Fly-ash cenosphere/clay blended composites for impact resistant tiles

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HIGHLIGHTS

• Blended fly-ash/clay composites were explored as potential material for roofing tiles.
• Microstructure, density and damage in four types of blended composites were compared.
• Clay + fly-ash composite manifested highest specific damage resistance to impact load.
• Specific damage resistance is the degree of damage normalized with composite density.
• Fly-ash + clay reduces the density (~36%) and improves the impact response (~26%).

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ABSTRACT

In this paper, blended fly-ash cenospheres/clay composites are explored as potential material for roofing tiles. In view of impact on roofs during hailstorms, the goal is to improve the impact resistance of roofing tile materials. It is hypothesized that the impact damage resistance of tiles can be improved while reducing their densities by incorporating hollow fly-ash cenospheres with base clay. Towards that, four types of composites, namely clay, clay + filler, clay + fly-ash and clay + filler + fly-ash are fabricated and evaluated under dynamic impact loading with 1 J impact energy. Changes in microstructure, densities and degree of damage in the four types of composites are compared. The addition of fly-ash cenospheres appears to improve the structural integrity of the tiles by reducing gaps observed in clay tiles. The highest reduction in density is observed in clay + fly-ash composites of approximately 36% as compared to clay samples. Whereas, a reduction in the degree of damage of about 22% and 26% are observed in clay + fly-ash and clay + filler + fly-ash specimens, respectively. Even though clay + filler and clay + filler + fly-ash manifested higher peak forces and lower degree of damage than clay + fly-ash composite, the reduction of densities are only 3.48% for clay + filler and 29.85% for clay + filler + fly-ash. Therefore, a combined consideration of degree of damage and density of composites reveals that clay + fly-ash composites are superior as compared to clay and other composites investigated in this study.

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1. Introduction

Hailstorms are significant causes for damaging the exterior of constructed environments, like roofs and external claddings on buildings. In the United States, hail and high-wind damage caused more than $3 billion in damage to crops and property in 2015 according to the National Oceanic and Atmospheric Administration’s (NOAA) National Centers for Environmental Information (NCEI) [1] in hail-prone regions. Organizations like the National Board of Catastrophe (from 1949 to 1964) and the United States Weather Bureau (from 1950 to 1960) have indicated that severe hailstorms occur predominantly in the central part of United States in areas covering Texas to Minnesota, and Colorado to Illinois [2]. Very often, roofing systems are damaged due to extreme hail impact events.

According to 2006 IBC, a roof must “serve to protect the building” and is expected to perform several functions, from preventing water intrusion to acting as a structural diaphragm during such events. Roofs must be designed to resist high and low temperatures, rain, high winds, exposure to ultraviolet radiation, snow, ice formation, and hail stones depending on the geographical location. In the current paper, hailstorms are of primary interest. Hail storms can vary from small sizes such as a pea (6 mm) up to the size of a softball (115 mm), as reported by NOAA [3]. Roof tiles are usually made of high-hardness and light-weight ceramics [4,5], which have poor impact resistance. In such situations, roof tiles are severely prove to hail impact damage.
The goal of the work presented here is to improve the impact resistance of roof tiles while reducing their density. Fly-ash cenosphere, which is a by-product generated in coal-power plants [6] is considered as an additive with clay to improve the effective impact response. Fly-ash, due to its pozzolanic properties, has been extensively studied [7,8] and frequently used as an inexpensive substitute for Portland cement in concrete production and has shown to improve the mechanical [9] and thermal [10] properties of concrete and the mixture easier to knead due to its small particles [11].

Previous researchers have investigated the possibility of using waste by-products like fly-ash in the brick industry to improve the quality of bricks manufactured [12–15]. Lingling et al. [16] reported that the addition of fly-ash in fired bricks in high volume ratio resulted in high compressive strength, low water absorption, no cracking due to lime, and high resistant to frost. Also, Cultrone et al. [17] reported that the cost of clay bricks can be reduced by partially replacing the clay in bricks with fly-ash [18]. Malik et al. [19] compared the compressive strength of bricks with rice husk and fly-ash, and concluded that the bricks with fly-ash had the highest compressive strength. In the dissertation by Porwal [20], fly-ash/clay bricks were compared against conventional bricks, and established that fly-ash bricks were suitable for use in building constructions due to their low weight, durability in aggressive environments and high strength. A study by Liu [21] reported that the cost of fly-ash bricks was 20% lower than clay bricks, which is attributed to the low-firing energy needed due to the presence of fly-ash.

In the case of ceramic tiles, seldom work has been reported on the use of fly-ash. Olgun et al. [22] investigated the addition of fly-ash and tincal ore waste (TW) to ceramic tiles, and stated that the firing strength of the tiles increased with the addition of fly-ash and TW. Mishulvich and Evanko [23] studied ceramic tiles with high-carbon fly-ash, and confirmed that the fly-ash tiles exhibited lower water absorption and manufacturing temperature in comparison to conventional tiles. Akhtar [24] investigated the influence of fly-ash with waste polythene fiber, and concluded that the strength of the tiles increased with the addition of these materials. In addition to all of these advantages, fly-ash is a waste from thermal power plants and re-using it in clay bricks or tiles aids in reducing environmental pollution [16].

This paper, a blended clay and fly-ash cenosphere composite is explored as potential material for roofing tiles. It is hypothesized that the fly-ash cenosphere/clay composite will yield higher strength and lightweight material with improved resistance to impact damage. The overarching goal of the research presented in this paper is to improve the damage tolerance and durability of roofing material systems when subjected to dynamic impact loading.

2. Methods

2.1. Material system

Four types of composites were investigated: clay, clay + fly-ash, clay + filler, and clay + filler + fly-ash, where “clay” refers to base clay material, “filler” is grog and “fly-ash” is fly-ash cenospheres. Further information regarding fly-ash cenospheres and base clay recipe are given in the following sections.

2.1.1. Fly-ash cenospheres

Free-flowing powder of hollow ceramic “fly-ash” spheres often referred to as fly-ash cenospheres with a size range of 10–106 microns were purchased from cenoster.com. CenoStar 3510 Grade cenospheres were chosen, which have low density (Bulk Density: 0.32–0.44/gcc; True Density: 0.85–0.95/g/cc), excellent durability during processing, as well as stability at high temperatures (Melting Point: 1200–1400°C). Typically, cenospheres are used for reducing densities, reducing VOC levels, increasing filler loadings, and improving viscosity in a variety of formulations. Other important physical and mechanical properties of fly-ash cenospheres are given in Table 1.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Material composition, physical and mechanical properties.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material composition [25]</td>
<td>Silica: 55.0–60.0; Alumina: 28.0–34.0; Iron: 1.5–5.0</td>
</tr>
<tr>
<td>Compressive strength, MPa [26]</td>
<td>2.8–55</td>
</tr>
<tr>
<td>Hardness, Moh's scale [25]</td>
<td>5–6</td>
</tr>
<tr>
<td>Thermal conductivity, W/mK [27]</td>
<td>0.93</td>
</tr>
<tr>
<td>Melting point, °C [28]</td>
<td>&gt;1400</td>
</tr>
</tbody>
</table>

2.1.2. Clay & filler

Dry base clay recipe is given in Table 2, which consists of Red Art (50%), Hawthorne Bond Fire Clay (350 mesh) (25%), Talc (15%) and Wollastonite (10%). Additional dry ingredients added along with the dry base clay recipe are gog and fly-ash cenosphere fillers. Filler or gog is fired clay that is ground to 40 × 28 mesh size, which represent the actual opening per linear inch horizontally and vertically.

2.2. Fabrication process

The process to fabricate clay and blended composite specimens is described next. The dry base clay and other dry ingredients (grog and fly-ash) were weighed as shown in Fig. 1(a) and stored individually in plastic containers.

Water was added while folding in and wedging the clay as shown in Fig. 1(b) and (c). Water was added while folding in and wedging the clay. The amount of water was maintained at approximately one fifth of the weight of dry clay batch (19.5%–22.5%) [29]. Water was added empirically until the clay could change its shape without rupturing, which was the desired plasticity in this case. Each batch (Fig. 1(d)) was placed in a separate container and then was individually flattened under a slab roller to obtain a thickness for each sample of approximately 15 mm, as shown in Fig. 1(e). At least five 100 mm long × 100 mm wide tiles were cut out from each batch of clay (Fig. 1(f)).

These tiles were then sandwiched between two plaster disks of 4 kg each and held until the tiles were completely dry and flat as shown in Fig. 1(g) and (h). The weight of only the top plaster disk was applied to the samples. Black stain was used to identify each tile corresponding to four types of composite specimens: clay, clay + fly-ash, clay + filler, and clay + filler + fly-ash.

Upon drying, the tiles were heated in an electric kiln to pyrometric cone 04 (1945°F) as displayed in Fig. 1(i) and (j). During the heating process, clay undergoes a process known as vitrification, during which the clay is fused to produce a weather resistant and hard product [10] and is accompanied by shrinkage of the final product. Five samples for each type of composite were fabricated. Table 3 shows the proportion by volume of fly-ash and gog (filler) used for each type of composite along with the average thicknesses of all the specimens. It is observed that the thickness of the samples with fly-ash and filler were higher as compared to clay samples. This is due to significant shrinkage that occurs in clay samples due to firing, which is lowered by the addition of fly-ash and filler in composite specimens.

2.3. Microstructure analysis and densities

Upon fabrication, one sample of 20 mm × 20 mm × 10 mm was cut from each of the 4 types of composites. These samples were polished and examined under a scanning electron microscope (SEM) in order to explore the influence of adding fly-ash and filler on the microstructure. Identical conditions with 12 kV potential were maintained for all samples.

In order to evaluate the hypothesis posed earlier that the addition of fly-ash to clay will reduce the density of the material along with improving the damage resistance to impact loading, the determination of composite densities is inevitable. Therefore, the average density of each composite type was determined by dividing the weight of each cube of a composite by its volume. A total of 4 cubes for each composite type were used to measure their densities.

2.4. Dynamic impact tests

The samples fabricated were subjected to dynamic impact loading to evaluate their structural integrity and durability. Drop-weight impact tests were conducted using an Instron CEAST 9340 drop tower impact system. To the best knowledge of
the authors, there is no current ASTM standard to evaluate the impact response of clay samples. Therefore, the specimen dimensions were based on the ASTM standard C647-04 “Standard Test Method for Breaking Strength of Ceramic Tile” [31], which requires that the length and width the tiles to be less than or equal to 108 mm. Schematic of the fixture used for holding the samples during these tests is shown in Fig. 2(a), which consists of two metal fixtures with a test area of 45.6 cm². A hemispherical impactor (striker) with a mass of 3.01 kg and diameter of 12.7 mm was used to impact each specimen at the center with a kinetic energy of 1 J. The kinetic energy was calculated based on the mass of the impactor and impact velocity, using the following equation:

$$E_k = \frac{1}{2}mv^2 = mgh$$

where, $E_k$ is the impact energy or kinetic energy, $v$ is the impact velocity and $m$ is the mass of the impacting object (striker), $h$ is the height of the striker in the impact drop tower and $g$ is the gravitational constant. To achieve a fixed impact energy of 1 J, mass of the striker was fixed at 3.01 kg which resulted in an impact velocity of 0.82 m/s. Impact velocity less than 10 m/s is typically categorized as low-velocity impact [32]. For each test, the impact velocity and striker falling height were adjusted internally by the impact machine to achieve an impact energy of 1 J.

Force-time, displacement-time and energy-time responses were recorded by the data acquisition system “CEAST DAS 8000 Junior” of the impact machine for each test.

**Table 3**
Composite composition and specimen dimensions.

<table>
<thead>
<tr>
<th>Material type</th>
<th>Volume (ratio)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clay</td>
<td>clay (100)</td>
<td>12.47 ± 0.35</td>
</tr>
<tr>
<td>Clay + filler</td>
<td>filler:clay (16:84)</td>
<td>12.90 ± 0.41</td>
</tr>
<tr>
<td>Clay + fly-ash</td>
<td>fly-ash:clay (16:84)</td>
<td>13.53 ± 0.80</td>
</tr>
<tr>
<td>Clay + filler + fly-ash</td>
<td>filler:fly-ash:clay (16:16:68)</td>
<td>14.79 ± 0.25</td>
</tr>
</tbody>
</table>

Fig. 1. Composite tile fabrication process.

Fig. 2. (a) Schematic of the impact fixture; (b) Typical energy-time response to dynamic impact loading.
The impact responses were evaluated in terms of visual damage as well as by calculating the degree of damage. A typical energy-time response is shown in Fig. 2(b), where the absorbed and impacted energies are highlighted. The “degree of damage” that quantitatively measures the overall extent of damage due to impact loading is defined in Eq. (2) as:

\[ D = \frac{\text{Absorbed Energy}}{\text{Impact Energy}} \]  

Optical images of the front and back faces of the impacted samples were obtained next to visually compare the extent of damage of the four composite types.

3. Results and discussion

3.1. Composite density

The densities of the four types of composites were determined and are reported in Table 4. The percentage relative change in densities were calculated for each type of composite with respect to the clay sample density, which appeared to manifest the highest density among all types. Composite with Clay and filler only showed a reduction in density by \( \approx 3.48\% \), and clay with fly-ash only exhibited the highest reduction in density of approximately 36\%. Whereas, composite with clay, filler and fly-ash exhibited a percent reduction of approximately 29.85%. It is evident from the density calculations that clay + fly-ash composite is the lightest among the four types.

3.2. Microstructural analysis

The influence of adding fillers and fly-ash on the microstructure of clay is discussed next. A typical microstructure of clay sample is shown in Fig. 3(a), which appear to possess voids that can result in the loss of material strength and undesirable fluid absorption (also shown by [33]). Fig. 3(b) shows the microstructure of a clay + filler composite sample, where gap between the clay material and a filler can be clearly seen, which hints that the composite is not compacted. Fig. 3(c) shows the microstructure of clay + fly-ash sample, where the fly-ash appears to have partially melted into the clay, thereby creating a bonding with the clay material. Previous researchers have reported that such bonding results in decreased water absorption [16]. In Fig. 3(d), the microstructure of a clay + filler + fly-ash sample clearly shows a good bonding between fly-ash as clay, however, a gap exists between grog filler and clay.

3.3. Impact response

Dynamic impact responses were evaluated in terms of visual damage and the degree of damage parameter. Fig. 4 shows the force (normalized by sample thickness)-time responses for one sample of clay, clay + filler, clay + fly-ash and clay + fly-ash + filler composite each for illustration. A total of twenty samples were tested with five samples for each type of composite. Clay samples experienced the lowest impact peak force as well as initial slope of the force-time response, while the other three composite types...
exhibited higher forces. This implies that the addition of fly-ash or filler increased the peak force and stiffness of the samples. Clay + filler composite samples experienced the higher peak forces as compared to all other types of composites.

Fig. 5 shows the energy-time response for one sample each of clay, clay + filler, clay + fly-ash and clay + fly-ash + filler composite. The post peak plateau region in the response for the clay sample is approximately equal 1 J (impact energy), which implies that it absorbed all the impact energy. Whereas, the clay + filler composite absorbed less energy in comparison to the other samples, which results in less damage in the sample. Even though the impacted samples showed cracks on the impacted and back faces, they were not completely broken (refer to Fig. 6(b)).

The degree of damage for these samples reduced by 45.97% as compared to the clay samples. Further, clay + filler + fly-ash and clay + fly-ash samples showed a reduction in the degree of damage by 26.44% and 21.84%, respectively. All the tested samples were broken as shown in Fig. 6(c) and Fig. 6(d). Adding fly-ash or filler appears to increase the impact resistance of roof tiles, however, the increase in damage resistance by the addition is not as significant as compared to adding fillers only. Further investigation into the combined influence on density and damage resistance is summarized in the following section.

Table 5 shows the average and percentage change in peak force values for the four composites with the clay composite as the baseline. The percentage increase in peak impact force for clay + filler composite is 67.57%, for clay + filler + fly-ash composite is 50.70% and for clay + fly-ash composite is 46.18%.

3.4. Specific degree of damage

This research focuses on increasing the damage tolerance without gaining extra weight, such that the design requirements for the supporting structures is reduced. For this reason, densities were also accounted for selecting the material that will not only increase the damage tolerance, but will also render the composite light-weight.
weight. As mentioned earlier, clay + filler composite has shown to decrease the degree of damage, however, is the one with the highest density. Due to this, a new term was devised called “Specific Degree of Damage”, \(D\), which is the value of damage scaled with the ratio of density of a composite/maximum density (clay). \(D\) was calculated for each type of composite as shown in Eq. (3):

\[
D = \frac{D_{\text{comp}}}{\rho_{\text{max}}} \tag{3}
\]

where, \(D\) is the degree of damage of a composite, \(\rho_{\text{comp}}\) is the density of the composite and \(\rho_{\text{max}}\) is the maximum density that corresponds to the baseline density, which is clay in the current paper.

In Eq. (3), \(D\) decreases and approaches 0 when both \(D\) and \(\rho_{\text{comp}}\) decrease, while holding \(\rho_{\text{max}}\) constant. This is the best-case scenario of higher damage tolerance (low degree of damage) and light weighting. With both \(D\) and \(\rho_{\text{comp}}\) in the numerator, their combined effect on the value of \(D\) is captured. Note here that the value of \(D\) is always between 0 and 1. \(D\) values are then plotted against normalized composite densities \(\left(\frac{\rho_{\text{comp}}}{\rho_{\text{max}}}\right)\) as shown in Fig. 7.

From Fig. 7, it is evident that clay + fly-ash samples performed the best with low scaled degree of damage and low normalized density as well. Even though, the values of \(D\) for clay + filler and clay + fly-ash + filler composites are comparable to that of clay + fly-ash, their normalized densities are higher. As a result, clay + fly-ash composites are suitable to be used in roof tiles with unique characteristics of reduced density and improved resistance to impact damage.

4. Conclusions

Blended fly-ash cenospheres/clay composites were explored in this paper as potential material for roofing tiles. The goal was to improve the damage resistance of tiles while reducing their densities. Hence, four types of composites, namely clay, clay + filler, clay + fly-ash and clay + filler + fly-ash were fabricated and evaluated under dynamic impact loading with 1 J impact energy. Changes in microstructure, densities and degree of damage in the four types of composites were noted. Key observations are as follows:

- From the micrographs of the fabricated composites, gaps/spacings between filler and the clay were observed, which disappear in the case of clay/fly-ash samples. This is attributed to the partial melting of fly-ash cenospheres into the clay sealing any gaps present. Therefore, the addition of fly-ash cenospheres appears to increase the stiffness of the material.
- Clay + fly-ash samples displayed the lowest density among all composites, with a reduction of approximately 36% as compared to clay samples. However, the reduction of density in clay + filler and clay + filler + fly-ash was 3.48% and 29.85%, respectively.
- From the impact tests, clay + fly-ash specimens showed an average increase of approximately 46% in peak load and a decrease of about 22% in the degree of damage as compared to clay samples. Also, clay + filler and clay + filler + fly-ash showed higher peak forces and lower degree of damage than clay + fly-ash composite.
- Clay + filler (\(\approx 46\%\)) and clay + filler + fly-ash (\(\approx 26\%\)) composites manifested lower degrees of damage than clay + fly-ash (\(\approx 22\%\)) composite. However, the specific degree of damage, which is the degree of damage normalized with composite density established clay + fly-ash composite as the one with the highest damage resistance to impact loading, while being lightweight.

To summarize, this study has shown very promising results of adding fly-ash with clay for reducing the density and improving the dynamic impact response for potential application in roofing tiles.

References


