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## Low temperature preparation of porous materials from TV panel glass compacted via hydrothermal hot pressing

Z. Matamoros-Veloza<sup>a,\*</sup>, J.C. Rendón-Angeles<sup>b,c</sup>, K. Yanagisawa<sup>d</sup>, E.E. Mejia-Martínez<sup>a</sup>,

<sup>a</sup>Technological Institute of Saltillo, Research and Graduate Division, Saltillo 25280, Mexico <sup>b</sup>Research Institute for Advanced Studies of the NPI, Campus Saltillo, Saltillo 25900, Mexico

<sup>d</sup>Research Laboratory of Hydrothermal Chemistry, Faculty of Science, Kochi University, Kochi 780-8520, Japan

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#### Abstract

The influence of aqueous solutions (deionized water or Na<sub>2</sub>CO<sub>3</sub> solutions, 0.1–0.5 M) and other processing parameters such as temperature (150-250 °C) for preparing hydrothermal hot-pressed TV panel waste glass compacts was investigated to produce porous glass specimens. Waste powdered glass (5 g) from old analog Philips television panels (Mexico) with particle sizes of < 38 μm was mixed with various amounts of the selected aqueous solution (5-20 wt%). The TV glass panel particles dissolved in all of the mineralizer solution that were employed, and the behavior was controlled by the amount of aqueous solution added to the glass powder densification stage. The reactions of the glass particles with water or Na<sub>2</sub>CO<sub>3</sub> solutions produced a solid glass phase that included water molecules or Na<sup>+</sup> and CO<sub>3</sub><sup>-2</sup> ions, respectively. The hydrothermal hot press (HHP)-treated glass compacts prepared at 200 °C for 2 h at a constant loading pressure of 20 MPa exhibited a marked expansion after heat treatment conducted at 700 °C for 1 h in an air atmosphere. The expansion of the HHP compacted TV panel glass specimens was further increased for the samples prepared with 0.5 M Na<sub>2</sub>CO<sub>3</sub> solution; these foamed glasses exhibited the lowest apparent densities that varied from  $0.309 \text{ to } 0.319 \text{ g/cm}^3$ .

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#### 1. Introduction

Porous materials prepared at low temperatures represent an important class of construction materials for their use in such applications as heat insulators, particularly the material processing, is environmentally friendly.

Particular interest has focused on used TV panel glass that may contain hazardous compounds in light of the increased number of digital electronic devices that are replacing older analog TV

\*Corresponding author.

Tel.: +52 844 438 9500x1204; fax: +52 844 438 9537.

E-mail addresses: zullyma@itsaltillo.edu.mx, renzuzy@gmail.com (Z. Matamoros-Veloza).

cathode tubes [1]. However, many analogs televisions can still be found in many developing countries, such a Mexico, which are being discharged as waste. Of the total of panel glass that is discarded (10,000 ton in 2011), only approximately 3% is recycled [2], which represents a significant environmental problem. Therefore, the implementation of recycling techniques to reduce the amount of this type of waste is essential.

Porous glass materials with open and/or closed pore networks are currently manufactured via a combination of solid state sintering and decomposition of organic compounds or calcium carbonate during the foaming process, which is generally conducted at elevated temperatures above 800 °C. Recently, new routes for preparing porous glass have been

<sup>&</sup>lt;sup>c</sup>Department of Nanoscience and Nanotechnology at the Research Institute for Advanced Studies of the NPI, Campus Mexico City, U. Profesional Adolfo Lopez Mateos, Mexico

Table 1 Chemical composition of waste TV glass powder determined by quantitative wet analysis.

Oxide	$SiO_2$	MgO	CaO	$ZrO_2$	SrO	BaO	MnO	PbO	$Fe_2O_3$	$Al_2O_3$	CoO	TiO <sub>2</sub>	Na <sub>2</sub> O	$K_2O$	$PO_4$	$SO_4$
wt%	78.05	0.25	0.24	3.49	0.47	0.48	0.17	< 0.05	0.32	3.12	0.38	0.05	0.99	9.35	2.124	0.06

implemented involving the reaction of glass with water under supercritical conditions, during which the water becomes extremely reactive due to the increased dissociation potential and the high mobility of molecules [3–7]. Other routes recently proposed established that the preparation of porous materials mainly proceeds via a phase separation process in the ternary system of Na<sub>2</sub>O–B<sub>2</sub>O<sub>5</sub>–SiO<sub>2</sub> due to thermal treatment. In addition, porous glass typically had been prepared using the sol–gel process, or the combination of glass powder and foaming agents such SiC and TiN, which are decomposed and oxidized to form gas at temperatures above 800 °C, thus producing pores [8,10].

Similar experiments conducted later by Yoshikawa et al., using 63 mass% SiO<sub>2</sub>–27 mass% Na<sub>2</sub>O–10 mass% B<sub>3</sub>O<sub>3</sub> glass reported that the formation of pores depends strongly on the water content and temperature [10]. However, the authors found that the initial foaming temperature was 200 °C, and although the porosity was not uniform, the obtained foam specimen could be used as a filter or a supporting material [13].

Under similar HHP pressing conditions, porous glass using  $50 \text{ wt}\% \quad \text{SiO}_2$ –24.14 wt% BaO–9.5 wt% Na<sub>2</sub>O–8.9 wt% K<sub>2</sub>O panel TV-glass was also successfully prepared. The expanded glass specimens were primarily composed of close porous cells and some large interconnected pores. A partial coalescence of the close pores and its devitrification were controlled by the water content and the chemical composition of the starting glass, in particular the presence of alkaline ions, which are thought to be responsible for the decreased transition temperature,  $T_{\rm g}$ , during the heating process and the maximum expansion that occurred at  $700 \, ^{\circ}\text{C}$ . The obtained porous product exhibited a relatively low thermal conductivity (0.021 W/cm  $^{\circ}\text{C}$ ), and thus can be used as a thermal insulator [12].

The aim of the present study was to evaluate the influence of the aqueous solution (water and  $Na_2CO_3$ ) on the preparation of porous materials from glass powdered derived from old analog Philips television glass (78 wt%  $SiO_2$ –9.4 wt%  $K_2O$ –3.5 wt%  $ZrO_2$ –3.0 wt%  $Al_2O_3$ –2.0 wt%  $PO_4^{-3}$ ) by hydrothermally hot-pressing the powder and subsequently applying heat treatment. The produced porous materials could have applications as a potential insulator [14]. In addition, the presence of  $ZrO_2$  and  $Al_2O_3$  oxides in the chemical composition of the glass, may improves the chemical durability of the material against water, as  $ZrO_2$  strengthens the silicate network and therefore increases the glass transition temperature and viscosity. The porous glass material could be used as a filter or insulator material [15].

#### 2. Experimental

Used TV panel glass was obtained by removing the glass from the panels of old analog Philips televisions (Mexico). The dismantled panel glass was crashed into 1–2 cm pieces on average, and the pieces were subsequently converted to powder by ball milling for

 $12\ h$  using a zirconia ball media. The particle size selected for the experiments was  $<38\ \mu m$ . The chemical composition was determined using inductively coupled plasma atomic emission spectrometry (ICP-AES). The details of the chemical composition of the glass determined via wet chemical analysis are shown in Table 1.

Hydrothermal hot pressing experiments were performed using 5 g of the powdered TV glass with a particle size less than 38 μm. The glass powder was mixed in an agate mortar with various contents of aqueous solution (5-20 wt%) [18]. Three solutions, including deionized water and 0.1 and 0.5 M Na<sub>2</sub>CO<sub>3</sub> solutions, were used as a mineralizer solution for the compaction experiments [24]. Each mixture sample was transferred into the main reaction chamber of a hydrothermal hot pressing autoclave, as shown in the schematic in Fig. 1 [16]. The autoclave contained a piston cylinder with an inner diameter of 20 mm. The pistons in contact with the powder include an open space for the liquid release, which occurs during the compaction process. The autoclave was closed and subsequently loaded at a pressure of 20 MPa, which was maintained constant throughout the compaction process, including the heating, holding and cooling stages. The autoclave was heated at a constant rate of 5 °C/min to the desired treatment temperature (200 °C). After hydrothermal hot pressing treatment, the autoclave was cooled to room temperature, and the compacted glass specimen was removed for physical and chemical characterizations.

In the second stage, some glass compacts prepared via HHP were heated in air for 1 h at 50-750 °C. These heat treatments were conducted at a constant heating rate of 5 °C/min. The weights of the compacts were measured before and after the heat treatment to determine the weight loss and to establish the maximum expansion temperature of the compacted glass specimens for this type of glass. Therefore, the porous glass materials were prepared via heat treatment at a fixed temperature of 700 °C for 1 h in an air atmosphere. The microstructure of the fractured surface of the porous specimens was observed using an FE-SEM scanning electron microscope (JEOL JSM-6500F). The apparent densities of the foamed glass specimens were determined based on Archimedes' principle using a helium pycnometer (Multipycnometer Quantachrome). Prior to the density measurements, the pycnometer was calibrated using a standard stainless steel sphere with a volume of 56.5592 cm<sup>3</sup>, which was located inside the measuring cell (volume 135 cm<sup>3</sup>). The foamed specimens were previously weighted, and the volume was measured three times at a helium pressure of 0.117 MPa.

#### 3. Results and discussion

# 3.1. Preparation of TV panel glass compacts under hydrothermal conditions

We recently reported a novel route for the preparation of porous glass-ceramics that involves a preliminary treatment

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