for specimens with different cenosphere contents.

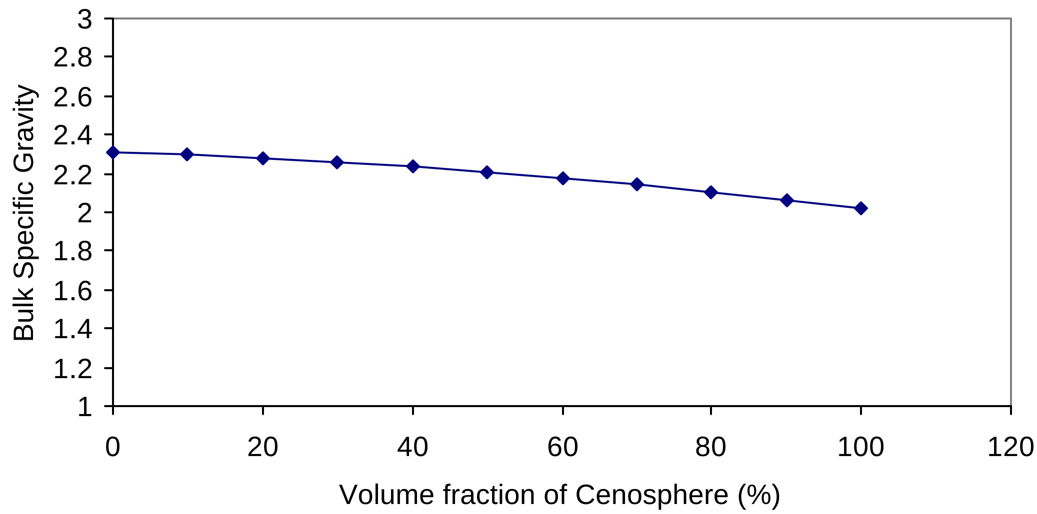

Figure 1.9 Change in Bulk Specific Gravity with percentage increase of cenospheres in Asphalt samples.

1.7 RESULTS

Results obtained from the standing wave apparatus are applicable for sound incident normally to the surface of the sample. Restrictions are placed on the use of the equipment to ensure that theoretical conditions are closely approximated during the practical operation. Currently, testing for acoustic properties of both Cement and Asphalt specimens is going on. Some of the results can be seen in Figure 1.10, 1.11 and 1.12 . Each experiment has been performed five times to check the repeatability and to ensure the correct results.

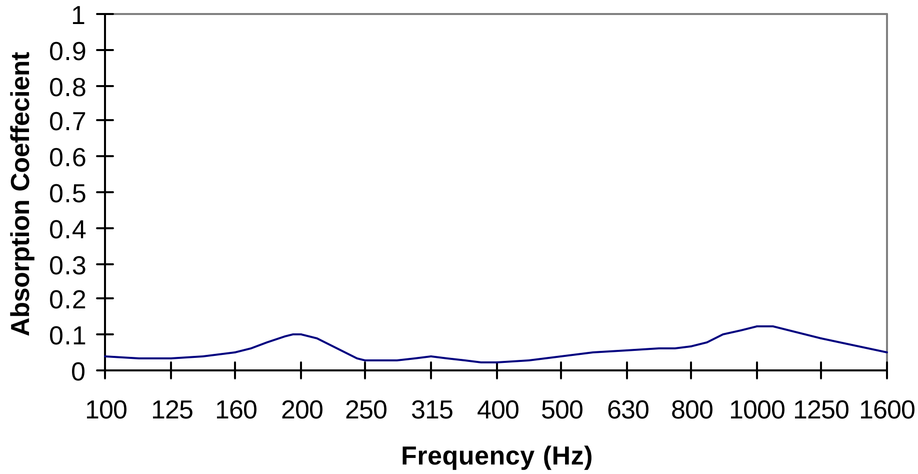

Figure 1.10 Variation of absorption coefficient as a function of frequency for two inch thick cement sample.

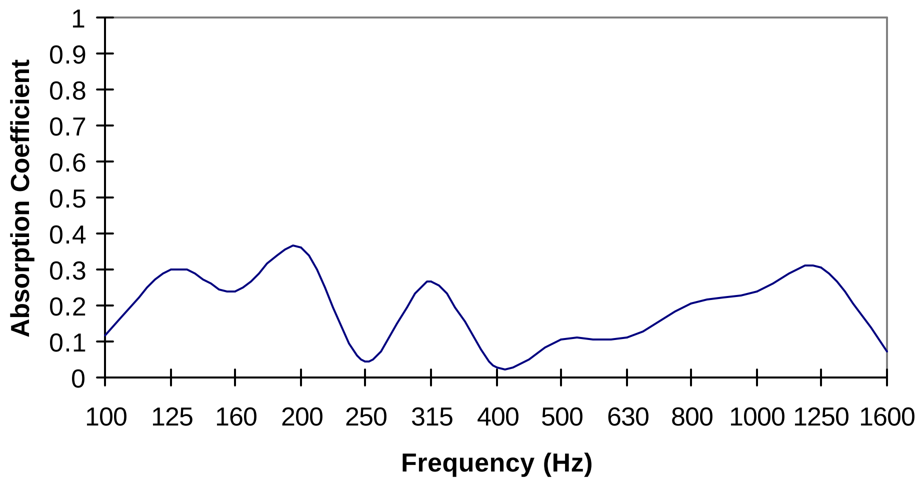

Figure 1.11 Variation of absorption coefficient as a function of frequency for one inch thick , 40% cenosphere. sample

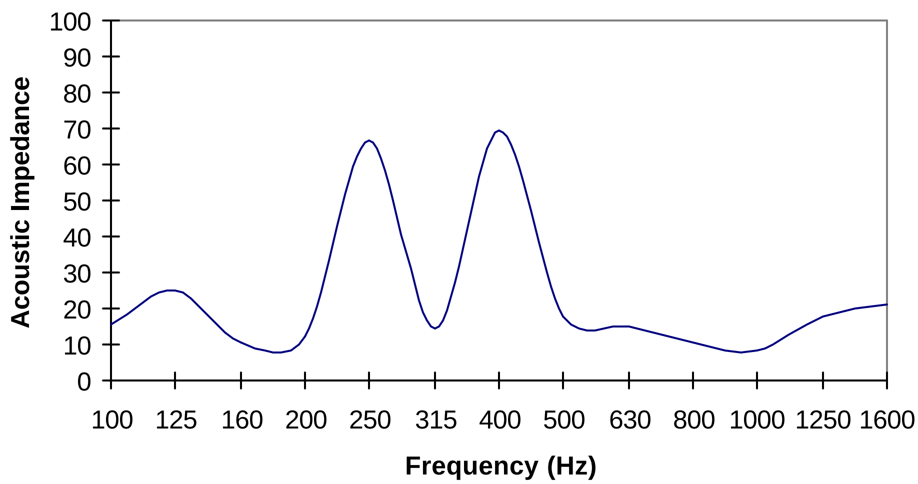

Figure 1.12 Acoustic impedance as a function of frequency for one inch, 20% cenosphere sample.

From the above Figures it can been seen that there is a certain change in acoustic charactersitcs of a cenosphere rich concrete with change in percentage of cenesopheres. More experiments will be done to in order to get a clear trend and to determine the cause of such variations. In many experiments it has been observed that ceneospheres help in improving the sound absorption properties of the materials which can be seen in figure 1.10 and figure 1.11.

1.8 CONCLUSIONS

Experimental studies on acoustical frequency response of cenosphere-rich material are going on, results seem promising and will help in detailed understanding of their behaviors under different frequencies. Our initial results indicate that addition of cenospheres in cement increases its sound absorption properties. Also the test on mass density of both cement and asphalt samples indicate appreciable reduction in density with increase in cenospheres content, where as there is not much change in the bulk specific gravity of Asphalt samples. The major accomplishments so far include establishing procedure to evaluate the acoustic properties, calibrating the test procedure and equipment. Further studies on the second year of the project will focus on the effect of particle distribution on the acoustic response of cement and asphalt in detail.

1.9 REFERENCES

1. Utsuno, H., Tanaka, T., and Fujikawa, T., "Transfer Function Method For Measuring Characteristic Impedance and Propagation Constant of Porous Materials", *Journal of Acoustic Society of America*, Vol. 86, No.2, pp. 637, 1989.
2. Dunlop, J.I., "Specific Acoustic Impedance Measurement by a Protruding Tube Method", *Journal of Acoustic Society of America*, Vol.79, No.4, pp. 1177 - 1179, 1986.
3. Smith, C. D., and Parrot, T. L., "Comparison of Three Methods For Measuring Acoustic Properties of Bulk Materials", *Journal of Acoustic Society of America*, Vol.74, No.5, pp. 1577 – 1582, 1983.
4. Yamaguchi, M., Nakagawa, H., and Mizuno, T., "Sound Absorption Mechanism of Porous Asphalt Pavement", *Journal of Acoustic Society of Japan*, Vol. E, No. 20, pp. 29 – 43, 1999.
5. Meiarashi , S., and Ishida, M., "Improvement in the Effect of Drainage Asphalt Road Surface on Noise Reduction", *Journal. of Applied Acoustics*, Vol 47, No. 3, pp.189-204, 1996
6. Watts, G. R., Chandler-wilde, S.N., and Morgan, P.A., " The combined effects of porous asphalt surfacing and barriers on traffic noise", *Applied Acoustic,* Vol. 58, pp. 351 –377, 1999.
7. Watts, G. R., and Godfrey, N.S., " Effects on roadside noise levels of sound absorptive materials in noise barriers", *Applied Acoustic,* Vol. 58, pp. 385 –402, 1999.

8. "Standard test method for impedance and absorption of acoustical materials by the impedance tube method", *Annual Book of ASTM Standards*. Designation C384-98.
9. " Standard test method for sieve analysis of fine and coarse aggregates", *Annual Book of ASTM Standards*. Designation C136-01.
10. "Standard test method for specific gravity and absorption of coarse aggregate", *Annual Book of ASTM Standards*. Designation C127-88.
11. "Standard test method for specific gravity and absorption of fine aggregate", *Annual Book of ASTM Standards*. Designation C128-97.

CHAPTER 2

LIGHTWEIGHT CONCRETE USING CERAMIC MICROBALLOONS: PROCESSING AND CHARACTERIZATION

2.1 ABSTRACT:

A study has been conducted in which a lightweight concrete was processed using ceramic microballoons, known as *cenospheres*, as a primary aggregate. The mechanical properties, including compressive strength, tensile strength, flexural strength and fracture toughness, were tested. For the sake of brevity, this paper will only focus on the compressive strength of the concretes evaluated. 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 producing a high-performance lightweight concrete.

2.2 INTRODUCTION

This study presents the development of a new type of lightweight concrete using ceramic microballoons 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, rock, sand and water. The rock 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. (1979) studied the effects of cork granules. Slate (1976) used coconut fibers, all with little benefit.

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. (1998) 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.

Silica fume is another compound, which has been studied extensively and is used in concrete today. Tazawa et al. (1984) have shown that silica fume can improve concrete strength, durability and corrosion resistance.

An important by-product of flyash is *cenospheres*, relatively large (10-300 μm) thin-walled microspheres produced during flyash formation. Clayton and Back (1989) show that cenospheres are formed during the coal burning process by evolution of gas becoming trapped in a viscous molten glass matrix. 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 (1996) has suggested many uses for this material, including using them as fillers in polymers and concrete.

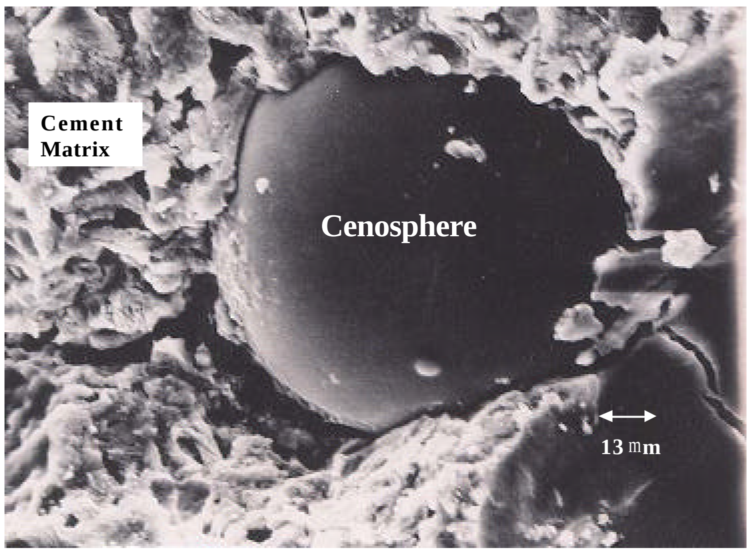

Figure 2.1 SEM micrograph of a cenosphere in fractured concrete.

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. An SEM micrograph of a cenosphere in concrete can be seen in Figure 2.1.

Initial cenosphere concretes were made by replacing the fine aggregate (sand) with cenospheres. Figure 2.2 shows the volume fraction of cenospheres used and the reduction in density that was achieved. Figure 2.3 shows the compressive strength of the cenosphere concretes. The compressive strength results are shown as a strength/density ratio to develop a criterion by which to compare performance. This figure shows that there is a 43% strength decrease with the addition of cenospheres.

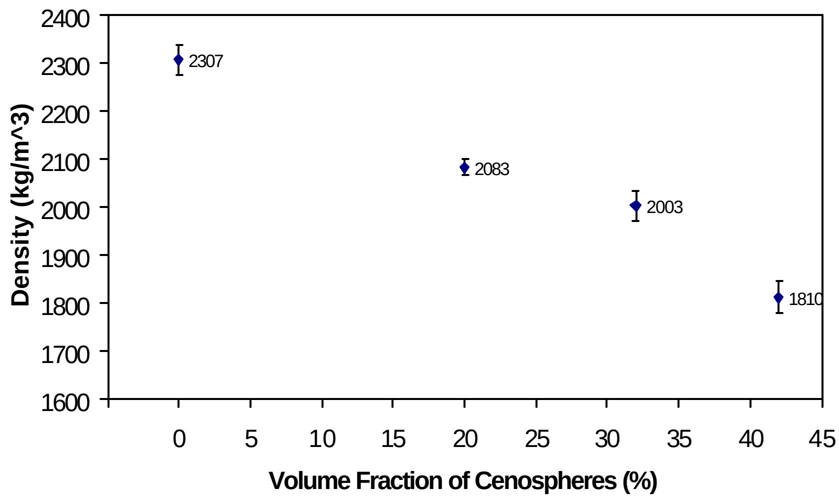

Figure 2.2 Density of various cenosphere concretes.

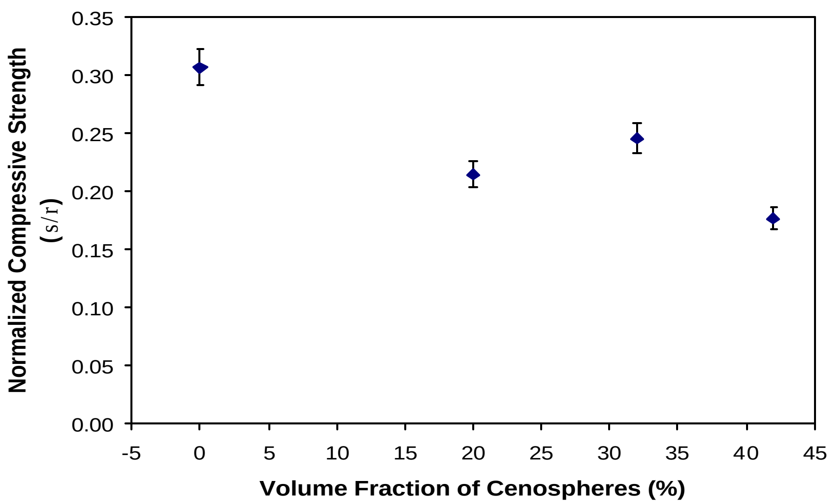

Figure 2.3 Compressive strength of various cenosphere concretes.

It was determined that this strength loss was due to the weak interfacial properties between the cenospheres and the cement binder. This can be seen in Figure 2.1 where the cenosphere shown has "popped out" as the crack passed around the cenosphere. This study will show how the interface properties of the concrete were quantified using the concept of bimaterial fracture mechanics, how these concepts were further used to locate a suitable interface modifier, and finally, how this modifier was used to create a high-performance lightweight concrete.

2.3 EXPERIMENTAL PROCEDURE

The interfacial strength of cenospheres and cement was evaluated using bimaterial fracture mechanics concepts. Similar experiments were also conducted to test the effectiveness of silica fume as an interface modifier.

BACKGROUND

A bimaterial system is defined as two dissimilar; linearly elastic materials bonded or cast together. Figure 2.4 shows a bimaterial system with a central crack geometry. Material 1 is the more compliant of the two materials.

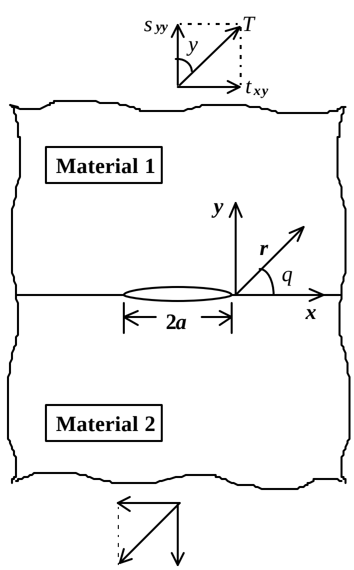

Figure 2.4 Geometry of a bimaterial fracture specimen.

The bimaterial fracture experiment is 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 developed by Rice (1988).

$$K = K_1 + iK_2 \qquad (1)$$

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 suggested by Rice (1988):

$$e = \frac{1}{2p} \ln \left(\frac{\frac{x_1}{m_1} + \frac{1}{m_2}}{\frac{x_2}{m_2} + \frac{1}{m_1}} \right) \qquad (2)$$

where m_i are the shear moduli and;

$$x_i = (3 - n_i)/(1 + n_i) \qquad \mathbf{(3)}$$

where n_i are the Poisson ratios.

Finally, for a uniaxially stress state $\left(T = s_{yy}^{\infty}\right)$ in a central crack geometry, K_1 and K_2 can be expressed in terms of the remote loading as suggested by Ricci et al. (1997):

$$K_1 = s_{yy}^{\infty} \sqrt{pa} \left[2e \cos(e \ln(2a)) + 2e \sin(e \ln(2a)) \right] \qquad (4)$$

$$K_2 = s_{yy}^{\infty} \sqrt{pa} \left[2e \cos(e \ln(2a)) - \sin(e \ln(2a)) \right] \qquad (5)$$

where $2a$ is the crack length.

SPECIMEN PREPARATION AND TEST SETUP

The bimaterial specimens were created by molding a cement paste on top of an aluminum silicate block. The cement paste was made with a water/cement ratio of 0.4.

The aluminum silicate represents the cenosphere. A piece of Teflon tape was placed as seen in Figure 5 to represent a central crack and then loaded into an Instron testing machine. These specimens were then loaded in tension until failure.

The specimens made with silica fume were made similarly, although 12% of the cement weight was replaced by silica fume. This same percentage of silica fume was added to one of the cenosphere concretes tested earlier and mixed according to ASTM C192-95. This concrete had a 42% cenosphere volume fraction, which was the highest volume fraction tested. The compressive strength of this concrete was tested according to ASTM C39-94.

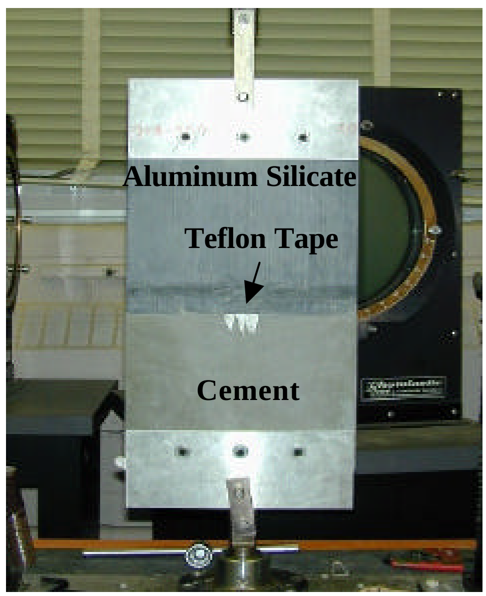

Figure 2.5 Experimental setup of a bimaterial fracture specimen.

2.4 RESULTS AND DISCUSSION

Following the experiment, the maximum load at failure was converted to stress and applied to equations (4) and (5) 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$^{1/2}$.The addition of silica fume showed some 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.

This improvement in the interfacial fracture toughness also increased the compressive strength as was suggested. The compressive tests showed that the cenosphere concrete with silica fume had a compressive strength of 36±1 MPa (5200±105 psi). This is an 80% improvement over the same cenosphere concrete without silica fume. When the density is considered as seen in Figure2.6, the performance of cenosphere concrete with silica fume is similar to control concrete with no cenospheres.

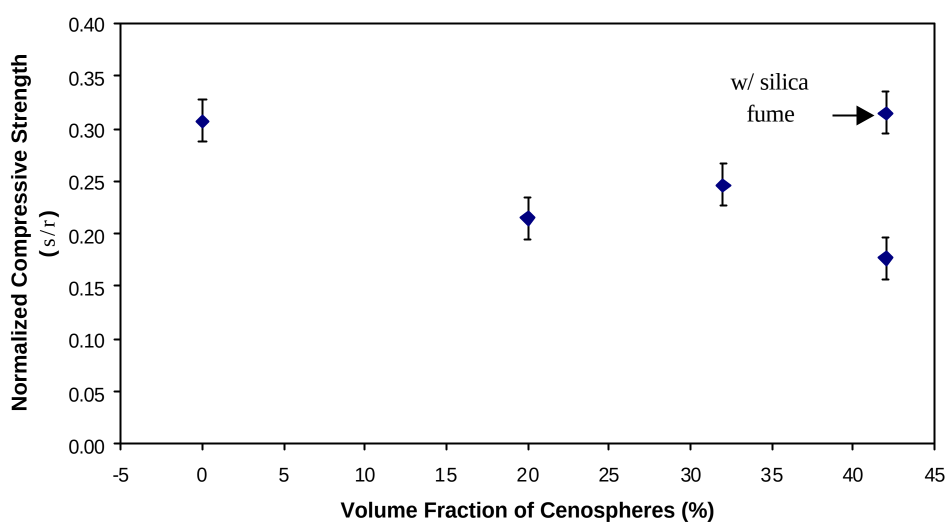

Figure 2.6 Compressive strengths of cenosphere concretes including silica fume type.

The mode of failure in compression was also different for the cenosphere concrete with silica fume. The control concrete failed in a conical fashion indicative of good aggregate bond strength. The subsequent cenosphere concretes all failed in a columnar fashion. The mode of failure returned to a conical type once silica fume was added, again showing good interfacial properties, which in turn gives better strength. These two types of failure can be seen in Figure2.7.

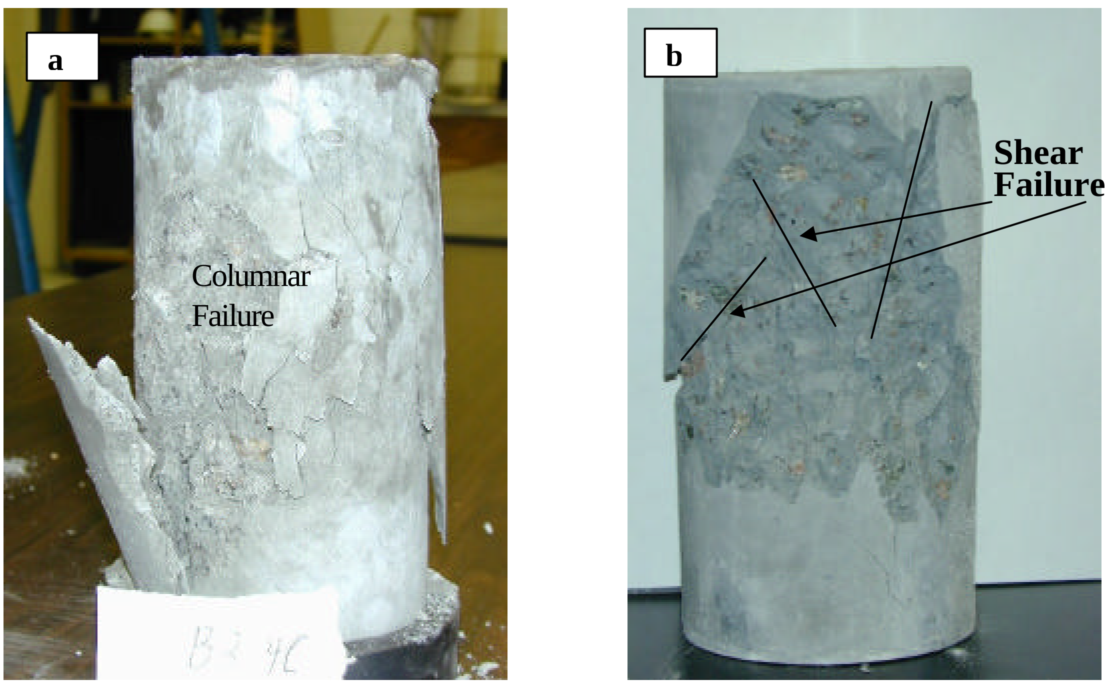

Figure 2.7 Failure patterns of cenosphere concretes (a) Columnar failure (without silica fume) (b) Conical failure (with silica fume).

2.5 CONCLUSIONS

A study has been conducted in which a new lightweight concrete using ceramic microspheres, called cenospheres, was investigated. This new concrete showed a dramatically lower density than normal concrete. Cenosphere concrete also showed some strength loss. This strength loss was attributed to a weak cenosphere/cement interface. The strength was regained by using a suitable interface modifier called silica fume, which was identified through the use of bimaterial fracture mechanics. The use of silica fume made it possible to create a high-performance lightweight concrete using cenospheres.

2.6 REFERENCES

Aziz, M.A., Murphy, C.K., Ramaswamy, S.D. 1979. Lightweight Concrete Using Cork Granules. *The International Journal of Lightweight Concrete.* 1:1: 29-33.

Clayton, R.M., Back, L.H. 1989. Physical and Chemical Characteristics of Cenospheres from the Combustion of Heavy Fuel Oil. *Journal of Engineering for Gas Turbines and Power*. 11: 679-684.

Naik, T.R., Singh, S. Ramme, B. 1998. Mechanical Properties and Durability of Concrete Made with Blended Flyash. *ACI Materials Journal*. 95:4: 454-462.

Ricci, V., Shukla, A., and Singh, R. P. 1997. Evaluation of Fracture Mechanics Parameters in Bimaterial Systems Using Strain Gages. *Engineering Fracture Mechanics*. 58: 4: 273-283.

Rice, J.R. 1988. Elastic Fracture Mechanics Concepts for Interfacial Cracks. *Journal of Applied Mechanics*. 55: 98-103.

Slate, F.O. 1976. Coconut Fibres in Concrete. *Engineering Journal of Singapore*. 3:1: 51-54.

Tazawa, Y, Nobuta, Y., Ishii, A. 1984. Physical Properties and Durability of High-Strength Lightweight Concrete Incorporating Silica Fume. *Transactions of the Japan Concrete Institute.* 6: 55-62.

Wandell, T. 1996. Cenospheres: From Wastes to Profits. *The American Ceramic Society Bulletin*. 75:6: 79-81.

CHAPTER 3

USE OF CENOSPHERES IN ASPHALT CONCRETE

3.1 ABSTRACT

A preliminary study has been conducted to investigate the influence of cenospheres (with and without coating of polymers) on the compressive strength of asphalt concrete. This study has just been initiated.

3.2 INTRODUCTION

Asphalt concrete is a composite material normally consisting of gravel and sand bound with asphalt. Before mixing with the asphalt, the aggregate can be separated by size. Therefore, an asphalt mix can specify a size distribution of aggregate that is to be used. Varying this distribution would affect how the material fits together once compacted. This would change the number of void spaces in the concrete and thus affect its strength and density.

These properties can also be changed if a different type of aggregate material is used. Cenospheres are chemically similar to sand, but they have a much lower density. Also, they are smooth round spheres which can more easily be slid past one another. Therefore, using cenospheres in an asphalt mix would lead to some expected changes in the concrete properties. Most obviously, the use of cenospheres would create a lighter concrete, but it may also lead to a weaker concrete.

In an asphalt concrete, there is weak bond between the asphalt and the aggregate. Therefore, the only resistance to deformation lies in the viscosity of the asphalt, which is a relatively weak resistive force, and the arrangement of the aggregate. Because the cenospheres are rounded spheres, there can be many slip planes in the concrete. The sand and gravel, because of their non-uniform shape, act as slip dislocations and create entanglements instead of slip. Therefore, it is to be expected that a normal asphalt concrete made with cenospheres instead of sand, would be weaker if nothing else is added to the mix.

Polymers have a good tendency to adhere to the surface of silicates, such as the cenospheres. Other polymers have been found to be miscible in asphalt. These two properties are rare in same polymer, though. But, a block copolymer can be used, one that has chain that bonds to the cenospheres and other chain that is miscible with the binder, to create a bond between the asphalt and cenospheres. Increased interfaced bonding would increase the resistance of the sample to deformation.

3.3 EXPERIMENTAL PROCEDURE

TESTING OF AGGREGATE MATERIAL

The mineral aggregate was obtained in four nominal size gradations. These included both the coarse aggregate; which included the ¾", ½", and the 3/8" size denominations; and the fine aggregate, which was denominated as sand. Each of these were subjected to a sieve analysis, (see American Society for Testing and Materials (ASTM) test designation C136 [1] or American Association of State Highway and Transportation Officials (AASHTO) test T27). The analysis consisted of separating a representative sample of each by a series of mesh sieves. The first sieve was one size higher than the nominal size of the sample and the sieves ran down in order until a number 8 sieve was used for the coarse aggregate and a number 200 sieve was used for the fine aggregate. Under the last sieve, a pan was placed to collect any filler in each sample. By this method a size distribution was obtained for the aggregate.

The aggregate samples were then also subjected to specific gravity tests, see ASTM C127 [2] and C128 [3] and AASHTO T85 and T84. The tests consisted of comparing three weights of each sample that were taken first when the sample was dry, second when the sample had been soaked with water overnight but surface dry, and lastly when the sample was placed under water. A comparison of the densities of the coarse and fine aggregate was thus obtained, and, as expected, they are all almost identical.

This information for the cenospheres was obtained from the supplier. The size distribution of the cenospheres was similar to the bottom half of the sand sieve analysis. In other words, if only the sand that passed through the number 500 sieve, which has a 300 micron mesh, was used to make a sample, then that sample would have nearly the same size distribution as the cenospheres. The specific gravity of the cenospheres was less than a third of the aggregates however.

SELECTION OF THE MIX DESIGNS

The design [4] for the standard specimens came from a CAMA [5] software program owned by the URI Department of Civil Engineering. The specifications of this mixture are modified slightly from one that is used by the Rhode Island DOT as a surface course mixture. However, the sieve analysis of the aggregate would not allow for a design to fit exactly within the specifications of this mixture. This is because there was relatively little filler in the sand that was obtained. Therefore, there is a slight deviation in the design from the specifications, but that occurs only at one end. The resulting design for the standard specimens is 25% from the ½" stockpile, 25% from the 3/8" stockpile, and 50% sand by volume.

As was mentioned earlier, the preliminary cenosphere asphalt design was to replace the sand with cenospheres. In order to have this design meet the first mixture specifications, only the sand that passed through the number 50 sieve was replaced. Therefore the second design consisted of 25% each of the ½" and 3/8" gravel, 30% sand (apportioned between the sand that was retained on the number 50 sieve or larger), and 10% cenospheres again by volume.

Since this resulted in only a small reduction of the weight of each sample, a third design was constructed to give an appreciable reduction in bulk specific gravity of the samples. Therefore, the design was to be made up of 40% cenospheres by volume, and the aggregate was proportioned according to the best fit to the original mix specifications. The rest of the aggregate was therefore divided as 20% from each of the stockpiles used in the first two designs. This distribution (Figure 3.1) was used for coated and untreated cenospheres. A graphical comparison of the mix designs is shown at the end of the report.

COATING OF CENOSPHERES

Two different block copolymer were used, a Styerene-Acrylonitrile polymer and Styrene-Butadiene polymer. Both were made up of approximately 50% of each monomer. The polymers were used at 0.25 wt% by weight of cenospheres. Four hundred grams of cenospheres were coated at one time; a 320ml of toluene were added to the cenospheres in the beaker. This was stirred until dissolved, about two hours. In the mean time, in a separate beaker, 320ml of denatured ethanol, 40ml of 1-butanol, 40ml of butyl cellosolve, and 40ml of deionized water were combined.

Once the copolymer was dissolved in the toluene, the solution was placed in a mixing bowl, and the mixer was turned on. The cenospheres were then slowly added to the bowl. The contents of the second beaker were then added, and the mixing was continued for about fifteen minutes. At this time, the slurry was then transferred to a shallow pan and placed in an oven at 90°C for an hour for drying.

PREPARATION OF TEST SPECIMENS

The rest of the procedure, including for the tests performed can be found in the MS-2 Handbook from the Asphalt Institute, "Mix Design Methods for Asphalt Concrete." The first step in making the test specimen was to weigh out the aggregate for each specimen. All the aggregate material had been previously separated by sieving. The mass of the aggregate in the standard specimen was 1200g. The volume fractions of this design could be considered weight fractions without any real error since the specific gravity of the aggregates were all nearly identical. Therefore, the total weight of aggregate from each stockpile was determined. This was divided among the separated aggregate according to the results from the sieve analysis.

Once the amounts of aggregate to be used, they were weighed out and combined in a stainless steel mixing bowl. The bowl was placed in an oven overnight along with the compacting mold, and the oven was set to 325 ^{o}F. The next day, approximately two hours before mixing, the asphalt is added to the oven and a hot plate is turned on to about 250-300 ^{o}F. The compacting hammer as well as a metal mixing spoon and spatula are placed on the hot plate.

Before mixing, the amount of asphalt used in each sample should be predetermined. Usually it is desirable to use approximately 6.0% Asphalt by weight of aggregate. Often, some samples are made with slightly more and some with slightly less asphalt. When ready to mix, the compacting mold is taken out of the oven and placed on the hot plate to keep it warm. Then the mixing bowl with the aggregate is taken out of the oven and placed on a scale, which is then tared. The correct amount of asphalt is added to the bowl, and the actual amount is recorded. The bowl is then immediately placed on the hot plate and the asphalt is mixed in with the aggregate with the spoon. The mixing should be done quickly, to avoid cooling, but thoroughly. When the asphalt is evenly mixed it is placed into the compacting mold.

First, a paper disk is inserted in the bottom of the mold to avoid sticking. The asphalt is then carefully placed into the mold. The spatula should be used to make sure all the asphalt and aggregate get out of the bowl and off the spoon and also to move the mixture away from the side of the mold and to form a cone on the top. The mold is then placed on the compacting stand.

The stand should have a notch which the mold can lock into and also a ring clamp to hold the top of the mold. The compacting hammer is then placed into position so that the compacting plate at the end of the hammer rests on top the asphalt mixture. The compactor should be set to deliver 50 even blows and should now be turned on.

After 50 blows, the hammer is removed and the bottom of the compacting plate scraped clean and returned to the hot plate until ready to be used again. The mold is also replaced on the hot plate and the bottom of the mold is removed. A new paper disk is place on the bottom of the mold. The mold is then flipped over and placed back on its bottom piece.

The mold is then placed back on the compacting stand. The asphalt mixture is compacted with another 50 blows to the other side. The mold is then removed from the stand and its bottom taken off again. With the spatula the paper disks should be removed if necessary and the sample marked with a grease pencil. The specimen is then placed aside to cool before it can be extruded. The next sample can now be mixed.

After all the samples have been mixed, the specimens are removed from the molds using a hydraulic extruder, and they are left to cool overnight.

3.4 TESTING THE SPECIMENS

The bulk specific gravity test (Figure 3.2) performed on the specimens is very similar to the bulk specific gravity test performed on the coarse aggregate. The largest difference is that the specimen need only be immersed in a water bath for a couple minutes before they can be considered saturated. Each sample is weighed and then placed into a water bath. Their apparent weight under water is then recorded as well. The specimens are then removed from the water, all excess water is removed, and the specimens are weighed again. The bulk specific gravity is then the dry weight of the samples divided by the difference between the saturated weight in air and the saturated weight in water.

The second test was the Marshall stability and flow test. The apparatus required for this test consists of a testing head, and a moving platform connected to a chart recorder. The testing head consisted of two pieces and would hold the specimen on its edge so the flat sides faced out. The bottom part of the head was a semicircular base to hold the specimen with two guide bars. The top piece was again a semicircle which would fit through the guide bars and rest on the top of the specimen without touching the semicircular part of the bottom piece. The testing platform was a stage that could be moved up or down. The stage had a top bar that would stop the top of the head from raising any more once it reached that. The top would then be pressed down as the bottom would continue to be moved up pressing them together. The apparatus had a sensor for detecting the amount of load exerted by the testing head on the sample.

Before the test, the samples were soaked in warm water, 60^{o}C, for about 40 minutes. The samples were then removed from the water bath and quickly dried before being placed in the testing head. The head was placed on the test apparatus, which was hooked up to an automatic chart recorder. The platform of the machine was raised so as to squeeze the two ends of the head together, thereby creating a stress on the sample. The apparatus was able to record the maximum load sustained by the sample before failing. Figure 3 shows the maximum sustainable load for different percentages of asphalt.

3.5 RESULTS

In the experiments concluded so far, Marshall stability tests on various asphalt concrete specimens have been conducted. The test is a destructive one which allows for a comparison of relative strength and performance for the test specimens. The specimens were also subjected to specific gravity tests first before being destroyed.

Four different types of test specimens were produced. The first type was the standard and it comprised of normal aggregate material and varying amounts of asphalt. The second type was designed the same as the first, except that the finer aggregates, the sand, were replaced with untreated cenospheres. The third design used more cenospheres and therefore less of conventional aggregate material. The final design specimens were made using copolymer-coated cenospheres. Otherwise, the design was the same as the previous.

The results of the experiments revealed that the density of the asphalt concrete decreases with the introduction of cenospheres. The relative strength as determined by the Marshall test also decreases (Figure 3.3); in fact, this effect is about twice as noticeable. The average density of the cenosphere-rich specimens was about 30% less than the standards, while the average maximum sustainable load for these samples was nearly 70% less.

The idea behind the coating of the cenospheres was to create a bond between the asphalt phase and the cenospheres. Two different copolymers were used, an acrylonitrile-styrene block copolymer and styrene-butadiene block copolymer. Both were added to the cenospheres at 0.25 wt% of the cenospheres. The experimental results demonstrated an effect opposite of what was theorized. The specimens with the coated cenospheres had a slightly decreased maximum sustainable load as compared to the samples made with untreated cenospheres. The density appeared unchanged, however.

Although the cenospheres have the chemical composition as the sand, they have different shapes. Whereas sand is jagged and irregular, the cenospheres are spherical, which allow easies deformation. The strength of asphalt lies in the inability of its particulates to slide past one another. Therefore, it also relies on an even distribution of the aggregates, as compared to cement, which is comprised of more filler. Therefore introduction of more cenospheres to reduce the density will also cause a reduction in the strength, which may be unrecoverable.

Further investigation should look at the reduction of strength associated with the coated cenospheres to determine if this is a recurring phenomenon. Also it may be desirable to look at other copolymers. These should include monomer chains that demonstrate good affinity toward absorption on silicates, such as poly-ethylene oxide and poly-vinyl pyridine.

Table 3.1 gives the results for both coated and uncoated cenospheres specimens.

Table 3.1: Sample Results at 6.0% Asphalt Content

		Original Mix Design		Preliminary Cenosphere Mix Design		Final Cenosphere Mix Design	
Trial	Asphalt Content %	Max Load (lb_f)	Bulk Specific Gravity	Max Load (lb_f)	Bulk Specific Gravity	Max Load (lb_f)	Bulk Specific Gravity
1	6.5	2350	2.30	1420	2.22	700	1.66
2	6.5	2060	2.29			680	1.66
3	6.6					710	1.67

3.6 CONCLUSION

Results from these experiments indicate that there is a substantive decrease in density of asphalts sample with the addition of cenospheres. However, the strength also decreases as the cenosphere content is increased.

Table 3.2 Design for surface course Class I-1

Sieve Size	Percentage Passing by Weight
3/4 ’’	100
1/2’’	80 – 100
3/8’’	70 – 90
#4	50 – 70
#8	35 – 50
#30	18 – 29
#50	13 – 23
#200	3 - 8

Table 3.3 Design used for making the specimens

Nominal Sample Size	1/2’’	3/8’’	Sand
pass 3/4 ret on 1/2	199.7		
pass 1/2 ret on 3/8	85.1	45.6	
pass 3/8 ret on #4	15.2	265.3	
pass #4 ret on #8			96.9
pass #8 ret on #30			254.2
pass #30 ret on #50			122.6
pass #50 ret on #100			70.1
pass #100 ret on #200			32.9
pass #200			12.4
		Total Weight	**1200.0 gm**

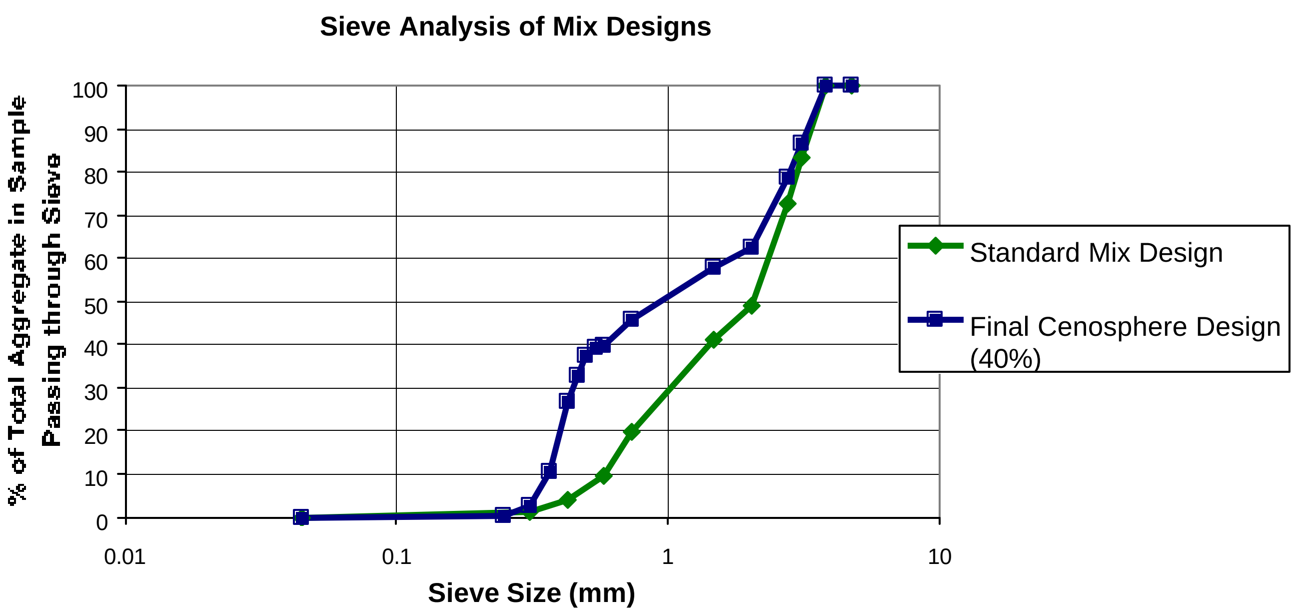

Figure 3.1 Mix Design for standard and cenosphere-rich asphalt samples.

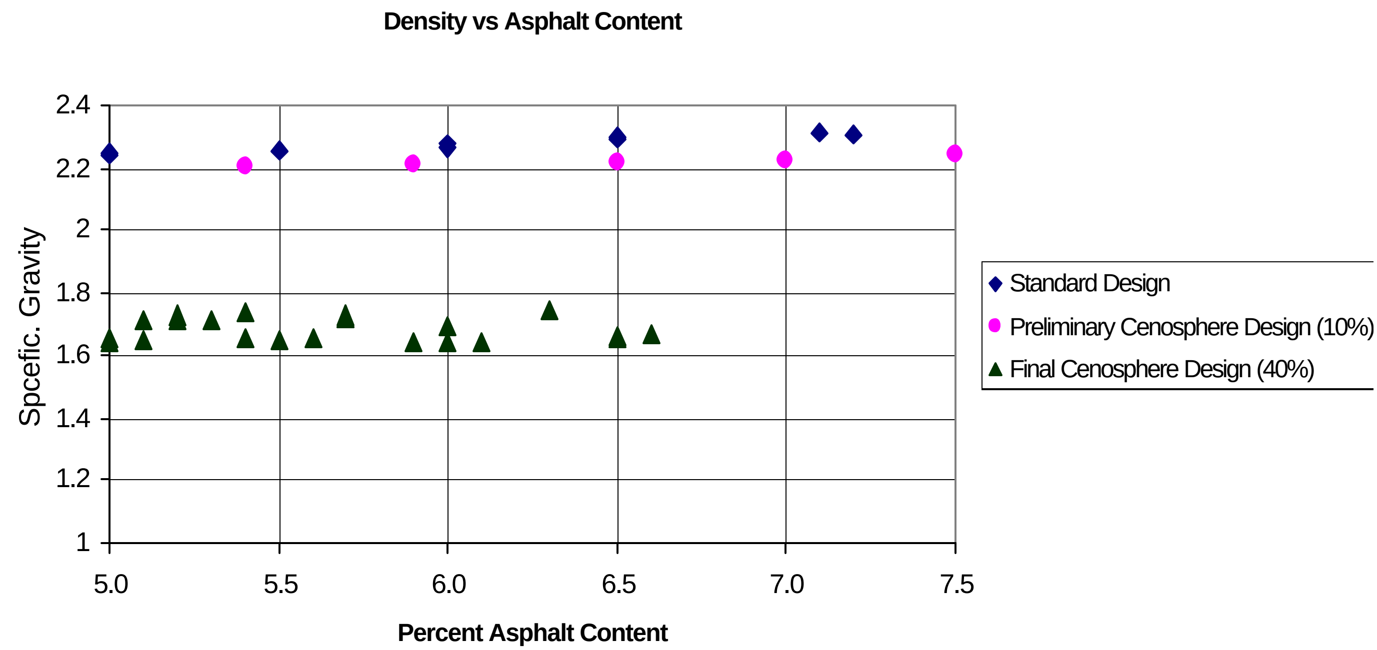

Figure 3.2 Variation of specific gravity with different percentage of asphalt.

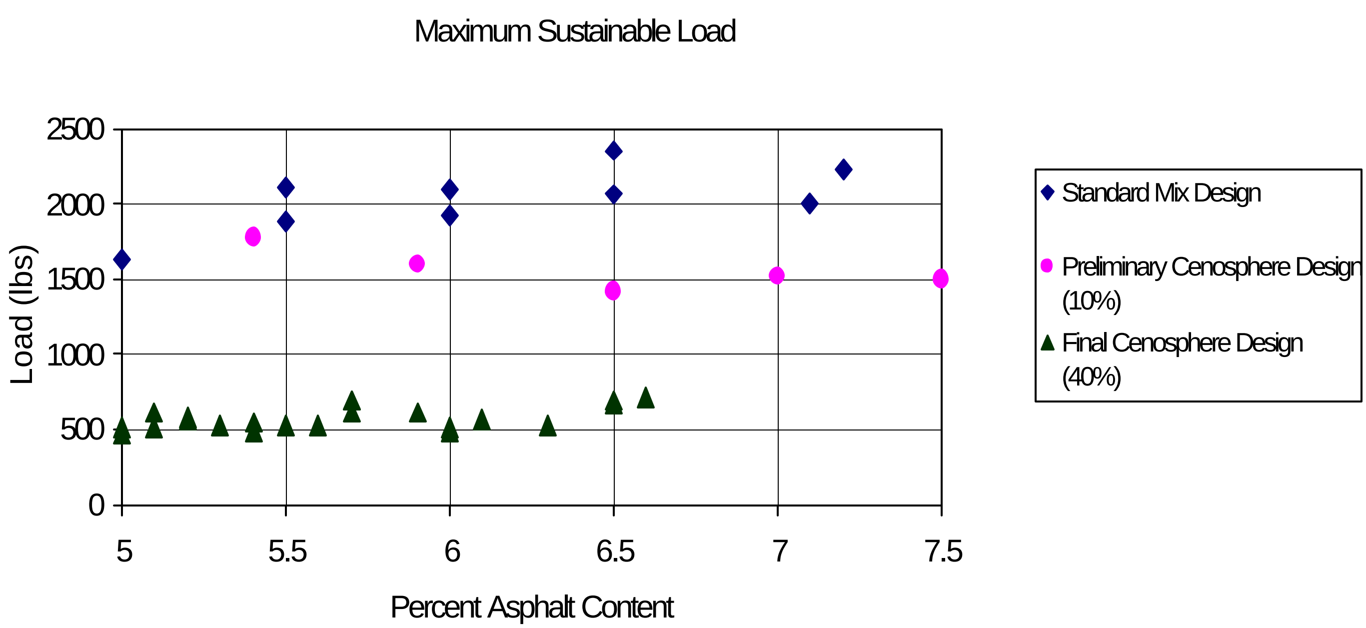

Figure 3.3 Variation of maximum sustainable Load with Percent

REFERENCES:

12. " Standard test method for sieve analysis of fine and coarse aggregates", *Annual Book of ASTM Standards*. Designation C136-01.

13. "Standard test method for specific gravity and absorption of coarse aggregate", *Annual Book of ASTM Standards*. Designation C127-88.

14. "Standard test method for specific gravity and absorption of fine aggregate", *Annual Book of ASTM Standards*. Designation C128-97.

15. Mix Design Methods, Sixth Edition; Asphalt Institute.

16. Computer-Assisted Mix Analysis, Asphalt Institute.