Study of graphite addition in SiO_2-Na_2O-CaO vitreous system to produce a foam glass intended for electromagnetic shielding

F. Benhaoua [a,*], D. Aoufi [b], N. Stiti [a], Y. Lamri [a], M. Toubane [a], R. Tala-Ighil [a], I. Ouldamri [a], F. Mouhouche [c]

[a] *Research unit Materials, Processes and Environment UR-MPE, Faculty of Engineering, University M'Hamed Bougara of Boumerdes, 35000 - Algeria*

[b] *Research Center in Industrial Technologies -CRTI- Echahid Mohammed Abassi, P.O. Box 64, Cheraga 16014 Algiers, Algeria*

[c] *Laboratory of Signal and Systems, Institute of Electrical and Electronic Engineering, University M'Hamed Bougara of Boumerdes, 35000 - Algeria*

In the present study, we have developed foam glass based on a waste glass of the SiO_2-Na_2O-CaO vitreous system, using calcium carbonate ($CaCO_3$) which is a natural porogen very abundant in nature, 1% of which ensures the formation of a homogeneous porous structure, formed thanks to its decomposition during sintering process at 800°C. Graphite is introduced into the raw material matrix by substituting 0, 3, 9, and 15 wt% of the waste glass. To analyze the performance of the foam glass obtained, we measured several properties such as density, porosity, coefficient of absorption, reflection, and transmission of electromagnetic waves. The results clearly show that graphite has no remarkable effect on the properties of materials up to 15 wt%. Beyond this, the substitution has a significant effect on density (increasing from 0.225 to 0.64 g/cm^3), porosity (decreasing from 91.01 to 73.59 %), and electromagnetic wave Absorption, which improves from 20 % for the sample with 0 wt % graphite (G1) to 91.37 % for the sample with 15 wt % graphite (G2).

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1. Introduction

Glass from the SiO_2-Na_2O-CaO vitreous system is highly sought after in many fields due to its excellent physical, chemical, optical, and electrical properties. However, its end-of-life poses a significant environmental challenge due to waste accumulation. Manufacturing foam glass from waste glass, and using a natural, synthetic, or waste-derived porogen (foaming agent) offers an effective solution, not only reducing waste volume but also potentially saving energy for heating/cooling in various types of buildings. This is because the porous structure imparts characteristic thermal and sound insulation properties to the materials [1-4].

In recent years, the obsession with absorbing electromagnetic waves has preoccupied many researchers, due to the harmful effects they cause on all living beings, including plants, animals and humans, and can also affect the operation of various devices [5-8]. This is due to the spectacular evolution of technologies and means of communication such as Wi-Fi, which is now used everywhere by all intelligent devices. This makes it necessary to find a way of isolating special premises from these waves [9-11].

The addition of elements absorbing electromagnetic waves in the raw material mixture such as carbon fibers, carbon nanotubes, and Fe_2O_3, allows expanding the applications of the porous material from the classic use to the isolation of electromagnetic waves [12-14].

In the present work, we have developed foam glass based on waste glass from the SiO_2-Na_2O-CaO vitreous system and $CaCO_3$, by substituting part of the glass with graphite powder with a particle size in the micrometre range. The characterization of the materials obtained by the ZNB

* Corresponding author: f.benhaoua@univ-boumerdes.dz
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network analyzer clearly shows the electromagnetic shielding effectiveness of waves by the materials obtained in the frequency range of 8 to 10 GHz (X band).

2. Experimental

2.1. Material preparation

For the present study, three types of micrometric raw materials with particle sizes less than 100 µm were used. The SiO_2-Na_2O-CaO vitreous system obtained from building waste constitutes the skeleton of the porous materials; the $CaCO_3$ is an economical, and naturally abundant porogen that forms the porous structure, the graphite composed of carbon with a hexagonal structure, is used as a filler to absorber electromagnetic waves. Table 1 represents the mass percentage composition of each variant. The mixture was well homogenized using a mortar and pestle then 40 g of each variant was pressed to form a 40 mm tablet under a pressure of 7.96 KN/cm² using a HERZOG-type mechanical press. The pellets were then placed in a NABERTHERME programmable furnace and subjected to a thermal treatment consisting of a linear temperature increase at a heating rate of 6.5 °C/min up to 800°C. The temperature was held constant for 20 minutes, after which the materials obtained were allowed to cool inside the furnace (very slow cooling) to room temperature.

Table 1. Chemical composition (wt%) *of the porous materials.*

Sample	glass Waste	Graphite	$CaCO_3$
G0	99	0	1
G1	96	3	1
G2	90	9	1
G3	84	15	1

2.2. Methods of characterization

The porous materials obtained (Fig. 1) underwent a succession of physico-chemical analyses to establish their effectiveness as materials that absorb electromagnetic waves. The apparent density of the foam glass was measured geometrically by dividing the mass by the total volume. The estimated porosity percentage was calculated using equation (1):

$$\% \text{ porosity} = 1 - (\text{bulk density} / \text{powder density}) \times 100 \quad (1)$$

SEM analysis was performed on 0.7 cm³ samples using a Philips ESEM XL 30 scanning electron microscope to observe the effect of graphite addition on the microstructure of the produced materials. X-ray diffraction (XRD) analysis was conducted on ground powders of the foam glass after processing, using a Philips PRO-MPD diffractometer.

Fig. 1. Photos of foam glasses.

Electromagnetic wave absorption, transmission, and reflection coefficients measurements were carried out in the X-band (8 to 12 GHz) using a ZNB network analyser.

3. Results and discussion

3.1. Density and porosity

The foam glasses obtained are characterized by considerable lightness. Density varies from 0.225 to 0.640 g/cm³, due to the porosity produced by the decomposition of the calcium carbonate ($CaCO_3$) during the thermal treatment. This decomposition occurs between 680 and 720 °C according to the TDA-TGA diagram, which corresponds to the softening range of the SiO_2-Na_2O-CaO vitreous system [4, 13]. This process imparts high porosity to the materials, ranging from 91.01 % for G1. Fig. 2 illustrates the development of density and porosity of the materials produced.

We also observed that the addition of graphite negatively affects the density and, consequently, the porosity. This is attributed to the high density of graphite compared to materials obtained density (which is 2.23 g/cm³ and 0.225 g/cm³ for graphite and G1 respectively). According to Fig. 1, the porous materials G2 and G3 exhibit the same pore size as G1. However, G4 displays non-uniform porosity due to the involvement of graphite in the pore formation process through its incomplete decomposition [15]. This adversely affects the homogeneity of the porosity in the porous vitreous system.

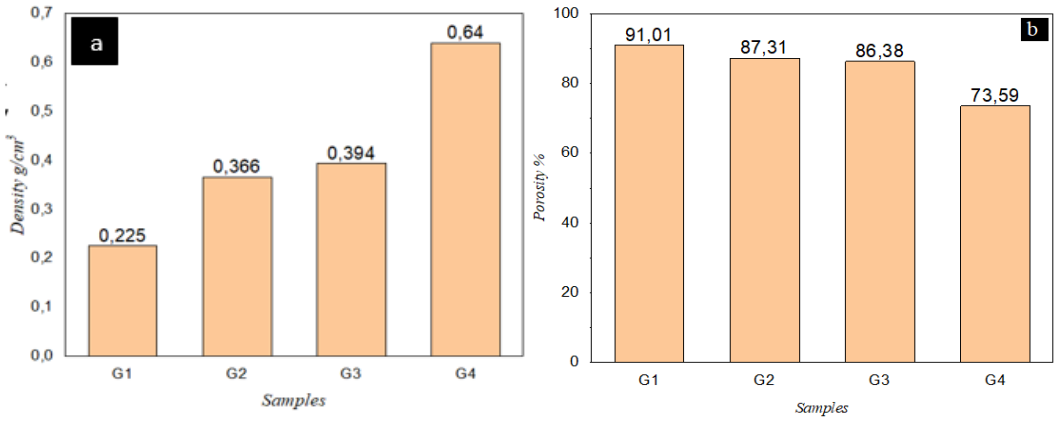

Fig. 2. Density (a) and porosity; (b) of foam glasses.

3.2. Microstructure

The microstructure of foam glasses was studied only on the G4 variant. That presents the highest fraction of graphite. SEM characterization (fig.3.a) confirmed the partial decomposition of graphite, which is located within the pore walls. Graphite is therefore inert, and it does not participate in the amorphous structure of the SiO_2-Na_2O-CaO vitreous system, but it plays a role in pore formation.

The microstructure of G4 also reveals a dual pore size distribution, with macrospores and microspores partially connected (fig. 3.b).

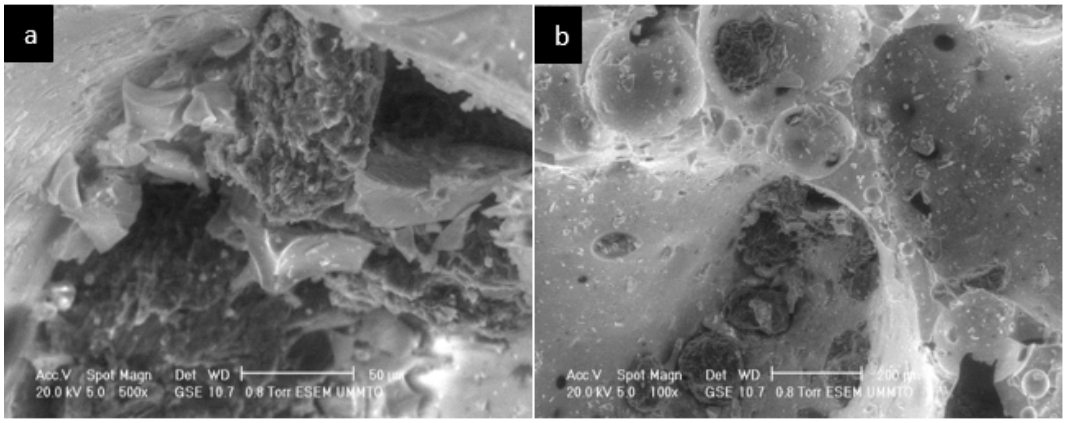

Fig. 3. SEM micrographs of foam glasses.

3.3. Structure

The result obtained from X-ray diffraction (XRD) of foam glasses analysis is shown in Fig.4:

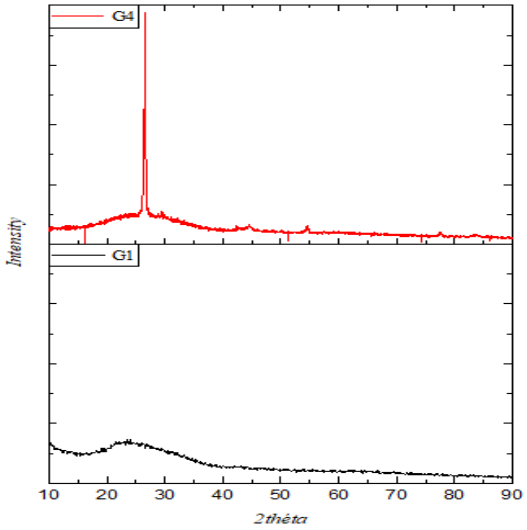

Fig. 4. X-ray diffraction patterns of foam glasses.

The X-ray diffraction (XRD) analysis was performed on two types of powder after preparing our porous materials, one without graphite (G1) and the other containing 15 wt% for graphite (G4). The X-ray diffraction spectrum shows the amorphous structure of both samples. However, G4 exhibits a partially crystalline amorphous structure, which is attributed to an intense peak at $2\theta = 26.56°$ and additional less intense peaks at 44.53°, 54.75° et 78.48°, indicating the presence of

graphite. This suggests that the graphite did not decompose completely, and therefore it does not participate in the formation of the vitreous network.

3.4. Electromagnetic rays shielding test

Fig.5: a, b, and c show the results of the absorption, reflection, and transmission coefficients of electromagnetic waves from foam glasses obtained and processed in the X-band (8 to 12 GHz).

From the results, it is evident that graphite positively affects electromagnetic wave absorption (Fig.5.a). However, with increasing graphite addition in the raw material powder mixture, the absorption coefficient increases, ranging from 0.2 for G1 with 0 wt% graphite addition to a maximum of 0.92 for G4 with 15 wt% graphite addition. This demonstrates a 4.6-fold increase in absorption.

The reflection coefficient result (Fig.5.b) of all the materials produced shows almost the same range of variation, from 0.5 to 0.01.

However, the transmission coefficient (Fig.5.c) decreases from 0.90 to 0.11 for materials made from G1 and G4 respectively, and from 0.27 at 8 GHz to 0.11 at 12 GHz for G4. This variation towards zero in transmission coefficient is very considerable for a material that electromagnetic waves absorber.

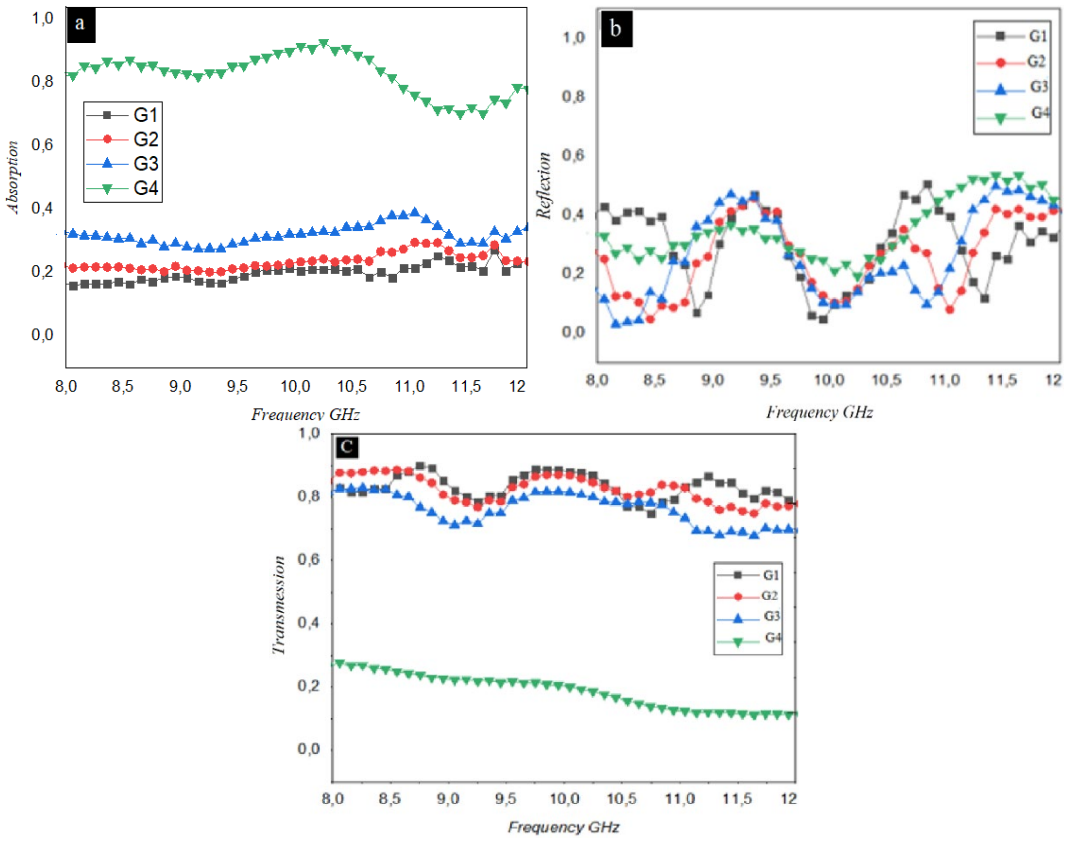

Fig. 5. Parameters of the electromagnetic response of foam glasses. Coefficients: a) absorption; b) reflection and c) transmission.

4. Conclusion

Materials developed from the waste of the SiO_2-Na_2O-CaO vitreous system and 1 wt% of a naturally abundant porogen are considered a positive step towards environmental protection by safeguarding nature against solid waste pollution. The porous structure also endows them with the potential to be promising materials in thermal insulation, thereby facilitating the reduction of energy

costs associated with air conditioning and consequently mitigating the greenhouse effect caused by carbon dioxide emissions.

The addition of graphite to the waste of the SiO_2-Na_2O-CaO vitreous system enhances the applicability of our materials in electromagnetic wave absorption. The G4 variant, with a 15 wt% addition of graphite, exhibits significant lightness and porosity, along with an electromagnetic wave absorption capacity exceeding 90% in the X-band (8 to 12 GHz). It also reduces transmission to values close to 11%, within the same frequency band, rendering it an effective material intended for electromagnetic rays shielding in this range.

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