Investigation of the Thermal Properties of Rice Husk Ash in Cement
A Major Qualifying Project in Collaboration with Worcester Polytechnic Institute

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**Abstract**

This project investigated the thermal properties and strength of cement with the addition of rice husk ash (RHA) and cenospheres in five mix designs tested against ordinary portland cement (OPC). Although research exists on how various percentages of RHA and cenospheres affect cement’s strength and durability, there is little information on the thermal impact of these additives. Understanding the thermal properties of the modified materials gives potential for a more resistant product that can be supplemented for ordinary portland cement.
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Authorship List

Throughout the duration of this project, all members of the team pooled their efforts and contributed to the final deliverable. All team members were present for the original mixing of the various samples. Each member authored individual sections, and edits were made in a collaborative group. Brendan focused on the cone calorimetry and cold plate testing, as well as the creation of the cementitious mix designs. Serena worked primarily with the compression and tensile testing. Abbey drafted the background section, as well as the X-ray diffraction sections. Daniel prepared the introduction, while also working primarily with the Fourier Transfer Infrared Spectroscopy information. All members of the team helped contribute to the final conclusions and recommendations of the paper, as well as the final edits to each section.
Executive Summary

Introduction & Background

Ordinary portland cement (OPC)-based concrete is the most widely used building material and is relied upon for its durability and life span, strength, and in some cases, its aesthetic effect. The goal of this project was to supplement OPC powder with rice husk ash (RHA) and expanded glass cenospheres to analyze the effects on the structural and thermal properties of the cement, and to determine if these additives create a more efficient product. Two RHA mixtures and two expanded glass cenosphere mixtures were compared to a baseline OPC mixture.

In the concrete industry, RHA can be used as a cement powder replacement. RHA derives from burned rice husks, is roughly 95% silica, highly porous and lightweight, absorbent, and possesses insulating properties beneficial for industrial applications. In finely divided form and in the presence of water, RHA reacts chemically to form compounds possessing cementitious properties. RHA can also be used in the manufacturing of general building blocks at a lower cost. OPC powder is relatively expensive, therefore, when the product is available, replacing portions of it with RHA makes the cementitious product more affordable. Research shows that different contents of RHA burned within the range 600°C to 850°C (1112°F to 1562°F) may influence the physical and mechanical properties of the concrete to which it is added.

Cenospheres are a byproduct of the combustion of coal products. Cenospheres are essentially a hollow cavity surrounded by a hard shell made up of primarily silicate and alumino-silicates. These spheres can be substituted for cement powder within a cementitious mix to add volume, but at a lower density and weight. Due to their spherical shape, cenospheres improve the workability of the mix, lowering the overall water consumption. Prior research in the ceramic field showed that cenospheres provided better heat resistance and possess better thermal characteristics than the ceramics with no additives. It was found that the presence of the cenospheres prevented heat from entering the ceramic material, thus improving the thermal conductivity of the material.

Thermal conductivity relates heat flowing through a sample to the temperature of the sample on opposite sides. Thermal resistance is the ability of a material to resist heat flow. Materials with a higher thermal resistance are more apt to remain at the same temperature when exposed to a change in temperature. Concrete structures have been shown to incur significant damage during fires. If an insulative additive can be included to a concrete structure, it could greatly improve the resistivity of the material. The structure would be more able to resist the change in temperature due to a fire condition. This improved heat resistance
would reduce the need for extra insulation protecting the concrete structure, while still providing a similar level of heat protection.

RHA and cenospheres have the potential to become useful, heat-resistant building materials in the construction industry due to their thermal properties. The goal of this project was to compare mixes with RHA to those with cenospheres and understand how they perform thermally as additives in cement.

**Methods**

Five unique mix designs were created to capture a spectrum of possible cementitious mixes. Two mixes contained RHA in two different quantities, 10% and 30%, while two other mixes contained cenospheres in the same quantities. The final mix was a control mix of pure OPC mortar. For the 10% substitutions, 1/10th of the required cement by volume was removed from the mix. This volume of cement powder was then replaced by the same volume of additive. The rice husks were burned at 650°C for a minimum of six hours then were left in the furnace, allowing them to slowly cool.

Each mix was created and poured into various molds for testing, consisting of 2 in. x 4 in. (5.08 cm. x 10.16 cm.) cylinders, 2 in. x 2 in. (5.08 cm. x 5.08 cm.) cubes, and a 7.87 in. x 7.87 in. x 1.97 in. (10 cm. x 10 cm. x 5 cm.) block. Compressive strength tests were conducted on 2 in. x 4 in. (5 cm. x 10 cm.) cylindrical samples of each mix design after curing for 7 and 28 days in a curing chamber with 100% humidity per ASTM C39. Tensile testing was conducted using the same methods as a standard compression test, except the sample was laid on its side, and an adaptive formula was used to compute tensile strength of the samples after 28 days of curing.

In order to evaluate the thermal conductivity and resistance of the various cementitious materials, the 10 cm. x 10 cm. x 5 cm. (7.87 in. x 7.87 in. x 1.97 in.) samples were exposed to an average heat flux of 50 kW/m² (15,845 Btu/hr/ft²) in accordance with the provisions of ASTM E1354. The samples were wrapped with aluminum foil on all sides except the top, which was directly exposed to the heat flux to simulate one-dimensional heat flow through the top of the sample. Thermocouples were attached to two faces of the cement samples; one on the side exposed directly to the heat flux, and one on the opposite side with no direct exposure. The temperature was recorded every second in both thermocouples throughout the 30-minute duration of the test.

Cubes with the dimensions 2 in. x 2 in. (5.08 cm. x 5.08 cm.) were created for thermal conductivity testing using a guarded longitudinal calorimeter apparatus in accordance with ASTM C177. The samples were
placed between two Pyroceram blocks with a known thermal conductivity, and thermocouples were placed between each layer. The program used for this series of experiments was an eight-step model, with equivalent temperature changes over a period of six hours.

Two more tests that were conducted, Fourier Transform Infrared Spectroscopy (FTIR) and Powder X-Ray Diffraction (pXRD), tested the chemical makeup of the samples. For the IR Spectroscopy testing, the 2 in. x 4 in. (5.1 cm. x 10.2 cm.) cylinders of concrete being tested were ground with a mortar and pestle into a fine powder and placed on that testing crystal. In the spectrometer, an IR beam passes through a beamsplitter and diffracts onto two different mirrors; one at a stationary distance and the other which changes its position throughout the test. The diffracted beams recombine at the beamsplitter and then proceed to pass through the sample. As the beams recombine, they create an interferogram (time vs. intensity) of constructive and destructive interference. The peaks at specific wavelengths correlate to specific R-groups known over the spectrum to represent the chemical makeup of the tested sample. The test was run for roughly 3.5 minutes as opposed to the typical 15-minutes, since the main observations being gathered from this test were to see if there were any outliers in the spectrum that did not show up in a typical cementitious mixture.

X-ray diffraction (XRD) is a technique used for identifying crystalline structures within a material. To be tested, each mix was ground into a powder and placed on a flat 1 in (2.5 cm.) diameter plate. The x-ray device moved through a spectrum of angles ranging from 10° to 42° in 0.05° increments, with each angle being held for two seconds for a total test time of 21 minutes. The moving x-ray device continuously sent x-rays through the powder sample, which diffracted as they hit crystalline structures within the powder. Where there existed a crystalline structure, the x-ray beams entered the receiver at a different frequency that correlated with specific crystalline structures at various angles. These frequency differences were recorded on a graph as peaks.

**Results**
The OPC mixture averaged a maximum compressive strength of 5,153 psi (36 MPa) with a standard deviation of 499 psi after seven days of curing, which was the highest compressive strength. The compression testing data suggests that after seven days, the substitution of cement powder with RHA or cenospheres provides no mechanical advantage. The sample with the maximum compressive strength at the 28-day mark was the 10% RHA mix, which failed at an average of 5,723 psi (39 MPa). The 30% RHA mixture was the weakest at the 28-day mark, likely because there was too much reactive silicate with not enough calcium hydroxide (Ca(OH)$_2$) to create strong bonds in the mixture. The compression testing suggested that the substitution of cenospheres added no benefit to the strength of cement when compared
to the control OPC. Tensile testing showed that the 30% cenosphere mix was the strongest sample with a maximum tensile stress of 455 psi (3 MPa).

From the FTIR test, a stacked plot of each sample interferogram was created. Each peak in the graph correlates to a specific chemical bond found in the sample at a specific wavelength. It was determined that each sample contained OH (hydroxide) stretching around 3400 cm\(^{-1}\), and either C-O asymmetric stretching or Gypsum around 1430 cm\(^{-1}\). Another major peak commonly found in all samples was either aluminosilicate bonding or the presence of calcium silicates around 1020 cm\(^{-1}\). The mixtures containing the cenospheres had lower peaks at the 1020 cm\(^{-1}\) mark. This is likely caused by cenospheres being hollow being an empty space.

The XRD graphs were determined to be inconclusive for the purpose of this experiment. If crystalline structures were present in any of the samples, large peaks would have showed up on the computer-generated graphs, indicating crystalline structures within the samples. The lack of major peaks can be explained by the lack of crystalline structures in the concrete samples, which is backed by the FTIR results. This showed that the addition of RHA to the cement mixture did not create any new phases in the samples.

From the cone calorimetry testing, the OPC mixture had the highest thermal conductivity, while the 10% RHA mixture had the lowest. The average thermal conductivity among the five samples was close to 8 W/m\(\cdot\)K (4.6 BTU/h\(\cdot\)ft\(\cdot\)\(\cdot\)°F), which is fairly typical for non-metallic solids. The values for thermal resistance fell between 0.0061-0.0071 K\(\cdot\)m\(^2\)/W (0.035-0.040 ft\(^2\)\(\cdot\)°F\(\cdot\)h/BTU). Thermal resistance calculations output a material’s 'R-value', which is its capacity to resist heat flow. The higher R-value a material has, the better it will resist heat flow, and the better it will serve as an insulator. From the cone calorimetry testing, it was determined that the addition of RHA or cenospheres provided marginally better thermal conductivity and thermal resistance.

The results from the cold plate testing showed that the OPC sample had the highest thermal conductivity at approximately 2.9 W/m\(\cdot\)K (1.67 BTU/h\(\cdot\)ft\(\cdot\)°F), while the 30% RHA sample had the lowest thermal conductivity at roughly 1.6 W/m\(\cdot\)K (0.93 BTU/h\(\cdot\)ft\(\cdot\)°F). The 30% RHA sample was therefore the most insulative, while the OPC sample was the least insulative. The addition of RHA or cenospheres was shown to impact the insulative qualities of the samples. As the rest of the sample cooled down, the room-temperature air in the pores of the RHA could slowly release the warmer air into the rest of the sample, thus slowing the cooling time of the samples. Similarly, the cenospheres could contain warm air that the sample
was originally held in prior to testing. As the cold plate test ran, the warm air in the cenospheres would slow the cooling process of the sample, demonstrating more insulative properties of the mix designs.

**Conclusions & Recommendations**
Data pertaining to the thermal conductivity of each sample were calculated in both the cone calorimetry and the cold plate tests. In general, cement is a poor thermal insulator, as it very easily transfers heat at a faster rate than other building materials such as wood. In both the cone calorimetry test and the cold plate test, the OPC mixture was determined to have the highest thermal conductivity, meaning it transferred heat at the fastest rate of all the samples. With the addition of RHA or cenospheres, a concrete structure can be expected to perform slightly better in a fire scenario. The structure would be more apt to resist the changes of heat within a space, as the additives would provide more insulative qualities. Using RHA or cenospheres in the concrete industry could reduce the risk of losing structural stability in buildings experiencing a fire.

One key recommendation for future work is to burn the rice husks at a temperature at or above 850°C (1562°F) as RHA has been determined to develop a crystalline structure when burned at this temperature. The crystalline structure may provide more resistance to compressive stresses, and could also serve as a better medium to resist heat transfer.

Another recommendation for future work is to run FTIR, XRD, and compression tests on the samples after being exposed to a one-dimensional heat flux in the cone calorimetry test. The FTIR and XRD tests would allow for comparison of the chemical makeup of the mixtures before and after exposure to a one-dimensional heat flow, mimicking a real life fire situation. Performing compression testing on the 10 cm. x 10 cm. x 5 cm. (7.87 in. x 7.87 in. x 1.97 in.) blocks before the cone calorimetry tests would provide a baseline strength value that could be compared to the samples after their exposure to the heat flux in the cone calorimetry test. This comparison could yield vital information to changes in strength of the various mixtures after a fire scenario, leading to better conclusions about the structural stability of the mixes.
1.0 Introduction

Cement-like materials can be traced back as far as the Egyptian era, when walls were constructed of hardened mud and straw bricks and coated with a wet layer of mud to stack onto one another to form walls (Blezard). Cement as it is known today did not come about until October of 1824 when Joseph Aspdin patented a material known as "ordinary portland cement" (OPC). This would then be refined by his son, William, in 1843, presumably by accident, into today's ordinary portland cement (Blezard). While there have been advances in the technology of concrete construction and the chemistry of cement has been revamped and improved, a majority of the processes and materials used today are still the same as those implemented in the erection of structures of the ancient world.

Today, OPC-based concrete is the most widely used building material. It is used in the foundations of various structures, retaining walls, stairways, and terraces. Concrete is relied upon for its durability and life span, its strength, and in some cases, its aesthetic effects. It has also been employed for its ability to retain water and to act as an insulator, both retaining and releasing heat. In the past, concrete was used for water storage, and can be seen today as the main material used to construct dams. A typical concrete sample can range in strength from 2,500 psi (17.2 MPa) all the way up to 10,000 psi (69 MPa) (National Ready Mixed Concrete Association). In addition to strength, concrete is used in construction because of its ability to last over extended periods of time. On average, a concrete bridge should be expected to last 50 years before requiring repair or maintenance (Weaver). Although concrete contains only a few simple ingredients – cement powder, water, and aggregate - it clearly has many beneficial properties.

Cement powder is the most expensive component when making concrete; therefore, reducing the amount of cement powder in the mix design will decrease the overall cost of the product. There are numerous additives that can be supplemented for cement powder that will reduce the overall cost of the mix, while maintaining or improving strength and resistance properties. On average, one cubic yard of concrete costs about $90, which appears costly when considering the limited ingredients of cement powder, water, sand, and rock. One must also factor in the 28-day time frame for the material to react and strengthen into functional concrete (Concrete Price Considerations – Cost of Concrete). When looking at cement as a whole, there is potential for an alternative product that can be supplemented for ordinary portland cement and perform as well or better at a lower cost.

The goal of this project was to supplement OPC powder with rice husk ash (RHA) and expanded glass cenospheres to see the effects on the structural and thermal properties of the cement and to determine if
these additives create a more efficient product. An efficient product in this sense means that its strength and thermal resistivity would be the same, or better, than concrete with only OPC powder. This was done by performing lab tests, specifically compression tests, tensile tests, calorimetry, x-ray diffraction, and infrared spectroscopy on various cement mixes. Each mix contained a different percentage of OPC powder substituted with RHA and cenospheres, and a different water to cement ratio. These RHA mixtures were compared to a baseline OPC mixture, and two mix designs containing different percentages of expanded glass cenospheres. The final comparison of all five mixes allowed for conclusions and comparisons to be made about the addition of RHA and cenospheres in cement. Based on preliminary research, it was thought that the substitution of RHA and cenospheres in the cement mixes would produce a product with better thermal properties, but may lack strength after the 28-day set period.
2.0 Background

Rice is one of the largest readily available biomass resources worldwide and covers 1% of earth’s land surface (Rice Husk Ash Market Study). Production is dominated by Asian nations, where rice is the primary food crop grown during the rainy season. Much like corn, rice develops within a thin fibrous membrane called a husk. During growth, rice plants absorb silica from the soil. It is this silica, concentrated by burning rice husks at high temperatures, thus removing other elements and contaminants, which makes RHA so valuable. RHA is roughly 95% silica, highly porous and lightweight, absorbent, and possesses insulating properties beneficial for industrial applications (Rice Husk Ash Market Study). The silica within RHA comes in multiple forms and can have a different chemical makeup depending on the temperature at which the husks were burned. Similarly, performance characteristics (specifically thermal conductivity) of the RHA differ based on the temperature and method by which they are burned. As husks are burned at higher temperatures, a more crystalline structure is developed and a higher silica content becomes available to the cement (Onojah et all).

The steel and concrete production industries are currently the two primary consumers of RHA. In the steel industry, RHA is used in the production of high quality flat steel, which is generally produced by continuous casting. This process involves molten steel that is solidified into a slab for rolling in finishing mills (Rice Husk Ash Market Study). RHA is used to insulate the tundish, an open container with holes in the bottom used to feed molten metal into molds to give a smoother flow in metal casting. The insulative properties of RHA prevent the rapid cooling of steel in the tundish and ensure uniform solidification in the molds (Kumar). RHA is used in the continuous casting process because of its insulative properties including low thermal conductivity, high melting point, and high porosity (Rice Husk Ash Market Study).

In the concrete industry, RHA can be used as a cement powder replacement. A study at the University of Malaya investigated the properties of RHA produced by using a ferro-cement furnace. When the husk was burned at a temperature below 690°C (1274°F), it was found that RHA was efficient as a pozzolanic material. This efficiency means that in finely divided form and in the presence of water, RHA reacts chemically to form compounds possessing cementitious properties (Habeeb, Mahmud). It was also determined that the strength of concrete increases with RHA fineness. The compressive strength of the blended concrete with 10% RHA increased 30.8% compared to the control mix that did not contain any RHA (Habeeb, Mahmud). RHA can also be used in the manufacturing of general building blocks at a lower cost. OPC powder is relatively expensive, therefore, when the product is available, replacing portions of it with RHA makes the cementitious product more affordable (Tashima). The addition of RHA to cement has
also been found to increase set-time, improve compressive strength, and improve resistance to acid attack. A study at São Paulo State University evaluated how different contents of RHA burned within the range 600°C to 850°C (1112°F to 1562°F) may influence the physical and mechanical properties of the concrete it is added to (Tashima). Figure 2.1 depicts the difference in appearance between unburned rice husk and burned RHA.

Cenospheres are similar to RHA in the sense that they are created through combustion, however expanded glass cenospheres are a byproduct of the combustion of coal products. Cenospheres are essentially a hollow cavity surrounded by a hard shell made up of primarily silicate and alumino-silicates (Fenelonov et al.). The hard shell is formed from easily melted inorganic compounds, while the hollow center is created from more heat resistant components that do not melt. When the resistant compounds are sealed by the hard shell, they begin to gasify and expand, forming the hollow core that defines the cenosphere (Fenelonov et al.). Once created by combustion, the fly ash product is electrostatically filtered out of exhaust gases from major manufacturing facilities, so the remaining exhaust can more safely be released into the environment. The recovered ash comes in a variety of densities and sizes, which can then be separated in a variety of ways. One separation technique involves adding the fly ash to a body of water such as a lagoon. The high density ash and broken cenospheres will sink to the bottom, while the light, hollow cenospheres float to the surface of the water. These cenospheres can then be separated from the water and the remaining ash in the lagoon. (Poraver)

![Figure 2.1: A) Unburned rice husk; B) RHA burned in a furnace.](image)
Cenospheres are low density because they are hollow, which is helpful in the separation process previously described. Cenospheres come in a variety of sizes which correlate to a variety of densities for the material, however a majority of cenospheres range from 1 - 2.9 g/cm$^3$ (0.04 – 0.10 lb./in.$^3$) (Tharaniyil). To put the size of cenospheres into perspective, one pound of standard cenospheres will equate to roughly the volume of 3.8 pounds (1.72 Kg) of typical sand (Tharaniyil). Cenospheres have been used in industry for the past 30 years for a variety of purposes including the manufacturing of low-density concrete. These spheres can be substituted for cement powder within a cementitious mix to add volume, but lower density and weight (Tharaniyil). This size differential reduces the weight of concrete, while still supplying the same volume of mix. Due to their spherical shape, cenospheres improve the workability of the mix, lowering the overall water consumption. The spheres roll during the mixing stage, allowing for better material packing, and a water reduction due to better flow characteristics (Tharaniyil). Figure 2.2 shows a sample of the cenospheres used for this project.

Lightweight cenospheres, along with providing better weight to volume relationships, may also be useful in the improvement of the thermal resistance of cement and concrete. A study conducted at the School of Mechanical and Building Sciences (SMBS) in Chennai, India looked into the thermal characteristics of cenosphere-filled ceramics. In this study, both fly ash and expanded glass cenospheres were added to ceramic materials, and thermal properties were evaluated. In both cases, the cenospheres provided better heat resistance and posed better thermal characteristics than the ceramics with no additives. It was found that the presence of the cenospheres prevented heat from entering the ceramic material. (Balaji, Sasikumar, & Elayaperumal). Comparing the results of cenosphere based mix designs with RHA based mix designs will give insight into the effect of RHA as a thermally resistant additive.

![Figure 2.2: A sample of cenospheres used in this experiment.](image)
Thermal conductivity and thermal resistance are two related concepts of heat transfer through a material. Thermal conductivity relates heat flowing through a sample to the temperature of the sample on opposite sides. Thermal conductivity helps to justify what a certain material feels cooler to the touch than another material. For example, concrete feels cool to the touch on a cool day, while wood does not feel as cold. This difference occurs because of the difference in thermal conductivity of the two materials. Concrete transfers heat faster than other materials, so on a cold day it will absorb heat from one's hand faster than another material (What is Thermal Conductivity?). Thermal resistance on the other hand is the ability of a material to resist heat flow. Materials with a higher thermal resistance are more apt to remain at the same temperature when exposed to a change in temperature (Thermal Resistance & Thermal Conductance). If thermocouples are attached to opposite sides of a sample, both the thermal conductivity and thermal resistance of the sample can be calculated.

In some cases, providing insulation on concrete structures has been proven to be beneficial to the structural integrity of the concrete. The insulation is able to protect the concrete structure from damage due to high temperatures. A normal-weight concrete surface will reach roughly 90% of the temperature of a fire after approximately 30 minutes (SFPE Handbook of Fire Protection Engineering). If an insulative additive can be added to a concrete structure, it could greatly improve the resistivity of the material. The structure would be more able to resist the change in temperature due to a fire condition. This improved heat resistance would reduce the need for extra insulation protecting the concrete structure, while still providing a similar level of heat protection.

RHA and cenospheres have the potential to become useful, heat-resistant building materials in the construction industry due to their thermal properties. The goal of this project is to compare mixes with RHA to those with cenospheres and understand how they perform thermally as additives in cement.
3.0 Methodology

The goal of this project was to investigate the thermal properties of cement mixtures with the addition of RHA or cenospheres. Five unique mix designs were created to capture a spectrum of possible cementitious mixes. Two mixes contained RHA in two different quantities, 10% and 30%, two other mixes contained cenospheres in the same quantities, and the final mix was a control mix of pure OPC mortar. These five mixes were created, and allowed to set for a full 28-day cure time to ensure they were fully functional for thermal and chemical testing.

Three parameters were altered that may have led to significant changes in thermal properties and chemical makeup. These variables included the percentage of RHA in the mixture, the temperature at which the RHA was burned, and the water to cement ratio. Each of these variables was assigned based on previous research and experiments to produce two unique mixes that would capture two different levels of strength and thermal characteristics. (Table 3.1).

Table 3.1: Makeup of five unique mix designs for testing.

<table>
<thead>
<tr>
<th>Mix Design</th>
<th>Percentage</th>
<th>W:C</th>
<th>Additive</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10%</td>
<td>0.35</td>
<td>RHA burned at 650°C</td>
</tr>
<tr>
<td>2</td>
<td>10%</td>
<td>0.4</td>
<td>Cenospheres</td>
</tr>
<tr>
<td>3</td>
<td>30%</td>
<td>0.4</td>
<td>RHA burned at 650°C</td>
</tr>
<tr>
<td>4</td>
<td>30%</td>
<td>0.35</td>
<td>Cenospheres</td>
</tr>
<tr>
<td>5</td>
<td>0%</td>
<td>0.35</td>
<td>None (OPC)</td>
</tr>
</tbody>
</table>

The values of the individual constraints were determined in a strategic way. In terms of percent substitution of RHA, two separate values were set; the first value was set at 30% and the second value was set at 10%. Past research shows that roughly 30% is the maximum percentage of RHA that can be substituted for cement powder before compressive strength is significantly lowered (Nair, Sivaraman, Thomas). The 10% value arose from the need for a significant difference in RHA substitution. Creating a small difference in RHA content would not show an overall change in thermal or strength properties, but this gap of 20% provided better results in determining the impact of adding RHA. The RHA was burned for an extended period of time ranging from six to eight hours, to ensure that the RHA had a uniform structure. Water to cement ratios for the RHA mixes varied between 0.35 and 0.4. These two values are essentially the minimum and maximum ratios to get a 'standard' quality of cement.
Alongside the RHA mixtures, two mix designs were created with the use of expanded glass cenospheres. Similar to the RHA mixtures, the cenospheres were used as a cement powder substitute. To maintain continuity in the experiment, cenospheres were added at 10% and 30% substitution for the two individual mixes. Temperature was not a factor, as the cenospheres came pre-packaged from Poraver. The mix designs used spheres ranging from 1-2 micrometers (3.9x10^{-5} - 7.8x10^{-5} in.) (as noted on the packaging from the manufacturer), having a density of roughly 0.26 g/cm³ (0.01 lb/in³). The cement used in this experiment came in 96-pound (43.54 kg.) bags of ordinary portland cement powder from a local hardware store. Aggregate came from a local quarry and contained rocks and pebbles of various sizes that were sieved for each design. Prior to sieving, the aggregate was baked in a furnace to ensure that it was dry and no extra water was present. The rice husks used in this study were shipped in a prepackaged bag from Nigeria.

Each mix was created and poured into various molds for testing. Molds consisted of 2 in. x 4 in. (5.08 cm. x 10.16 cm.) cylinders, 2 in. x 2 in. (5.08 cm. x 5.08 cm.) cubes, and a 7.87 in. x 7.87 in. x 1.97 in. (10 cm. x 10 cm. x 5 cm.) block. The five mix designs were created by combining quarry sand, cement powder, water, and substituted additive (if applicable) according to their weights. A summary of each mix design can be found in Appendix A.

The bottom row of the table found in Appendix A represents the amount of additive that was substituted for cement powder in each mix design. Mix designs 1 and 3 both utilized the substitution of RHA, while mix designs 2 and 4 used the expanded glass cenospheres. The substituted additive was determined by volume, then by mass. For the 10% substitutions, 1/10th of the required cement, by volume, was removed from the mix. This volume of cement powder was then replaced by the same volume of additive. As volume can be difficult to precisely measure, specific gravity and density of the two additives were used to calculate the mass associated with the replacement volume. For example, RHA was determined to have a specific gravity of 2.075 based on two studies (Nair; Habeeb, Mahmud). These two studies proposed the specific gravity of RHA to be between 2.11 and 2.04. Taking an average of these two specific gravities gives the value of 2.075, used for this experiment. The density of the RHA was determined using the calculated specific gravity volume, multiplied by the density of water at 16.89°C (62.4°F). With a density of 1 g/cm³ (0.04 lb/in³), the density of water was multiplied by the RHA specific gravity of 2.075 to get a RHA density of 2.075 g/cm³ (0.075 lb./in.³). Multiplying the density of RHA by the volume of RHA to substituted, the mass of RHA to be added in replacement of cement powder was determined.
Similar to RHA, the mass of cenospheres to replace a certain volume of cement powder was also determined using densities. From the manufacturer’s technical data sheets, the density of expanded glass cenospheres was determined to be 0.26 g/cm³ (0.01 lb./in.³) (Poraver). Taking 10% and 30% of the cement volume, and multiplying them by the density of the cenospheres, outputted the mass of cenospheres required to replace the volume of cement powder in each mix.

Distilled water was used during mixing to minimize the possibility of outside elements in the municipal water contaminating the samples. Sand was sifted in the lab using varying sieve sizes (#4, #8, #16, #30, #50, and #100), weighed separately in buckets, and then added to the cement mixing bowl. Cement powder was also weighed then added directly to the mixing bowl along with the sieved sand. Half of the dry mix was taken out of the bowl to provide easier mixing of the mortar. Approximately half the allotted weight of distilled water was added to the mixing bowl then the remaining dry ingredients and water were slowly added into the bowl. The addition of water and dry ingredients continued until the correct weights required by the mix design were added, then the batch was allowed to mix evenly for approximately 5 minutes. The cementitious mixture was poured into the various molds. The molds were filled halfway then tamped down with a rod to minimize the amount of air bubbles. More mortar was then added to the molds until they were full then they were tamped down again. This process was done to fill 14 cylinder molds, three 2 in. x 2 in. (5.08 cm. x 5.08 cm.) blocks, and one 10 cm. x 10 cm. x 5 cm. (7.87 in. x 7.87 in. x 1.97 in.) block. Compressive testing occurred at the 7 and 28 day marks using three cylinders per test. Three cylinders provide for an average compressive strength for the mix at each test point. After the 28-day mark, tensile testing of cylinders was conducted, as well as chemical makeup tests and the calorimetry tests.

The batches of rice husk were burned at 650°C for a minimum of six hours. Various pans were tested to burn the husk, and after multiple trials two steel pans were selected. Aluminum pans deformed and melted at the high temperatures, but the steel pans were able to withstand the elevated temperatures in the furnace. The husks were left in the furnace after burning, allowing them to slowly cool to a temperature that was tolerable to handle. The first batches were weighed prior to incineration, and the husk was found to have an 80% mass loss after burning at 650°C. The first batch of husk weighed roughly 465 grams (1.03 lbs.) before being burned in the furnace overnight. After the husk cooled, it weighed just under 90 grams (0.20 lbs.). The proceeding batches of RHA followed a similar pattern of mass loss, averaging roughly 80% loss. After enough rice husk was burned, mix designs 1 and 3 were created with the incinerated RHA. Mix designs 2 and 4 were created using the expanded glass cenospheres after mix designs 1 and 3 were created. With only one set of molds, the mixes had to be created on a staggered schedule.
3.1 Compression & Tensile Tests

Compressive strength tests were conducted on 2 in. x 4 in. (5 cm. x 10 cm.) cylindrical samples of each mix design after curing for 7 and 28 days in a curing chamber with 100% humidity per ASTM C39. A tensile test of each sample mix was conducted after a curing period of 28 days. The tensile testing was conducted using the same methods as a standard compression test, except the sample was laid on its side, and an adaptive formula was used:

\[ T = \frac{2P}{\pi DL} \]

This equation relates P, the applied load; D, the diameter of the sample; and L, the length of the cylindrical sample to compute T, the tensile strength of the samples.

3.2 Fourier Transform Infrared Spectroscopy (FTIR) & Powder X-Ray Diffraction (pXRD) Tests

Two more tests that were conducted, Fourier Transform Infrared Spectroscopy (FTIR) and Powder X-Ray Diffraction (pXRD), tested the chemical makeup of the samples. If there were any major variances in strength or thermal resistivity/conductivity throughout the other tests, understanding the chemistry of the samples, more importantly the bonds and R-groups, might give more insight as to what was causing these variances, more so than just knowing the different RHA/cenosphere percentages and water-cement ratios.

In the spectrometer, in the case of this project a Bruker Vertex 70 instrument with Specac Golden Gate ATR accessory, an IR beam passes through a beamsplitter and diffracts on to two different mirrors; one at a stationary distance and the other which changes its position throughout the test. As the diffracted beams bounce off their respective mirrors, they recombine at the beam splitter and then proceed to pass through the sample. The ability of the one mirror to move and change position allows the “spectrum” to be created. As the beams recombine, they create an interferogram (time vs. intensity) of constructive and destructive interference. The infrared beam then passes through the sample, and as it absorbs the interference, the software subtracts from the interferogram, and the differences are reported. The computer then runs these differences through the Fourier transform equation:

\[ A(r) = \Sigma X(k)e^{-2\pi ikr/N} \]
In this equation, \( A(r) \) is the frequency domain point and \( X(k) \) is the time domain point of a number of \( N \) spectral points, and gives the data necessary to convert the interferogram to an intensity vs. frequency graph as seen in Figure 3.2.1. The peaks shown at specific wavelengths on this graph correlate to specific R-groups known over the spectrum, and allow the tester to see what is in the tested sample.

Most FTIR spectrometers will utilize the above equation automatically and plot the data simultaneously. For this procedure, the OPUS software integrated into the Bruker device was used. In the software, the result spectrum was set to absorbance, or “peaks up”. The beam path was set to be continuous from source to detector and the amplitude of the signal was between 1500-1600 units. The device was setup to run 256 scans over a wavelength of 600-4500 cm\(^{-1}\). Typically, the device runs 1,024 scans, and takes about 15 minutes. Since most of the chemical components of the samples were known from a previous “Materials of Construction” course at WPI, Andy Butler, the Associate Director of the Life Sciences and Biochemistry Department, assured that the test could be run over fewer scans to save time. The test was cut down to about 3.5 minutes, since the main observations being gathered from this test were to see if there were any “outliers” in the spectrum that didn’t show up in a typical cementitious mixture.

A background scan must be run before any sample is placed onto the testing crystal (what the sample rests on while being tested). This ensures that any obstructions are being accounted for when testing the actual sample. After this was done, the 2 in. x 4 in. (5.1 cm. x 10.2 cm.) cylinder of concrete being tested was ground with a mortar and pestle into a fine powder. This was then scooped onto the testing crystal until the pile was about 5 mm (0.2 in.) high to assure there was enough to cover the entire crystal surface. The anvil of the gate attachment was then lowered and clamped into place, the compartment lid was closed, and the test was executed. After this test finished, the surface was wiped off with an acetone-soaked Kimwipe to assure that no traces of this sample were left to contaminate the next test. This process was then repeated for each of the samples being tested.

![Figure 3.2.1: General interferogram showing light intensity vs. mirror position (Herschel).](image)
Powder X-ray diffraction (XRD) is a technique used for identifying crystalline structure within a material. To be tested, each mix was ground into a powder and placed on a flat 1 in (2.5 cm.) diameter plate. The machine used for this test had two x-ray devices; one sent an x-ray beam at the powder sample, while the other served as a receiver for the x-ray beams. The x-ray device moved through a spectrum of angles ranging from 10° to 42° in 0.05° increments, with each angle being held for 2 seconds. Each test cycled for a total of 21 minutes. The moving x-ray device continuously sent x-rays through the powder sample, which diffracted as they hit crystalline structures within the powder. If the beam bounced off of the sample and was received at the same intensity, this indicated that there was no crystalline structure present. Whereas if the x-ray was shot at the sample and the beam was received at a higher intensity, meaning it was diffracted, then this indicated that there is a crystalline structure present.

Where there existed a crystalline structure, the x-ray beams entered the receiver at a different frequency that correlated with specific crystalline structures at various angles. These diffracted X-rays were then detected, processed and counted. "By scanning the sample through a range of angles, all possible diffraction directions of the lattice are obtained" (Dutrow). After the program calculated the number of diffracted X-rays, a graph was generated, similar to Figure 3.2.2. The peaks indicate at which angle a crystalline structure was found.

![XRD graph of Zinc Oxide](image)

Figure 3.2.2: XRD graph of Zinc Oxide, showing crystalline peaks (Akhtar, et. al.)
3.3 Cone Calorimetry Test

In order to evaluate the thermal conductivity and resistance of the various cementitious materials, the 10 cm. x 10 cm. x 5 cm. (7.87 in. x 7.87 in. x 1.97 in.) samples were exposed to an average heat flux of 50 kW/m² (15,845 Btu/hr/ft²) in the fire lab at WPI in accordance with the provisions of ASTM E1354. ASTM E1354 calls for a 60-minute test duration, but in the case of samples that are not expected to ignite a 30-minute duration is acceptable. One face of the cement sample was exposed to this 50 kW/m² (15,845 Btu/hr/ft²) flux for a duration of 30 minutes to simulate an environment in which a concrete wall would be exposed to one dimensional heat flow, such as a foundation wall experiencing a fire inside of a basement. To simulate more realistic situations, the cone calorimeter is programmed to output an average heat flux, so the actual heat flux at any point varies by roughly 5 kW/m² (1,585 Btu/hr/ft²). In the case of this project, the cone calorimeter heat flux ranged from 45-55 kW/m² (14,265 - 17,435 Btu/hr/ft²).

The samples were wrapped with aluminum foil on all sides except the top, which was directly exposed to the heat flux. The reflective surface of the aluminum foil was able to direct any radiant heat away from the sides and bottom of the samples to ensure one-dimensional heat flow through the top of the sample. The samples were then placed into a metal frame under the cone calorimeter for the 30-minute duration of the test. Thermocouples were attached to two faces of the cement samples; one on the side exposed directly to the heat flux, and one on the opposite side with no direct exposure. The temperature was recorded every second in both thermocouples throughout the 30-minute duration of the test. Sample masses were measured both before and after exposure to the heat flux to record the percent loss of mass due to the heating. A diagram of the cone calorimeter set up can be seen in Figure 3.3.1.

![Diagram of the cone calorimeter test set-up.](image)

Figure 3.3.1: Diagram of the cone calorimeter test set-up.
3.4 Cold Plate Test

2 in. x 2 in. (5.08 cm. x 5.08 cm.) cubes were created for thermal conductivity testing using a guarded longitudinal calorimeter apparatus in accordance with ASTM C177. The samples were placed between two Pyroceram blocks known as 'meter bars'. These Pyroceram blocks have a known thermal conductivity that was used to calculate the thermal conductivity of the cementitious samples. The mortar samples and Pyroceram blocks were arranged into a stack with thermocouples placed between each layer. The stack was then inserted into a block of foam insulation that was placed on top of the cold plate apparatus. The program used for this series of experiments was an eight-step model, with equivalent temperature decreases over a period of six hours. The temperature of the sample continuously dropped from 25°C (77°F) for three hours until it reached 0°C (32°F); after which the temperature was increased for three hours until the sample reached 25°C (77°F) again. Throughout this six-hour test duration, 65 temperature readings from each thermocouple were recorded. A sample diagram of the cold plate apparatus can be seen in Figure 3.4.1:

![Diagram of the cold plate apparatus used for testing.](image)
4.0 Results

4.1 Compression & Tensile Results

The compressive strengths of the various mixtures varied with the amount of substituted materials. The values for compressive strength are necessary for any construction purposes to ensure stability in a structure. The average maximum stress for all mixtures are reported in Figure 4.1.1. Ideally, the longer a cement sample cures, the higher its compressive strength should be due to the more calcium silicate hydrate (C-S-H) bonds that forms in the hydration process.

![Average Compressive Strength](image)

Figure 4.1.1: Average Compressive Strength of 2”x4” Cylinders

The OPC mixture averaged a maximum compressive strength of 5,153 psi (36 MPa) with a standard deviation of 499 psi after seven days of curing, which was the highest compressive strength. The weakest seven-day compressive day strength was the 30% RHA mix. This low strength could be due to the water content within the 30% RHA mixture, which used the most water in the mixing process. The 10% RHA and 10% Cenosphere samples had much less strength than the control OPC sample. The 30% Cenosphere mix had nearly the same strength as the control OPC sample. These data suggest that after 7 days the substitution of cement powder with other materials provides no mechanical advantage.

The 28-day compressive strength test indicates the highest strength for concrete and, therefore, used for determining strength in design projects. The details for the three samples per each mix design can be found in Appendix B. The sample with the maximum compressive strength was the 10% RHA mix that failed at
an average of 5,723 psi (39 MPa). RHA and cenospheres are pozzolans: materials with silicates and aluminates that, when reacted with cementitious material, form C-S-H. The silica in pozzolans react with the calcium hydroxide from the portland cement. This could explain why the substitution of 10% of cement powder with rice husk ash was stronger after allowing the reactions to occur over 28 days. The 30% RHA might be the weakest because there was too much reactive silicate with not enough calcium hydroxide (Ca(OH)$_2$) to create the strong bonds in cement. The 30% cenosphere mixture was stronger than the 10%, with strengths at 4,577 psi (32 MPa) and 4,156 psi (29 MPa), respectively. Both cenosphere samples were weaker than the OPC sample, suggesting that the substitution of this pozzolanic material adds no benefit to the strength of cement when compared to the control OPC. Although the OPC mixture was the strongest for the 7-day strength test, it was weaker at 28 days. An explanation for this could be that two batches were made for OPC since the first batch was not tested at 7-days as originally planned. This weakness at the 28-day mark could also be from the mixing process. If the samples were not homogenous, there could be spaces of reactive silicate that did not bond with the necessary chemicals to form the bonds necessary for adhesion and leaving weak spots for cracking.

The tensile strength of the cylindrical samples indicates the maximum tensile strength this construction material can withstand. Figure 4.1.2 shows the average tensile stress at which the sample failed. The 30% cenosphere mix was the strongest sample with a maximum tensile stress of 455 psi (3 MPa) which was greater than the control OPC sample. There was only one sample crushed per tensile test therefore there was no standard deviation to show. The remaining samples crushed under a smaller loading than the control sample, as shown in Figure 4.1.2.
4.2 Fourier Transform Infrared Spectroscopy (FTIR) & Powder X-Ray Diffraction (pXRD) Results

After each sample was run through the Bruker testing device, the data taken by the OPUS software were exported and a graph for each sample was generated. All graphs were combined as seen in Figure 4.2.1 at the same scale. This allows the individual graphs to be stacked on top of each other in order to better compare them.

Figure 4.2.1: FTIR results for the five cement mixtures.
Each peak in the graph correlates to a specific chemical bond found in the sample at a specific wavelength. Some of these wavelengths and their associated bonds are shown in Table 4.2.2:

<table>
<thead>
<tr>
<th>Location (cm⁻¹)</th>
<th>Origin</th>
</tr>
</thead>
<tbody>
<tr>
<td>3650</td>
<td>Hydrated Minerals (i.e. Ca(OH)_2)</td>
</tr>
<tr>
<td>3600-3100</td>
<td>S-O (Gypsum)</td>
</tr>
<tr>
<td>3400</td>
<td>OH Stretching (H_2O)</td>
</tr>
<tr>
<td>2930,2850</td>
<td>Calcite Harmonic; S-O (Gypsum)</td>
</tr>
<tr>
<td>1650</td>
<td>H-O-H Bending (H_2O)</td>
</tr>
<tr>
<td>1430</td>
<td>C-O Asymmetric Stretching; S-O (Gypsum)</td>
</tr>
<tr>
<td>1100</td>
<td>Si-O-Si and Al-O-Si Asymmetric Stretching</td>
</tr>
<tr>
<td>1035-1030</td>
<td>'aluminosilicate bonding'</td>
</tr>
<tr>
<td>1010-1000</td>
<td>Calcium Silicates</td>
</tr>
<tr>
<td>960-800</td>
<td>Si-O, Al-O Stretching</td>
</tr>
<tr>
<td>872</td>
<td>C-O Bending</td>
</tr>
<tr>
<td>480</td>
<td>Si-O-Si and O-Si-O Bending</td>
</tr>
</tbody>
</table>

From Table 4.2.2, it was determined that each sample contained OH (hydroxide) stretching around 3400 cm⁻¹, and either C-O asymmetric stretching/gypsum around 1430 cm⁻¹. A third major peak commonly found in all samples was either aluminosilicate bonding or the presence of calcium silicates around 1020 cm⁻¹.

In the 30% RHA sample, the larger peak at the 3400 cm⁻¹ mark indicates that this sample contains more OH stretching than the rest of the samples. It also contains more C-O asymmetric stretching/gypsum because of its larger peak around 1430 cm⁻¹. The mixtures containing the cenospheres had lower peaks at the 1020 cm⁻¹ mark. This is likely caused by cenospheres being hollow being an “empty space”. Since cement has aluminosilicates present, the volume of the concrete taken up by the hollow cenospheres means that there is less cementitious product in the concrete, and therefore less abundance of the aluminosilicate group.

The graphed XRD results are located in Appendix C. If crystalline structures were present in any of the samples, large peaks would have showed up on the computer-generated graphs, as seen previously in Figure 3.2.2. The graphs generated by these tests had no definitive peaks but many erratic ones, which were confirmed to be simply due to mechanical noise. If the X-ray graphs were to show sharp, distinct peaks, they would indicate crystals in the sample. The lack of major peaks can be explained by the lack of crystalline structures in the concrete samples, which is backed by the FTIR results. This showed that the addition of RHA to the cement mixture did not create any new phases in the samples.
4.3 Cone Calorimetry Results

Thermal conductivity relates heat flowing through a sample to the temperature at each surface, while thermal resistance displays a material's ability to resist heat flow. The thermal conductivity and thermal resistance of each sample were calculated from the results of the cone calorimetry test, using:

\[ K = q \left( \frac{L}{\Delta T} \right) \]

Thermal conductivity \([K]\) relates the heat flux \([q, \text{[w/m}^2; \text{BTU/hr/ft}^2]\)] to the thickness of the material \([L, \text{[m; ft.]}]\) and the change in temperature \([T, \text{[K;°F]}]\) of the material. The formula for thermal resistance \([R]\) relates the thickness of the material \([L, \text{[m; ft.]}]\) to the thermal conductivity \([K, \text{[w/m•K; BTU/h•ft•°F]}]\). All samples yielded similar values in terms of average thermal conductivity and average thermal resistance as shown in Table 4.3.1.

Table 4.3.1: Average thermal conductivity and average thermal resistance of each cementitious sample.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Average Thermal Conductivity [W/m•K]</th>
<th>Average Thermal Conductivity [BTU/h•ft•°F]</th>
<th>Average Thermal Resistance [R] [k•m²/W]</th>
<th>Average Thermal Resistance [ft.²•°F•h/BTU]</th>
</tr>
</thead>
<tbody>
<tr>
<td>10% RHA</td>
<td>7.2</td>
<td>4.2</td>
<td>0.0071</td>
<td>0.0400</td>
</tr>
<tr>
<td>30% RHA</td>
<td>8.4</td>
<td>4.8</td>
<td>0.0062</td>
<td>0.0351</td>
</tr>
<tr>
<td>10% Cenospheres</td>
<td>7.7</td>
<td>4.4</td>
<td>0.0070</td>
<td>0.0395</td>
</tr>
<tr>
<td>30% Cenospheres</td>
<td>8.2</td>
<td>4.8</td>
<td>0.0063</td>
<td>0.0358</td>
</tr>
<tr>
<td>OPC</td>
<td>8.4</td>
<td>4.9</td>
<td>0.0061</td>
<td>0.0346</td>
</tr>
</tbody>
</table>

The OPC mixture had the highest thermal conductivity, while the 10% RHA mixture had the lowest. The higher thermal conductivity value correlates to a less insulative substance. A higher thermal conductivity means that the sample more readily transfers heat to its surroundings. The average thermal conductivity among the five samples was close to 8 W/m•K (4.6 BTU/h•ft•°F). For non-metallic solids, the average thermal conductivity typically ranges from 0.5-50 W/m•K (0.29-28.9 BTU/h•ft•°F) (SFPE Handbook of Fire Protection Engineering). The mortar samples fell within this typical window. The other column of Table 4.3.1 depicts the average thermal resistance of the samples. These values fell between 0.0061-0.0071 K•m²/W (0.035-0.040 ft²•°F•h/BTU). Thermal resistance varies with the thickness and density of the concrete. As the samples were all the same thickness, they can be evenly compared. Thermal resistance calculations output a material's 'R-value', which is its capacity to resist heat flow. The higher R-value a material has, the better it will resist heat flow, and the better it will serve as an insulator. As all the thermal resistances shown in Table 4.3.1 are relatively the same, and all small, none of the samples are standout.
insulators. A standout insulator would have a notably higher R-value, such as a one inch (2.5 cm.) block of polyurethane foam, which has an R-value of 6.25 ft²•°F•h/BTU (1.1 K•m²/W) (*R-Values of Insulation and Other Building Materials*).

Observations were made throughout the duration of the cone calorimetry test. The first sample tested was the OPC mixture. After a few minutes of heat exposure, there was noticeable popping and spalling from the sample. This was likely due to rapid water evaporation from the mix. The excess water in the mix transforms into steam and expands outward from the inside of the sample. After the conclusion of the test, there was a faint color gradient from the top of the sample, likely caused from the heat exposure. The side exposed to the heat flux had a slightly darker gray/brown color that faded into a light gray color that was typical for the rest of the sample. The bottom of the sample had noticeable dull yellow spots on it, which were likely from the decomposition of calcium sulfate (CaSO₄) in the mix.

After the OPC was tested, the 10% RHA sample was prepared for the cone calorimeter. Once the heat flux was initiated, similar popping to that of the OPC sample occurred. Unlike the OPC sample, the 10% RHA sample formed craters and cavities where the popping occurred. This left a non-uniform surface on the top of the sample that was exposed directly to the heat flux. After the 30-minute duration of the test, a more clear color gradient was evident in the 10% RHA sample. The gradient was similar to that of the OPC mix, with a darker gray/brown color on the side exposed to the heat flux and a lighter gray on the sides that were not directly exposed to the heat flux. Though similar to the OPC, the 10% RHA sample had more intense brown/gray coloring, and brighter white/light gray coloring. Again, a faint yellow color was found on the bottom of the sample in small patches that were likely due to the decomposition of calcium sulfate (CaSO₄) in the mix.

The 30% RHA, 10% cenosphere, and 30% cenosphere samples all had similar reactions during the cone calorimeter test. In all three samples, no popping was observed as in the OPC and 10% RHA samples. The color gradient in these three samples was not very evident, and was most similar to that of the OPC mix. There was also no trace of the faint yellow substance that appeared on the OPC and 10% RHA samples. The lack of spalling in these samples could have arisen from the addition of RHA and cenospheres. The empty space within the cenospheres could have served as a space for steam to be held while the excess water in the samples evaporated. The steam could enter the small cavities within the cenospheres as opposed to reaching the surface of the sample. Similarly, the porous RHA could create space for steam to settle during the testing. The higher percentage of RHA would provide more space for the steam to travel to, as
opposed to the 10% RHA sample that experienced spalling. Both samples containing cenospheres did not experience spalling, showing that the steam entering the small cavities is a plausible deduction.

Before each sample was inserted into the cone calorimeter, it was weighed to determine the mass loss after exposure to the heat flux. Mass losses were likely caused by evaporation of water from the samples, as they had been stored in the curing room with 100% humidity until the day of the test. The spalling from the OPC and 10% RHA removed small portions of samples that also reduced the mass of the samples during testing. Table 4.3.2 depicts the percent loss of the samples before and after the cone calorimetry test.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sample Mass Before Burning (g)</th>
<th>Sample Mass After Burning (g)</th>
<th>Percent Loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPC</td>
<td>852.5</td>
<td>789.0</td>
<td>7.4%</td>
</tr>
<tr>
<td>RHA 10%</td>
<td>856.0</td>
<td>796.0</td>
<td>8.1%</td>
</tr>
<tr>
<td>RHA 30%</td>
<td>716.5</td>
<td>639.0</td>
<td>10.8%</td>
</tr>
<tr>
<td>Cenosphere 10%</td>
<td>819.0</td>
<td>757.0</td>
<td>7.6%</td>
</tr>
<tr>
<td>Cenosphere 30%</td>
<td>816.0</td>
<td>756.0</td>
<td>7.4%</td>
</tr>
</tbody>
</table>

Graphs displaying the thermocouple data captured during testing can be found in Appendix D.

4.4 Cold Plate Results

In order to calculate the thermal conductivity of the cement samples, it was first necessary to calculate the heat flow through both Pyroceram meter blocks using the equation (Sharifi et al.):

$$Q_{xy} = \frac{A(\lambda_{xy})(T_x - T_y)}{d_{meter}}$$

The above equation relates A, the cross-sectional area of the meter block; \( \lambda \), the thermal conductivity of the meter bar; \( T_x \) and \( T_y \), the temperatures recorded by the respective thermocouples; and \( d_{meter} \), the thickness of the meter bar. The cross-sectional area of the Pyroceram blocks was known to be 4 in.\(^2\) (0.00258 m\(^2\)). Similarly, the thickness of the meter bars was known to be 1 in. (0.00254 m). For the blocks of Pyroceram, the thermal conductivity was determined to be 4.18 W/m•K (2.42 BTU/h•ft•°F) (Sharifi et al.). After the heat flow of both Pyroceram blocks was determined, the thermal conductivity of the sample could be determined using (Sharifi et al.):
The thermal conductivity formula above relates $Q_{1-2}$ and $Q_{3-4}$, the heat flow through each Pyroceram block; $d_{\text{sample}}$, the thickness of the cement sample; $A$, the cross-sectional area of the sample; and $T_3$ and $T_2$, the temperature at the corresponding thermocouple. Graphs of the thermal conductivity of each sample were created using the above formula. The graphs show 'noise' in the first and last 10 data points as the temperature gradient stabilized. The graphs of each sample flattened out at their respective steady state thermal conductivities as seen in Figure 4.4.1. Individual sample graphs can be seen in Appendix E.

Figure 4.4.1: Thermal conductivities of each sample from the cold plate test. A) Stacked plot of thermal conductivities. B) Plot isolating the steady state region of each sample’s thermal conductivity.
As described above, the flatter portion of Figure 4.4.1 shows the point at which the samples reached a point of steady state thermal conductivity. Each sample is represented with a different color on the graph and each sample had relatively similar thermal conductivities, summarized in Table 4.4.1.

Table 4.4.1: Summary table of thermal conductivities from the cold plate testing.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thermal Conductivity [W/m•K]</th>
<th>Thermal Conductivity [BTU/h•ft.•°F]</th>
</tr>
</thead>
<tbody>
<tr>
<td>10% RHA</td>
<td>2.0</td>
<td>1.2</td>
</tr>
<tr>
<td>30% RHA</td>
<td>1.6</td>
<td>0.9</td>
</tr>
<tr>
<td>10% Cenospheres</td>
<td>1.9</td>
<td>1.1</td>
</tr>
<tr>
<td>30% Cenospheres</td>
<td>2.6</td>
<td>1.5</td>
</tr>
<tr>
<td>OPC</td>
<td>2.9</td>
<td>1.7</td>
</tr>
</tbody>
</table>

As seen in the above table, the OPC sample had the highest thermal conductivity at approximately 2.9 W/m•K (1.67 BTU/h•ft.•°F), while the 30% RHA sample had the lowest thermal conductivity at roughly 1.6 W/m•K (0.93 BTU/h•ft.•°F). The 30% RHA sample was therefore the most insulative, while the OPC sample was the least insulative. The RHA and cenosphere samples proved to have lower thermal conductivities than the OPC mixture, showing that the additives impacted the insulative qualities of the mix. The RHA in the samples could improve conductivity by storing warmer air in the porous ash. As the rest of the sample cooled down, the room-temperature air in the pores of the RHA could slowly release the warmer air into the rest of the sample. This would slow the overall cooling process of the RHA mixtures, meaning they were more resistant to temperature changes. As the RHA is a pre-burned substance, it is likely to be less susceptible to temperature changes in general. The substance, being so porous, is made up of mostly air. As the temperature in the sample changes, the RHA would not be as impacted by the change. The 30% RHA samples had the lowest thermal conductivity, which may indicate that a larger percentage of RHA could provide even better insulative qualities. Similarly, the cenospheres could contain warm air that the sample was originally held in prior to testing. As the cold plate test ran, the warm air in the cenospheres would slow the cooling process of the sample, demonstrating more insulative properties of the mix designs.
Conclusions/Recommendations

It was found that the addition of RHA or cenospheres to OPC slightly improved the thermal conductivity, but reduced the compressive strength of the mix. If the cementitious materials used in this study were implemented in real structures, certain catastrophes could potentially be avoided. For example, pre-stressed concrete bridges become structurally unstable in fire scenarios. Their compressive and shear strength drastically decrease at temperatures greater than 700°C (Wright et al.). If a more insulative cementitious mix were to be used in the bridge design, such as a mixture containing RHA or cenospheres investigated in this project, the structure would be more apt to resist the transfer of heat from a potential fire. The higher resistance to the fire temperatures would lead to less temperature change within the structure. By slowing the heating process, firefighting teams would have more time to battle the blaze, and extinguish the fire before the structure heated to a point where the compressive and shear strengths would be significantly impaired.

The use of RHA in more designs for structures would also resolve the wasteful nature of rice husks. Currently, the production of rice generates nearly 770 million tons of husk in Asian nations alone (Santiaguel). The rice husk used in this project experienced a mass loss of approximately 80%, meaning more husk had to be burned to achieve a higher mass of RHA. Substituting a small percentage of RHA for cement powder in a large structure, such as a foundation wall or a bridge, would greatly reduce the amount of rice husk waste found throughout the world.

In order to further develop the cementitious materials investigated within this project, research should be conducted with RHA burned at a higher temperature and compression, FTIR, and XRD tests should be performed on the samples exposed to the cone calorimeter. Similarly, testing should be performed on other percentages of substituted additive, as this project investigated only 10% and 30% substitution of both RHA and cenospheres.

RHA has been determined to develop a crystalline structure when burned at roughly 850°C (1562°F) (Xiong), but the facilities used for this project did not have the capability of burning the husks at this high of a temperature within the timeframe of the project. At this elevated temperature, the RHA should develop a more crystalline structure, which could improve the strength characteristics of each cementitious mixture with RHA substituted. The crystalline structure may provide more resistance to compressive stresses, and could also serve as a better medium to resist heat transfer, but would be less likely to react with Portlandite to form C-S-H. Running similar tests with RHA burned at a higher temperature could yield different
strength and thermal properties of the cementitious mixtures. The 850°C (1562°F) RHA would be better for the XRD testing as well, because more crystalline structures could be present.

The FTIR and XRD tests would allow for comparison of the chemical makeup of the mixtures before and after exposure to a one-dimensional heat flow, mimicking a real-life fire situation. The cementitious samples could develop a crystalline structure under the heat flux applied during the cone calorimetry testing, which would appear in the FTIR and XRD tests. Performing compression testing on the 10 cm. x 10 cm. x 5 cm. (7.87 in. x 7.87 in. x 1.97 in.) blocks before the cone calorimetry tests would provide a baseline strength value that could be compared to the samples after their exposure to the heat flux in the cone calorimetry test. This comparison could yield vital information to changes in strength of the various mixtures after a fire scenario, leading to better conclusions about the structural stability of the mixes.
Acknowledgments

Throughout the duration of this project, we received endless advice and support from many people. We would like to extend our gratitude to everyone who played a role in this MQP, and those that made it all possible.

We would like to acknowledge those that helped us with setting up and conducting our various tests on each mix design. We would like to thank Russ Lang for assisting us with all our mix designs and ensuring that all our experiences in the lab were educational and successful. We would also like to thank Andy Butler, Daryl Johnson, and Ray Ranellone for teaching us about their respective lab procedures and the testing methods involved with them.

Most importantly we would like to thank our advisor, Aaron Sakulich, for his continued support and desire to ensure that our project was a learning experience. With his enthusiasm and drive to create a great project, our MQP experience was unmatched. His knowledge of all things concrete was very helpful throughout the duration of this project, and without his help our project would not have been possible.
Design Statement

The design problem for this project was to supplement ordinary portland cement (OPC) powder with rice husk ash (RHA) or expanded glass cenospheres to see the effects on the structural and thermal properties of the cement. Each mix contained a different percentage of OPC powder substituted with RHA and cenospheres, and a different water to cement ratio. These RHA mixtures were compared to a baseline OPC mixture, and two mix designs containing different percentages of expanded glass cenospheres. The primary testing involved compressive strength, cone calorimetry, and cold plate apparatus to determine the strength, heat release, and heat resistance of the mixtures. Additional testing was performed to determine the tensile strength, chemical makeup, chemical bonding and crystalline structure. Assembling this information allowed the proper assessment of whether or not these additives created a more efficient product.

This project originally used a 'design of experiment' method to create the five separate cement mix designs. A 'design of experiment' is a mathematical system that aids in the determining effect of individual variables on a system. The 'design of experiment' was chosen to model the cement mix designs to see the impact of adding strictly RHA in different quantities. With a properly conducted design of experiment, the optimal mix of RHA in a batch of cement could be determined to maximize the thermal properties the sample. Originally, three parameters were set to alter the properties of each mix including: the percentage of additive in the mixture, the temperature at which the rice husk was burned at, and the water:cement ratio. Each of these values was assigned a 'high' and 'low' value and the Taguchi L4 partial factorial design method was used. This method originally aided in creating four unique RHA mix designs that mathematically covered all possible combinations of the three variables outlined above. Using a full factorial model would involve the same high and low values for three variables, but would yield 8 total mixtures; \((2 \text{ values})^3 \text{ variables} = 8\) mix designs. The Taguchi L4 method uses the same mathematical strategies, but reduces the number of mix designs to only four. The fifth mix design for this project was the control mix of OPC.

After the initial burns of the rice husk, the 'design of experiment' model had to be abandoned. The equipment in the lab used to conduct the rice husk burns did not contain a furnace that could reach the 'high' temperature of 850°C (1562°F). This left only two variables for the 'design of experiment'; the quantity of RHA in the mix, and the water to cement ratio. The Taguchi L4 method could not be carried out under these restrictions.
Professional Licensure Statement

In the profession of Civil Engineering, a professional engineering (PE) license is a distinguishing feature and signifies a standout engineer at any company or firm. Each state regulates the practice of engineers through the PE license. Each state also has unique standards and characteristics associated with the PE license, so it is crucial that each candidate understand the specifics in regard to their state of application. For example, in the Commonwealth of Massachusetts, applicants are to follow the provisions of 250CMR (Code of Massachusetts Regulations for the registration of professional engineers and land surveyors). Professional licenses are regulated by an individual state’s Board of Registration of Professional Engineers to ensure that registered engineers are practicing in a safe and competent manner.

In order to obtain a civil engineering PE license, there are four steps that must first be taken. The first step is to complete a Bachelor of Science degree in civil engineering at an accredited university or from an accredited program. Once this education portion is complete, the candidate is expected to take and pass the Fundamentals of Engineering (FE) exam. With the passing of this exam comes the title of Engineer in Training (EIT). The second step in the journey to a PE license comes from professional practice under a registered professional engineer. In order to be qualified for a PE license, a candidate must work for at least four years under the supervision of another PE to gain relevant and proper experience with real world engineering solutions. Step three involves learning the individual state requirements for obtaining a PE license. For example, in the Commonwealth of Massachusetts, an applicant is required to send five letters of reference; three of which must be from other PE’s familiar with the work of the applicant, and one must be from the direct supervisor of the applicant. As each state has unique requirements, it is essential to understand what is needed for the state of application. The final step in obtaining a PE license is to study for, and take the Principles of Practice and Engineering exam. The exam takes place in two sessions, totaling eight hours. After passing the exam, the applicant can register their official Professional Engineer stamp to be able to officially approve plans and submittals. It is possible to apply for reciprocity in other states and become licensed in more areas than just the state where the exam was taken.

To maintain licensure, it is essential for engineers to continue growing in knowledge and practice. Organizations such as the National Society of Professional Engineers (NSPE) offer online workshops and information bulletins to ensure that safe and competent engineering is always being practiced. Engineers must also reapply for licensure after an allotted amount of time, as the PE license does expire.

There are numerous advantages to being a registered Professional Engineer, especially in the field of civil engineering. With a PE license, an engineer is able to legally approve and stamp plans and drawings, making
them a valuable asset to any firm. A PE license also demonstrates a sense of responsibility and knowledge of engineering concepts. A diligence to education and learning is essential to obtain a PE license, which shows good character and determination of an individual. With a PE license, engineers essentially vow to protect the public in their designs, and promote a safe and healthy environment for all.
## Appendix A: Mix Design Table

### Table A.1: Summary of Mix Designs

<table>
<thead>
<tr>
<th></th>
<th>0%</th>
<th>10%</th>
<th>20%</th>
<th>30%</th>
<th>40%</th>
<th>50%</th>
<th>60%</th>
<th>70%</th>
<th>80%</th>
<th>90%</th>
<th>100%</th>
<th>Sand</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gravel</td>
<td>0</td>
<td>24</td>
<td>48</td>
<td>72</td>
<td>96</td>
<td>120</td>
<td>144</td>
<td>168</td>
<td>192</td>
<td>216</td>
<td>240</td>
<td>216</td>
<td>1080</td>
</tr>
<tr>
<td>Water</td>
<td>1</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td>7</td>
<td>8</td>
<td>9</td>
<td>10</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>Cement</td>
<td>0</td>
<td>24</td>
<td>48</td>
<td>72</td>
<td>96</td>
<td>120</td>
<td>144</td>
<td>168</td>
<td>192</td>
<td>216</td>
<td>240</td>
<td>240</td>
<td></td>
</tr>
</tbody>
</table>

Note: The total mixture includes all components with the specified percentages.
## Appendix B: Compressive & Tensile Test Result Graphs

Table B.1: Summary table of tensile strength tests.

<table>
<thead>
<tr>
<th></th>
<th>Day</th>
<th>Peak Load (lbf)</th>
<th>Diameter (in)</th>
<th>Length (in)</th>
<th>Pi</th>
<th>Tensile Stress (lb/in²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPC</td>
<td>Post 28</td>
<td>5598</td>
<td>2</td>
<td>4</td>
<td>3.141593</td>
<td>445.47</td>
</tr>
<tr>
<td>RHA 10%</td>
<td>Post 28</td>
<td>5231</td>
<td>2</td>
<td>4</td>
<td></td>
<td>416.27</td>
</tr>
<tr>
<td>RHA 30%</td>
<td>Post 28</td>
<td>4876</td>
<td>2</td>
<td>4</td>
<td></td>
<td>388.02</td>
</tr>
<tr>
<td>Cenosphere 10%</td>
<td>Post 28</td>
<td>5209</td>
<td>2</td>
<td>4</td>
<td></td>
<td>414.52</td>
</tr>
<tr>
<td>Cenosphere 30%</td>
<td>Post 28</td>
<td>5722</td>
<td>2</td>
<td>4</td>
<td></td>
<td>455.34</td>
</tr>
</tbody>
</table>

Table B.2: Summary table of compressive strength and peak loads.

<table>
<thead>
<tr>
<th></th>
<th>Average</th>
<th>Day</th>
<th>Compressive Strength (psi)</th>
<th>Peak Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPC</td>
<td></td>
<td>7</td>
<td>5153</td>
<td>16302</td>
</tr>
<tr>
<td></td>
<td></td>
<td>28</td>
<td>5046.33</td>
<td>17464</td>
</tr>
<tr>
<td>RHA 10%</td>
<td></td>
<td>7</td>
<td>3452</td>
<td>10946.33</td>
</tr>
<tr>
<td></td>
<td></td>
<td>28</td>
<td>5723.33</td>
<td>18165.33</td>
</tr>
<tr>
<td>RHA 30%</td>
<td></td>
<td>7</td>
<td>1228</td>
<td>3888.33</td>
</tr>
<tr>
<td></td>
<td></td>
<td>28</td>
<td>2595</td>
<td>8152.33</td>
</tr>
<tr>
<td>Cenosphere 10%</td>
<td></td>
<td>7</td>
<td>4090</td>
<td>12849.67</td>
</tr>
<tr>
<td></td>
<td></td>
<td>28</td>
<td>4156</td>
<td>13056.33</td>
</tr>
<tr>
<td>Cenosphere 30%</td>
<td></td>
<td>7</td>
<td>5062</td>
<td>15902.33</td>
</tr>
<tr>
<td></td>
<td></td>
<td>28</td>
<td>4576.67</td>
<td>14378</td>
</tr>
</tbody>
</table>
Figure B.1: OPC 7-day compressive strength.

Figure B.2: OPC 28-day compressive strength.
Figure B.3: 10% RHA 7-day compressive strength graph.

Figure B.4: 10% RHA 28-day compressive strength graph.
Figure B.5: 30% RHA 7-day compressive strength.

Figure B.6: 30% RHA 28-day compressive strength.
Figure B.7: 10% Cenosphere 7-day compressive strength.

Figure B.8: 10% Cenosphere 28-day compressive strength.
Figure B.9: 30% Cenosphere 7-day compressive strength.

Figure B.10: 30% Cenosphere 28-day compressive strength graph.
Appendix C: X-ray diffraction graphs

Figure C.1: OPC x-ray diffraction graph.

Figure C.2: 10% RHA x-ray diffraction graph.
Figure C.3: 30% RHA x-ray diffraction graph.

Figure C.4: 10% Cenosphere x-ray diffraction graph.
Figure C.5: 30% Cenosphere x-ray diffraction graph.
Appendix D: Cone Calorimetry Test Graphs

Figure D.1: OPC cone calorimetry thermocouple output.

Figure D.2: 10% RHA cone calorimetry thermocouple output.
Figure D.3: 30% RHA cone calorimetry thermocouple output.

Figure D.4: 10% Cenosphere cone calorimetry thermocouple data.
Figure D.5: 30% Cenosphere cone calorimetry thermocouple output.
Appendix E: Cold Plate Thermal Conductivity Graphs

Figure E1: OPC cold plate thermal conductivity graph.

Figure E2: 10% RHA cold plate thermal conductivity graph.
Figure E3: 30% RHA cold plate thermal conductivity graph.

Figure E4: 10% Cenosphere cold plate thermal conductivity graph.
Figure E5: 30% Cenosphere cold plate thermal conductivity graph.
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