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A Comparative Study of Production of Glass Microspheres by using Thermal Process

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Abstract

Microspheres are spherical particles that can be distinguished into two categories; solid or hollow. Microspheres typical ranges from 1 to 200 μm in diameter. Both solid and hollow microspheres can be produced from glass, ceramic, carbon or plastic. Solid glass microspheres are usually made from soda-lime glass due to the low melting point and chemical inertness of soda-lime glass. The conventional method in producing solid glass microspheres is by the In-Flame Spheroidisation Method where a continuous controlled flow of powdered glass is fed to a gas flame. In contrast, hollow glass microspheres are produced by adding a blowing agent to glass powder. Blowing agent such as sodium silicate decomposes to multiple gases when burned, causing the microsphere to form with a hollow structure. Glass microspheres applications as fillers in syntactic foams resulting in reduction of material density, compaction and heat conductivity. Developed Vertical Thermal Flame (VTF) process has potential to produce cenosphere from fly ash with high yields. In the VTF process, the raw materials are fed into a vertical tube via a funnel and the raw material will come in contact with the...
flame located at the bottom of the vertical tube. The burned particles are collected and cooled in a beaker via a collector plate before proceeding to the particle characterization study [5]. This paper aims to study the potential production of microspheres from glass by using the VTF process.

2. Methodology

2.1. Preparation of feed sample
Recycled glass was used as the primary raw material (Ecosand, supplied by Day Group Ltd, London). This was commercially ball milled to form a powder with a mean particle size of 45μm. Polyvinyl alcohol (PVA) was dissolved in water form a solution. The milled glass was then mixed with a small volume of the binder solution to form spherical glass agglomerate particles with size between 90 to 125μm. PVA is an organic compound that acts as a binder and blowing agent to aid in formation of hollow glass microsphere. When the agglomerated powdered soda-lime glass is burned, the PVA decomposes and resulted in the gaseous evolution of acetic acid, water and hydrogen bromide. The gases evolved within the feed material structure develops the hollow cavity in the glass microspheres.

2.2. Burning Process
The VTF process was done by setting up a vertical blue flame by adjusting the propane gas-to-air ratio. Pre-treated soda-lime glass powders were fed into the quartz tube with a different dimension - length 35cm and width 20mm via a stainless steel filter funnel. The burned particles were collected at the bottom using a collector plate [5]. The particles undergoes second pass for comparison study.

2.3. Particle Characterization and Density Analysis
The powdered soda-lime glass and the respective thermally treated particles were characterized using a table-top Scanning Electron Microscope (SEM). SEM imaging was used to study the morphology of the powdered soda-lime glass and the respective thermally treated particles. A qualitative analysis is performed to determine if there is a density reduction of the pre-burned and burned soda-lime glass powder. Pre-burned soda-lime glass powder and burned soda-lime glass powder are placed in two
different beakers (filled with distilled water). Floating particles in the beaker would show that there is density reduction of the burned particles.

3. Results and Discussion

Effects of thermal process on morphology of glass microsphere. A total of three samples between particle size selected are between 90 to 125 μm are evaluated with single and second passes respectively. The effect of PVA as binding agent on the morphology of the glass microspheres was studied.

Figure 2 shows the morphology of pre-treated and treated particles. After the vertical thermal process, the morphology of some of the agglomerated soda-lime glass particles was observed to have transformation to spherical particle after the single pass of burning in the VTF. Figure 2 (a) shows the non-treated glass powder and Figure 2 (b) shows the morphology of agglomerated soda-lime glass powder with binder. Figure 2 (d) shows the second pass thermal treatment of the glass particles where it has better transformation to spherical particle as compared to the single pass thermal treatment of glass particles shown in Figure 2 (c). By undergoing two passes, it is found that the particles have spheroidized further. This is due to the longer overall contact time between the flame and particle, causing further spheroidization of the particles. This results in a more spherical particle as shown in Figure 2 (d).

![Figure 2](image_url)

**Figure 2.** Morphology of thermally treated glass powders: (a) powdered soda-lime glass (b) pre-treated soda-lime glass (c) formation of glass microspheres in single pass thermal treatment (d) formation of glass microspheres in two times pass thermal treatment
The formation of smooth spherical particle is dependent on the residence time of feed material in the flame produced. At the flame, the glass powder softens and spheroidizes. This is due to the effect of surface tension resulting in the formation of the spherical particle shape. As the temperature increase, the viscosity of the glass decreases. It is believed that when the glass is fed into the flame, the glass powder melts and gases are evolved forming the hollow cavity in the microsphere. As the glass powder exits the high temperature zone, the glass powders are cooled rapidly, conforming to the spherical particle shape.

Soda-lime glass have a relatively low melting point. The flame temperature supplied by the propane gas is sufficient to melt the soda-lime glass and decompose the gas in the glass particle resulting in the morphology change of the soda-lime glass from irregular surface to smooth spherical microspheres as shown in Figure 2 (a) to (d). The temperature produced by the propane gas ranges between 1000°C to 1500°C. The melting point of the soda-lime glass is at 1400°C. At this temperature, the glass melts and thus, forming the smooth spherical particles.

Similarly, Poirier et.al [6] produced glass microspheres by using oxy-acetylene flame that burns at a higher temperature and successfully produced smooth spherical particles. By comparison, oxy-acetylene gas burns at a higher temperature as compared to propane gas. Figure 2 shows production of glass microspheres by using the VTF set-up with propane gas which burns at a lower temperature than oxy-acetylene gas. This would suggest that glass microspheres can be produced using propane gas.

A comparison study is made with the production of hollow glass microsphere from amber glass frit by flame spraying method using oxy-acetylene gas. Dalai et.al [3] incorporated urea as blowing agent in modification of the microsphere wall thickness and porosity. Addition of urea as blowing agent showed there are changes the glass morphology. The morphology of the glass microsphere in Figure 1 (c) shows similar results to the hollow glass microsphere by Dalai et al. The successful production of glass microsphere as shown in Figure 1 (c) and Figure 1 (d) suggests that PVA can be used as a binder and blowing agent to aid in the development of the hollow cavity in the glass microsphere.

**Qualitative analysis of pre-burned and burned particles on density reduction.** Glass microspheres usually have density less than density of water [1]. Based on the literature reviews, thermally treated glass powders will have density less than 1000 kg/m³ thus allowing it to float on water [7]. In contrast, pre-burned soda-lime glass powders with typical density of 2720 kg/m³ [8] will sink in water. This would suggest that the glass microspheres will float on the surface of water. In Figure 3 (b), it is observed that the burned particles floats in a beaker of water as compared to the pre-burned particles in Figure 3 (a) where the pre-burned particles sinks at the bottom of the beaker. It is believed that there is a density reduction of the pre-burned particles. Therefore, it is believed that the VTF process have successfully formed hollow glass microsphere. The reduction in the density of the thermally treated glass particles depicts that the particles are hollow. These hollow particles can be used as lightweight fillers in syntactic foam or used as hydrogen gas storage in the pharmaceutical industry [3].

![Figure 3.](image-url) (a) Sink agglomerated soda-lime glass powder before undergoing the VTF process (b) floated burned particles after the VTF process
4. Conclusion

The morphology changed of the binded powdered soda-lime glass with PVA from an irregular surface to smooth spherical surface was observed from the SEM analysis. Hence, vertical thermal flame process is a feasible process to produce smooth spherical particle from powdered soda-lime glass. Other analytical test such as density measurement is recommended. A quantitative analysis would prove if there is a density reduction in the microspheres formed, proving if the microspheres formed are hollow or solid. Further improvements of the VTF process is suggested to increase the transformation of glass microspheres by using a vacuum system in the set-up or using a different source of heat (plasma or oxy-acetylene flame gun).

5. Acknowledgement

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