

Recycling of Automotive Vehicle's Waste Glass to Develop Sustainable Glass Foam using a Curing-Sintering Process

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Abstract: In Australia, yearly, around 22,500 Tons of waste glasses (WG) are discarded from end-of-life vehicles being landfilled because of the costly and convoluted recycling process. This research is designed for recycling the automotive WG to produce sustainable glass foam by a modified method. Nonuniformity in size and distribution of pores within glass foam are major challenges in the traditional powder sintering method. Therefore, there is a continuous quest for searching sustainable and eco-friendly pore stabilizing chemicals. This research aimed to enhance pores' stability and uniformity through the curing of glass powder without any chemical stabilizing agent. The glass powder, additives, and foaming agent were mixed and hydrated using water. Then after curing, it was sintered (800°C). Due to the pozzolanic reactivity of glass, hydration products were formed during curing. These hydration products provided stability to the glass particles during sintering; therefore, the stability, uniformity in distribution, and pores' sizes were improved. Consequently, the strength and thermal insulation performance of glass foam were enhanced. Thus, the modified curing-sintering process shows the advancement in glass foam manufacturing. The sustainability of glass foam manufacturing is enhanced through the recycling of automotive waste glass. Additionally, the uses of chemical stabilizing agents and emissions from them are also eliminated. Besides, the curing process makes the glass foam sintering more convenient for large-scale industrial applications, which is complex in the powder sintering process.

Keywords: Recycling; waste glass; hydration; insulation; sustainability.

1. Introduction

The number of vehicles that reach the end of their lives was about 40 million globally in 2010 [1]. In Australia, yearly more than 750,000 numbers of vehicles reach the end of their lives which includes 22,500 Tons of waste glass (WG) [2,3]. These WG are discarded as automotive shredder residue (ASR) from the end-of-life and damaged vehicles and being landfilled because of the costly and complicated recycling process [3]. Most of those glasses are laminated windshields and tempered window glasses. A very few studies were done considering the ASR WG in concrete construction [4,5]. However, due to the high risk of expansion due to alkali-silica reaction, the optimum WG amount in the concrete industry is around 30% [6]. Besides, to recover silicon and synthesis of ferrosilicon alloy, the ASR WGs are now being considered; however, this process involves high-temperature synthesis (>1500°C), which could result in the emission of CO gas [3,7,8]. Also, the recovery rate is very low (~25%) comparing to the cost [9]. In this study, this complex WG is being considered as the main raw material for making glass foam applying low synthesis temperature. Glass foam composite is used for thermal and sound insulation purposes, which exhibits porosity, apparent density, thermal conductivity, and compressive strength values of about 85–95 vol.%, 0.1–0.3 g/cm³, 0.05–0.25 W/(m.K), and 0.4–6 MPa, respectively [10–14]. For the high insulation properties, high resistance to thermal and fire exposure, the manufacturing of sustainable glass foams is now gaining attention from researchers and engineers [15]. The production of a completely different and demanded product “glass foam” by using WG as the main raw material is normally termed up-cycling [16,17].

The industrial method of glass foam manufacturing requires melting of glass and decomposition of a foaming agent, which requires high energy for high melting temperature (~1200°C) and a particular arrangement of melting and annealing of glass cullet [18,19]. Besides, the newly developed method of glass foam manufacturing is the sintering of pressed glass powder at 800–1200°C with a chemical foaming agent (generally carbon-based) [12,14]. However, the sintering process requires complex arrangements for pressing the powder ingredients and transfer them into the furnace for sintering [10]. For large-scale industrial production of glass foam panels, the demolding of pressed powder is not practically convenient. Moreover, researchers recommend 1–2% stabilizing agents like Na₂HPO₄,

Na_2CO_3 , $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, Sb_2O_3 , NaSiO_3 to reduce the sintering temperature and keep uniformity in pore distribution in glass foam [14,19]. These chemical additives cost an extra charge in glass foam production and produce greenhouse gases that are being captured inside the pores of glass foam (80–90% of its volume) [11–13,20]. Hence, there is a continuous quest for replacements of the chemicals by the waste-and by-products to make them sustainable and eco-friendly.

As the WG particles are typically amorphous, and their primary constituents are silica, therefore, pozzolanic reactivity of glass particles can be helpful for pore stabilization [16]. Besides, fly ash (FA) is a by-product generated from coal-fired power stations, which contains silica and alumina. Therefore, it is suitable as pozzolanic material in glass foam mix. Due to the pozzolanic reaction of WG powder and FA, they produce bonds within the raw mix after hydration and curing [21]. After curing, the raw mix will be sufficiently strong to be moved in the sintering system. Thus, the pelletization stage in powder sintering can be eliminated. Besides, these reaction products are calcium silicate hydrates (C-S-H and C-H) which are stable up to high temperatures ($\sim 450^\circ\text{C}$) and transformed into stable wollastonite (CaSiO_3) above 800°C [22,23], thus, will be effective for pore stabilization. In this study, a curing-sintering approach of glass foam making was applied to stabilize the pore structure and to eliminate the usage of additional chemical pore stabilizers. Therefore, this study aims to develop sustainable insulation materials using complex ASR WG, utilizing minimal energy, and chemicals and proposes a convenient and sustainable method for sintering glass foam on an industrial scale. To justify the method and properties of the developed glass foam, their physical, mechanical, and thermal insulation properties were evaluated and compared with the traditional process.

2. Experimental program

2.1 Materials selection and specimen preparation

The ASR WG was collected from a local automotive vehicle repair workshop. The glass cullets were a mix of tempered and laminated broken glasses of the windows/windshields of automotive vehicles. The as-received and processed WG are shown in **Figure 1** (a-b). The WGs were manually separated from the other parts of automotive wastes (rubber, plastic, wood dust, fibers). The polyvinyl butyral plastic layer in WG was also separated manually. After washing using water, the WG was dried in an oven for 24 hours at a temperature of 105°C to remove the organic impurities and water. After cooling to room temperature, the WG cullets were ground for 20 minutes at 300 rpm speed using a planetary ball mill machine. The ground WG powders (**Figure 1b**) were used for characterization and developing glass foam. The D_{50} and D_{90} of WG were $25\ \mu\text{m}$ and $125\ \mu\text{m}$ respectively, with a Brunauer–Emmett–Teller (BET) specific surface area of about $893\text{m}^2/\text{kg}$. The particle size distribution curve is shown in **Figure 2**. Based on the X-ray fluorescence (XRF), the prepared WG contains 72.03% SiO_2 . The detailed X-ray fluorescence (XRF) data are shown in **Table 1**. Besides, the X-ray diffraction (XRD) pattern (**Figure 3**) of the WG powder shows that the glass powder sample is amorphous.

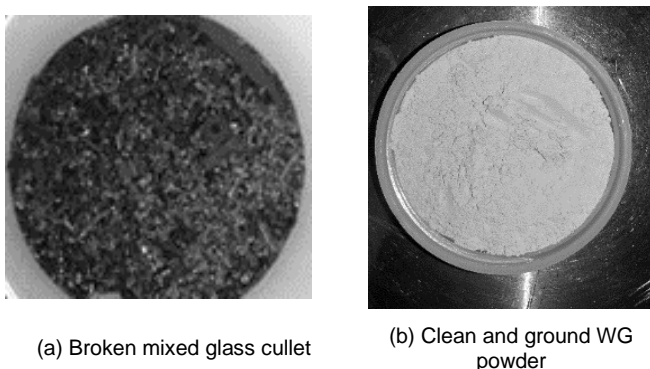


Figure 1 ASR waste glass sample.

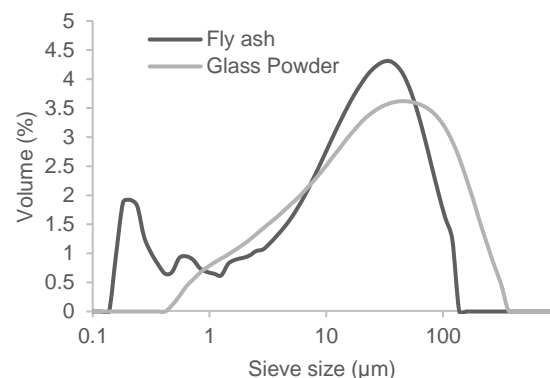


Figure 2 Particle size distribution of glass powder and fly ash.

Eight different mixes were designed to prepare glass foam for investigating the effectiveness of the proposed curing-sintering method (**Table 2**). Six mixes were prepared using FA additive of different proportions to accelerate the hydration reaction and reduce the unreacted glass particle [17]. The particle size distribution and chemical composition of FA are shown in **Figure 2** and **Table 1**, respectively. The specific surface area of the FA was $1063\ \text{m}^2/\text{kg}$ with the D_{50} and D_{90} of $18.5\ \mu\text{m}$ and $68\ \mu\text{m}$, respectively (**Figure 2**). The foaming agent was laboratory reagent grade CaCO_3 with $>98\%$ purity. The detailed mix ratio and the sintering condition are listed in **Table 2**. The WG, FA, and CaCO_3 of the designed amount were mixed and homogenized for 20 minutes at 200 rpm speed, then 30% water was mixed to complete the hydration of the glass mix (**Figure 4**). The glass mix was cast in a $25\ \text{mm} \times$

25 mm cubic mold and then was placed for hardening at room temperature for 24 hours. After hardening, demoulded raw glass foams were placed for curing at room temperature for 7 days in sealed condition. During curing, some reaction products and bonds were developed within the glass foam mix. The whole process is shown in **Figure 4**.

Table 1 Chemical composition of Glass and Fly Ash

Chemical constituent (wt%)	SiO ₂	Na ₂ O	MgO	Al ₂ O ₃	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	Cr ₂ O ₃	Fe ₂ O ₃	CuO	ZnO	MnO	LOI*
Waste glass	72.03	14.21	3.5	0.77	0.01	0.13	0.31	8.66	0.06	0.022	1	0.011	0.033	-	0.29
Fly ash	66.56	0.58	0.65	22.47	0.11	0.1	1.75	1.64	0.88	-	3.54	-	-	0.06	1.66

*LOI: loss in ignition

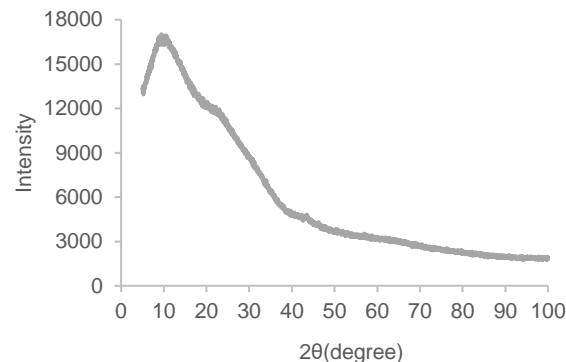


Figure 3 XRD pattern of waste glass.

The 7 days cured glass foams were placed in alumina crucibles and sintered in a muffle furnace, where the CaCO₃ was decomposed and produced gas bubbles to produce porous glass foam. The sintering temperature (800°C) was selected based on the glass transition point of WG and the decomposition starting temperature of CaCO₃ (600°C). The heating rate was 5°C/minute in a normal air environment. Sintered samples were cooled to room temperature inside the furnace naturally (slowly). The glass foam samples of different sizes were prepared for testing. The main difference in the applied method for glass foam manufacturing is the hydration and curing process instead of the pelletization of powder that has been done by applying pressure in the traditional method [12].

Table 2 Mix Proportion, Sintering Condition, and Properties of Sintered Glass Foam.

Mix ID	Raw mix (wt%)		Foaming agent CaCO ₃ (wt% of the raw mix)	Curing time (Days)	Sintering condition
	WG	Fly ash			
M1	100	-	1	7	800°C for 10 min
M2	100	-			800°C for 45 min
M3	90	10			800°C for 45 min
M4	80	20			
M5	70	30	3		800°C for 60 min
M6	70	30			800°C for 45 min
M7	70	30	1	2	800°C for 45 min
M8	70	30			800°C for 45 min

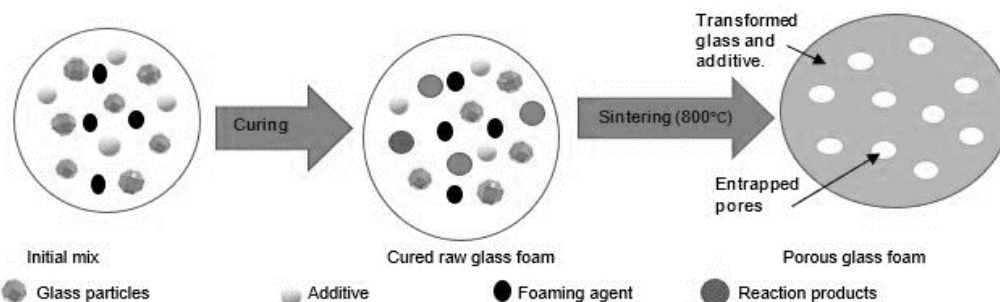


Figure 4 The curing-sintering method of porous glass foam development by using the carbonate-based foaming agent.

2.2 Methods

To characterize the raw materials and manufactured glass foams, XRD, XRF, particle size distribution, BET specific surface area analyses were done. The XRD was done using PANalytical Xpert Multipurpose X-ray Diffraction System at Mark Wainwright Analytical Centre at the University of New South Wales, Australia, using Cu–K α radiation, operated at 40 mA, and within the scan range of 5–100°. The BET was done using NOVAtouch 4LX equipment where absorbate gas was Nitrogen at a pressure of 11 psi. The density, compressive strength, porosity, thermal conductivity properties were measured to evaluate the performance of manufactured glass foam. The geometric density (ρ_g) was measured by taking the weight and volume of sintered glass foam. The true powder density (ρ_t) and porosity (P) of glass foams were measured using measuring pycnometer (water pycnometer) following ASTM C128-15 [24] and equation 1:

$$\text{Total porosity, } P (\%) = 1 - \frac{\rho_g}{\rho_t} \quad (1)$$

The sintered glass foams were cut to 15-25 mm cubes for the compressive strength test. The compressive strength test was carried out using a universal testing machine (Instron) at a rate of 0.5 mm/min. Thermal conductivity was measured by using a C-therm machine (Trident Thermal Conductivity) and Modified Transient Plane Source (MTPS) sensor following ASTM D 7984 [25]. All the test results were taken from at least three measurements. Microscopic images were taken from the cross-section of the porous samples by a Leica automated microscope.

3. Results and discussions

3.1 Properties of developed glass foams

The density, total porosity, compressive strength, and thermal conductivity of the developed glass foams are tabulated in **Table 3**, corresponding error bars are shown in **Figures 6-Figure 8**. The microscopic images of the surface of glass foam products are presented in **Figure 5**. The density and porosity of the glass foam products varied between 0.213-1.67 g/cm³ and 31-90%, respectively depending on the mix proportion. The thermal conductivity of the glass foams was around 0.083-0.309 W/m.K. For application in building insulation panels, the recommended density and thermal conductivity of glass foam are around or below 0.30 g/cm³ and 0.25 W/m.K, respectively [26]. Therefore, in this study, the developed glass foam M1, M2, M3, M4 are fulfilling these criteria for application in the building industry. However, comparing the density, strength, and thermal conductivity of glass foams developed in literature at this temperature [27–29], the glass foams M6 and M7 have desirable properties as insulation material.

Table 3 Properties of Developed Glass Foam

Mix ID	Density (g/cm ³)	Porosity (%)	Compressive strength (MPa)	Thermal conductivity (W/m.K)
M1	0.213	89.7	0.44	0.309
M2	0.24	88.5	0.54	0.117
M3	0.36	83.7	0.75	0.097
M4	0.55	77.5	1.57	0.083
M5	1.67	31.7	10.91	0.147
M6	0.83	66.5	3.51	0.254
M7	0.63	73.5	4.12	0.196
M8	0.91	60.3	4.28	0.172

3.2 Pore stabilization through the curing-sintering process

The raw material, ASR WG, was amorphous and contained ~72% of SiO₂. The glass contained 8.66% CaO and a very negligible amount of Al₂O₃. The glass powder was amorphous as found in XRD pattern (**Figure 3**). Amorphous silica shows high pozzolanic reactivity and produces calcium-silicate-hydrate (C-S-H) products after curing [30], thus, the ASR WG is suitable for the curing-sintering process of glass foam making. The amorphous silica in WG produces bonds of C-H and C-S-H gels within raw glass foam after hydration during 7 Days curing [16]. Therefore, the requirement of pressing and pelletization of powders are eliminated, and the cured raw glass foams can be moved to the sintering system conveniently, which is required for large-scale industrial production. Besides, those bonds hold the glass particle in position during sintering up to their decomposition temperatures (~450°C) and transformed into stable calcium silicate products above 800°C [22,23]. When decomposition of the bonds and reactions products starts, the softening of the glass mix starts which causes new network development between glass particles. At peak temperature, crystallization of the sintering body occurred. Thus, only pore expansion takes place due to the release of CO₂ from CaCO₃ without collapsing the pores. Therefore, comparatively uniform pore structures were observed in glass foam (**Figure 5**).

The density and strength of the glass foam were higher with the increasing amount of FA (**Figure 6**). The pore distribution and pore sizes in FA blended glass foam were more uniform and finer. FA improves hydration and formation of C-S-H and C-H bonds during the curing [16], therefore, FA provides more stability to the soft glass mix during sintering. Consequently, pore stability and uniformity are improved. Due to the higher glass transition temperature of FA [12], it increases the viscosity of the mix during sintering, prevents the expansion of bubbles, and resists pore collapse. Therefore, a denser matrix of sintered glass foam was obtained for the higher volume FA added glass foam. The amount of Al_2O_3 increases with the increasing amount of FA, which increases the required sintering temperature and time for the mix [12,19]. Moreover, when the sintering time increased to 60 minutes (M6), the porosity of glass foam was 66.5%, which is much lower than the glass foam with 20% FA sintered for 45 minutes. Besides, with the increasing curing time from 2 days (M8) to 7 days (M5), the density and strength increased significantly due to the more pozzolanic reactions.

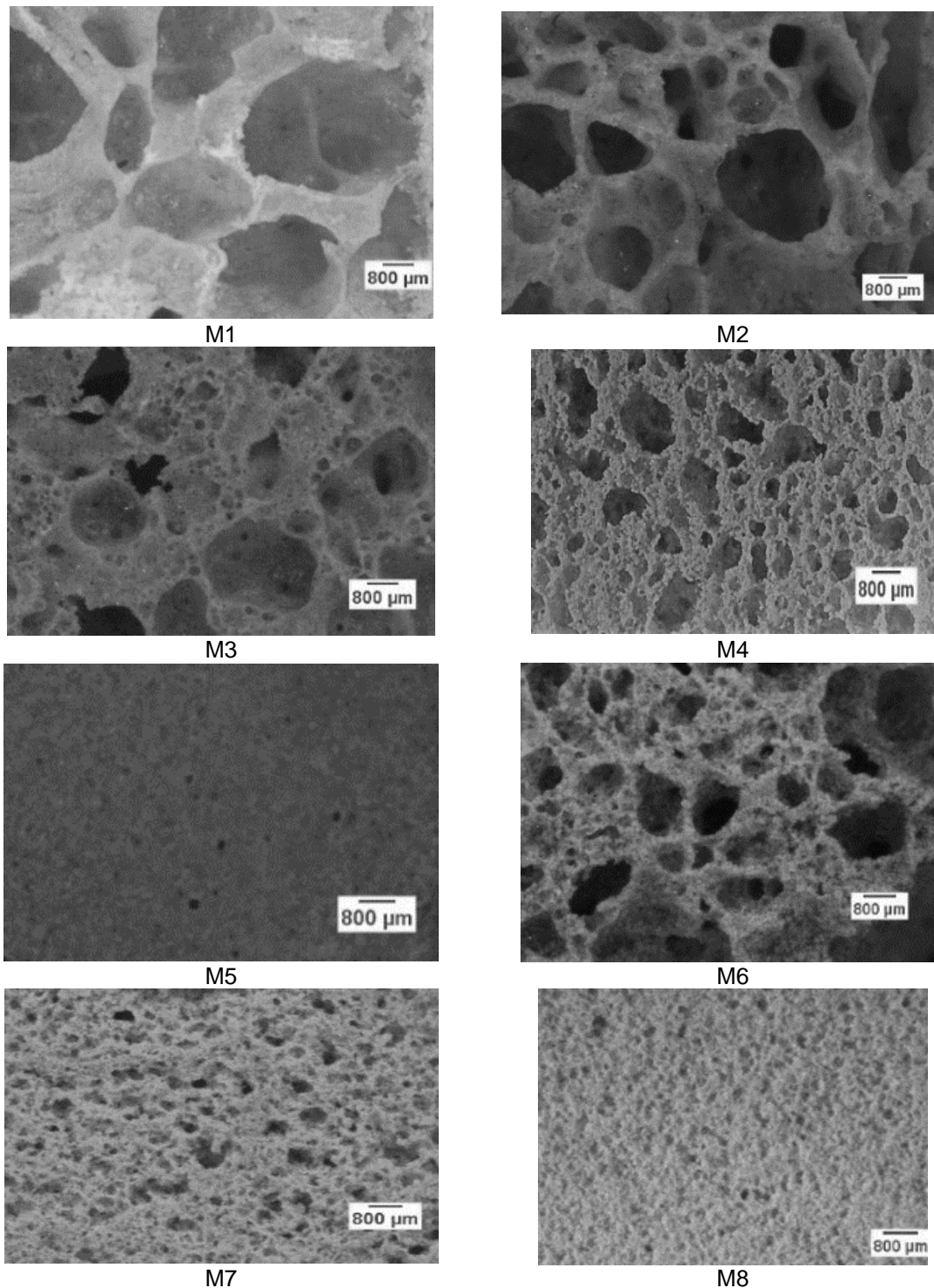


Figure 5 Microstructural details of glass foam developed from different mix proportions.

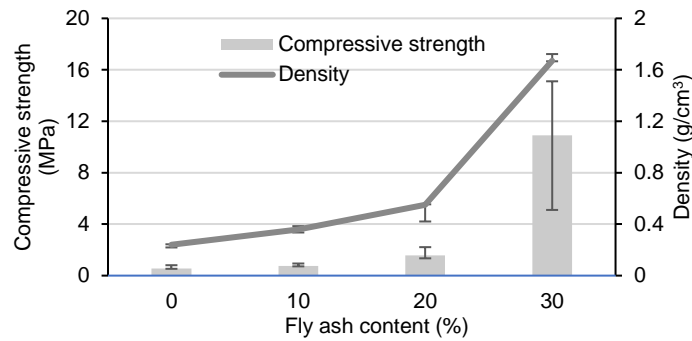


Figure 6 Variation in density and compressive strength of glass foam with fly ash content

The porosities of glass foam sintered with 1% and 3% CaCO_3 were 31.7% and 73.5% respectively. Unrealistically, with more than 2 times porosity, the glass foam M7 has thermal conductivity more than the M5 (**Table 3**). This is attributed due to the non-homogenous pore distribution in the M7 glass foam and unreacted FA [20]. However, the typical relationship between the porosity and thermal conductivity of sintered glass foam is shown in **Figure 7** and **Figure 8**. The higher porosity does not always ensure lower thermal conductivity, as the distribution and additives also influence the insulation properties. As shown in **Figure 5**, most of the pores in glass foams (M1 and M2) are open and the walls of the large pore contain many very small micropores. Thus, being highly porous, their thermal conductivities are higher than that of M3 and M4.

The main factors for open and large pore formation are the escape of CO_2 and rapid pore expansion due to the high pressure in gas bubbles during sintering. However, with the increasing sintering time from 10 minutes (M1) to 45 minutes (M2), the total porosity decreases, and some open pores in glass foam turn to closed type (Figure 5). When sintering temperature reached 800°C , the soft glass particles are bonded to each other and get stabilized as a new phase. Extending the sintering time from 10 minutes to 45 minutes provided more stabilization time and thus, the soft glass particle starts to densify and gets closer to each other. However, in the case of more sintering time, the gas pressure may increase heavily and some of the pores may get collapsed [10]. Due to the large and open pores, the heat flow increases, consequently a high thermal conductivity was observed for samples M1 and M2 despite having very high porosity. In this case, the FA acts as a stabilizer of the pore in the glass foam [12]. Therefore, more uniform size and distribution of pores have been observed in M3, M4, M5, M6, M7, and M8 glass foams. The most ideal results were obtained for M4 glass foam, which contained 20% FA and possesses 77.5% porosity and 0.083 W/m.K thermal conductivity. This is clearly showing the importance of uniform size, homogeneous distribution, and closed pores for thermal insulation performance, which is prominent in this curing-sintering method.

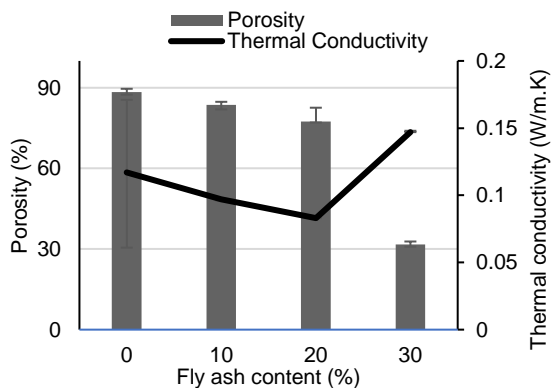


Figure 7 Variation in porosity in glass foam with fly ash content

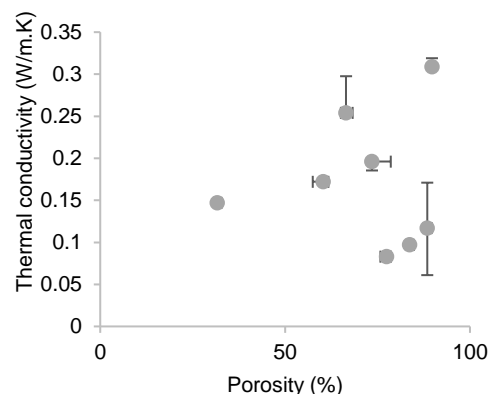


Figure 8 Relationship of the porosity and thermal conductivity of sintered glass foam

3.3 Comparative sustainability

The main raw material for this study is the automotive vehicle's waste glasses. These glasses are being considered for silicon recovery technology, which requires high-temperature synthesis ($\sim 1550^\circ\text{C}$), thus not sustainable [3,7]. In this study, the maximum adopted sintering temperature was 800°C , and the properties of developed glass foams were satisfactory. Therefore, the developed method is suitable for industrial applications to recycle automotive vehicle's waste glasses sustainably and conveniently. Besides, Australian coal-fired power stations produce 12 million tonnes of ash per year from burning

coal, where only 44% of ash is saved from the dump, and half of them is recycled in the construction industry as FA [31]. This research proposes an additional path to recycle FA in the construction industry.

Additionally, the conventional melting and annealing of glass foaming manufacturing require high energy; the powder sintering method also requires stabilizing agents and is not practically convenient for large-scale production [10,14,18,19]. Therefore, both the melting and sintering methods are still under investigation. Additionally, the melting of ASR WG above 1000°C temperature releases toxic CO gases [3]. Besides, the powder pelletization system is complicated and inconvenient for large-scale sintering. Meanwhile, the most important findings of this study are the improvement of the pore's structure, uniformity, and distribution by curing WG and the elimination of using pelletization stage and stabilizing agent. The chemical bonds (developed during hydration) act as stabilizers and keep the pore uniform and homogenous by resisting pore collapse during sintering with the easily decomposable CaCO₃ foaming agent. Consequently, the proposed curing-sintering process is comparatively a more convenient and sustainable solution of glass foam manufacturing, which need to be explored in large scale.

4. Conclusions and future recommendations

This study aimed to explore the utilization of waste glasses of automotive vehicles' windows/windshields for preparing sustainable glass foam at low sintering temperatures with minimal environmental impact. A modified process was adopted for stabilizing the pore structure within glass foam. In general, it can be concluded that the glass foam developed in this study is suitable for thermal insulation and acoustic application in the building industry which possesses a density around 0.213-0.63 g/cm³ with a porosity ranged between 66.5-89.7% and corresponding thermal conductivity of 0.083-0.309 W/m.K. The compressive strength of these glass foams was around 0.44-4.12 MPa. The results revealed that 1% of CaCO₃ foaming agent and 20% fly ash additives are an optimized composition for glass foam sintering at 800°C for 45 minutes. However, the ultimate findings of this study are low cost and convenient processing of the automotive waste glasses and making the glass foam manufacturing process more convenient, eco-friendly, and sustainable. This method needs to be explored on large scale for industrial manufacturing of glass foam, where recycling of glass and fly ash will contribute to sustainability in the construction industry. To accelerate the reactions between glass particles and reduce the sintering temperature, a low temperature and pressure curing system can be applied, where the pore stability and eco-friendliness will be enhanced. Besides, instead of carbonate-based foaming agents, the eco-friendly surfactant can be utilized in future studies.

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