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# Effect of Chicken Bone as Foaming Agent on the Structural and Mechanical Properties of Glass Foam

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## ABSTRACT

Amber glass is widely used to pack a large number of industrial products. However, its waste is disposed of into landfills, which results in serious environmental concerns, and only a small amount of it is either reused or recycled. Recycling the glass offers reduction in the landfills and hence minimizes the adverse environmental impact. This study follows the concept of recycling amber glass to produce glass foam that possesses high strength. The hard glass foam is produced by sintering pulverized pharmaceutical amber glass and chicken bones at 860°C and 10 min. Chicken bones were used for the first time as a recycling material for producing glass foam. In this work different characterization techniques were used to investigate the mechanical and structural properties of the produced glass foam. The glass foam produced resulted into high values of flexural strength ranging from 15.90 ± 2.34 to 28.31 ± 1.62 MPa. The prepared glass foam, due to its unique properties such as high strength, sound absorption, heat insulation and shock-wave absorption, offers valuable insights into the world of material science.

## INTRODUCTION

Pharmaceutical amber glass is widely used as a packing material for the protection of medicine from harmful effects on life (Polley *et al.*, 1998). It is normally composed of silica, carbon and oxides of calcium, iron and sodium. Amber glass has the ability to adsorb several bands of light and prevent the direct interaction of pharmaceutical products with light (Morsi *et al.*, 2015; Mosch, 1998). Therefore, a large amount of pharma amber glass is produced on a daily basis to meet the demand of the pharmaceutical industry. Only a small amount is reused or recycled, and the rest of the glass waste is usually disposed of in landfills. Only a small amount is reused or recycled, and the rest of the glass waste is generally disposed of in landfills (Shayan & Xu, 2004). The glass disposed of in landfills results in social and environmental concerns (Burnley, 2001). In contrast, the recycling of waste material helps in lowering the approach of landfilling. Recently, global environmental factors have caused a rise in the recycling of different wastes, including pharmaceutical amber glass (Arulrajah *et al.*, 2015). Therefore, to solve the social and environmental concerns, the waste amber glass needs recycling techniques.

One possible way to recycle it is to convert it into powder form and mix it with the concrete but as it has some sodium contents which causes cracking in the concrete and hence affecting the mechanical strength of the concrete (Polley *et al.*, 1998; Akai *et al.*, 2005). For that reason, waste glass with low sodium contents could be used for concrete mixing method. Another possible way is to heat the amber glass and perform hydrofluoric etching to convert it into porous silica for research or commercial use (Chen *et al.* 2006; Minakuchi *et al.*, 1996, 1997). This technique is suitable for borosilicate glass, which is a phase-separated glass, and therefore, it is not appropriate for amber glass because it has no phase separation. Besides this the etching process generates a lot of waste and is not an eco-friendly process (Chen *et al.*, 2006). Thus, a potential way to recycle pharmaceutical amber glass is using it in the production of glass foams (Andreola *et al.*, 2007; Bernardo & Albertini, 2006; Bernardo *et al.*, 2005, 2007; Ducman & Kovacevic, 1997; Guo *et al.*, 2010a, 2010b; Hicks *et al.*, 2005; Konig *et al.*, 2014; Ponsot *et al.*, 2015). As glass foams has an edge of lower operating temperature as compared to other techniques of waste glass treatment (Minakuchi *et al.*, 1996; Andreola *et al.*, 2007; Ducman & Kovacevic, 1997; Guo *et al.*, 2010a, 2010b; Konig *et al.*, 2014). This low temperature is advantageous both environmentally as well as economically as ceramic and glass sector spends 8.9% of the total energy of global production (Ponsot *et al.*, 2015). Such a huge amount of energy input is mainly spent in heat production for melting and sintering processes (Gong *et al.*, 2016). Thus, foaming process for the preparation of glass foam is a cost-efficient and eco-friendly recycling process for waste treatment. Other advantages of glass foam are its usage in various construction and building applications due to their properties of heat insulation, shock wave and sound absorption, light weight and its resistance to fire and moisture (Guo *et al.*, 2010a). Because of the porous structure and high flexural strength of the glass foam it is used for construction applications. Its mechanical strength can be increased by improving its abilities of shock wave absorption (Guo *et al.*, 2010a). The porosity in glass foam is typically achieved by melting the glass in the presence of a foam agent. The foam agent releases gases at designated high sintering temperatures. Different foam agents used are alkali-carbonates (Konig *et al.*, 2014;

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Chen *et al.*, 2012; Fernandes *et al.*, 2009) carbides (Guo *et al.*, 2010a) and some other chemicals (Chen *et al.*, 2009; Llaudis *et al.*, 2009). However, the foaming agents are normally expensive, and therefore glass foams produced are not always cost effective (Kingery *et al.*, 1976). To address this issue a simple, cheap and obtainable foaming agent is of high need and interest.

Here in this work, high strength glass foam was successfully sintered from waste material: pharmaceutical amber glass and chicken bones. The chicken bone was taken as it was not reported as a recycling material in glass foam production according to the authors' best knowledge. The produced foams were examined thermally, structurally and mechanically for finding the relationship between the material properties and the processing techniques of the foam glasses. This recycling method helps in reduction of food waste in the food industry. Besides addressing the environmental concerns related to food industry it could lower the production cost of glass foams by using only waste materials.

## Experimental work

### Raw materials

Waste amber glass and chicken bone powder was obtained by applying different physical operations. The amber glass powder used in the preparation of foam glass was obtained from waste amber glass bottles. About twenty bottles were obtained from local scrap store and washed three times with hot water and detergents. The labels were removed with knife and the metallic strips were cut down by using a metal cutter. After thoroughly washing with water and then drying it in sunlight, the glass bottles were subjected to size reduction. Hammer mill was used for the initial breaking of the bottles. The course glass cullet obtained from the hammer mill was pulverized in a Gyro Mill. The powder obtained was separated in a sieve shaker and the average size of glass powder collected was approximately 150 µm. The powder is then cleaned in ultrasonic bath with acetone for 15 min. The wet powder was dried in a vacuum at 90°C (Burnley, 2001). The final glass powder was kept for further operation as shown in Figure 1.

The bone powder used was obtained by pulverizing chicken bones  The bones were taken from a local chicken slaughter house and washed cleanly with hot water. The clean bones were then cooked slowly at 90° C. For drying purposes, it was kept in sun light for few days and then further dried at 90° C in an oven for 3 hours. The as dried bones were cut into small pieces with hand saw for further operations. In order to reduce the size of small pieces, these were ground in Disk Mill. The product of the Disk Mill was put in Ball Mill for fine size reduction. The bone powder obtained from the Ball Mill operation was sieved through 100-mesh. Then, the bone powder was treated with acetone for 24 hr. This was done to remove any organic compound form the bone powder. After 24 hr the powder was separated from the mixture by using a filter paper. Vacuum was used for drying purposes.

### Sample preparation

In order to get different compositional batches both the powders were combined, by weight, in different proportions. Digital mass balance was used for weighing purpose. Following were the compositional batches: 98%-2%, 94%-6%, 92%-8%, 80%-20%. The amount of bone added to the glass powder is typically in the range of 1% to 20 % by weight (Burnley, 2001; Chen *et al.*, 2006). The powders were mixed thoroughly in an electrical stand mixer machine at 300 rpm.

The as mixed powder was then pressed into small pellets under a compaction pressure of 30 MPa in cylindrical mould. Fig. 1 shows the mechanical pressing machine and prepared compact pellets. The prepared compacts, having 10 mm diameter and 6 mm thickness for the foam expansion, were prepared without using a binder.

From the Differential Thermal Analysis (DTA) report the sintering temperature was fixed at 860°C. The as prepared pellets were then put into an electrical furnace for foaming process as shown in the Figure 1.

The heating rate of the sample was set 5°C/min but after 200°C to 250°C the rate decreased to an average of 1.4°C/min. After achieving 860°C the temperature was maintained for 10 minutes. The produced foam samples were kept in an oven for one hour. All the samples were cooled at the rate of 15°C/min to resist the micro structural evolution. The glass foams were further cooled slowly at about 0.5 °C/min to impart the annealing effect.


**Figure 1:** Different stages of sample preparation of glass foam

### Characterization techniques

### Differential Thermal Analysis

Before the heating process some of the as prepared powders from each sample were thermally analyzed using a SDT Q 600, V20.9 Build 20 TA/DTA Instrument over a range of 25˚C to 1000˚C. Heating rate used in all the measurements was 10˚C /min.

### Mechanical strength test

The glass foams samples for compression testing were prepared by foaming 10mm x10mmx 6 mm size compacts.

The compacts were made by applying a pressure of 30 MPa in a steel die of 10 mm x10mm dimensions. The compact specimens were then placed in a closed steel mould coated with Kaolin clay. The clay prevented the reaction between the mould and the foamed glass. The sintering temperature was set at 860°C. After the foam formation, the samples were prepared for 3-point load bend test by cutting the specimens with the help of Disc cutter in Gem and Jewellery Centre of Excellence as shown in Figure. 2. Each sample was cut into 10 mm x 4 mm x 3 mm dimensions and a 3-point bend test was performed at each specimen. To avoid the uncertainty in dimensions and minimizes the surface defects in the pellets all the samples were ground and polished to 1200 grade with Silicon Carbide grinding paper. By using Instron, 8874 Axial-Torsion fatigue testing system the strength was calculated at a cross-head rate of 2 mm/min. To measure the flexural strength (σ), 6 test bars were measured for each sample. Geometric loading area was calculated for the measurement of flexural test. Standard error was measured as follows:

$$\sigma_{error} = \frac{\sqrt{(\sigma_1 - \sigma_{ave})^2 + \cdots + (\sigma_n - \sigma_{ave})^2}}{n}$$

Where, n shows number of samples whereas σ is used for showing the strength.


**Figure 2:** Image of the disc grinder and produced glass foam.

### Phase identification

All the prepared glass foam pellets and the as cleaned chicken bone were subjected to X-ray diffraction. The reading was obtained from 10° to 70° 2θ with Angstrom Advanced Inc. D2 Phaser XRD (Source Kα, Emission current 10mA and voltage 30kV). MDI/JADE XRD software was used for identification of phase with ICDD pdf-4 data base.

### Micro-structural characterization

After the flexural strength test the broken foam glass surfaces were investigated by using Scanning Electron Microscope (JSM-IT100, JEOL, Japan. A lower value of vacuum of 1.1 x 10-2 psi with an emission current equal to 272 μA and accelerating voltage of 20 kV. Five images of each sample were taken from back scattered electrons (BSE). For each sample the area % porosity was statistically obtained with the help of SEM images, according to the (ASTM, 2010). the magnification was set 100x (ASTM, 2010).

## RESULTS AND DISCUSSION

### Differential Thermal analysis

DTA was done for finding the effect of the quantity of foaming agent on the foaming temperature. Fig. 3 shows the endothermic peaks for each wt% of bone added. The peaks were present at different sintering temperatures of 880°C for 20 wt%, 970°C for 8 wt% and 960°C for 6 wt% bone addition, each. The endothermic peaks for 3 wt% bone addition was invisible due to low amount of the bone and its peak was seemed at higher temperature ranges. Different features, including water evaporation, glass transition, pyrolysis of organic matter, were observed below 550°C. The endothermic peaks being observed were considered as the foaming temperature for their respective samples of different compositions. However, the study was aimed to find out the result of the quantity of chicken bone on the mechanical strength of the produced foam glass. Therefore, the foaming temperature was set at 860°C to neglect the influence of temperature on other physical properties of the glass foam, following practices in the literature (Konig *et al.*, 2014). The viscosity of the pulverized glass has a key role in the foaming process at the foaming temperature (Guo *et al.*, 2010a). The glass matrix absorbs the maximum heat at the endothermic peaks, which results in reduction in viscosity at these peaks (Gong *et al.*, 2015). It means lower viscosity causes increase in the area % porosity if large quantity of bone is present at the sintering temperature.

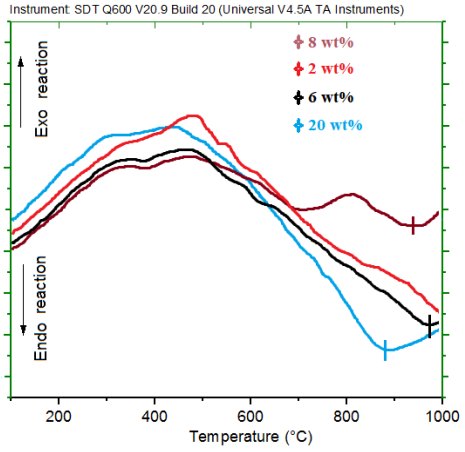


**Figure 3:** DTA curves of all samples of as produced glass foam.

### Microstructural analysis

The BSE images of the sintered foam glass are shown in the Fig. 4. In general, all the samples show porous nature, which is not uniform in distribution and size at the focusing area. Observed pores are spherical,shaped and their distribution varies with change in the amount

of foaming agent. The particles were not homogeneous in size therefore the non-uniform structure is attributed to the non-homogeneity of particle size and the interconnected pores (Fernandes *et al.*, 2009). The calculated area percentage porosity by using ImageJ Software is 60.0 ± 1.4% , 45.0 ± 1.9%, 41.6 ± 0.9% and 21.9 ± 1.1% as shown in (Table 1)for 20wt%, 8wt%, 6wt% and 2wt% bone addition, each. The trend shows that area % porosity is decreased with the decrease of foaming agent.

Figure 4(a) shows the backscattered electron image of the, 20 wt% bone addition, produced glass foam. The micro-graph shows a high porous structure of the foam which is looking non-uniform in size. The non-uniform and high porous micro-graph suggests that large amount of the chicken bone as a foaming agent is not suitable for producing quality glass foams. Figure. 4b and 4c show representative BSE images of the 8wt% and 6wt% bone addition glass foam samples. It is observed that both the micro-graphs resulted in almost same area percentage porosity values of 45.0 ± 1.9% and 41.6 ± 0.9% respectively. The similarity between the two samples suggests an identical porous structure within the range of these two samples. Another sample having 2wt% of bone addition is less porous as shown in the Figure. 4d which has a high density.

**Table 1:** Quantification of area % porosity using 100x Mg. scanning electron micrographs

| Sample | 20 wt% Bone Addition | 8 wt% Bone Addition | 6 wt% Bone Addition | 2 wt% Bone Addition |
|---|---|---|---|---|
| Micrograph 1 | 55.7 | 45.4 | 42.6 | 25.3 |
| Micrograph 2 | 61.4 | 38.1 | 40.8 | 20.2 |
| Micrograph 3 | 59.9 | 50.3 | 39.7 | 21.1 |
| Micrograph 4 | 64.6 | 44.6 | 40.2 | 19.2 |
| Micrograph 5 | 58.8 | 46.7 | 45.1 | 24.1 |
| Average | 60.08 | 45.02 | 41.68 | 21.98 |
| Standard Deviation | 3.279786578 | 4.44150875 | 2.20386025 | 2.60710567 |
| Sample Size | 5 | 5 | 5 | 5 |
| √ of the Sample size | 2.236067977 | 2.23606797 | 2.23606797 | 2.23606797 |
| SEM | 1.466765148 | 1.98630309 | 0.98559626 | 1.16593310 |

The calculations give a statistical consistency in the area percentage porosity among all the micro-graphs. Thus, it is concluded that area percentage porosity in the produced foam glass is associated with the amount of bone powder added. The high number of pores decreases the density of the glass foam and hence the strength of the foam decreases. The same result is reported in the literature (Burnley, 2001).

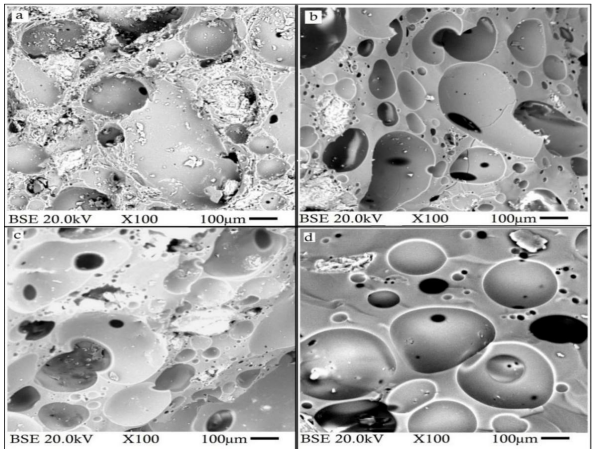


**Figure 4:** SEM photographs of the glass foam samples with (a) 20 wt% (b) 8 wt% (c) 6 wt% (d) 2 wt%

**Mechanical property**

The sample with 2wt% bone addition gave the maximum flexural strength value of 28.31 ± 1.62 MPa, whereas samples with 6wt% and 8wt% bone addition had flexural strengths of 22.07 ± 3.12 MPa and 21.63 ± 1.72 MPa, respectively. The sample with a 20 wt% bone possessed the lowest flexural strength, of 15.90 ± 2.34 MPa as shown in the Figure 5. In comparison to values published in previous research (Guo *et al.*, 2010a), the mechanical strength values achieved in this study were comparatively high. Furthermore, the increase in the amount of bones added decreased the flexural strength, which might be due to increase in the rate of produced gas during sintering process (Bernardo & Albertini, 2006; Bernardo *et al.*, 2007; Clark & Reed, 1986; Müller *et al.*, 2000; Swift 1947). Consequently, glass foams with small amount of bones were less porous and therefore they showed higher

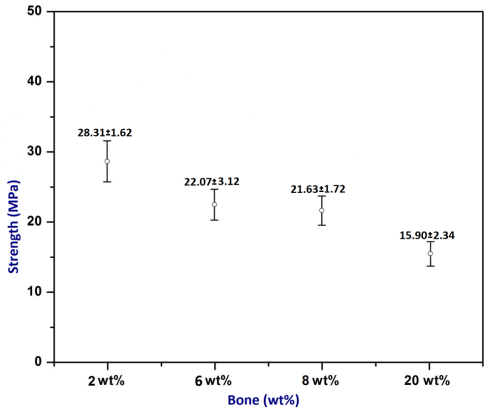


**Figure 5:** Flexural strength values of produced foam samples vs. the foaming agent

values of flexural strength (Bernardo *et al.*, 2007). Due to their comparable micro structures, the 6 wt% and 8 wt% glass foam samples had almost similar flexural strengths. This work highlighted the direct relation between amount of bone added and the gas released with the mechanical strength of the produced glass foams.

**Phase identification**

In this work, decomposition of the bone as a foaming agent was qualitatively measured by using X-ray diffraction. All the prepared glass foam samples including cleaned bone were subjected to XRD. The presence of hydroxyapatite was observed by the diffraction patterns (003), (211), (112), (305), (315), (212) and (219) as shown in the Fig. 6. These are identical to hydroxyapatite (JCPDS74-0566, PDF-4, ICDD). Moreover, the intensity of (211) and (112) peaks were identified by using Jade software. These peaks were observed in each measurement which confirms the plane of hydroxyapatite in all the produced glass foams. The shift in (211) peak to a higher position by increasing the foam agent showed an increase in is loss of structural water, which may be associated with the foaming process (Gong y et. 2015) The produced glass foam has potential impact in the field of material as it has less porosity and good mechanical strength as other different other studies reported in the past (Bernardo and Albertini, 2007; Chen *et al.*, 2009; Colombo *et al.*, 2003; Guo *et al.*, 2010a, 2010b; Konig *et al.*, 2014; Llaudis *et al.*, 2009).

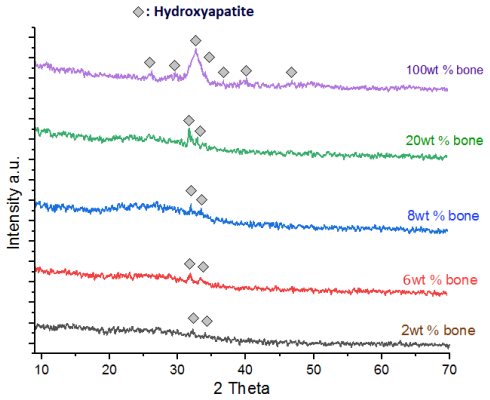


**Figure 6:** XRD diffractogram of the produced samples

## CONCLUSIONS

In this particular work, high-strength and fast sintered glass foams with high flexural strengths ranging from 15.90 ± 2.34 to 28.31 ± 1.62 MPa were effectively produced using waste pharmaceutical amber glass and chicken bones. Different amounts of the bones were added to study the mechanical and structural analyses of the glass foam. According to this study, adding more foaming agent causes increase in area % porosity but decrease in density and flexural strength. Therefore, higher the % porosity lower will be the mechanical strength. Moreover, it is investigated that the foaming process is the result of disintegration of hydroxyapatite, which caused the loss of structural water. The production of the glass foam by using only waste material which are easily obtainable can appreciably reduce the cost associated with the preparation of the glass foam. Porous nature and high mechanical strength show that the glass foam might be used for constructional purposes. Significantly, this research provides useful insights for recycling of waste materials.

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