Production and characterization of foam glass from container glass waste

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PRODUCTION AND CHARACTERIZATION OF FOAM GLASS FROM CONTAINER GLASS WASTE

A Thesis submitted to
The Mechanical Engineering Department

In partial fulfillment of the requirements for the degree of
Master of Science

By Dina Mohamed Abdel Alim

B.Sc. Mechanical Engineering

Under the supervision of
Dr. Salah M. El Haggar

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A Thesis Submitted by
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to the Mechanical Engineering Department

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in partial fulfillment of the requirements for the degree of

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Abstract

Foam glass with excellent properties was produced from container glass waste. The processing technique depended on the powder sintering approach using sodium silicate solution as a foaming agent. The morphology, density and compressive strength were studied in relation to different processing parameters: sintering temperature, amount of foaming agent, soaking time, powder particle size and glass powder color. Foam glass was sintered in the range (750-900 °C) for 30 minutes with the incorporation of 12 and 19 wt. % sodium silicate solution. At lower sintering temperature (750-800 °C), the foam has denser structure (bulk density ranged from 0.37-0.61 g/cm$^3$) along with high compressive strength (ranged from 2.29-18.68 MPa). As the sintering temperature increased, higher levels of porosity were achieved. At 850 °C with 12 wt. % sodium silicate solution, lightweight (bulk density = 0.25 g/cm$^3$), highly porous (% of porosity = 90 %) foam glass was achieved. It had relatively high compressive strength (1.62 MPa), compared to other insulating foams, along with low thermal conductivity (0.078 W/m.$^\circ$C) and the most homogeneous pore morphology. Significant change in foam glass properties took place with changing the amount of foaming agent. As the amount of foaming agent increased, the density of the foam decreased till it reached a minimum of (0.25 g/cm$^3$) that corresponded to 12 wt. % sodium silicate solution. Further addition of foaming agent caused the density to re-increase and the pore morphology to coarsen. Sintering foam for different soaking times had a slight effect on changing the foam glass properties. The morphology of all the foam glass produced at different soaking times was comparatively homogeneous. The compressive strength of the produced foam was
relatively high (1.6 MPa at 40 min and 3.13 MPa at 10 min). The powder particle size had a major effect on foam glass properties. As the particle size increased, the bulk density of the the foam increased and the morphology became less homogenous. Increasing the sintering temperature for the larger particle size did not succeed in increasing the foam structure homogeneity. The glass color also had an effect on the foam glass properties. The properties and the morphology of the green and brown glass samples were approximately the same (bulk density = 0.38, 0.37 MPa and compressive strength = 2.05, 1.97 MPa respectively). However, they differed from the morphology and properties of the white glass (bulk density = 0.25 g/cm$^3$ and compressive strength = 1.62 MPa). The specific compressive strength of the white glass foam ($6.48 \times 10^{-3}$ MPa m$^3$/Kg) was higher than that of green and brown glass ($5.39 \times 10^{-3}$, $5.32 \times 10^{-3}$ MPa m$^3$/Kg respectively). EDX analysis was performed for the white, green and brown powder. It showed that they had more or less the same compositions (except the presence of chromium element in green glass and titanium in brown glass which are coloring additives). They had the same main elements but with different weight percentages.

The optimum processing parameters for producing foam glass for thermal insulation was to use sintering temperature 850 °C, amount of foaming agent 12 wt. %, soaking time 30 min and glass powder particle size 75 µm.
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Figure A.4: A Thermal brick carved to accommodate 2 samples.................158
# Nomenclature

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>LCD</td>
<td>Liquid crystal display</td>
</tr>
<tr>
<td>GPS</td>
<td>Global positioning system</td>
</tr>
<tr>
<td>EPA</td>
<td>Environmental protection agency</td>
</tr>
<tr>
<td>CRT</td>
<td>Cathode ray tubes</td>
</tr>
<tr>
<td>LNG</td>
<td>Liquefied natural gas</td>
</tr>
<tr>
<td>$\rho_b$</td>
<td>Bulk density</td>
</tr>
<tr>
<td>$\rho_t$</td>
<td>True density</td>
</tr>
<tr>
<td>$\rho_r$</td>
<td>Relative density</td>
</tr>
<tr>
<td>DTA</td>
<td>Differential thermal analysis</td>
</tr>
<tr>
<td>DTG</td>
<td>Differential thermal gravimetry</td>
</tr>
<tr>
<td>TG</td>
<td>Thermogravimetry</td>
</tr>
<tr>
<td>EDX</td>
<td>Energy dispersive x ray spectroscopy</td>
</tr>
</tbody>
</table>
1.1 Glass

1.1.1 Glass properties

Glass is a ceramic material which is made from inorganic materials that are fused at high temperature then cooled to a rigid condition without crystallization which makes glass a noncrystalline material that has an amorphous structure. Because glass is noncrystalline material, its molecules are not arranged in a repetitive long-range order. Its molecules change their orientation in a random manner [1]. Among lots of engineering materials, glass has a range of special properties that makes it indispensable for many engineering application. It is hard and transparent at room temperature along with excellent corrosion resistance to most of the normal working environment; it is also electrical insulator. These properties made glass widely used in many fields such as [1]:

- The construction field
- The automotive industry mainly as vehicle glazing
- The electronics and the electrical industries extensively use glass because it is an insulating material that provides a vacuum tight enclosure mainly for electron tubes and lamps
- The chemical industry uses glass because it is a material with high chemical and corrosion resistance. Glass is used in laboratory apparatus and in liners for pipes and reaction vessels
Most inorganic glasses are constituted of a network of ionically covalently bonded silica (SiO₂) tetrahedra. Other oxides are added to glass to give it a range of properties that suit many applications. For example, the addition of Na₂O, K₂O, CaO and MgO oxides to glass modifies the basic silica network and lowers the glass melt viscosity. That makes glass more workable and easy to be formed [1]. Some of the common types of commercial glasses are listed in table 1.1 along with their compositions and applications.

Soda-lime glass: is the most widespread type of glass, it represents approximately 90% of total production of glass. It is prepared by melting sodium carbonate (soda), limestone (lime), dolomite, silicon oxide (silica), aluminum oxide, in addition to other fining agents at temperature around 1675 °C. It is used in applications such as:

- Containers
- Flat glass (used for windows)
- Pressed and blown ware
- Lighting products (where high heat resistance and chemical durability are not needed)

Additives such as Na₂O and CaO are added to soda lime to reduce the softening point of the glass from 1600 to 700 °C to make it more workable. MgO is added to prevent devitrification (a condition in the firing process where glass develops wrinkles on its surface instead of a smooth glossy surface [2]). Al₂O₃ is added in a small percentage to increase the glass durability [1].

Lead glasses: lead oxide is added in order to use glass as a shield from high energy radiation. This property is important for applications such as fluorescent lamp envelopes, television bulbs and radiation windows. High lead glasses have also high refractive indexes which make them good candidates for some optical glasses [1].
Permanent dyes are added to glass in order to give it different colors. Colors in glass are made by adding coloring ions and colloids. Metals and metal oxides are added during the manufacturing process of glass in order to give it specific colors. For
example, ferrous oxide is added to glass to give it a bluish-green color. Adding chromium with iron oxide gives the glass a green color [3].

1.1.2 Viscous deformation of glasses

The solidification behavior of glass which is non crystalline solid differs from that of a crystalline solid. Figure 1.1 shows the solidification behavior of a crystalline vs. a noncrystalline material where Tg is the glass transition temperature and Tm is crystalline material melting temperature. The crystalline solid (for example a pure metal) as it cools down, it crystallizes at its melting temperature with a significant decrease in its specific volume as shown by path ABC [1]. When glass cools down it follows a path like AD. The liquid glass becomes more viscous as it cools down, it transforms from a soft rubbery state to a rigid brittle state in a narrow temperature range. It is noticeable from figure 1.1 that the slope of the curve decreases slightly as the temperature decreases; the point of intersection of these two slopes is the glass transition temperature (Tg). Glass transition temperature is a transformation point in the glass melting process and it is structure sensitive. It can be defined as the center of a range of temperature in which glass changes from being brittle to being viscous and soft [1]. Glass acts as a supercooled liquid above the transition temperature. As the temperature increases above the glass transition temperature, the glass melt viscosity decreases and the viscous flow takes place easier. Figure 1.2 shows the effect of temperature on the viscosity of the common commercial types of glasses [1].
Figure 1.1: The solidification behavior of a crystalline vs. a noncrystalline glassy material [1].

Figure 1.2: The effect of temperature on the viscosity of commercial types of glasses [4].
The horizontal line denotes several viscosity reference points. These points are [1]:

- **Working point**: at this temperature the working of glass is easy to take place.
- **Softening point**: at this temperature glass flows considerably under its own weight. This point cannot be defined precisely because it varies according to the glass surface tension and density.
- **Annealing point**: at this temperature the internal stresses of the glass are relieved
- **Strain point**: below this temperature the glass is rigid and the stress relaxation process takes place at a very slow rate.

The interval between the annealing and strain points is the annealing range of glass [1].

**1.1.3 Glass Manufacturing Procedures:**

- **Forming plate glass**

This is done by a process called the float process. The glass is melted in a furnace then a ribbon of glass is moved out of the furnace and fed to a bath of molten tin where it floats on its surface. Tin is suitable for that process because tin is immiscible and it has a high specific gravity. Glass cools down as it moves on the molten tin surface. When the surface is hard enough, the sheet of glass moves to an annealing furnace in order to remove the residual stresses as shown in figure 1.3 [1].
Blowing, pressing and casting of glass

In order to fabricate the deep items like containers and light bulb envelopes, the glass is shaped by blowing where a flow of air is blown to the molten glass to force it into molds [1]. The pressing method is used to fabricate the flat items such as optical lenses. The molten glass is put in a mold; then a plunger presses the molten glass to take the shape of the mold (figure 1.4) [1].
Casting is another forming technique for glass. In order to fabricate large simple shaped objects like a large telescope mirror of 6 m diameter. Molten glass is casted into an open mold. Centrifugal casting is used for other types of objects that are hollow and have uniform wall thickness like television tubes. The molten glass is poured into a spinning mold that makes glass flow to the wall of the mold forming a glass layer of approximately uniform thickness [1].

1.1.4 Glass recycling

With a rising global population and increasing levels of consumption among developed and developing countries, pressure on the earth ecosystems will continue to increase in the foreseeable future. Societies are faced now with a real need to increase the efficiency of resources (material and energy) especially in developed countries. It is no longer accepted that raw materials are used for producing products and after completing their life cycle, they are disposed off as wastes to be landfilled or incinerated. The recycling of products at the end of their lifecycle is becoming a crucial worldwide demand.

The amount of municipal solid wastes in Egypt is 21,000,000 Tons/year. The percentage of glass waste is not usually high with respect to other types of wastes. However, the weight percentage of glass waste is a significant figure. Table 1.2 shows the composition of the solid waste in Egypt.
Table 1.2: Composition of solid waste in Egyptian cities [5]

<table>
<thead>
<tr>
<th></th>
<th>(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Organic Waste</td>
<td>60</td>
</tr>
<tr>
<td>Paper</td>
<td>10</td>
</tr>
<tr>
<td>Plastic</td>
<td>12</td>
</tr>
<tr>
<td>Glass</td>
<td>3</td>
</tr>
<tr>
<td>Metals</td>
<td>2</td>
</tr>
<tr>
<td>Textiles</td>
<td>2</td>
</tr>
<tr>
<td>Other</td>
<td>11</td>
</tr>
</tbody>
</table>

The typical composition of solid wastes of USA and Britain but in weight percentage is shown in table 1.3 for comparison:

Table 1.3: Solid waste composition in USA and Britain as a percentage of weight [5]

<table>
<thead>
<tr>
<th></th>
<th>USA</th>
<th>Britain</th>
</tr>
</thead>
<tbody>
<tr>
<td>Food</td>
<td>7</td>
<td>20</td>
</tr>
<tr>
<td>Yard Waste</td>
<td>18</td>
<td>4</td>
</tr>
<tr>
<td>Plastics</td>
<td>8</td>
<td>7</td>
</tr>
<tr>
<td>Glass</td>
<td>7</td>
<td>10</td>
</tr>
<tr>
<td>Metals</td>
<td>8</td>
<td>10</td>
</tr>
<tr>
<td>Miscellaneous</td>
<td>12</td>
<td>8</td>
</tr>
<tr>
<td>Other</td>
<td>-</td>
<td>8</td>
</tr>
</tbody>
</table>

The glass containers represent about 6-8 % of the UK household waste; the recycling rate for the glass containers is approximately 30% that is almost 500 000 tonnes of cullet every year for industry and household wastes. In the UK, around 700 000 tonnes of flat glass for window and door glazing are produced yearly; 15 % of which is recycled [6]. Due to more organized and effective collection methods in most of the European countries, the glass recycling process is well established in Europe. Some European countries achieve recycling rates higher than 75 %. USA recycles 25 % of its glass containers; however, container glass represents only 1.5 % of its municipal solid waste because alternative containers materials competitive to glass are nowadays used [6].
1.1.5 Types of glass wastes

1.1.5.1 Electronics glass wastes:

**Fluorescent Light Bulbs**

It is defined as a “gas-discharge bulb that uses electricity to excite mercury vapor. The excited mercury atoms produce short-wave ultraviolet light that causes a phosphor to fluoresce, producing visible light. Mercury is an essential component of all fluorescent light bulbs, and allows these bulbs to be energy-efficient light sources” [7]. Because mercury is a hazardous material, Fluorescent Light Bulbs are considered to be hazardous wastes. Manufacturers in the US succeeded to reduce the amount of mercury used per Fluorescent Light Bulbs by 14 % from 2001 to 2004. However, the total amount of mercury used in compact Fluorescent Light Bulbs increased to 70 % in the same period of time because of the increased amount of sales [7]. The Fluorescent Light Bulb contains from 10 mg of mercury up to 100 mg depending on its power capacity. While the compact Fluorescent Light Bulb that has less amount of mercury per lamp contains from 5 mg to 10 mg of mercury. Since 2004, considerable increase happened in the production and use of electronic devices that use Fluorescent Light Bulbs for illumination in screen displays. Liquid-crystal display (LCD) screens have backlighting that contains fluorescent bulbs. These screens are enlightened when the mercury is electrically energized. LCD screens are included in:

- Computers
- Flat panel televisions
- Global positioning systems (GPS) units
• Digital cameras

• Hand-held entertainment and communication systems

Recycling of burned Fluorescent Light Bulbs is the best way to prevent the mercury from being released to the environment through landfill and incinerators. That is why The United States Environmental Protection Agency (EPA) is currently cooperating with the manufacturers and major retailers to develop and implement recycling opportunities [7].

**CRT (cathode ray tubes)**

A CRT is the display component of the television and computer monitor. It contains between 15 and 90 pounds of glass. Some types of CRT contains 25% lead oxides which is added to CRT in order to protect the user from the x rays generated within the CRT during its operation [8]. Because lead is a hazardous material, CRT should not be disposed off in trash or in municipal landfills; it must be disposed in landfill for hazardous wastes. However, recycling CRT is the best option for managing CRT wastes.

1.1.5.2 Container Glass Wastes

Container glass is the glass used for making jars and bottles like soft drink bottles, beer bottles, mayonnaise and baby food jars, wine and liquor bottles and lots of other containerized food and beverages [9]. Container glass is the only type of glass that is recycled in large quantities at the present time. Other types of glass like window panes, light bulbs, mirrors, glassware, crystal, ovenware, cathode ray tubes and fiberglass are not recyclable with the container glass; they are considered to be
contaminants in container glass recycling [9]. Container glass makes up over 90 percent of the amount of glass waste, while the remaining 10 percent comes from other types of glass [10]. Permanent dyes are used to give different colors for container glass (figure 1.5). The most common colors are green, brown and white (colorless). In industry, the green glass is called emerald, brown glass is called amber and colorless glass is called flint [9]. In order for recycled bottles and jars to meet certain manufacturing specifications, only amber or emerald cullet (crushed glass) can be used to make brown and green bottles respectively [9]. That is why a special consideration in collecting container glass is the need for color separation (figure 1.6).

A USA company had developed a methodology for overcoming the problem of the permanent glass colors and the need for color separation. It covered the clear glass with colored organic coating that melts away while the glass is recycled. This will allow only one type of glass to be produced, which is the clear glass, since the color is not a constituent of the glass ingredients anymore. That extends the limit of the amount of cullet that might be recycled and ease the recycling process [6]. Another development reached a solution for the containers labels made of paper, foil, and plastic that must be removed during the recycling process. A photographic quality ink was developed to label containers; it is applied directly to the glass [6].

![Figure 1.5: White, green and brown glass containers](image)
Glass waste does not represent a threat to the environment, it is not hazardous because it is inert, and it is not biodegradable. When exposed to weathering forces, glass breaks down into small particles of silica (basic beach sand) that is one of the most common elements on earth [9]. Glass recycling has grown considerably in recent years because the municipal solid waste collection is becoming more organized and glass manufacturers increased their demands for recycled glass. Today, most glass manufacturers rely on a steady supply of cullet to supplement their raw material [5].

Container glass is made from common inert raw materials including silica sand, soda ash and limestone. Slag, salt cake, feldspar, aragonite and cullet are other ingredients that are used in container glass manufacturing. The mixture is heated to a temperature of 1675 °C. The melted glass is shaped by a forming machine where it is pressed into the desired shape or blown. The glass containers are left to cool slowly in an annealing furnace [9]. Cullet from recycled container glass is always part of the
recipe for new glass container production; sand is the only material used in greater volume than cullet in glass manufacturing [5]. The amount of cullet used in glass containers differs from one manufacturer to another; but it is increasing. The average amount of cullet used is approximately 25 to 35 percent of the raw material needed for glass container manufacturing. Manufacturers expect to increase cullet use to 50 percent. However, bottle manufacturers have the capability to use up to 100 percent cullet. But this is virtually impossible in the production process because that requires a consistent supply of cullet that must be completely clean, free of metals and color sorted. It is the rigidity of the quality control process that is reducing the percentage of cullet that is used by containers manufacturers. The glass maker must be sure that the cullet used in the production process is of known type and color. In the production process when a batch is started, chemicals must be added in order to compensate for impurities and color distortion of cullet [14]. Glass manufacturers are conservatives in specifying the percentage of cullet used because the supply of cullet is not steady [9].

The basic raw materials of the glass (like silica sand, limestone and sodium carbonate) are cheap; however, the energy needed to make glass is huge. Recycling glass will have a great advantage in reducing the energy needed to produce new glass products. Using cullet in new glass container manufacturing has the following advantages:

- **Conserves energy and reduces the manufacturing costs.** Energy is conserved because cullet melts down at temperature lower than that required in melting the virgin raw material that is used in glass making. This also increases the furnace life which can be extended by as much as 15 to 20 percent depending on the amount of cullet being used. The conservation of
energy will also conserve natural resources such as fossil fuel. It was found that for every 1 percent increase in the use of cullet, 0.25 percent of the needed energy is saved [9].

- **Reduces the green house gas emissions.** Less energy needed by the furnace means reduced emissions of green house gases mainly CO\textsubscript{2}. It was found that 315 Kg of CO\textsubscript{2} is saved for every tonne of glass melted if recycled glass was used for new container manufacturing (that after accounting for transportation and processing) [15]. Moreover, the virgin material looses almost 15% of its input weight as waste gases, while no waste gases are released from cullet [6]. In general, energy reduction enables the manufacturing plants to run more efficiently and to reduce the operation costs.

- **Reduces the demand for raw material.** The glass raw materials are abundant in nature but still they must be extracted from our landscape. It was found that 1.2 tonnes of raw materials are preserved for every tonne of recycled glass used [15].

- **Reduces the amount of waste glass that needs to be landfilled.** Although glass is inert and it does not represent a direct threat to the environment but loosing it as a possible input for other industries and making it occupy large spaces in landfills is a waste of resources and is not a sustainable solution.

The container glass is unique in the recyclables manufacturing industry. A 12 ounce glass bottle, melted down and reformed yields a 12 ounce bottle with the same quality and with no by-products. Furthermore, the same glass can be recycled repeatedly while retaining its strength [9]. This feature makes glass 100 percent recyclable; that means glass should never reach a landfill.
1.1.6 The stages of Glass Container recycling

Glass containers are usually recovered from drop-off centers that receive solid wastes. Figure 1.7 shows a drop-off center in El Gouna recycling center located near El Gouna touristic village. Glass containers are collected in these drop-off centers either separated at the source or they are separated in the center upon receipt [9].

Figure 1.7: Mix of green and white glass containers left in a drop off area in El Gouna recycling center [16].

The processing of glass containers to cullet may or may not take place in the drop-off center. The glass containers recycling consists of:

- **Manual sorting**: glass containers are separated by color (green, brown, and mixed). Workers remove any large objects such as ceramic, stone, and plastic as the glass passes on a conveyor belt (figure 1.8) [17].

- **Crushing and screening**: glass containers are crushed by steel rollers to small pieces between 10 and 50 mm. After crushing, the crushed glass is screened by
a vibrating bar screen that removes corks, papers and any other larger items [17].

- **Ferrous metal removal**: the screened cullet passes over a large magnet that rotates on a drum whose main function is to remove any ferrous metals such as bottle caps, wires and steel [17].

![Figure 1.8: The glass bottles are collected manually from the conveyor belt](image)

- **Stone and ceramic removal**: cullet passes through another screen with a mesh base (about 19mm square) where stone and ceramic small items passes through the mesh [17].

- **Vacuuming**: cullet passes through a strong vacuum duct that sucks any paper like bottle labels [17].
• **Non-ferrous metal removal**: metals such as aluminum and lead that comes from the bottle's lid are removed. Cullet drops over a metal detector head that directs a strong air jet fire to the non ferrous metals directing them away from the cullet stream side [17].

• **Small ceramic and stone removal**: a laser beam is directed to the cullet. The beam is able to pass through the glass, but when it hits a piece of ceramic or stone it is reflected. When the laser beam is reflected, a computer fires an air jet that directs the small pieces of stone and ceramic away from the cullet stream through a split chute that is split into cullet stream and waste stream [17].

Some of the processing steps take place at the manufacturers' site or all the steps might take place at recycling centers that sells high quality cullet to the market.

Any foreign materials left with cullet such as metals, ceramics, and stones reduce the quality of the cullet. These foreign objects do not melt in the furnace with the glass and they form bubbles and stones in the glass bottles that weaken their walls. It is impossible to remove every single foreign material from cullet but glass manufacturers can accept a minimum amount of foreign materials. It is stated by a glass recycling company in the United Kingdom that sells cullet to the market that their quality control allows only 20 g of foreign materials in one ton of cullet (In order for recycled bottles and jars to meet certain manufacturing specifications, only amber or emerald cullet (crushed glass) can be used to make brown and green bottles respectively [17]).

Breaking glass before separation is an undesirable step because as mentioned earlier, in order to meet certain manufacturing specifications, only amber or emerald cullet can be used to make brown and green bottles respectively. So the broken glass
that cannot be color-sorted from the waste stream is of no value for the glass containers manufacturers. This type of mixed glass might be used in other applications. It can be used as a component of a composted waste product. Glass has the same physical properties as sand; that is why glass particles can be used in compost [9]. Another use for recycled glass that does not meet the manufacturing specifications is to use it as aggregate for glassphalt. Glassphalt is the same as the conventional asphalt except that some of the aggregates are replaced with crushed recycled glass [18]. Recent research studies proved that mixed cullet can be used to produce foam glass, which is an insulating material with excellent properties [19].

1.1.7 Usage of glass cullet

Although the fact that glass containers manufacturing is the primary market for cullet, glass cullet can be used in other industries [9]:

- Soil conditioner where glass is used to improve drainage and moisture distribution.
- Glassphalt for road pavement where crushed glass substitute a percentage of the aggregates.
- Glass wool as an insulating material.
- Reflective paints that are used in road signs.
- Artificial sand for beaches.
- Fiberglass.
- Abrasives.
- Lightweight aggregates in concrete.
- In glass polymer composites.
- Construction materials such as tiles, clay bricks and glascrete.
Foam glass for insulation and construction boards

1.2 Foam Glass

Foam glass is one of the promising products that can be made fully out of recycled glass. It is a valuable product that can be used in a range of engineering applications due to its cellular structure. Foam glass is a lightweight glass material with cellular structure. It is a heterophase system that consists of a solid phase and a gaseous phase. The solid phase is glass that constitutes the thin walls of the cells; the wall thickness is several micrometers thick. Inside the cells, there is the gaseous phase. Foam glass has a very low density (120 Kg/m$^3$) yet a relatively high compressive strength and dimensional stability [20]. These characteristics make foam glass a good candidate for thermally and acoustically insulating construction materials.

The foam glass consists of millions of sealed glass cells where every cell represents an insulating space. This makes it a highly effective insulating material. The closed cells structure of foam glass makes the material watertight since no diffusion can take place; this makes foam glass an efficient barrier against soil humidity [20]. Most importantly, foam glass can be manufactured fully out of waste glass with minimum addition of virgin materials; the thing that makes it an exemplary process for recycling wastes based on an industrial basis. Foam glass has many applications. However, the most prevalent application is as a heat insulating material. Its service parameters as a heat insulator surpass many other organic types of foam. It has a relatively high mechanical strength that facilitates its installation. It is resistant to water, moisture impermeable, incombustible, and it is biologically resistant because it does not putrefy or get moldy. In addition, it is chemically inert. All these properties
ensure the constancy of the thermal conductivity value of foam glass with time [20]. Foam glass insulation is an environmentally sustainable solution for recycling glass wastes. Figure 1.9 explains the foam glass processing technique adopted by Pittsburgh Corning Company for foam glass insulation [20]. Silica sand in addition to other additives are melted to produce glass. Glass is extruded in hollow tube then mixed with 66% post consumer glass. Then glass is milled to fine powder and mixed with carbon. The mixture is put in moulds and heated in furnace at 1000 °C. Foam glass is produced with millions of sealed pores that are two-thirds vacuum and one-third CO$_2$. Foam glass blocks are finally annealed to remove the internal stresses. Figure 1.10 shows a block of foam glass when it comes out of the sintering furnace. Figure 1.11 shows the Pittsburgh Corning foam glass products; they include slabs, boards, elbows and piping insulation segments.
Figure 1.9: Foam glass processing technique [20]
Foam glass is known as a thermal insulation material since the middle of the last century [21]. However, growing interest in foam glass nowadays is due to the energy problem. Fuel costs have tremendously increased. Heating residences is crucial for cold countries like Russia, North America and Western Europe. In Russia, 500-600 kW.h/m^2 of living space is consumed for heating residences. Heat loss means energy loss. Using effective thermal insulation will reduce fuel consumption for
heating by a minimum of 25% [21]. It will also decrease the harmful emissions generated from burning fuel. In 2007, the volume of heat insulating materials that were used in construction was 23-25 million m³; it was estimated that this amount will increase to 45-50 million m³ by 2010 (this amount includes industry and engineering systems) [21]. Table 1.4 summarizes the technical characteristics of different types of insulating materials including foam glass.

Table 1.4 states that the commercial value of the foam glass thermal conductivity is around (0.05-0.08) W/m.K. Comparing foam glass to other insulating material proves that foam glass has high compressive strength (> 0.7 MPa), higher water vapor impermeability, it has high maximum temperature of use (up to 450 °C) and most importantly incombustible. The data in table 1.4 states that mineral wool and plastic foam, widely used in construction, are combustible and release toxic emissions when burned. The heat insulating materials contain more than 5% polymeric binder that release toxic gases when burned [21]. On the other hand, foam glass in a fire resistant insulating material that is totally environmental friendly.

The thermal resistance of plastic foams drops by 25% after six months. They might absorb water vapor that result in corrosion, degradation and further decrease of the thermal resistance [21]. Table 1.4 states that the service life of mineral wool and plastic foam is less than 12 years, which is less than the life time of buildings and structures. However, foam glass has unlimited service life and it keeps its characteristics throughout its life time. The excellent service parameters make foam glass an effective insulating material in many applications.
Table 1.4: The technical characteristics of different types of heat insulating materials [21]

<table>
<thead>
<tr>
<th>Articles</th>
<th>Density, kg/m³</th>
<th>Compressive strength, kPa</th>
<th>Thermal conductivity, W/(m·K)</th>
<th>Maximum temperatures of use, °C</th>
<th>Water vapor permeability, mg/(m²·h·Pa)</th>
<th>Combustibility</th>
<th>Environmental evaluation</th>
<th>Lifetime, years</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral wool products:</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>heat-insulating tiles made of mineral wool on synthetic binders</td>
<td>200</td>
<td>40 – 120</td>
<td>0.066 – 0.060</td>
<td>100</td>
<td>0.380 – 0.600</td>
<td>Difficulty combustible</td>
<td>Toxic when burned (7 – 8% binder)</td>
<td>7 – 10</td>
</tr>
<tr>
<td>high-rigidity mineral-wool tiles</td>
<td>200</td>
<td>100</td>
<td>0.045</td>
<td>–</td>
<td>0.580</td>
<td>&quot;</td>
<td>Toxic when burned (10% binder) and inhaled</td>
<td>10 – 12</td>
</tr>
<tr>
<td>glass staple fiber tiles</td>
<td>175 – 200</td>
<td>–</td>
<td>0.049</td>
<td>180</td>
<td>–</td>
<td>&quot;</td>
<td>Toxic when burned (6% binder) and inhaled</td>
<td>7 – 10</td>
</tr>
<tr>
<td>Foam plastics</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>polystyrene foam</td>
<td>20 – 40</td>
<td>50 – 150</td>
<td>~ 0.038</td>
<td>70</td>
<td>0.050</td>
<td>Combustible</td>
<td>Toxic when burned</td>
<td>10 – 12</td>
</tr>
<tr>
<td>polyurethane foam</td>
<td>40 – 60</td>
<td>120 – 200</td>
<td>0.035</td>
<td>200</td>
<td>–</td>
<td>&quot;</td>
<td>Same</td>
<td>10 – 12</td>
</tr>
<tr>
<td>Heat-insulating tiles made of reol resins</td>
<td>50 – 100</td>
<td>50 – 200</td>
<td>0.040 – 0.046</td>
<td>130</td>
<td>–</td>
<td>&quot;</td>
<td>Harmful emissions</td>
<td>10 – 12</td>
</tr>
<tr>
<td>Cellular inorganic materials (foam concrete)</td>
<td>350</td>
<td>700 – 800</td>
<td>0.090 – 0.100</td>
<td>350</td>
<td>0.230</td>
<td>Incombustible</td>
<td>Environmentally clean</td>
<td>~ 10 (when protected from moisture)</td>
</tr>
<tr>
<td>Foam glass</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>blocks</td>
<td>120 – 200</td>
<td>≥ 700</td>
<td>0.050 – 0.080</td>
<td>450</td>
<td>0.001 – 0.004</td>
<td>&quot;</td>
<td>&quot;</td>
<td>Unlimited</td>
</tr>
<tr>
<td>gravel</td>
<td>80 – 100*</td>
<td>500 – 1000</td>
<td>0.030 – 0.050</td>
<td>450</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td></td>
</tr>
<tr>
<td>Keramzit (gravel)</td>
<td>250 – 350</td>
<td>500 – 1500</td>
<td>0.210 – 0.230</td>
<td>450</td>
<td>0.21</td>
<td>&quot;</td>
<td>&quot;</td>
<td>Long-lived</td>
</tr>
</tbody>
</table>

* Specific bulk mass.
1.2.1 Construction applications for foam glass thermal insulation

Foam glass is used for insulating walls, roofs, floors of buildings whether in cold or hot regions (foam glass is also a sound insulating material) [21]. Figure 1.12 shows layer of foam glass in concrete roof deck. The foam glass slab is fixed on the primed concrete by hot bitumen. The foam glass layer represents a secondary waterproof layer below the asphalt layer.

![Figure 1.12: Layers of concrete roof deck where foam glass slabs are used for insulation [20]](image-url)
Figure 1.13 shows internal wall insulated with foam glass. Foam glass reduces the effect of thermal bridges and prevent condensation damage that affect the interior finish. Foam glass boards are fixed to the wall by adhesives and mechanical fixings.

![Figure 1.13: Interior wall insulated with foam glass](image)

**1.2.2 Industrial applications for foam glass thermal insulation**

Foam glass is used for insulating systems where the operating temperature is below ambient; in addition to cryogenic processes systems. It is used also for insulating hot systems. The operating temperature ranges from -268 °C to 482 °C [20]. Examples are as follows:

- Industrial refrigerators and ships
- Low temperature pipelines
- Chilled water lines
- Low temperature storage spheres
- High temperature pipelines and equipments
- Underground piping
- Vertical storage tank walls and roofs [20]

Foam glass is suitable for underground applications because it is strong, resistant to moisture and corrosion. Having inorganic nature, it resists vermin, insects, microorganisms and it does not putrefy. Figure 1.14 shows the installation of two layers of foam glass insulation around a liquefied natural gas (LNG) pipe in Isle of Particle LNG phase 2 project in the UK. The project had a strategic national importance; that is why a reliable insulating material was chosen. Foam glass was chosen for insulating the LNG pipes because it is fire and vapor resistant with extended lifetime and can be installed rapidly with more flexibility [20].

Figure 1.14: LNG pipe insulated by two layers of foam glass [20]
Another possible application of foam glass insulation is in atomic energy applications where fire safety requirement is indispensable [21]. Table 1.5 summarizes the production volumes and prices for heat insulating materials in different countries. The price of 1 m$^3$ of foam glass ranges between 150-500 US dollars depending on the quality of the foam glass and the supply and demand. The Pittsburgh-Corning Europe (Belgium) is the largest company that produces foam glass in Europe. It produces 860,000 m$^3$/year [21].

Table 1.5: The production volumes and prices for heat insulating materials in different countries [21]

<table>
<thead>
<tr>
<th>Country</th>
<th>Type of heat insulation</th>
<th>Production volume per 1000 inhabitants, m$^3$</th>
<th>Price of 1 m$^3$, US dollars</th>
</tr>
</thead>
<tbody>
<tr>
<td>Russia</td>
<td>All types</td>
<td>53</td>
<td>47 – 54*</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>78 – 120**</td>
</tr>
<tr>
<td></td>
<td>Including foam glass</td>
<td>0.05</td>
<td>120 – 140***</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>300 – 400****</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>500****</td>
</tr>
<tr>
<td>Belarus</td>
<td>Foam glass</td>
<td>48</td>
<td>100</td>
</tr>
<tr>
<td>Japan</td>
<td>All types</td>
<td>350</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>Including foam glass</td>
<td>50</td>
<td>300</td>
</tr>
<tr>
<td>Finland</td>
<td>All types</td>
<td>400</td>
<td>–</td>
</tr>
<tr>
<td>USA</td>
<td>All types</td>
<td>500</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>Including foam glass</td>
<td>120</td>
<td>300 – 400</td>
</tr>
<tr>
<td>Germany</td>
<td>All types</td>
<td>70</td>
<td>–</td>
</tr>
<tr>
<td>Sweden</td>
<td>Same</td>
<td>600</td>
<td>–</td>
</tr>
<tr>
<td>Belgium</td>
<td>All types</td>
<td>350</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>Including foam glass</td>
<td>85</td>
<td>150 – 200*****</td>
</tr>
</tbody>
</table>

* Polystyrene foam
** Mineral wool tiles.
*** In Moscow.
**** In Siberia.
***** In Europe.
1.2.3 Other Applications for foam glass

Other than thermal insulation, foam glass can be used for many applications such as:

- Light weight gravel and filling material: the foam glass gravel might be the scrap generated during the foam glass blocks cutting process or they might be produced through a separate process. In this case the annealing step is not required [21]. Foam glass can be used as low weight filling material in construction projects (figure 1.15). It was used in road fill over a tunnel along one of the major roadways in Norway (road E6) [22]. Moreover, it can be used in filling trenches around embedded pipes [23].

![Figure 1.15: spreading light weight foam glass as a filling material [24]](image)

- Abrasive material: foam glass blocks might be used in stone washing industry; such as washing jeans. They can also be used for coating removal and surface preparation [22].
• Biological filter: a biological filter consists of a bed of rock, gravel or foam media over which wastewater flows downward causing a layer of microbial slime. The organic matter in the wastewater is adsorbed and absorbed by the layer of microbial slime that covers the media bed. The diffusion of the wastewater over the media is important in order to provide oxygen that the slime microbial layer needs for the biochemical oxidation of the organic matter [25]. Foam glass, as a highly porous material, has large surface area that will be highly efficient when used as a bed media for water treatment [24].

Foam glass production is affected by many processing parameters. The parameters mainly affect the foaming process and the properties of the developed foam glass. Sintering temperature is an important processing parameter. Sintering temperature is the temperature at which the glass powder/foaming agent mix is transformed into foam glass. Moreover, the amount of the foaming agent is a parameter that certainly affects the foaming process. Another important parameter is soaking time which is the time that the mix is left in the furnace, at the sintering temperature, in order to be transformed into foam. Glass powder particle size is another parameter that affects the properties of the developed foam glass. The color of the glass certainly has an effect on the properties of the foam glass because different glass color means different starting material since different glass colors have different compositions.

The objective of this research work is to develop the technology for transforming the waste container glass from MSW into high quality foam glass using sodium silicate solution as a foaming agent. The physical and mechanical properties of the developed foam glass will be studied. The relation between the experimental variables: sintering temperature, amount of foaming agent, soaking time, glass powder
particle size, glass powder color and the properties of the foam glass produced (thermal conductivity, compressive strength, density, pore morphology) will be thoroughly analyzed.
CHAPTER 2
LITERATURE REVIEW

2.1 Cellular solids

A cellular solid is a type of material that consists of "an assembly of cells with solid edges or faces, packed together so that they fill space" [26]. Cellular solids exist such as cork, wood, sponge, and coral in nature (figure 2.1). Moreover many foods are types of foams like bread, meringue, junk food crisp, and cake.

Figure 2.1: a) Cork b) Balsa c) Sponge d) Cancellous bone e) Coral f) Cuttlefish bone g) Iris leaf h) Stalk of a plant [26]
Man has used the natural cellular solids for centuries. Recently, man has made different types of cellular solids. Foaming techniques were developed for different types of materials such as polymers, metals, ceramics and glasses [26]. A familiar example of a polymeric foam is the disposable tea and coffee cup. Different types of foams are increasingly used in structural applications for thermal and acoustic insulation, for cushioning, and for absorbing kinetic energy from impacts. The unique combinations of properties that cellular solids provide are mainly derived from their cellular structure [26]. There are three structural types for cellular solids: two dimensional, three dimensional with open cells, three dimensional with closed cells. The first type is a two dimensional array of polygons packed in a plane; this structure is called honeycomb. The second type is polyhedron cells that fill space in three dimensions. Such three dimensional cellular material is called foam. If the cells connect through open faces, the foam is open celled. If every cell is sealed off from the neighboring cell, the foam is close celled. Some foams are partly open and partly closed (figure 2.2) [26].
Figure 2.2: a) 2D honeycomb b) 3D foam with open cells c) 3D foam with closed cells [26]

One of the most important features of the foam is its relative density ($\rho_b/\rho_t$) where $\rho_b$ is the bulk density of the foam and $\rho_t$ is the true density of the solid material from which the cell walls are made [26]. The relative density of cork is about 0.14; polymeric foams used for packaging, insulation and cushioning have relative densities between 0.05 and 0.2. Special ultra-low density foam can be made with a relative density of 0.001. When the relative density increases that means the walls of the cells are getting
thicker and the pore space shrinks. This means that the material is changing from a cellular structure to solid containing isolated pores as shown in figure 2.3. This transition happens at a relative density above 0.3 [26].

![Cellular structure vs solid containing isolated pores](image)

Figure 2.3: Cellular structure (left) vs. solid containing isolated pores (right) [26]

There are different techniques for foaming different types of materials. Polymers are foamed by introducing gas bubbles into the hot polymer. The bubbles grow and stabilize then the mixture is solidified by cooling or by cross linking [26]. The gas is introduced to the hot polymer by one of two methods; either by mechanical stirring or by mixing blowing agent into the polymer. Blowing agents are either physical or chemical. Physical blowing agents are inert gases such as Nitrogen that are forced as solution into the hot polymer at high pressure then they expand into bubbles by reducing the pressure. A low melting point liquid might be used alternatively; it is mixed into the hot polymer and volatilize on heating forming vapor bubbles. Chemical blowing agents are additives that combine to chemically react and release gases or that decomposes once heated. Glass
foam is foamed by techniques that are similar to those for polymers; mainly by the use of blowing agents [26]. The foaming process extends the range of properties of cellular solids. Figure 2.4 compares between the physical (density), mechanical (Young's modulus and strength), and thermal properties of foams vs. true solids:

Figure 2.4: The range of properties of foams (a) density (b) thermal conductivity (c) Young's modulus (d) compressive strength [26]
The extension of the properties range extends the engineering applications for foams that might not be achieved by using the true solids. The low density of foams makes it suitable for light, stiff components such as sandwich panels, also for any floatable component. The low thermal conductivity makes foam a good candidate for cheap, reliable thermal insulation. The low stiffness allows foams to be used as cushioning material. The low strength with large compressive strains makes foams suitable for energy absorbing applications. That is why they are used as a protecting material around computers and other devices [26].

2.2 Foam glass preparation

The production of foam glass might be done through two different processes. The first, consists of blowing gases into the molten glass. The second consists of foaming sintered fine glass powder by adding a foaming agent [26]. The glass powder: foaming agent ratio percentage (by weight) is usually optimum in the range (97-99.5) : (1.5-3) [27-30]. Foaming agents are additives that combine to chemically react and release gases or that decompose once heated. The main role of the additive is to create the gaseous phase during heating; it is added to the glass powder in small quantities. The additive is called pore-forming, gas-forming, blowing agent or foaming agent. Currently, most of the foam glass production is done using the foaming agent technique (the second method) [27-33]. The foaming agent technique is much less expensive and requires less sophisticated technology.
To produce foam glass through the powder method, the glass powder and foaming agent must be heated to a temperature above the glass softening point. During the thermal treatment of the glass powder and additives mixture, several processes take place. When the temperature reaches the softening temperature of glass, the glass powder starts to sinter and form a continuous sintered body. At that stage, the particles of the gas-forming agents are insulated by the softened glass. They start to emit gases after a certain temperature frothing the melted glass [34]. The emission of gases creates the insulated pores throughout the glass melt; this occurs where the particles of the gas-forming agents are blocked inside the softened glass. The foaming reaction must start only after the glass particles are sintered, otherwise the gas will escape from the glass powder. The gas evolution due to the chemical reaction between the glass powder and the foaming agent creates bubbles inside the glass melt. The bubbles will grow due to the increase of the gas pressure inside the bubbles. That takes place due to the chemical reaction that generates gases and due to the increase of the sintering temperature inside the furnace. As the temperature increases, the surface tension of the glass melt decreases. Consequently, the external pressure exerted on the bubbles (by surface tension) decreases enhancing the bubble growth. The growth rate of the bubbles mainly depends on the glass melt viscosity and surface tension. The viscosity acts as a resistance against bubble growth [35]. The characteristics of the foam glass obtained and the shape of the pores mainly depend on the amount and type of the foaming agent. During sintering, the glass particles sinter together enclosing the voids inside which the foaming agent is enclosed. Initial small bubbles are formed; then the bubbles grow and the glass surrounding the bubbles must
expand forming a thinner lamella surrounding the gas bubble (as illustrated in figure 2.5). Transforming glass powder into foam glass takes place through a number of steps: Heating (drying the mixture of the glass powder and foaming agent), sintering, and pre-cooling (annealing).

![Figure 2.5: Schematic representation of the bubble formation and growth in a mixture of glass/carbon powder mixture upon sintering to produce foam glass [35].](image)

2.3 Foaming agents: types and mechanisms

Foaming agents are of two types: neutralization and redox agents. Neutralization agents include salts (carbonates or sulphates) that when heated decompose emitting \( \text{CO}_2 \). The gas release is intense so that it breaks the walls of the isolated pores during its decomposition making the internal structure look like a maze system of merged cavities. This type of foam glass has high water absorption and soundproof parameters. The neutralization foaming agent ensures high water absorption of approximately 50-70 % [34]. On the other hand, the redox agents such as carbon containing materials like coke, soot, anthracite, graphite, carbon black, and less frequently silicon carbide passes through an oxidation reaction by the gases dissolved in the glass melt such as oxygen and sulfur anhydride. The oxidation process releases gases that produce foam glass inside which
sealed pores prevail. That type of foam glass is mainly used as a heat insulating material. Using redox foaming agent ensures lower water absorption of approximately 10-15% [34]. Foam glass with sealed pores has lower thermal conductivity than foam glass with communicating pores. Thermal conductivity depends on the type of porosity: its shape, size, and its distribution inside foam glass. The thermal conductivity depends mainly on a ratio between the solid and the gaseous phase. The pores inside foam glass, which represent the gaseous phase, exceed 90% and gases in general have low thermal conductivity. That is why foam glass has low thermal conductivity in general and lower thermal conductivity values when its pores are sealed [34].

Carbonaceous materials were used as foaming agents in various studies [21, 30-33]. Bernardo et al. [32] reported that commercial carbon black was used as foaming agent with the amount of 0.5 wt % at a sintering temperature of 850 °C for 30 min. The produced foam glass had closed porosity (around 95%) and a density lower than 0.3 g/cm³. SiC is an effective foaming agent that was used by many researchers [30-31, 33]. Wu et al. [30] produced foam glass with uniform pore morphology by adding 2 wt. % SiC and heat treatment at T=1000°C for 5 min. The compressive strength of the produced foam glass was in the range 1.2-1.7 MPa. The only drawback of SiC is that it decomposes at high temperature (950-1150 °C), and thus increases the production cost despite the short time (5 min).

Carbonates have been used as foaming agents in various studies [28, 32]. For example, CaCO₃ was used as foaming agent in [32]. The decomposition of CaCO₃ leads to extensive foaming resulting in open cell morphology. CaCO₃ usually agglomerates
when mixed with glass powder. That is why the mixture needed long-standing mixing for 0.5 hour to achieve homogeneous distribution of pores. Fernandes et al. [28] used CaMg (CO$_3$) and calcite based sludge derived from the marble cutting process (consists of about 99 % CaCO$_3$) as foaming agents. The foams that comprised 2 wt. % CaMg(CO$_3$) and 2 wt. % marble waste (separate samples) had low densities (around 0.36 g/cm$^3$) and compressive strength (around 2.4 MPa) at sintering temperature = 850°C. Fernandes et al. [28] achieved better mechanical properties (compressive strength around 2.4 MPa) at low sintering temperature (850 °C) than Wu et al. [30] that produced foam glass, using SiC as a foaming agent, with lower mechanical properties (maximum compressive strength 1.7 MPa) at much higher temperature (1000 °C).

2.3.1 Sodium silicate solution as a foaming agent

Sodium silicate is the common name for sodium metasilicate (Na$_2$SiO$_3$). It is available in solid state (anhydrous form) and in aqueous solution (hydrates). There are number of hydrates that have the formulae Na$_2$SiO$_3$.nH$_2$O (n=5, 6, 8, 9). Water glass is the commercial name of sodium silicate in aqueous solution; its chemical formula is Na$_2$SiO$_3$.9H$_2$O. It is a glassy and colorless alkaline solution [36]. Crystal like lumps with white or grayish white color of sodium silicate (as shown in figure 2.6) is dissolved in water to form a dense liquid of sodium silicate solution (water glass). Sodium silicate is originally formed by the reaction [36]:

$$Na_2CO_3 + SiO_2 \rightarrow Na_2SiO_3 + CO_2$$
Figure 2.6: Sodium silicate Na$_2$SiO$_3$ [36].

The sodium silicate is best dissolved in water if it is heated with water under pressure [37].

The water glass has excellent binding properties. It is used in automotive repair to seal leaks at the head gasket. A jar of water glass is poured inside the radiator. The water glass will circulate with the radiator water and reach the hotspot of the motor. At $T = 99$-$104 \, ^\circ$C, the sodium silicate will lose water molecules and become a very powerful sealant that will not remelt below $815 \, ^\circ$F [36].

The shape and size of the pores in foam glass is mainly determined by the amount of the foaming agent as well as by its type and state. Using solid foaming agent will not make it easy to achieve fine homogeneous isolated pores because the foaming agent agglomerates inside the powder mix. However, liquid foaming agent will not create such problem and it can be used with high efficiency to create micrometer pores in powder compacts [30]. Water glass might be used as a liquid foaming agent. Once it is mixed and sintered with glass powder, it will create ultrafine isolated pores.
Manevich et al. [21] reported that sodium silicate solution was used as a foaming agent for foam glass production. The foaming agent consists of 85% sodium silicate solution, 12% water, 3% glycerin. The glass powder: foaming agent ratio percentage is 93.5 : 6.5. Other authors [19, 38-39] reported the use of water-soluble glass as a foaming agent to produce foam glass granules with high porosity from container and building glass cullet. The sodium silicate, in the form of aqueous solution acts as a binder. It is indicated in [19,40] that treating glass with alkali decreases the melting temperature of glass by 50-100 °C. The glass is destructed by alkali in a complex physicochemical process that causes the formation of an intermediate layer on the glass surface. This layer has a composition that differs from the composition of initial glass.

Eidukyavichus et al. [19] reported that that upon adding sodium silicate solution to the glass powder, they chemically react (already at room temperature). Consequently, Silicates are formed containing bound water (Bound water is chemically bounded by becoming part of a crystal lattice). The bound water is released at temperature range (600-620 °C) facilitating frothing the foam glass. Figure 2.7 compares the Differential Thermal Analysis (DTA), Differential Thermal Gravimetry (DTG), Thermogravimetry (TG) of non treated cullet and cullet treated with sodium silicate solution.
DTA, DTG, TG of non treated cullet (1), cullet treated with sodium silicate solution (2) [19]

The DTA curve has endothermic effect at T = 100, 620, 760, 960 °C. The points at which endothermic effect takes place along with weight loss denotes the removal of adsorbed water (dehydration of water glass) and they are resulted also from the formation of the products of water glass reacting with cullet. This endothermic effect is seen at T= 100 °C. The endothermic effect at 100 °C is wider for the cullet treated with water glass than the non treated cullet. At T= 600-620 °C, the chemically bonded water is released. At T=760 °C, the water glass had melted and reacted with glass. At T=960 °C, the glass that did not react had melted.
The foaming process of the glass treated with water glass was further investigated by a high-temperature microscope [19]. Figure 2.8 shows the high-temperature microscope photos taken for glass powder treated with sodium silicate solution at different sintering temperature.

Figure 2.8: High temperature microscope photos for glass powder treated with sodium silicate solution [19]
The sample volume increase in temperature range (600-750 °C) creating fine pores foam glass. At T= 760 °C, the volume of the sample does not further increase, but the water glass melts. At T=795 °C, the pores of the cellular structured foam glass grow creating coarser pore cellular structure. Then at T=880 °C, the sample shrinks in size because the water glass and the products of the reaction of glass with water glass are completed melted.

2.4 Annealing of foam glass

After reaching the foaming temperature and completing the required soaking time, the foam glass must be thermally treated (annealed). If the foam glass was taken out of the oven to room temperature directly, it will crack. When the glass is quickly cooled from elevated temperature, thermal stresses develop inside the material due to the high rate of cooling. Consequently, thermal contraction takes place between the surface of glass and the interior regions. Annealing relieves the internal stresses that exist inside the material after sintering by cooling the glass at slow rate. After annealing, the produced foam glass is stronger and more stable. Annealing point is the temperature at which the atomic diffusion is rapid enough such that any thermal stresses inside the material will be removed within approximately 15 min [4]. From figure 1.2, the softening point of the soda lime glass is around 700 °C. The annealing point is around 500 °C.
Bernardo et al. [32] performed the annealing process by drastically cooling the produced foam glass at a rate more than 10 °C/min from the sintering temperature 950 °C to 600 °C then slowly cooled to 500 °C at a rate approximately 1 °C/min then quickly cooled to room temperature at a rate approximately 5 °C/min. The cooling of the foam glass from the annealing point to the strain point must take place at a very slow rate (0.2-0.8 °C/min) to avoid any conservation of thermal tension inside the foam glass body [35]. Another research [27] states that the foam glass (using soda lime glass / SiC mixture) was annealed at T = 600 °C for 30 min then slowly cooled to room temperature. Another experiment [27] states that the foam glass (using soda lime glass / carbon black mixture) was annealed by cooling it at room temperature at very slow rate, approximately 2 °C/min.

2.5 Use of cullet from different chemical compositions in foam glass production

The utilization of the glass waste in most of the industrial application requires a strict level of purity. For producing conventional (container glass), only cullet with well-controlled composition is acceptable. The use of glass waste in engineering applications where no strict requirement of purity is needed is highly required in order to achieve 100% recycling of glass wastes. Foam glass production is an exemplary process for fully recycling large amount of waste glass to produce high quality foam. Eidukyavichus et al. [19] used cullet of different chemical compositions (mixture of window and container glass) to produce foam glass. Sodium silicate in the form of aqueous solution of water
glass (1 part water glass: 1.5 parts distilled water) was added to the mixture to homogenize the chemical composition of the glass cullet batch in addition to its technological properties. Adding 10 % water glass to glass cullet decreased the glass softening temperature by 15-19 %. It was reported in [21] that the initial glass for producing foam glass might be in-house or purchased cullet or a mixture of both. The initial raw material might be vertically drawn sheet glass, float glass, rolled, colorless bottle, green glass. While the use of brown containers, electric lamps and lead crystal requires additional processing, adding sheet glass and container glass (colorless and green) to the initial material of foam glass is proposed to achieve high quality and low cost foam glass [21].

2.6 Reducing sintering temperature and soaking time for energy conservation in foam glass production

It is a concern to minimize the thermal treatment temperature range and soaking time (suitable for the selected raw material) during foam glass production in order to conserve energy and to speed up the processing cycles. The authors of [29, 33] report that reducing the firing temperature, produced a more homogenous foam glass with better mechanical properties. Liaudis et al. [29] worked on reducing both firing temperature and soaking time. They reported that increasing the soaking time from 7 to 15 min at T=850 °C did not decrease significantly the density of the foam glass. However, the compressive strength decreased significantly. By limiting the soaking time, the cell coalescence and the transformation of the cell size distribution from monomodal to bimodal will be
limited. Mear et al. [33] stated that by sintering foam glass at $T = 750 \, ^\circ C$, more homogeneous samples with narrow pore size distribution was obtained.

### 2.7 Coalescence phenomenon, pore morphology and strength of foam

The pore size, shape and distribution inside foam glass is affected by many factors including type and amount of foaming agent, sintering temperature, soaking time and glass powder particle size. Wu et al. [30] used SiC to foam a mixture of 80 wt.% bottle glass cullet and 20 wt.% coal pond ash. They reported that as the sintering temperature increases, the pores agglomerate or coalesce leading to larger pores. Since the amount of material (solid glass) is constant, the prevalence of larger pore means that the total number of pores decreases. The walls separating the pores are getting thicker as the sintering temperature increases and the number of pores decreases. This effect is illustrated by figure 2.9.

![Figure 2.9](image_url)

**Figure 2.9**: The effect of sintering temperature on wall thickness, roundness, and pore diameter of foam glass with 2 wt% SiC [30].
The pore size distribution for foam glass at different sintering temperature is shown in figure 2.10.

![Pore size distribution graph](image)

Figure 2.10: Pore size vs. number of pores for foam glass sintered at different sintering temperature [30].

It is noticeable from figure 2.10 that the foam glass sintered at lower temperature had smaller spread of size range. They have larger number of pores with a narrower pore size range. As the sintering temperature increases, the spread of size range is getting larger; i.e. lower number of pores with wider pore size range, see the distribution at T=1100 °C [30].
An important factor that affects the pore morphology is the particle size of the glass powder. The homogeneity of the pore size distribution is affected by the glass particle size. A research study [27] reported that as the particle size of glass powder increases, the average cell size increases (as shown in figure 2.11) and that using fine glass powder achieves higher homogeneity in pore size distribution.

Figure 2.11: Pore diameter as function of particle size of glass powder (soda lime glass powder / 5 wt. % SiC ) [27].

It is usually reported that as the foam glass density decreases, the compressive strength decreases, as a general rule [27, 41]. However, some research studies reported the opposite. It was observed in [27] that when high sintering temperature was used (heating soda lime glass to 950 °C and holding for 30 min then heating to 1000 °C and holding for 30 min), the distribution of the pore size was inhomogeneous (keeping the particle size of the glass powder constant). The high foaming temperature produced foam with coarser structure; that means larger pores and thicker struts. That coarse-structure
foam had higher density but lower resistance due to the existence of strength-decreasing large pores in the foam. In addition, large voids existed inside the struts; the thing that weakened its resistance. The strut strength decreases as its thickness increases. Figure 2.12 illustrates that observation.

![Figure 2.12: Strut thickness vs. compressive strength and density of foam glass [27].](image)

The compressive strength of the foam glass does not only depend on its density. It depends also on the pore size, shape and distribution inside the foam. It was concluded by [41] that the smaller the cell size, the stronger the foam. It was reported in [27] that foam glass samples with comparable densities have distinct mean pore size (see figure 2.13).
From the figure, it shows that samples might have comparable densities but different compressive strength. Furthermore, for some samples, as the density increases, its compressive strength decreases.

The density of foam glass is affected by many factors including sintering temperature, soaking time, type and amount of foaming agent, and glass powder particle size. It was observed by [27] that as the soaking time increases the density decreases till it reaches a minimum value at a critical soaking time; then the density re-increase (see figure 2.14).
Concerning the relation between the density of the foam and the amount of foaming agent. It was reported in [32] that a minimum relative density of 0.08 is reached at 12.5 wt. % SiC based waste. However, as the percentage of SiC based waste increased beyond 12.5 wt. %, the relative density started to increase due to the coalescence phenomenon (as shown in Figure 2.15).
2.8 The compressive properties of cellular materials and foam glass

The most important mechanical property of foam glass is the compressive strength because it is a brittle fragile material. Gibson and Ashby [26] explained the
mechanism with which brittle foam deform during compression as shown in figure 2.16.

The deformation follows three regimes:

- **Linear elastic**: during first loading the cell walls bend in a linear elastic behavior (that means that the material that constitutes the cell wall is linear elastic).

- **Plateau of approximately constant stress**: once a critical stress is reached, the cells begin to collapse by brittle fracture (this deformation is not recoverable).

- **Steeply rising stress (densification)**: at high strains, the cell walls are collapsed to the extent that the opposing cell walls touch or the fragments of the cell walls are packed on each other. Further compression will compress the material of the wall itself giving the densification of the final regime, which is a steep rising of the stress-strain curve.

Figure 2.16: Compressive stress-strain curves for elastic brittle foam [26].
The larger the thickness of the cell walls, the larger the relative density of the foam and the higher the material resistance to collapse. As the relative density of foams increase, the stress plateau goes up and the strain, at which the densification starts, decreases [26].

2.8.1 Sample size, crosshead speed and compressive strength calculation

The ASTM standard for cellular glass thermal insulation (ASTM C 552-07) [42] states that the test specimen to be compressed must be a half-block with minimum acceptable dimensions 200 by 200 mm. Preparing samples with 200 by 200 mm will consume a huge amount of raw material. It is suitable in a commercial scale but in research scale it is unpractical. Kitaigorodskii et al. [43] prepared specimens for compression as cubes or rectangles of size below 3x3x3 cm. Kitaigorodskii et al. [43] prepared samples as cubes of size between 1.5-3 cm. Wu et al. [30] compressed cubic samples of size between 1-1.2 cm at crosshead speed of 5 mm/min. In [29], rectangular samples with average size 8x8x3 mm were compressed. Mear et al. [33] used rectangular samples of size 5x5x12.5 mm for compression tests. Bernardo et al. [32] compressed cubic samples of approximately 30 mm edges with crosshead speed of 0.5mm/min. Bernardo et al. [32] used samples of about 15x15x10 mm with crosshead speed of 2 mm/min. The C165-07 Standard Test Method for Measuring Compressive Properties of Thermal Insulations [44] states that the crosshead speed must not exceed the range (0.25 to 12.7 mm/min) for each 25.4 mm of specimen thickness.
Fernandes et al. [28] stated that the crushing strength was calculated by dividing the maximum load of the first plateau of the stress-strain curve by the cross sectional area. The ASTM standard for cellular glass thermal insulation (ASTM C 552-07) [42] states that the compressive strength is calculated by dividing the load at the failure point or at a definite yield point by the cross sectional area.

### 2.9 The properties of recently produced foam glass

The Pittsburgh Corning foam glass company [20] determines the properties of foam glass for high load bearing applications such that the average density, for all grades, is (0.12-0.16 g/cm$^3$), the compressive strength is (0.8-1.6 MPa) and thermal conductivity at T=10 °C is (0.043-0.048 W/(m.K)).

The recently produced glass foam has density in the range (0.18-0.3 g/cm$^3$) and compressive strength (0.7-2.5 MPa) [28, 30, 32]. The typical range of thermal conductivity of the recently produced foam glass is (0.05-0.08 W/(m.K)) [21]. Table 2.1 shows the properties of some recently produced foam glass samples.
Table 2.1: The properties of foam glass samples [45]

<table>
<thead>
<tr>
<th>Sample No.*</th>
<th>Foaming temperature, °C</th>
<th>Foaming agent</th>
<th>Mass content, %</th>
<th>Compressive strength, MPa</th>
<th>Porosity, %</th>
<th>Average pore size, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>910</td>
<td>Chalk</td>
<td>2.5</td>
<td>373</td>
<td>2.7</td>
<td>85.0</td>
</tr>
<tr>
<td>2</td>
<td>910</td>
<td>&quot;</td>
<td>2.5</td>
<td>340</td>
<td>2.9</td>
<td>86.4</td>
</tr>
<tr>
<td>3</td>
<td>910</td>
<td>&quot;</td>
<td>2.5</td>
<td>152</td>
<td>1.9</td>
<td>94.0</td>
</tr>
<tr>
<td>4**</td>
<td>No data</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>910</td>
<td>Chalk</td>
<td>2.5</td>
<td>241</td>
<td>2.7</td>
<td>90.0</td>
</tr>
<tr>
<td>6</td>
<td>790</td>
<td>Carbon, carbon black</td>
<td>0.9</td>
<td>210</td>
<td>2.0</td>
<td>92.0</td>
</tr>
<tr>
<td>7</td>
<td>910</td>
<td>Chalk</td>
<td>2.5</td>
<td>395</td>
<td>2.1</td>
<td>84.0</td>
</tr>
<tr>
<td>8</td>
<td>900</td>
<td>Carbon black</td>
<td>0.2</td>
<td>329</td>
<td>3.1</td>
<td>86.7</td>
</tr>
<tr>
<td>9</td>
<td>910</td>
<td>Chalk</td>
<td>2.5</td>
<td>238</td>
<td>2.2</td>
<td>90.5</td>
</tr>
</tbody>
</table>

* In all cases, the specific surface area of the batch was 400 m²/kg.

** Sample from Foamglass Co.
CHAPTER  3

EXPERIMENTAL PROCEDURE

This chapter discusses the experimental method that was used to develop foam glass and to study the effect of different parameters on its properties. The chapter is divided into three parts. The first part deals with characterizing all the starting materials used in the experimental work and their preparation steps. The second part deals with the sampling technique. The third part deals with the experimental methodology followed to produce foam glass. The fourth part deals with the testing procedures for evaluating foam glass properties.

3.1 Materials

3.1.1 Raw material

The starting material used in the experimental process was waste container glass with different colors (white, green, brown). The foaming agent used was sodium silicate solution (water glass). Water was used, with some compositions, as a binder.

3.1.1.1 Waste container glass

Waste container glass was used as the starting material (true density = 2.5 g/cm³). The container glass is collected from the municipal solid waste (MSW). The recycled container glass is usually contaminated from the MSW and it must be washed prior to crushing.
3.1.1.2 Sodium silicate solution

Sodium silicate solution (Na$_2$SiO$_3$.9H$_2$O) in the form of water glass was used as the foaming agent

**Sodium silicate composition:**

Na$_2$O = 7.8-8.2 wt.%

SiO$_2$ = 26.6-27 wt.%

Silica modulus (molar ratio of SiO$_2$ to Na$_2$O) = 3.41-3.51

Density = 1.426 g/cm$^3$ (measured at room temperature around 32°C)

3.1.1.3 Water

Regular tap water was added to the compositions that had $\leq$ 12 wt. % sodium silicate solution to enhance the binding properties of the mixture. Water was added (for all samples that needed additional water) in the amount of 6 wt.%.

3.1.2 Material preparation

The recycled container glass needed some preparation steps before use. It was washed, dried, crushed and milled, sieved with different sizes. All these preparation steps were repeated separately for white, green and brown glass. Figure 3.1 shows a process flow diagram that illustrates the steps of preparation for glass powder.
Figure 3.1: Process flow diagram for container glass preparation
3.1.2.1 Washing glass container

The glass container was washed prior to crushing to clean it from organic wastes or any other type of contaminants. A regular washing machine was used for washing (figure 3.2). A basket made out of steel network was fitted inside the washing machine to hold the glass container (figure 3.3). The main role of the basket was to keep tiny cullet from entering the fan or the discharge pipe of the washing machine. It allowed the water with detergent to access the glass container and eased the collection of the washed container glass.

Figure 3.2: Regular washing machine (left), steel basket that holds glass container (right).
3.1.2.2 Drying and crushing glass container

The washed glass container was left to dry in the air for 24 hours then crushed manually by a hammer.

3.1.2.3 Grinding Glass cullet

Clean glass cullet, after drying and crushing, was fed to a ball mill. The ball mill rotates at 40 rpm to grind glass cullet into fine powder (figure 3.4).
3.1.2.4 Sieving glass powder

The glass powder was sieved into 3 particle sizes: size 1 = 75 µm, size 2 = 150 µm, size 3 = 250 µm (figure 3.5). Any contaminants that were remaining with the glass powder, like pieces of papers or metals, were separated and removed in this stage. The contaminants remained at the top of the sieves; it was further collected and removed.

Figure 3.5: Set of sieves that correspond to sizes 75, 150, 250 µm fixed on a shaker

All the previous preparation steps were repeated separately for white, green and brown container glass.
3.2 Sampling

Mixes of glass powder, foaming agent (sodium silicate solution) and water (when needed) were prepared. Different variables such as glass color, amount of foaming agent, glass powder particle size, sintering temperature and soaking time were varied with different compositions to study their effect on the characteristics of the produced foam glass. Twenty two compositions were prepared for analysis. Table 3.1 describes the variables associated with every composition.

Table 3.1: The compositions of foam glass prepared with different variables

<table>
<thead>
<tr>
<th>Composition number</th>
<th>Glass color</th>
<th>sodium silicate solution wt. %</th>
<th>Particle size µm</th>
<th>Sintering temperature °C</th>
<th>Soaking time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>white</td>
<td>12</td>
<td>75</td>
<td>750</td>
<td>30</td>
</tr>
<tr>
<td>2</td>
<td>White</td>
<td>12</td>
<td>75</td>
<td>800</td>
<td>30</td>
</tr>
<tr>
<td>3</td>
<td>White</td>
<td>12</td>
<td>75</td>
<td>850</td>
<td>30</td>
</tr>
<tr>
<td>4</td>
<td>White</td>
<td>12</td>
<td>75</td>
<td>900</td>
<td>30</td>
</tr>
<tr>
<td>5</td>
<td>White</td>
<td>19</td>
<td>75</td>
<td>750</td>
<td>30</td>
</tr>
<tr>
<td>6</td>
<td>White</td>
<td>19</td>
<td>75</td>
<td>800</td>
<td>30</td>
</tr>
<tr>
<td>7</td>
<td>White</td>
<td>19</td>
<td>75</td>
<td>850</td>
<td>30</td>
</tr>
<tr>
<td>8</td>
<td>White</td>
<td>19</td>
<td>75</td>
<td>900</td>
<td>30</td>
</tr>
<tr>
<td>9</td>
<td>White</td>
<td>6</td>
<td>75</td>
<td>850</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10</td>
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<td></td>
<td>Color</td>
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<td>13</td>
</tr>
<tr>
<td></td>
<td></td>
<td>White</td>
<td>White</td>
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<td>White</td>
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<tr>
<td></td>
<td></td>
<td>32</td>
<td>12</td>
<td>12</td>
<td>12</td>
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<td></td>
<td>30</td>
<td>10</td>
<td>20</td>
<td>40</td>
</tr>
</tbody>
</table>

Tab water was added to the compositions that had $\leq 12$ wt. % sodium silicate solution to enhance the binding properties of the mixture.

- Compositions 1, 2, 3, 4, 5, 6, 7, 8 were prepared to study the effect of sintering temperature. They were prepared with two different amounts of foaming agent (sodium silicate solution) (12 and 19 wt. %) and subjected to sintering
temperature that ranged from 750-900 °C with increment of 50 °C, using white glass with particle size 75 µm, soaking time 30 min.

- Compositions 9, 3, 7, 10 were prepared to study the effect of the amount of foaming agent. They were prepared with different amounts of sodium silicate solution (6, 12, 19, 32 wt% sodium silicate solution), using white glass with particle size 75 µm, sintering temperature 850 °C, soaking time 30 min.

- Compositions 11, 12, 3, 13 were prepared to study the effect of soaking time. They were prepared with different soaking time (10, 20, 30, 40 minutes), using white glass with particle size 75 µm, sintering temperature 850 °C, 12 wt. % sodium silicate solution.

- Compositions number 3, 14, 15, 4, 16, 17, 18, 19, 20 were prepared to study the effect of glass powder particle size. They were prepared with three particle sizes (size 1 = 75 µm, size 2 = 150 µm, size 3 = 250 µm) and sintered at different sintering temperature (850, 900, 920 °C), using white glass with 12 wt. % sodium silicate solution, soaking time 30 min.

- Compositions number 3, 21, 22 were prepared to study the effect of container glass color on foam glass. They were prepared with different colored glass powder (white, green, brown container glass), using particle size = 75 µm, 12 wt. % sodium silicate solution, sintering temperature 850 °C, soaking time 30 min.
3.3 Experimental Procedure

The glass powder, sodium silicate solution and water were first weighted using a digital laboratory balance (maximum capacity 1000 g, readability 0.01 g, Adam, England) (figure 3.8). The sodium silicate solution was dissolved first in the water (water was needed only with samples that contains less than 19 wt% of sodium silicate solution) before adding it to the glass powder. The mixture of glass powder, sodium silicate solution and water were then mixed for 5 minutes in a rotary mixer (220 V, 800 W, 350-4306 rpm, Moulinex, France) (figure 3.9). Figure 3.6 (photo 1 and 1) shows the glass powder and the wet glass powder/sodium silicate solution mixture. The mixture was then cold compacted at 10 Tons, using a uniaxial laboratory hydraulic press (figure 3.10) for 20 min into rectangular slab of 15 x 15 x 1.5 cm (figure 3.7, photo 3). The slabs were dried in an oven (figure 3.11) for 1 hour at T=200°C. The dried slabs were sintered, in an electric laboratory furnace (figure 3.12); they were put directly at temperature in the range (750-920 °C). After completing the required soaking time, samples were severely cooled by natural convection at a rate (40°C/min) until the temperature reaches 600 °C in order to freeze the evolution of the microstructure. Then the foam glass slabs were slowly cooled from 600 °C to 500 °C at a rate (1 °C/min) to be annealed in a temperature close to, but slightly lower, than the soda lime glass softening temperature (700 °C); then cooled to room temperature at a rate (25 °C/min) (figure 3.7, photo 4, shows a foam glass slab after annealing). Figure 3.6 illustrates the processing steps for foam glass production.
Foam glass samples were machined using a bench-type circular saw 600 W (2800 rpm) with carbide saw blade (figure 3.13); cooling of the cutting tool was not needed. Figure 3.14 shows foam glass pieces with different sizes cut with high precision.
Figure 3.7: (1) glass powder (2) glass powder/sodium silicate solution mix (3) pressed slab prior to drying (4) foam glass slab

Figure 3.8: Digital laboratory balance (Adam, England)
Figure 3.9: Rotary-type mixer (Moulinex, France)

Figure 3.10: Laboratory-type hydraulic press
Figure 3.11: Oven for drying pressed pellets

Figure 3.12: Furnace for sintering foam glass
One of the major problems that was faced during the initial experimentation stage was choosing the suitable type of molding that best suits foam glass. Appendix A shows the trials that were done.
3.4 Testing

3.4.1 Density

The bulk density of foam glass was measured by dividing the mass by the total volume of the foam glass. The volume of the foam was measured geometrically (because if the foam was immersed in any fluid, it may absorb some of the fluid due to its permeable porous nature, which will give wrong results). Standard deviation value for bulk density was less than 0.05.

3.4.2 Compressive strength

Compressive strength was measured using a screw-driven universal testing machine (Instron 5569, USA) (figure 3.15) using a 50 KN load cell, with a crosshead speed of 2mm/min employing cubic samples with average size 2 cm (figure 3.16). The samples were compressed to a deformation 5 mm. Three samples from every composition were tested. For samples having irregular surfaces, they were rubbed on silicon carbide abrasive paper to obtain flat parallel surfaces prior to compression. Standard deviation value for compressive strength was less than 3.
Figure 3.15: Mechanical screw-driven universal testing machine, Instron (left), cubic foam glass sample between compression plates (right).

Figure 3.16: Cubic sample of foam glass prior to compression.
3.4.3 Thermal conductivity

Samples size was 3x3x10 cm. They were dried at 110 °C for 24 hours before testing. The environmental test conditions were: temperature = 24 °C and relative humidity = 25 %. The thermal conductivity was measured by the Housing and Building National Research Center according to ASTM D5334 “standard test method for determination of thermal conductivity of soil and soft rock by thermal needle probe procedure”. Morphology

The morphology of selected samples of foam glass was studied using a scanning electron microscope (SEM) (Zeiss Leo Supra55, Germany).

3.4.4 The energy dispersive X-ray spectroscopy (EDX)

The elemental composition of the foam glass was analyzed using EDX (Instruments Inca, Oxford, England).
CHAPTER 4
RESULTS AND DISCUSSION

When sodium silicate solution (Na$_2$SiO$_3$.nH$_2$O) was mixed with glass powder and dried in the oven for 1 hour at 200 °C, all the adsorbed water was removed. During the sintering process, the chemically bounded water started to be released at temperature around 620 °C [19]. The glass transition temperature for soda-lime glass is around 570 °C [32], slightly lower than the foaming reaction temperature of the sodium silicate solution. At 570 °C the glass particles started to sinter together enclosing tiny voids. The formed liquid phase material hampers the release of the gaseous phase (that occurs at 620 °C). The voids started to grow into bubbles when the foaming reaction started to release gases. As the temperature increased, the viscosity of the glass melt decreased and the pressure inside the bubbles increased, which caused the bubbles to grow. The intensive release of the bounded water, along with the low viscosity of the glass melt (associated with the increase of the sintering temperature) were responsible for frothing the glass melt.

4.1 Compressive strength behavior and calculation

The model of the elastic-brittle foam compression behavior [2.17] described by Gibson and Ashby [26] was not exactly detected in the compression curves of the foam glass samples. The regimes, explained by Ashby for elastic-brittle foam, were detected in
somewhat in a different pattern in the foam glass compression curves. The Ashby’s regimes for compression behavior of elastic-brittle foam were explained in chapter 2.

Figure 4.1 shows the compression stress-strain curve for foam glass with sodium silicate solution amount 12wt. %, sintered at T=850 °C for 30 min with particle size = 75 µm (composition 3). The compression curve is divided into the typical regimes that characterized all the compression curves of the tested foam glass.

![Compression stress-strain curve for composition 3](image)

Figure 4.1: Compression stress-strain curve of foam glass (composition 3) with the typical regimes.
The three regimes that were clearly detected in the stress-strain curves (figure 3.16) were:

- **The linear elastic**: during first loading the cell walls bend in a linear elastic behavior

- **The plateau of brittle crushing**: as the material reached a critical stress, the walls around the pores started to bend and collapse in an unrecoverable way responding in a series of approximately constant stresses that constituted a plateau. The highest load of that plateau is the compressive strength of the material (the highest load that the material can withstand before catastrophically fails in a brittle mode). The fluctuations in the plateau took place because of the cellular nature of the material. The cell walls carried the stress till they reached a critical value of stress then they failed, directly after their failure the load drops till the compression plates settled on another set of cell walls.

- **Densification**: beyond reaching the compressive strength of the material, the cell walls were completely collapsed and the fragments of the cell walls were the carriers of the load. That is why the last regime of the stress-strain curve was approximately a plateau of equal stresses till complete failure took place. The densification in the case of foam glass was not represented by a steep rising of the stress-strain curve (as explained by Ashby’s model) because the fragments of the cell walls are made of glass, which is a very fragile and brittle material. Mostly, the specimens’ cell walls were full of critical flaws in the form of tiny strength-
decreasing pores. Figure 4.2 shows the typical failure mode of foam glass after compression.

![Figure 4.2: Typical failure mode of foam glass due to compression.](image)

For the foam glass compositions that had considerably high density, they deformed in a pattern similar to the compression behavior of solid glass. Figure 4.3 shows the compression stress-strain curve of some brittle materials including glass.
To give an example for the compression behavior of the compositions that had high densities, the compositions with different particle sizes were considered. As the glass powder particle size increased, keeping other variables constant, the density of the foam glass as well as its compressive strength increased. The table (4.1) lists the particle size and bulk density ($\rho_b$) of the compositions that had particle sizes 75, 150, 250 µm (with white glass, sodium silicate solution amount 12wt. %, sintered at $T=850$ °C for 30 min).
Table 4.1: The bulk density of foam glass with increasing particle size

<table>
<thead>
<tr>
<th>Composition</th>
<th>Particle size (µm)</th>
<th>$\rho_b$ (g/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>75</td>
<td>0.30</td>
</tr>
<tr>
<td>14</td>
<td>150</td>
<td>0.74</td>
</tr>
<tr>
<td>15</td>
<td>250</td>
<td>1.02</td>
</tr>
</tbody>
</table>

Figure (4.4, 4.5, 4.6) show the compression stress-strain curves of compositions 3, 14, 15 that correspond to particle sizes 75, 150, 250 µm (with white glass, sodium silicate solution amount 12wt. %, sintered at $T=850$ °C for 30 min).

Figure 4.4: Compression stress-strain curve of foam glass with particle size 75 µm (composition 3).
Figure 4.5: Compression stress-strain curve of foam glass with particle size 150 µm (composition 14).

Figure 4.6: Compression stress-strain curve of foam glass with particle size 250 µm (composition 15).
It is noticeable from figure (4.4, 4.5, and 4.6) that as the particle size increased and the bulk density of the material increased, the foam glass compression behavior shifted gradually to the compression behavior of solid brittle glass. From composition 3 to 15 to 16, the compressive strength of the material increased considerably; while failure took place at lower strain percentage.

The foaming process and the resulting foam glass characteristics are controlled by many variables. Among these variables is the sintering temperature, the amount of the foaming agent, the soaking time, the glass powder particle size and the color of the glass powder. The influence of all these variables will be analyzed in this chapter.

4.2 The influence of sintering temperature

The influence of the sintering temperature on the foam glass was studied by subjecting compositions that contain 12 and 19 wt. % sodium silicate solution to sintering temperature that ranged from 750-900 °C with increment of 50 °C (using white glass with particle size 75 µm, soaking time 30 min).

4.2.1 Density

The bulk density (\(\rho_b\)) of the produced foam was measured by dividing its mass by its total volume (the volume was measured geometrically). The relative density (\(\rho_r\)) was calculated by dividing the bulk density by the true density (\(\rho_t\)) of the solid that constitute the cell wall of the foam, which is the soda lime glass (\(\rho_t = 2.5\) g/cm\(^3\)).
The fraction of space occupied by the pores inside the foam is the porosity. The percentage of porosity equals:

\[
\% \text{ of Porosity} = (1 - \frac{\rho_r}{\rho_t}) \times 100
\]  

Table 4.2 and 4.3 show the variation of the bulk density, relative density and % of porosity for the compositions that contain 12 and 19 wt. % sodium silicate solution (respectively) both subjected to sintering temperature that ranged from 750-900 °C with increment of 50 °C (using white glass with particle size 75 μm, soaking time 30 min). Figure 4.7 plots the bulk density as function of sintering temperature for the data shown in table 4.2 and 4.3.
Table 4.2: The variation of bulk density, relative density and % of porosity with different sintering temperature at 12 wt. % sodium silicate solution.

<table>
<thead>
<tr>
<th>Sintering Temperature (°C)</th>
<th>Composition</th>
<th>Sample</th>
<th>ρb (g/cm³)</th>
<th>STDEV</th>
<th>ρr</th>
<th>% of Porosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>750</td>
<td>1</td>
<td>0.62</td>
<td>0.56</td>
<td>0.65</td>
<td>0.61</td>
<td>0.049</td>
</tr>
<tr>
<td>800</td>
<td>2</td>
<td>0.43</td>
<td>0.42</td>
<td>0.40</td>
<td>0.42</td>
<td>0.015</td>
</tr>
<tr>
<td>850</td>
<td>3</td>
<td>0.23</td>
<td>0.30</td>
<td>0.22</td>
<td>0.25</td>
<td>0.045</td>
</tr>
<tr>
<td>900</td>
<td>4</td>
<td>0.20</td>
<td>0.26</td>
<td>0.20</td>
<td>0.22</td>
<td>0.036</td>
</tr>
</tbody>
</table>

Table 4.3 The variation of bulk density, relative density and % of porosity with different sintering temperature at 19 wt. % sodium silicate solution.

<table>
<thead>
<tr>
<th>Sintering Temperature (°C)</th>
<th>Composition</th>
<th>Sample</th>
<th>ρb (g/cm³)</th>
<th>STDEV</th>
<th>ρr</th>
<th>% of Porosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>750</td>
<td>5</td>
<td>0.54</td>
<td>0.55</td>
<td>0.52</td>
<td>0.54</td>
<td>0.017</td>
</tr>
<tr>
<td>800</td>
<td>6</td>
<td>0.36</td>
<td>0.38</td>
<td>0.38</td>
<td>0.37</td>
<td>0.011</td>
</tr>
<tr>
<td>850</td>
<td>7</td>
<td>0.30</td>
<td>0.32</td>
<td>0.24</td>
<td>0.29</td>
<td>0.043</td>
</tr>
<tr>
<td>900</td>
<td>8</td>
<td>0.34</td>
<td>0.33</td>
<td>0.29</td>
<td>0.32</td>
<td>0.028</td>
</tr>
</tbody>
</table>
Figure 4.7: The variation of bulk density with different sintering temperature at 12 and 19 wt. % sodium silicate solution.

The data shown in table 4.2 and 4.3 indicates that the sintering temperature has a considerable effect on the bulk density and the degree of foaming achieved in the produced foam glass; this effect is shown in compositions that contain 12 or 19 wt. % sodium silicate solution. Generally, as the sintering temperature increased from 750 °C to 900 °C, the bulk density decreased while the percentage of porosity increased (as shown in table 4.2 and 4.3).

Foam glass with 12 wt. % sodium silicate solution

Table 4.2 shows that the bulk density of the produced foam decreased continuously as the sintering temperature increases. The foam glass has a relatively high
bulk density ($\rho_{b}=0.61 \text{ g/cm}^3$) at 750 °C; its relative density ($\rho_r=0.24$) is to some extent high for a foam. It is stated by Gibson and Ashby that foams must have relative densities less than 0.3 [26]. The bulk density dropped by approximately 31% from 750 °C to 800 °C (0.61 to 0.42 g/cm$^3$). Further considerable drop of bulk density (approximately 40 % from 0.42 to 0.25 g/cm$^3$) was achieved as the temperature increased from 800 °C to 850 °C. As the temperature increased from 850 °C to 900 °C, the bulk density dropped by only around 12 % (from 0.25 to 0.22 g/cm$^3$).

Foam glass with 19 wt. % sodium silicate solution

Table 4.3 shows that the bulk density of the foam glass decreased as the sintering temperature increased till it reached a minimum of 0.29 g/cm$^3$ at 850 °C then it increased to 0.32 g/cm$^3$ at 900 °C. As the temperature increased from 750 to 800 °C, the bulk density dropped by around 32% (from 0.54 to 0.37 g/cm$^3$). Further increase of the temperature from 800 to 850 °C decreased the bulk density by around 21%. However, the bulk density increased from 0.29 to 0.32 g/cm$^3$ as the temperature increased from 850 to 900 °C (see figure 4.7). This phenomenon was detected and explained by the research study [27]. As the temperature increased, the pore size increased; smaller pores tended to dissolve in larger pores to decrease the surface energy of the whole system. This feature is called the coalescence phenomenon and it is responsible for the coarsening of the structure of the foam glass produced (see figure 4.11). As the size of the pores increases, the total number of pores decreases and the thickness of the struts separating them increase (because the total amount of the material is constant). Figure 4.6 shows that as
the sintering temperature increased from 800 to 850 °C, the size of the pores increased and the struts thickness increased. Foams with thicker struts are associated with higher bulk density and weaker structure. This feature took place in the compositions that had 19 wt. % sodium silicate solution because probably the higher amount of foaming agent led to extensive foaming inside the material, which amplified the coalescence phenomenon.

4.2.2 Morphology

Foam glass with 12 wt. % sodium silicate solution

Figure 4.8 shows the morphological evolution of foam glass with 12 wt. % sodium silicate solution at sintering temperature ranged from 750 to 900 °C (using white glass with particle size 75 µm, soaking time 30 min). Figure 4.9 shows a higher magnification for the compositions sintered at 850 and 900 °C.

The evolution of the morphology of the foam glass with increasing sintering temperature, as shown in figure 4.8, proves that as the sintering temperature increases, the foaming process inside the material increases. The pore size increases as the temperature increases. As the temperature increases, the viscosity of glass melt decreases and the pressure of the gas entrapped inside the pores increases as well. Consequently, the pore size increases as the sintering temperature increases [28].
Figure 4.8: The morphological evolution of foam glass with 12 wt.% sodium silicate solution at sintering temperature equals (1) 750 °C, (2) 800 °C, (3) 850 °C, (4) 900 °C.

The most homogeneous structure is for the foam glass sintered at 850 °C; below 850 °C and beyond 850 °C, the pore size distribution is comparatively inhomogeneous. As the temperature increases pores coalesce; they tend to combine forming larger pores as previously explained. This resulted in a coarser structure with larger pore size and inhomogeneous distribution of pores.
Figure 4.9: Higher magnification of the morphological evolution of foam glass with 12 wt. % sodium silicate solution at sintering temperature equals (1) 750 °C, (2) 800 °C, (3) 850 °C, (4) 900 °C.

Photo 3 and 4 (in figure 4.9) shows that as the sintering temperature increased from 850 to 900 °C, the structure coarsened. The coarser structure was also associated with struts that had larger thickness (as shown in figure 4.10). The morphology of the foam glass sintered at T = 900 °C shows the coalescence phenomenon took place extensively forming larger interconnected pores (see circles in figure 4.8, photo 4 depicting coalescence of pores).
Figure 4.10: Higher magnification of the morphology of foam glass with 12 wt. % sodium silicate solution at sintering temperature equals (1) 850 °C, (2) 900 °C.

Foam glass with 19 wt. % sodium silicate solution

Figure 4.11 shows the morphological evolution of foam glass with 19 wt.% sodium silicate solution at sintering temperature ranged from 750 to 900 °C (using white glass with particle size 75 µm, soaking time 30 min). Figure 4.12 shows a higher magnification for the compositions sintered at 800 and 850 °C. Figure 4.13 shows a higher magnification for the composition sintered at 900 °C.
Figure 4.11: The morphological evolution of foam glass with 19 wt.% sodium silicate solution at sintering temperature equals (1) 750 °C, (2) 800 °C, (3) 850 °C, (4) 900 °C

Figure 4.11 shows that as the sintering temperature increased, the pore size increased and their number decreased. The circle in photo 3 depicts two pores that were approaching each others to coalesce. As the sintering temperature increased, lots of small size pores were formed inside the struts (see figure 4.12 and 4.13). These pores are critical flaws inside the struts that sharply decrease the strength of the foam.
Figure 4.12: Higher magnification of the morphology of foam glass with 19 wt.% sodium silicate solution at sintering temperature equals (1) 800 °C, (2) 850 °C
Comparing figure 4.8 and 4.11 shows that the morphology of the foam glass with 12 wt.
% sodium silicate solution is more homogenous and contain less critical flaws than that
of the foam glass with 19 wt. % sodium silicate solution. The higher magnification shown
in figure 4.10 shows that the foam with 12 wt. % sodium silicate solution, even at higher
sintering temperature (850 and 900 °C), had no small pores in the struts. While figure
4.12 shows that the foam with 19 wt. % sodium silicate solution, sintered at 800 and 850
°C, had lots of multi-size smaller pores concentrated inside and around the struts. This
explains why the mechanical strength of the foam glass with 12 wt. % sodium silicate
solution was higher than that of foam glass with 19 wt. % sodium silicate solution (see figure 4.14) in the next section.

4.2.3 Compressive strength

Table 4.4 and 4.5 show the variation of the compressive strength for the compositions that contain 12 and 19 wt. % sodium silicate solution (respectively) both subjected to sintering temperature that ranged from 750-900 °C with increment of 50 °C (using white glass with particle size 75 µm, soaking time 30 min). Figure 4.14 plots the compressive strength as function of sintering temperature for the data shown in table 4.4 and 4.5.

Table 4.4: The variation of compressive strength with different sintering temperature at 12 wt. % sodium silicate solution.

<table>
<thead>
<tr>
<th>Sintering temperature °C</th>
<th>Composition</th>
<th>Sample</th>
<th>Compressive strength (MPa)</th>
<th>STDEV</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>750</td>
<td>1</td>
<td>16.60</td>
<td>17.69</td>
<td>21.74</td>
</tr>
<tr>
<td>800</td>
<td>2</td>
<td>4.91</td>
<td>2.97</td>
<td>5.41</td>
</tr>
<tr>
<td>850</td>
<td>3</td>
<td>1</td>
<td>1.8</td>
<td>2.07</td>
</tr>
<tr>
<td>900</td>
<td>4</td>
<td>0.59</td>
<td>0.93</td>
<td>0.91</td>
</tr>
</tbody>
</table>
Table 4.5: The variation of compressive strength with different sintering temperature at 19 wt. % sodium silicate solution.

<table>
<thead>
<tr>
<th>Sintering temperature °C</th>
<th>Composition</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
<th>Compressive strength (MPa)</th>
<th>STDEV</th>
</tr>
</thead>
<tbody>
<tr>
<td>750</td>
<td>5</td>
<td>7.38</td>
<td>7.95</td>
<td>7.36</td>
<td>7.56</td>
<td>0.336</td>
</tr>
<tr>
<td>800</td>
<td>6</td>
<td>2.46</td>
<td>2.10</td>
<td>2.30</td>
<td>2.29</td>
<td>0.181</td>
</tr>
<tr>
<td>850</td>
<td>7</td>
<td>0.88</td>
<td>0.91</td>
<td>0.93</td>
<td>0.91</td>
<td>0.025</td>
</tr>
<tr>
<td>900</td>
<td>8</td>
<td>0.80</td>
<td>0.78</td>
<td>0.70</td>
<td>0.76</td>
<td>0.053</td>
</tr>
</tbody>
</table>

Figure 4.14: The variation of compressive strength with different sintering temperature at 12 and 19 wt. % sodium silicate solution.
Foam glass with 12 wt. % sodium silicate solution

Figure 4.14 shows that the compressive strength decreased as the sintering temperature increased. Table 4.4 shows that the composition that had 12 wt% sodium silicate solution and sintered at 750 °C had the highest value of compressive strength (18.68 MPa). This was expected because it has lower amount of foaming agent and low sintering temperature and consequently, the foaming of the sample was relatively low (it has a % of porosity of 76%). The compressive strength dropped tremendously by around 76% (from 18.68 to 4.43 MPa) from the sintering temperature 750 to 800 °C (as the % of porosity increased from 76 to 83%). Further drop occurred from 800 to 850 °C by around 63% (from 4.43 to 1.62 MPa). The minimum compressive strength achieved equaled 0.81 MPa; it corresponds to 900 °C and $\rho_b = 0.22$ g/cm$^3$.

As the density of the foam glass decreases, its compressive strength decreases as normally expected [27]. This relation is illustrated by the experimental results of the foam with 12 wt. % foaming agent. Figure 4.15 compares between the behavior of the bulk density and the compressive strength of the foam having 12 wt. % sodium silicate solution as function of the sintering temperature. It shows that as the sintering temperature increased, the bulk density of the foam glass decreased and its compressive strength decreased too.
Figure 4.15: The relation between the bulk density and compressive strength of the foam glass that has 12 wt. % sodium silicate solution as a function of the sintering temperature

Foam glass with 19 wt. % sodium silicate solution

Figure 4.14 shows that the compressive strength of the foam glass decreased as the sintering temperature increased (similar to the compression behavior of the foam with 12 wt. % sodium silicate solution). Table 4.5 shows that at 750 °C, the foam glass had a maximum compressive strength of 7.56 MPa. This value is much lower than that achieved for the foam glass with 12 wt. % sodium silicate solution sintered at the same sintering temperature. This is because increasing the amount of foaming agent from 12 to
19 wt. % increased the degree of foaming of the foam glass (the porosity increased from 76 to 79 % respectively); the thing that decreased the mechanical strength of the foam. As the temperature increased from 750 to 800 °C, the compressive strength dropped by around 70%. The relation between the bulk density of the foam glass and its compressive strength is illustrated in figure 4.16.

Figure 4.16: The relation between the bulk density and compressive strength of the foam glass that has 19 wt. % sodium silicate solution as a function of the sintering temperature.

As the bulk density decreased, the compressive strength tended to decrease till sintering temperature 850 °C. At 900 °C, the bulk density of the foam increased and the compressive strength decreased. As previously explained, the coalescence phenomenon
takes place at higher temperature and it is responsible for the coarsening of the foam structure creating denser foam with lower compressive strength [27]. The pore size increased with an inhomogeneous distribution inside the foam (see figure 4.11, photo4). These strength-decreasing large pores are responsible for the drop of the compressive strength of the foam. In addition, the larger the strut thickness (associated with the coarser structure), the weaker the foam is because the thick struts are full of pores with different sizes (as shown in figure 4.13). The foam with thick struts and larger pores is weaker than the foam with homogeneously distributed small pores separated by thin struts. The thick struts are associated with critical flaws represented by the numerous pores with different sizes that exist in the body of the strut. These multi-size pores weaken the strength of the struts and the foam as a whole. In brittle foam, such as foam glass, the failure takes place because a crack nucleates at a weak strut or a pre-existing critical flaw. The crack propagates very fast leading to catastrophic brittle failure [5].

4.2.4 Thermal conductivity

The thermal conductivity was measured for the foam glass that has 12 wt. % sodium silicate solution subjected to sintering temperature that ranges from 750-900 °C with increment of 50 °C, using white glass with particle size 75 µm, soaking time 30 min. Table 4.6 shows the variation of thermal conductivity and % of porosity of the compositions with sintering temperature and figure 4.17 plots the variation of thermal conductivity and % of porosity as function of the sintering temperature.
Table 4.6: The variation of thermal conductivity and % of porosity with different sintering temperature at 12 wt. % sodium silicate solution.

<table>
<thead>
<tr>
<th>Sintering temperature °C</th>
<th>Composition</th>
<th>Thermal conductivity (W/m.°C)</th>
<th>% of porosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>750</td>
<td>1</td>
<td>0.092</td>
<td>76</td>
</tr>
<tr>
<td>800</td>
<td>2</td>
<td>0.090</td>
<td>83</td>
</tr>
<tr>
<td>850</td>
<td>3</td>
<td>0.078</td>
<td>90</td>
</tr>
<tr>
<td>900</td>
<td>4</td>
<td>0.053</td>
<td>91</td>
</tr>
</tbody>
</table>

Figure 4.17: The variation of thermal conductivity and % of porosity as function of the sintering temperature for foam glass with 12 wt. % sodium silicate solution.
The materials that have thermal conductivity less than 0.25 W/m.°C are classified as insulating materials [33]. All the measured samples had thermal conductivity lower than 0.25 W/m.°C. The thermal conductivity was found to vary from 0.092 W/m.°C (foam glass sintered at 750 °C) to 0.053 W/m.°C (foam glass sintered at 900 °C), which is close to the value of the thermal conductivity of recently developed foam glass (0.05-0.08 W/m.K) [21]. It is slightly higher than the thermal conductivity of the commercial foam glass produced by Pittsburgh Corning which equals (0.043-0.048 W/m.K) [20]. The thermal conductivity was found to decrease with increasing porosity percentage. It reached the minimum (0.053 W/m.°C) at percentage of porosity equals 91 %. This relation between the thermal conductivity and the percentage of porosity is expected because as the porosity increases inside the foam, its thermal conductivity tends to the thermal conductivity of air (thermal conductivity of air at room temperature is around 0.023 W/m.°C) [33]. It is noticeable that the values of the thermal conductivity of the first two compositions (at 750 °C and 800 °C) are approximately similar (0.092-0.090 W/m.°C). However, thermal conductivity dropped significantly from 800 °C to 850 °C (0.090-0.078 W/m.°C) and further drop took place at 900 °C; it reached 0.053 W/m.°C.

From all the previous analysis for the bulk density, compressive strength, thermal conductivity and morphology of the produced foam glass, it was found that the sintering temperature has a great effect on the physical and mechanical characteristics of the foam glass. Foam glass with different combination of service parameters can be produced at different sintering temperature depending on the required properties and characteristics. For applications that requires foam with relatively high structural characteristics along
with good thermal conductivity, the foam sintered at lower temperatures (750-800 °C) at 12 wt.% sodium silicate solution will be more suitable because it has high compressive strength (4.43-18.68 MPa) along with low thermal conductivity (around 0.09 W/m.°C). Considering the thermal insulation application, excellent combination of properties was achieved at sintering temperature 850 °C and sodium silicate solution amount 12 wt.% (ρb = 0.25 g/cm³, compressive strength= 1.62 MPa, Thermal conductivity = 0.078 W/m.°C, % of porosity = 90%) along with a homogeneous morphology with pore size around 0.5 mm. The typical technical characteristics of foam glass for insulation application range around 0.12-0.20 g/cm3, compressive strength > 0.7 MPa and thermal conductivity 0.05-0.08 W/m.K [21]. The highest sintering temperature which 900 °C did not produce foam glass with satisfactory characteristics because although the thermal conductivity of the foam was low (0.053 W/m.°C); however, the mechanical resistance of the foam was very low (0.81 MPa) and the morphology was inhomogeneous, full of coarse interconnected pores. Moreover, the density and percentage of porosity did not increase significantly from 850 °C to 900 °C (0.25-0.22 g/cm³) (90%-91%) respectively. Therefore, it is not recommended to use sintering temperature above 850 °C because beyond 850 °C, the foam glass properties are not satisfactory. Most importantly, increasing the sintering temperature beyond 850 °C will consume more energy and increases the production cost. The foam glass produced had properties that are superior to the foam glass recently produced and at much lower sintering temperature. The research [27] recycled glass from soda lime glass, it reported that the foam glass produced had ρb = 0.55-0.65 g/cm³ and thermal conductivity = 0.124-0.136 W/m·K and that the optimum
sintering temperature was 950 °C. Another research [30] that also recycled foam glass from soda lime glass states that the foam glass produced had ρ_b = 0.2-0.4 g/cm³ and compressive strength= 1.5 MPa and that the optimum sintering temperature was in the range 1000-1050 °C.

It is not recommended to increase the sintering temperature to 900 °C because at that temperature, the structure of the foam was coarse with large interconnected pores and it was mechanically weak. The optimum sintering temperature is 850 °C for preparing foam glass for thermal insulation.

4.3 The influence of the amount of foaming agent

The influence of the amount of foaming agent on the produced foam glass was studied by preparing compositions with 6, 12, 19, 32 wt.% sodium silicate solution.

4.3.1 Density

Table 4.7 shows the variation of bulk density, relative density and % of porosity with different amount of foaming agent (using white glass with particle size 75 μm, sintering temperature 850 °C, soaking time 30 min). Figure 4.18 shows the variation of bulk density with sodium silicate solution amount while figure 4.19 shows the variation of % of porosity with sodium silicate solution amount.
Table 4.7: The variation of bulk density, relative density and % of porosity with different amount of foaming agent.

<table>
<thead>
<tr>
<th>Wt.% sodium silicate solution</th>
<th>composition</th>
<th>Sample</th>
<th>$\rho_b$ (g/cm$^3$)</th>
<th>STDEV</th>
<th>$\rho_r$</th>
<th>% of porosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>9</td>
<td>0.38</td>
<td>0.43</td>
<td>0.34</td>
<td>0.38</td>
<td>0.047</td>
</tr>
<tr>
<td>12</td>
<td>3</td>
<td>0.23</td>
<td>0.30</td>
<td>0.22</td>
<td>0.25</td>
<td>0.045</td>
</tr>
<tr>
<td>19</td>
<td>7</td>
<td>0.30</td>
<td>0.32</td>
<td>0.24</td>
<td>0.29</td>
<td>0.043</td>
</tr>
<tr>
<td>32</td>
<td>10</td>
<td>0.33</td>
<td>0.38</td>
<td>0.35</td>
<td>0.35</td>
<td>0.023</td>
</tr>
</tbody>
</table>

Figure 4.18: The variation of bulk density with different amount of foaming agent
Figure 4.19: The variation of % of porosity with different amount of foaming agent

The density of the foam glass decreased with the progressive increase of sodium silicate solution amount till it reaches a minimum of 0.25 g/cm$^3$ (% of porosity 90 %) at the composition having 12 wt % sodium silicate solution then the density starts to re-increase. From the density curve, it is noticeable that the composition having 6 and 32 wt. % have approximately the same density and % of porosity (0.38-0.35 g/cm$^3$ and 85-86 % respectively). Increasing the amount of foaming agent resulted in decreasing the density of the foam glass until it reaches a critical amount beyond which the density will not decrease continuously with increasing the amount of foaming agent. That is caused by the coalescence phenomenon reported in the literature [27, 32]. Increasing the amount of foaming agent result in extensive foaming. The small pores tend to dissolve in larger
pores in order to decrease the surface energy of the whole system. That phenomenon result in coarsening the cellular structure, i.e. the pores are larger in size, smaller in number and the thickness of the cell walls increases. As a result, the density of the foam increases; however, the compressive strength decreases continuously with increasing the sodium silicate solution amount. The larger thickness of the cell walls increases the probability of critical flaws existence making it weaker than thinner walls. The coarse structure with large pores is a weak structure. This phenomenon is confirmed by the compressive stress curve that shows that the progressive increase of the sodium silicate solution amount cause a continuous decrease of the compressive strength of the foam glass samples (as shown in figure 4.17).

4.3.2 Morphology

Figure 4.14 shows the morphological evolution of foam glass as the amount of sodium silicate solution increases: 6, 12, 19, 32 wt.% (using white glass with particle size 75 µm, sintering temperature 850 °C, soaking time 30 min). Figure 4.15 shows higher magnification of the morphology at 12 and 19 wt.% sodium silicate solution. Figure 4.16 shows higher magnification of a strut full of critical flaws at 32 wt. % sodium silicate solution.
Figure 4.20: The morphological evolution of foam glass as the amount of sodium silicate solution increases (1) 6 wt.%, (2) 12 wt.%, (3) 19 wt.%, (4) 32 wt.%.

Figure 4.14 shows that the foam glass having 6 wt.% sodium silicate solution had a dense structure and a fairly homogenous structure. The foam glass with 12 wt.% sodium silicate solution has a more homogeneous structure with equally sized pores (around 0.5 mm) with no smaller pores in the struts. As the amount of sodium silicate solution increases to 19 wt.%, the structure appeared denser again because the cell walls are getting larger and the pores size are in homogeneously distributed. Also the struts are full of small strength-
decreasing pores (see figure 4.15). As the sodium silicate solution amount increased to 32 wt.%, the pore size increased tremendously with inhomogeneous distribution, the strut thickness increased as well. The structure is coarse and mechanically weak (compressive strength =0.33 MPa as shown in table 4.7). Figure 4.16 shows the critical flaws that fill the strut.

Figure 4.21: Higher magnification of the morphology of foam glass with 19 wt.% sodium silicate solution amount
4.3.3 Compressive strength

Table 4.8 shows the variation of compressive strength with different amount of foaming agent: 6, 12, 19, 32 wt. % sodium silicate solution (using white glass with particle size 75 µm, sintering temperature 850 °C, soaking time 30 min). Figure 4.23 shows the variation of compressive strength with water glass amount.
Table 4.8: The variation of compressive strength with different amount of foaming agent.

<table>
<thead>
<tr>
<th>Wt.% of sodium silicate</th>
<th>Composition</th>
<th>Sample</th>
<th>Compressive strength (MPa)</th>
<th>STDEV</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>9</td>
<td>1</td>
<td>3.03</td>
<td>2.90</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>2.94</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>2.73</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>3</td>
<td>1</td>
<td>1.00</td>
<td>1.62</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>1.80</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>2.07</td>
<td></td>
</tr>
<tr>
<td>19</td>
<td>7</td>
<td>1</td>
<td>0.88</td>
<td>0.91</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>0.91</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>0.93</td>
<td></td>
</tr>
<tr>
<td>32</td>
<td>10</td>
<td>1</td>
<td>0.25</td>
<td>0.33</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>0.34</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>0.40</td>
<td></td>
</tr>
</tbody>
</table>

Figure 4.23: The compressive strength as function of the amount of foaming agent.
The compressive strength, unlike the bulk density, continuously decreased as the sodium silicate solution amount increased. As the sodium silicate solution amount increased from 6 to 12 wt. %, the compressive strength decreased by around 44% (from 2.9 MPa to 1.62 MPa). Further increasing sodium silicate solution amount from 12 wt. % to 19 wt. % dropped the compressive strength by the same amount around 44% (from 1.62 to 0.91 MPa). Increasing the sodium silicate solution amount from 19 to 32 wt. % caused dramatic decrease of the strength of the produced foam; it dropped by around 64% (from 0.91 to 0.33 MPa). Figure 4.24 compares between the foam prepared with different amount of foaming agent in terms of bulk density, compressive strength and morphology. Although the foam glass compositions that had sodium silicate solution amount 6 and 32 wt.% had approximately the same density (0.38-0.35 g/cm$^3$ respectively), they had totally different compressive strength (2.9-0.33 g/cm$^3$ respectively). That is because although the percentage of porosity for both samples are in the same order of magnitude (85-86 %), the much smaller pore size that are homogeneously distributed, that corresponds to the compositions having 6 wt.% sodium silicate solution, resulted in much higher compressive strength (as shown in figure 4.24). This proves that the compressive strength of the foam glass does not only depend on the density of the foam but also on the inner structure of the material. It depends mainly on the pore size, shape and the extent of homogeneity in their distribution inside the material. Also, it depends on the strut thickness and the amount of critical flows inside them.

It is not recommended to increase the amount of the foaming agent beyond 12 wt.% because beyond that amount, the structure of the foam was getting coarser,
denser and mechanically weaker. The optimum amount of foaming agent is 12 wt. % for preparing foam glass for thermal insulation.

Figure 4.24: The relation between the bulk density, compressive strength and morphology as function of the amount of sodium silicate solution
4.4 The influence of soaking time

The influence of soaking time on the foam glass was studied by sintering compositions at 10, 20, 30, 40 minutes.

4.4.1 Density

Table 4.9 shows the variation of the bulk density, relative density and % of porosity with soaking time (using white glass with particle size 75 µm, sintering temperature 850 °C, 12 wt. % sodium silicate solution). Figure 4.25 shows the variation of the bulk density with soaking time.

Table 4.9: The variation of the bulk density, relative density and % of porosity with soaking time.

<table>
<thead>
<tr>
<th>Soaking time (min)</th>
<th>composition</th>
<th>Sample</th>
<th>$\rho_b$ (g/cm$^3$)</th>
<th>STDEV</th>
<th>$\rho_r$</th>
<th>% of porosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>11</td>
<td>1</td>
<td>0.48</td>
<td>0.44</td>
<td>0.050</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>0.46</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>0.38</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>12</td>
<td>1</td>
<td>0.31</td>
<td>0.31</td>
<td>0.001</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>0.31</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>0.31</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>3</td>
<td>1</td>
<td>0.23</td>
<td>0.22</td>
<td>0.045</td>
<td>0.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>0.30</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>0.30</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>13</td>
<td>1</td>
<td>0.23</td>
<td>0.24</td>
<td>0.007</td>
<td>0.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>0.24</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>0.24</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The results showed that the soaking time have a significant effect on the properties of the produced foam glass. As the soaking time increased, the bulk density of the foam glass decreased. Sintering foam glass for 10 min resulted in a dense structure with $\rho_b = 0.44$ g/cm$^3$. Increasing the sintering temperature to 20 then 30 min dropped the bulk density to 0.31 then to 0.25 g/cm$^3$ respectively. However, further increasing the soaking time to 40 min did not significantly decreased the bulk density (from 30 to 40 min, bulk density dropped from 0.25 to 0.24 g/cm$^3$ with same % of porosity (= 90 %).

Figure 4.25: The variation of the bulk density with soaking time
4.4.2 Morphology

Figure 4.26 shows the evolution of the morphology of foam glass at different soaking time (using white glass with particle size 75 µm, sintering temperature 850 °C, 12 wt. % sodium silicate solution). Figure 4.27 shows higher magnification for compositions soaked for 30 and 40 minutes.

Figure 4.26: The morphological evolution of foam glass as the soaking time increases (1) 10 min, (2) 20 min, (3) 30 min, (4) 40 min.

The morphology of the foam glass sintered at different soaking time is quite similar with no dramatic change from one soaking time to the other. They are all more or less homogenous with equal size pores. The foam glass sintered at 10 min has relatively
smaller pore size and appear as a denser microstructure (see photo 1). The morphology of the foam glass sintered at 30 min is quite similar to that sintered at 40 min with slight increase in the pore size of the foam sintered at 40 min (figure 4.27).

Figure 4.27: Higher magnification of foam glass sintered at soaking time (1) 30 min, (2) 40 min.
The higher magnification of the foam glass sintered at 30 and 40 min showed that in both structures, the struts thicknesses are not high and the amount of strength-decreasing small pores that exist in the struts is not high. That means that the structure is mechanically strong.

### 4.4.3 Compressive strength

Table 4.10 shows the variation of the compressive strength with different soaking time: 10, 20, 30, 40 min (using white glass with particle size 75 µm, sintering temperature 850 °C, 12 wt. % sodium silicate solution). Figure 4.28 shows the variation of the compressive strength with soaking time.

Table 4.10: The variation of the compressive strength with soaking time.

<table>
<thead>
<tr>
<th>Soaking time (min)</th>
<th>Composition</th>
<th>Sample</th>
<th>Compressive Strength (Mpa)</th>
<th>STDEV</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>11</td>
<td>3.30</td>
<td>3.40</td>
<td>2.70</td>
</tr>
<tr>
<td>20</td>
<td>12</td>
<td>3.10</td>
<td>2.70</td>
<td>3.20</td>
</tr>
<tr>
<td>30</td>
<td>3</td>
<td>1.00</td>
<td>1.80</td>
<td>2.07</td>
</tr>
<tr>
<td>40</td>
<td>13</td>
<td>1.10</td>
<td>2.10</td>
<td>1.60</td>
</tr>
</tbody>
</table>
Figure 4.28: The variation of the compressive strength with soaking time.

Figure 4.28 shows that the compressive strength of the foam glass sintered at different soaking times was high. It ranged around 1.6-3.13 MPa. This is caused by the homogeneity of the morphology and the lack of critical flaws in the cell walls (see morphology). The compressive strength for the foam glass sintered at 10 and 20 min was approximately the same (3.13 and 3 MPa respectively). Then, increasing the soaking time from 20 to 30 min caused significant drop in the compressive strength (from 3 to 1.62 MPa). However, the resistance of the foam glass did not decrease much (from 1.62 to 1.6 MPa) by increasing the soaking time from 30 to 40 min. From the results, it appeared that increasing the soaking time from 30 min to 40 min did not have significant effect on the foam glass properties (as shown in figure 4.28). So soaking foam glass for beyond 30 min
will consume more energy and increase the production cost without adding any significant improvement for the properties of the foam produced.

Comparing the effect of sintering temperature vs. soaking time the morphology of the foam glass (figure 4.29) showed that the sintering temperature had a more significant effect on the pore morphology. Increasing the sintering temperature form 800 to 900 °C increases tremendously the size of the pores because increasing the temperature has a more considerable effect on decreasing the viscosity of the glass melt. It is more responsible also for increasing the coalescence phenomenon that coarsens the pore structure. All these factors decrease the strength of the foam glass. On the other hand, changing soaking time, does not cause dramatic increase in pore size. It only increases the homogeneity of the pore size distribution. That is why increasing soaking time does not decrease considerably the mechanical resistance of the foam. However, beyond a certain value, further increasing soaking time does not have significant effect on foam characteristics.

Reducing the sintering temperature to the most suitable value is the most important parameter because it achieves foam glass with good characteristics and reduces the energy consumption in addition to the production cost. Reducing the soaking time to the appropriate value also is an important parameter because it saves energy; in addition to reducing the production cost by reducing the time for the processing cycles. It is important, to stop the manufacturing process once the optimum characteristics are achieved to interrupt the morphology evolution and to save energy and cost. The
experimental results showed that using sintering temperature beyond 850 °C and soaking time beyond 30 min does not have significant effect on improving foam glass properties.

It is not recommended to increase the soaking time beyond 30 min as this did not have a significant effect on improving the properties of the developed foam glass. The optimum soaking time is 30 min.

Figure 4.29: The evolution of the foam glass morphology with increasing sintering temperature (from 800 to 900 °C) and soaking time (from 20 min to 40 min).
4.5 The effect of particle size

The influence of the particle size on the foam glass was studied by preparing compositions from glass powder with 3 particle sizes: size 1 = 75 µm, size 2 = 150 µm, size 3 = 250 µm sintered at different sintering temperature: 850 °C, 900 °C, 920 °C. Sintering temperatures beyond 850 °C were used in order to give the chance to the larger particles sizes to be foamed.

4.5.1 Density

Table 4.11 shows the variation of the bulk density, relative density and % of porosity with the glass powder particle size at different sintering temperature (using white glass with 12 wt. % sodium silicate solution, soaking time 30 min ). Figure 4.30 shows the bulk density as function of powder particle size at different sintering temperature.

From the results shown in table 4.11, it appeared that regardless of the sintering temperature, the bulk density of the foam glass increased with increasing particle size although the sensitivity of that increase was reduced at the highest sintering temperature 920 °C. The density of the compositions prepared from particle size 1 had low density throughout the different sintering temperature 850, 900, 920 °C (0.25, 0.22, 0.24 g/cm³ respectively). The density decreased from 850 to 900 °C then it re-increased at 920 °C (figure 4.30). This is because at 920 °C the structure of foam is tremendously coarse with thick struts (see figure 4.31, photo 3).
Table 4.11: The variation of the bulk density, relative density and % of porosity with powder particle size and sintering temperature.

<table>
<thead>
<tr>
<th>Sintering temperature °C</th>
<th>Particle size</th>
<th>Composition</th>
<th>Sample</th>
<th>( \rho_b ) (g/cm(^3))</th>
<th>STDEV</th>
<th>( \rho_r )</th>
<th>% of Porosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>850</td>
<td>1</td>
<td>3</td>
<td>0.23</td>
<td>0.30</td>
<td>0.22</td>
<td>0.25</td>
<td>0.045</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>14</td>
<td>0.56</td>
<td>0.64</td>
<td>0.62</td>
<td>0.61</td>
<td>0.042</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>15</td>
<td>1.00</td>
<td>1.05</td>
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<td>1.02</td>
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</tr>
<tr>
<td>900</td>
<td>1</td>
<td>4</td>
<td>0.20</td>
<td>0.26</td>
<td>0.20</td>
<td>0.22</td>
<td>0.036</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>16</td>
<td>0.46</td>
<td>0.38</td>
<td>0.37</td>
<td>0.40</td>
<td>0.049</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>17</td>
<td>0.78</td>
<td>0.72</td>
<td>0.69</td>
<td>0.73</td>
<td>0.047</td>
</tr>
<tr>
<td>920</td>
<td>1</td>
<td>18</td>
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<td>0.23</td>
<td>0.24</td>
<td>0.24</td>
<td>0.006</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>19</td>
<td>0.38</td>
<td>0.29</td>
<td>0.32</td>
<td>0.33</td>
<td>0.047</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>20</td>
<td>0.51</td>
<td>0.53</td>
<td>0.51</td>
<td>0.52</td>
<td>0.012</td>
</tr>
</tbody>
</table>

The lower viscosity of glass melt at 920 °C along with the coalescence of pores resulted in the increase of the pore size. The small size of the particle also caused the pore size to tremendously increase. In general, the sintering rate is strongly affected by the powder particle size. The smaller the particle size, the higher the sintering rate. The finer particles sintered earlier than the larger particles. The density of compositions with particle size 2 and 3 had dropped considerably throughout the increasing sintering temperature 850, 900, 920 °C (size 2: 0.61, 0.40, 0.33 g/cm\(^3\); size 3: 1.02, 0.73, 0.52 g/cm\(^3\) respectively). The higher temperature affected the density drop for the particle size 2, 3 more than it did for
particle size 1 because particle size 1 is finer it reached lower viscosity earlier that is why its density decreased then re-increased when the structure coarsened. On the other hand, size 2 and 3 were still in the process of decreasing glass melt viscosity and increasing pore size.

Figure 4.30: The bulk density as function of powder particle size at different sintering temperature.
4.5.2 Morphology

Figure 4.31 shows the evolution of the foam glass morphology with increasing powder particle size (size 1 = 75 µm, size 2 = 150 µm, size 3 = 250 µm) and sintering temperature (850, 900, 920 °C).

Figure 4.31: The evolution of the morphology of foam glass prepared from different particle size and at different sintering temperature
It appears from the morphology (shown in figure 4.31) that, at the same sintering temperature, as the particle size increases, the pore size distribution is becoming less homogeneous and the size of the pore decreases (except the case of density drop of size 1 from 850 to 900 °C check figure 4.31, photo 2). Checking the morphology at 850 °C, it is noticeable that size 1 had the larger pore size but the most homogenous structure (figure 4.31, photo 1,4,7). Size 3 had a dense structure where the ratio between the area occupied by the cell struts and the total area is high. It had relative density 0.41, which is very high for a foam (figure 4.31, photo 7). It was stated by Gibson and Ashby [26] that beyond relative density of 0.3, the material is transformed from foam to solid that contain isolated pores. Even at 900 °C, size 3 still appears as dense structure with small pore size and inhomogeneous distribution of pore size (figure 4.31, photo 8). The high homogeneity in pore size distribution associated with finer particle size was probably due to the fact that when larger particles were mixed with the foaming agent, the latter agglomerated inside the mixture because of the large voids caused by the large particles (although liquid foaming agent was supposed to reduce that effect in comparison with powder foaming agent) [27]. In general, it appears from the morphology that as the particle size increases, the homogeneity of the foam structure decreases even with increasing temperature. Figure 4.32 shows higher magnification of compositions with particle size 150 µm at 850 and 900 °C.
Figure 4.32: Higher magnification of foam glass with particle size 2 (150 µm) sintered at (1) 850 °C, (2) 900 °C

Higher magnification of composition prepared from size 2 showed that it had multi-size pores (figure 4.32, photo 1); in addition to strength-decreasing pores in the struts that appeared at higher sintering temperature (900 °C) (figure 4.32, photo 2).
4.5.3 Compressive strength

Table 4.12 shows the variation of the compressive strength with the glass powder particle size at different sintering temperature (using white glass with 12 wt. % sodium silicate solution, soaking time 30 min). Figure 4.33 shows the compressive strength as a function of powder particle size and sintering temperature.

Table 4.12: The variation of the compressive strength with powder particle size and sintering temperature.

<table>
<thead>
<tr>
<th>Sintering temperature °C</th>
<th>Particle size</th>
<th>Composition</th>
<th>Sample</th>
<th>Compressive strength (MPa)</th>
<th>STDEV</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>850</td>
<td>1</td>
<td>3</td>
<td>1.00</td>
<td>1.80</td>
<td>2.07</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>14</td>
<td>3.80</td>
<td>6.20</td>
<td>4.60</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>15</td>
<td>7.90</td>
<td>7.00</td>
<td>12.50</td>
</tr>
<tr>
<td>900</td>
<td>1</td>
<td>4</td>
<td>0.59</td>
<td>0.93</td>
<td>0.91</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>16</td>
<td>0.75</td>
<td>0.67</td>
<td>0.80</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>17</td>
<td>5.55</td>
<td>6.00</td>
<td>4.70</td>
</tr>
<tr>
<td>920</td>
<td>1</td>
<td>18</td>
<td>0.33</td>
<td>0.32</td>
<td>0.36</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>19</td>
<td>0.32</td>
<td>0.31</td>
<td>0.32</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>20</td>
<td>0.85</td>
<td>0.81</td>
<td>0.60</td>
</tr>
</tbody>
</table>
Figure 4.33: The compressive strength of the foam glass as function of the glass powder particle size at different sintering temperature.

Table 4.12 shows that, as the particle size increased, the compressive resistance of the foam increased except the 2 cases that correspond to size 2 whose compressive strength dropped at 900 and 920 °C. The foam glass prepared from size 1 had relatively good compressive strength 1.62 MPa at 850 °C. Further increasing sintering temperature decreased its resistance till it drastically decreased to 0.34 MPa at 920 °C. Although, the bulk densities of the foam prepared from size 1 sintered at 850 °C and 920 °C are approximately equal (0.25, 0.24 g/cm³); however, the one sintered at 920 °C is much weaker (figure 4.33). This is caused by the inhomogeneous structure of the foam sintered...
at 920 °C with respect to the foam sintered at 850 °C (as shown by the morphology in figure 4.31, photo 1 and 3). In addition, the foam sintered at 920 °C had much coarser structure with strength-decreasing large pores.

The foam prepared from size 2 had a dense structure at 850 °C (figure 4.31, photo 4) that is why its compressive strength is relatively high (4.87 MPa). However, at higher sintering temperature, its compressive strength dropped severely to (0.74 MPa at 900 °C then to 0.32 MPa at 920 °C). This was expected because the structure of the foam is highly inhomogeneous with large pores and full of strength-decreasing small pores located around and inside the struts (figure 4.32, photo 2). The foam prepared from size 3 was dense whether sintered at 850 or 900 °C, it had large resistance (9.13, 5.42 MPa respectively). However, its strength decreased sharply at 920 °C (0.75 MPa) because the structure is inhomogeneous (figure 4.31, photo 9). All the compositions lost their mechanical resistance at 920 °C, because at such high temperature, the viscosity of the glass melt was low. The coalescence of pores was amplified and the pore size was large. Consequently, the mechanical strength of all the foam sintered at 920 °C was low.

As the particle size increases, higher sintering temperature will be needed to transform the glass powder into foam. But still at higher temperature, the large particle size result in a weak inhomogeneous foam structure full of defects and critical flaws. Although, grinding glass powder to finer size will use more energy and cost, however, the energy consumed to decrease the particle size will be much less than the energy used to increase
the sintering temperature. In addition, it will produce relatively stronger foam glass with homogeneous structure.

It is not recommended to increase the glass powder particle size beyond 75 µm because beyond that size, the structure of the foam was inhomogeneous and dense. The optimum particle size was 75 µm.

4.6 The effect of using glass powder with different colors

Glass with different color consists of different composition, which might affect the properties of the produced foam glass. The effect of container glass color on the properties of the produced foam glass was investigated by preparing samples using green and brown container glass and to compare them with those prepared using white container glass.

Table 4.13 shows the variation of the bulk density, compressive strength and specific compressive with the color of the glass (using particle size = 75 µm, 12 wt. % sodium silicate solution, sintering temperature 850 °C, soaking time 30 min). Figure 4.34 compares between the bulk density, compressive strength and specific compressive strength with respect to the color of the glass.
Table 4.13: The variation of the bulk density, compressive strength and specific compressive strength with the color of the glass.

<table>
<thead>
<tr>
<th>Glass color</th>
<th>Composition</th>
<th>$\rho_b$ (g/cm$^3$)</th>
<th>Compressive strength (MPa)</th>
<th>Specific compressive strength (MPa m$^3$/Kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>White</td>
<td>3</td>
<td>0.25</td>
<td>1.62</td>
<td>6.48*10^{-3}</td>
</tr>
<tr>
<td>Green</td>
<td>21</td>
<td>0.38</td>
<td>2.05</td>
<td>5.39*10^{-3}</td>
</tr>
<tr>
<td>Brown</td>
<td>22</td>
<td>0.37</td>
<td>1.97</td>
<td>5.32*10^{-3}</td>
</tr>
</tbody>
</table>

The specific compressive strength values * 10^{-3}

Figure 4.34: The relation between the bulk density, compressive strength and specific compressive strength with respect to the color of the container glass.
The specific compressive strength is the ratio between the compressive strength and the bulk density. It describes the strength of the material with respect to its density. This property was calculated for the foam glass in the research study [29].

Table 4.13 shows that the properties of the green and brown glass samples are approximately the same; but they differ significantly from the white glass samples. The white glass samples had the lowest density and compressive strength (0.25 g/cm³, 1.62 MPa respectively). However, it had the highest specific compressive strength i.e. it is strong with respect to its low density (figure 4.34). The green and brown glass samples had bulk densities in the same order of magnitude (0.38, 0.37 MPa respectively) and also close compressive strength (2.05, 1.97 MPa).
4.6.1 Morphology

Figure 4.29 shows the morphology of foam glass prepared from white, green, brown container glass (using particle size = 75 µm, 12 wt. % sodium silicate solution, sintering temperature 850 °C, soaking time 30 min).

Figure 4.35: The morphology of foam glass prepared from (1) white, (2) green, (3) brown container glass; (4) The surface morphology of the white, green, brown foam glass (from left to right)

Figure 4.35 shows that the 3 colors have different morphology. The pore size of the white foam glass was slightly smaller than that of the green and brown. Figure 4.36 shows high magnification (of 120 and 300 times) of the three colors.
Figure 4.36: Higher magnification for the morphology of the glass prepared from white (1)(2), green (3)(4), brown (5)(6) container glass.

The higher magnification showed that the white foam glass had equally sized pored with thinner struts (see photo 1 and 2). The green and brown foam glass had denser structure with less homogeneous pore size, especially the brown (see photos 3 and 5). Further magnification showed that green and brown foams had thicker struts that are full of small
pores (see photos 4 and 6). This explained why the white foam glass had higher specific compressive strength with respect to green and brown foam glass. The structure of the white foam glass is generally more homogeneous.

4.7 The energy dispersive X-ray spectroscopy (EDX) analysis of the glass powder samples

The EDX analysis is used for the elemental chemical analysis of a sample. It shows only the main elements that constitute the sample. The EDX was done for glass powder with different colors: white, green, brown (with particle size = 75 µm).

4.7.1 The EDX results for white glass powder

The analysis started by defining an area on which the EDX analysis will be performed. The rectangle that appears in figure 4.37 contours the area for which the chemical composition was analyzed. Table 4.14 shows the chemical analysis of the white glass powder.
Figure 4.37: the spectrum defined for the EDX analysis of white glass powder

Table 4.14: The analysis of the main elements of white glass powder.

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>54.37</td>
</tr>
<tr>
<td>Si</td>
<td>27.33</td>
</tr>
<tr>
<td>Na</td>
<td>10.03</td>
</tr>
<tr>
<td>Ca</td>
<td>5.75</td>
</tr>
<tr>
<td>Fe</td>
<td>1.20</td>
</tr>
<tr>
<td>Al</td>
<td>0.66</td>
</tr>
<tr>
<td>Mg</td>
<td>0.53</td>
</tr>
<tr>
<td>K</td>
<td>0.12</td>
</tr>
<tr>
<td>Totals</td>
<td>100.00</td>
</tr>
</tbody>
</table>
The elemental composition of the white glass powder showed that oxygen represented the highest wt. % followed by silicon (table 4.14). This was expected because glass consists mainly of oxides where the major constituent oxide is SiO$_2$. The rest of the elements are the additives that are typically added to soda lime glass to improve its property and workability. Figure 4.38 shows the spectrum of the elements that constitute the white glass powder.

![Spectrum 1](image)

Figure 4.38: Spectrum that show the main elements that constitute white glass powder.

### 4.7.2 The EDX results for green glass powder

The rectangle that appears in figure 4.39 contours the area for which the chemical composition was analyzed. Table 4.15 shows the chemical analysis of the green glass powder.
Figure 4.39: The spectrum defined for the EDX analysis of green glass powder

Table 4.15: The analysis of the main elements of green glass powder.

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>51.73</td>
</tr>
<tr>
<td>Si</td>
<td>28.22</td>
</tr>
<tr>
<td>Na</td>
<td>9.21</td>
</tr>
<tr>
<td>Ca</td>
<td>6.97</td>
</tr>
<tr>
<td>Fe</td>
<td>1.17</td>
</tr>
<tr>
<td>Al</td>
<td>1.08</td>
</tr>
<tr>
<td>Mg</td>
<td>0.96</td>
</tr>
<tr>
<td>K</td>
<td>0.51</td>
</tr>
<tr>
<td>Cr</td>
<td>0.14</td>
</tr>
<tr>
<td>Totals</td>
<td>100.00</td>
</tr>
</tbody>
</table>
The elemental composition of the green glass powder showed that the main elements that constitutes the green and the white glass are the same (except for chromium), with slight variation in the weight % (table 4.15). In addition to, tiny amount of chromium (0.14 wt.%) was detected in the green glass. Normally chromium is added along with iron oxide to container glass to give it a richer green color [46]. Figure 4.40 shows the spectrum of the elements that constitute the green glass powder.

![Spectrum 1](image)

Figure 4.40: Spectrum that shows the main elements that constitute green glass powder.

### 4.7.3 The EDX results for brown glass powder

The rectangle that appears in figure 4.41 contours the area for which the chemical composition was analyzed. Table 4.16 shows the chemical analysis of the white glass powder.
Figure 4.41: The spectrum defined for the EDX analysis of brown glass powder

Table 4.16: The analysis of the main elements of brown glass powder.

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>55.12</td>
</tr>
<tr>
<td>Si</td>
<td>26.56</td>
</tr>
<tr>
<td>Na</td>
<td>9.83</td>
</tr>
<tr>
<td>Ca</td>
<td>5.70</td>
</tr>
<tr>
<td>Mg</td>
<td>0.97</td>
</tr>
<tr>
<td>Al</td>
<td>0.71</td>
</tr>
<tr>
<td>Fe</td>
<td>0.68</td>
</tr>
<tr>
<td>K</td>
<td>0.31</td>
</tr>
<tr>
<td>Ti</td>
<td>0.12</td>
</tr>
<tr>
<td>Totals</td>
<td>100.00</td>
</tr>
</tbody>
</table>
The elemental composition of the brown glass powder showed that the main elements that constitutes the brown and the white glass are the same (except for Titanium), with slight variation in the weight % (table 4.16). Tiny amount of Titanium was detected (0.12 wt. %). Titanium is normally used to give the container glass the yellowish-brown color. Usually titanium is used by glass makers to brighten other coloring additives [46]. Figure 4.42 shows the spectrum of the elements that constitute the brown glass powder.

Figure 4.42: Spectrum that shows the main elements that constitute brown glass powder.
CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The production of foam glass insulation from MSW container glass is a promising technique for achieving sustainable recycling of waste container glass. The waste container glass can be fully recycled into foam glass, which is a valuable product with marketable value.

A wide range of highly porous foam glass with excellent properties was successfully produced from waste soda-lime container glass. The addition of the sodium silicate solution, as foaming agent, to the milled cullet particles reduced the sintering temperature by around 100 °C, compared to the sintering temperature used by the recent research studies. It was proved that the foaming process is influenced by the sintering temperature, the amount of foaming agent, soaking time, glass powder particle size and the color of the glass. The sintering temperature had significant effect on the foam glass physical, mechanical and thermal properties. At lower sintering temperature (750-800 °C), the foam glass produced (at 12 wt.% sodium silicate solution) had high compressive strength (4.43-18.68 MPa) along with low thermal conductivity (around 0.09 W/m.°C). As the sintering temperature increased, higher levels of porosity were achieved accompanied by lower bulk density and thermal conductivity. The foam sintered at 850 °C (at 12 wt. % sodium silicate solution) had lower thermal conductivity (0.078 W/m.°C) with lower bulk density (0.25 g/cm³) and high porosity (90%). These properties are
excellent for thermal insulation application because the thermal conductivity of the foam is low; in addition to, good compressive strength (1.62 MPa) with respect to other thermal insulation foam. Increasing the sintering temperature to 900 °C led to extensive porosity. High levels of porosity in foam glass were associated with cell coalescence, which led to inhomogeneous coarse structure with large pores and thick struts. The foam sintered at 900 °C was mechanically weak (compressive strength = 0.81 MPa) and its morphology was full of coarse interconnected strength-decreasing pores. It is recommended not to use sintering temperature beyond 850 °C because higher temperature will consume more energy and cost that is not justifiable by enhancement in foam glass properties.

The amount of foaming agent had a significant effect on the properties of the foam glass produced. The bulk density of the foam decreased with increasing foaming agent amount till it reached a minimum value of 0.25 g/cm³ (at 12 wt. % sodium silicate solution) then it re-increased. Sort of saturation took place in the foam upon introducing amount of foaming agent beyond 12 wt. %. The foam with 6 and 32 wt. % had approximately the same density (0.38-0.35 g/cm³ respectively). However, they had totally different compressive strength (2.9-0.33 g/cm³ respectively). Increasing the amount of foaming agent to 32 wt. % lead to extensive foaming, which lead to a coarse structure with thick struts full of critical flaws. This structure is denser but mechanically weaker. That proved that the mechanical strength of the foam glass is greatly influenced by the homogeneity of the foaming that in turn depends on the amount of foaming agent.
The variation of the soaking time had a slight effect on the properties of the foam glass. In general, the morphology of all the foam glass produced at different soaking times was homogeneous. Consequently, the compressive strength of all the produced foam was relatively high (1.6 MPa at 40 min and 3.13 MPa at 10 min). Increasing the soaking time from 30 to 40 min did not have a significant influence on the foam glass properties; the bulk density changed from 0.25 to 0.24 g/cm$^3$ while the compressive strength changed from 1.62 to 1.6 MPa. It is recommended not to use soaking times more then 30 min because this will consume more energy and cost without being justified by enhancing foam glass properties.

The properties of the foam glass was strongly influenced by the glass powder particle size. As the particle size increased, the bulk density of the foam increased (regardless of the sintering temperature). Larger particle size (150 and 250 µm) had inhomogeneous morphology regardless of the sintering temperature. As the particle size decreases, the sintering rate increases. Consequently, as the particle size decreased, the size of the pores increased (this effect was shown in all sintering temperatures) because all the stages of foaming: sintering, bubble formation and bubble growth took place earlier at finer particle size. The finer particle size (75 µm) had the lowest density throughout the sintering temperature variation (850, 900, 920 °C corresponded to 0.25, 0.22, 0.24 g/cm$^3$ respectively). However, the highest particle size (250 µm) had the highest density throughout the sintering temperature variation (850, 900, 920 °C corresponded to 1.02, 0.73, 0.52 g/cm$^3$ respectively). Increasing the sintering temperature for the larger particle size did not succeed in increasing the pore size homogeneity. It is
recommended to grind the glass powder to finer sizes because the energy consumed in
decreasing the particle size is justifiable by the major enhancement that takes place in
foam properties due to the lowering of the particle size.

The optimum processing parameters for producing foam glass for thermal
insulation was to use sintering temperature 850 °C, amount of foaming agent 12 wt. %,
soaking time 30 min and glass powder particle size 75 µm.

The foam glass morphology and properties varied significantly with changing the
color of the glass powder. The properties and the morphology of the green and brown
glass samples were approximately the same (bulk density = 0.38, 0.37 MPa and
compressive strength = 2.05, 1.97 MPa respectively). However, they differed from the
morphology and properties of the white glass (bulk density = 0.25 g/cm³ and compressive
strength = 1.62 MPa). White glass foam had a more homogeneous distribution of pores
with slightly smaller pore size. The green and brown foam had larger pores with lots of
small pores inside the struts. The specific strength of the white glass foam (6.48*10⁻³
MPa m³/Kg) was higher than that of green and brown glass (5.39*10⁻³, 5.32*10⁻³ MPa
m³/Kg respectively). The white glass foam was stronger relative to its own weight. The
EDX analysis for the white, green and brown powder showed that they had more or less
the same compositions (except the presence of chromium element in green glass and
titanium in brown glass). They had the same main elements but with different weight
percentage. The chromium element in green glass is normally added to give the glass the
green color; while the titanium element in brown glass is normally added to give the glass
the yellowish-brown color.
5.2 **Recommendations for future work**

- Study the parameters associated with using green and brown glass in producing foam glass.
- Produce foam glass from a mixture of glass wastes (different colors) to reduce the need for separation and to make use of a huge amount of glass waste mixed in the form of cullet with different color.
- Produce foam glass for other applications such as light weight aggregates, biological filters and sound insulation. The foam glass properties can be tailored to suit a range of applications.
- Recycle hazardous glass products such as CRT and fluorescent lamps into foam glass and analyze whether this will minimize the risk of heavy metals release.
- Perform a cost benefit analysis for producing foam glass on an industrial scale.
- Study the effect of the factor of material inconsistency associated with using recycled raw material in general and the possibility of using 100% recycled glass container in larger scale mass production.
- Study the effect of changing the type of foaming agent (such as graphite, coal, silicon carbide, calcium carbonate) and define the optimum processing parameters that correspond to every foaming agent. Also recycled foaming agent might be used to achieve a fully recycled foam glass like Aluminum slag and marble waste (mainly calcium carbonate).
• Study in more depth the effect of the shape, size and distribution of the pores inside the foam on its thermal and mechanical properties.
References


[17] “How it is done”, on Glass Recycling UK [cited 2007 February 6], Available: [http://www.glassrecycle.co.uk/](http://www.glassrecycle.co.uk/)


Appendix A : Molding Trials

One of the problems that was faced during the primarily experimentation was choosing the suitable type of mould that best suits foam glass; a mold that will be subjected to a temperature around 950 °C and still keeps its dimensional stability. Also ejecting the sample, after its sintering, from the mold without crushing it was a concern. At first, a circular ceramic mold was used with 5 cm in diameter (Figure A.1). Since it is ceramic, it supported the high temperature. However, every time the mold must be broken in order to get the sample out of it. That was an unpractical and uneconomic mold.

![Figure A.1: ceramic mold](image)

Alternatively, steel mould was used (20 x 5 x 10 cm) and wall thickness 1 cm. 2 sides of the mold can be dismantled to get out the samples and it was filled with a considerable amount of the glass powder/foaming agent mixture. That mould had two drawbacks. The first is that the sample used to stick to its walls. The second is that the walls of the mold at that high temperature warped slightly. In order to avoid these
drawbacks, the thickness of the walls increased to 2 cm and the mold was filled with sand where cavities were carved in this sand according to the number and size of the foam samples needed. The powder mixture is then put in these cavities and the mold is put in the oven (figure A.2). Ejecting the foam from that mold was easy, however, still the walls of the mold warped (figure A.3). In addition, for every batch, fresh sand must be used. So that mold was unpractical and uneconomic solution.

Figure A.2: steel mold filled with sand and curved to accommodate 3 samples

Figure A.3: foam glass once taken out of the furnace (notice the warped sides of the steel mold)
Afterwards, a thermal brick was used as a mold for two reasons. First because it is a ceramic material that is designed to withstand high temperature. Second because the samples can be ejected from it easily. 2 cavities were carved in every brick to accommodate 2 samples (figure A.4). That mold was successful in terms of thermal durability and ease of ejection. However, because it is thermally insulating material, it prevented the lower part of the sample (the base) to be effectively sintered. Consequently, the lower part, contained by the thermal brick wall, was not expanded efficiently.

![A Thermal brick carved to accommodate 2 samples](image)

Figure A.4: A Thermal brick carved to accommodate 2 samples

Finally, samples were pressed, through hydraulic press, into rectangular pellets in order to get rid of the restrictions imposed by the molds. The pressed slabs can be fed to the sintering furnace without molds.