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Y. Lamri, R. Benzerga, A. Ayadi, L.L. Gendre, A. El-Assal. Glass foam composites based on tire's waste for microwave absorption application. *Journal of Non-Crystalline Solids*, 2020, 537, pp.120017. 10.1016/j.jnoncrysol.2020.120017 . hal-02535372

HAL Id: hal-02535372

<https://univ-rennes.hal.science/hal-02535372>

Submitted on 20 May 2022

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Glass foam composites based on tire's waste for microwave absorption application

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Abstract

Waste glass and tire based glass-foams have been studied to produce a low-cost microwave absorbing material that could be used in building industry. These composites show, firstly, an heterogeneous structure induced by the degradation of the tires during the heat treatment, and secondly, low dielectric loss partially induced by their low densities. In order to avoid the heterogeneity of the samples, two different composites, using tire waste, have been tested. For the first composite, tires were coated with a refractory material, for the second, tires were pre-calcined before composite preparation. The use of calcined tires, which contain more than 99wt.% of carbon, has led to homogenous composites with higher dielectric loss ($\tan\delta=0.33@10\text{GHz}$). The absorption performance simulation of these composites shows encouraging results. A low reflection (close to -30dB @ 11.90GHz) and a large absorption bandwidth have been obtained for carbon (calcined tires) loaded foam composite with a thickness of 12mm.

Key words:

Glass foam composites; microwave absorbers; wastes; cullet; tires; dielectric properties.

1. Introduction:

The increasing use of electromagnetic (EM) waves for communication (mobile phone, WiFi, etc.) and electronic equipment, expose people to an omnipresent EM pollution. Today, both the population and the equipment manufacturers and suppliers, become sensitive to this issue. For this, development of EM absorbing materials is needed to protect human health and devices from this increasing invisible pollution [1,2]. Consequently, great attention has been given to the development of new microwave absorber materials in order to meet the recurring specifications, a broad band absorption frequency, a low density and a low production cost [3].

Usually, absorbing materials are classified in two broad categories according to the different loss mechanisms involved: dielectric loss materials [4] and magnetic loss materials [5]. Dielectric absorbers are often produced from dielectric matrices containing carbon in its different forms: graphite [6], graphene [7], carbon nanotubes [8], etc. These materials present a high permittivity and dielectric losses, leading to the EM attenuation. Materials with magnetic losses are often based on ferrites MFe_2O_4 with ($M= Ni, Co, Cu, Zn, Mg \dots$) [9-11] or metal carbonyls (for example carbonyl iron [12-14]). However, their high density ($d > 2 \text{ g.cm}^{-3}$) limits their use in a number of mobile or building applications [15]. From these two absorber categories and as the EM waves are composed of electrical and magnetic fields, some absorbing materials combine these different loss mechanisms. Different studies have shown that the association of magnetic and dielectric losses led to an effective EM attenuation [16-18].

Concerning the shaping of these absorbing materials, different techniques are used depending on the material composition and the targeted application. Absorbers exist as thin films (often based on heavy ferrites) [19], thick films or plate [20,21], or in the form of foams.

Indeed, porous materials have been widely used as EM absorbers: polymer foams [22,23], ceramic foams [24,25], carbon foams [26,27] and silicon carbide foams [28].

Otherwise, glass foam made from cullet (glass waste) is often studied for thermal and acoustic [29,30] domestic insulation. Other studies showed that glass foams could be also used as photo-catalyst for hydrocarbons degradation [31]. In recent years, some studies have shown the potentiality of using glass foams for EM applications. Different loads have been associated with this dielectric foam matrix such as carbon particles [6,32,33], carbon fibers [34] or silicon carbide SiC [28].

Furthermore, several tons of tires are thrown away each year [35]. These used tires are often stocked in inappropriate places or are burned leading to serious dangers where their destruction is the source of toxic and harmful emissions [36,37]. In recent years, the idea of recycling this waste has emerged and has given rise to several attempts to reuse these materials after treatment. One of the important procedures of used tire treatment is the tire grinding where used tires are converted into granular materials that can be used later and give a second life to this waste. Different applications are tested, using these tires as granulates, such as for playgrounds [38] and athletics race tracks [39]. Another tested solution is the use of these tire granulates as composites, in combination with other materials, such as concrete [40,41], mortars [42] or resins [39].

In this work, used tires were envisaged as a load for electromagnetic absorber composites because they are mainly composed of natural and synthetic rubber that contains a significant amount of carbon [39,41]. Moreover, they often contain high percentages (between 20 and 40 wt.%) of carbon black particles [43,44], which is often used to develop electromagnetic absorbers [4,45].

In this article, we propose to study the potentiality of associating the glass foam matrix (elaborated from glass waste) with waste of tires to produce EM absorber composites. This

association could lead to the development of a new way of recycling for the glass waste and used tires.

This article is organized as follows: the next part presents the preparation of glass foam composites, loaded with tire granulates, and the characterization techniques used. Then, results of structure and morphology of glass foam composites and their dielectric properties are presented. In the last part, the absorption performance of glass foam composite is simulated and discussed.

2. Material and methods

2.1. Preparation of glass foam composites

2.1.1. Tire granulates / glass foam composite

The glass foams were prepared from soda-lime glass waste provided by Mediterranean Float Glass (Algeria). The composition of this glass waste, provided by the manufacturing company, is shown in Table 1. This waste is mainly composed of SiO₂ (~71.7 wt.%), Na₂O (~13.5 wt.%) and CaO (~8.7 wt.%), as well as other oxides in lower percentage.

Elements	SiO ₂	Na ₂ O	CaO	MgO	Al ₂ O ₃	K ₂ O	Fe ₂ O ₃
wt.%	71.73	13.49	8.69	4.33	0.98	0.60	0.07

Table 1. Composition of the soda-lime glass waste

The grinding of the cullet was carried out using a planetary ball mill RETSCH PM 100 (Haan, Germany), with a rotation speed of 300 rpm for 30 minutes. After grinding, the cullet was sieved at 100 μm particle size. After grinding and sieving, the glass powder was mixed with 1 wt.% of the foaming agent (calcium carbonate CaCO₃, from CETIM, Boumerdes, Algérie) and using different percentages (Table 2) of the tire granulates. The latter have diameters ranging between 1 mm and 2.5 mm. The mixture of the different compounds

(cullet, foaming agent and tire granulates) was then humidified with 15 wt.% of water, placed in a refractory steel mold and heated at 800°C for 30 minutes in a furnace Thermolyne 46100 (Dubuque, USA). In these conditions, a gas emission, produced by the reaction of the foaming agent ($\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$), associated with a glass softening (at 800°C), traps the produced gas in the softened matrix and allows the production of the foamed glass. The latter is a porous material with a density that depends on the nature and the percentage of the foaming agent [33], the glass composition and the heating temperature of the mixture [46].

Table 2 summarizes the composition of the powder mixtures and the resulting foam densities. We can note that the densities range between 0.22 and 0.43. The sample elaborated with the largest tire granulates fraction (20 wt.%) presents the highest density.

Sample	Smp-1	Smp-2	Smp-3	Smp-4	Smp-5	Smp-6
Tire granulates (wt.%)	0	4	8	12	16	20
Cullet (wt.%)	99	95	91	87	83	79
CaCO_3 (wt.%)	1	1	1	1	1	1
Density (g.cm^{-3})	0.28	0.22	0.30	0.27	0.37	0.43

Table 2. Batch compositions and resulting foam densities

2.1.2. Tires embedded in a refractory cement / glass foam composite

The glass foam composite loaded with refractory cement-coated tire granules was made with the same process as that used for the standard glass foams presented above. Here, the tire granules were coated with refractory cement, which can resist temperatures up to 1500°C before degrading. The chosen coating process was simple. First, used tire aggregates have been treated with a saturated aqueous solution of NaOH for 20 minutes to improve their adhesion with cement [40]. Then, a mixture of 10 wt.% tire granules and 90 wt.% refractory mortar paste (Pyrofeu, Apt, France) was produced. After stirring for 10 minutes, beads with a

near-spherical shape of 8-10 mm in diameter were hand-made from this mixture. To remove the binder, the resulting balls were air dried for one day before being used as fillers to produce glass foam composites. A weight percentage of 12 wt.% of those tires embedded in refractory mortar granules was used for the elaboration of the foam composite.

2.1.3. Calcined tires / glass foam composite

The carbon based glass foam composite was prepared using calcined tire granules. To produce carbon powder from the calcination of tires, the granules of these tires were heated to 500°C under air atmosphere. The powder produced by this calcination was characterized by an EDS analysis which showed a carbon composition of more than 99%. This powder was then milled and mixed with the cullet and the calcium carbonate, as a foaming agent, to achieve loaded glass foam. The same heating treatment as before (800°C for 30 minutes) was used. The carbon powder, obtained from the calcination of the equivalent of 12 wt.% of tires, was used for the achievement of glass foam/carbon composite sample.

2.2. Characterization technics and simulation

2.2.1. Morphological and thermal characterization

The structure of the samples was observed by an optical microscope Leica DM 2500M (Mannheim, Germany) and a scanning electronic microscope (SEM) JEOL 5600 (Tokyo, Japan). The chemical composition was measured by an EDS OXFORD instruments 51-ADD 0076 (Bristol, UK).

The density of the glass foams was calculated by measuring the mass and the volume of each sample. A measurement uncertainty of ± 0.01 was estimated and was taken into account for the presented results.

The thermogravimetric analysis (TGA) was carried out with SDT Q600 equipment (TA Instrument, New Castle, USA) with a heating rate of 5°C/min under controlled atmosphere (N₂ and air).

2.2.2. Dielectric characterization

The complex permittivity ($\epsilon^* = \epsilon' - j\epsilon''$) of the samples was measured in the frequency range from 6 to 16 GHz, with an Agilent 85070E coaxial probe linked to an Agilent 8510C vector network analyzer (Keysight, Ratingen, Germany). This measure gives direct access to the real (ϵ') and imaginary (ϵ'') parts of the permittivity and the dielectric loss ($\tan\delta = \epsilon''/\epsilon'$) of the composites.

For this characterization, samples were cut to the dimensions 3x3x3 cm³ and polished, to obtain plane surfaces in contact with the probe and to limit the contribution of air gap during measurements that leads to underestimation of the dielectric properties. In order to minimize measurement uncertainty, 8 measures were carried out for each sample; a mean value is calculated and presented here.

2.2.3. Electromagnetic absorption simulation

In order to estimate the absorption performance of our composite, the simulation of reflectivity (R), see Supplementary File 1, using CST Microwave Studio software, was done in the normal incident of EM wave on the sample backed by a perfect electric conductor (PEC). The reflectivity was simulated using the dielectric properties of the composite material with the following equations [6,21]:

$$R \text{ (dB)} = 20 \log \left| \frac{Z_{in}-1}{Z_{in}+1} \right| \quad \text{Eq. 1}$$

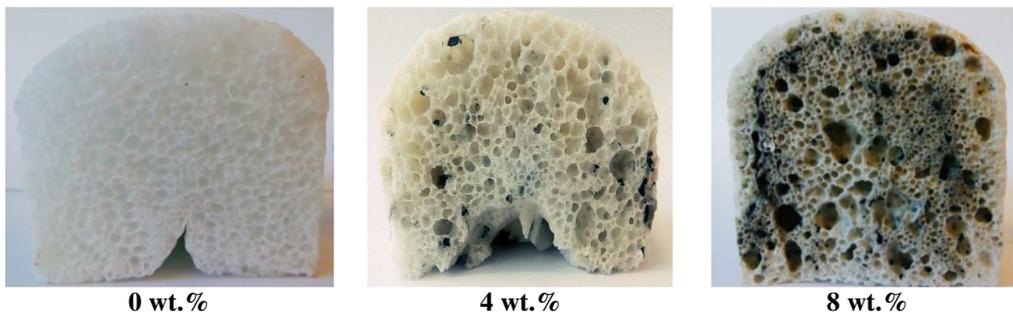
$$Z_{in} \text{ (\Omega)} = Z_0 \sqrt{\mu_r/\epsilon_r} \tanh \left((j2\pi f d/c) \cdot (\sqrt{\mu_r \epsilon_r}) \right) \quad \text{Eq. 2}$$

Here, Z_{in} is the normalized input impedance, Z_0 is the impedance of air, c is the light velocity in vacuum, f is the microwave frequency, d is the thickness of the absorber, ϵ_r and μ_r are the complex permittivity and permeability of samples, respectively. In this case, μ_r could be taken as 1 due to the non-magnetic aspect of our composites.

3. Results

3.1. Structure and morphology of glass foams

Fig. 1 shows the pictures of the foams elaborated with different contents of tire granulates. The figure shows, that the unloaded sample (0 wt.%) can be considered homogeneous. In fact, a weak gradient of porosity dimensions should be noted between the bottom and the top of this sample. This behavior, often observed in the literature [47,48], can be explained by the foaming which only occurs in uniaxial direction. For the samples loaded with tire granulates, a heterogeneity in structure (size and distribution of the porosity) has to be noted with pores ranging from 1 mm to 5 mm in diameter (Fig. 1). In addition, a non-homogeneous distribution of the resulting calcined/carbonaceous load in these composites can also be observed. This phenomenon is clearly visible on the sample loaded with the lowest percentage of tires (4wt.%). This inhomogeneity is due to the initial distribution of the tires load in the mixture, before the heat treatment, due to a difference in dimensions between the particles of the cullet powder (micrometric dimension) and those of the tire granulates (millimetric dimension).



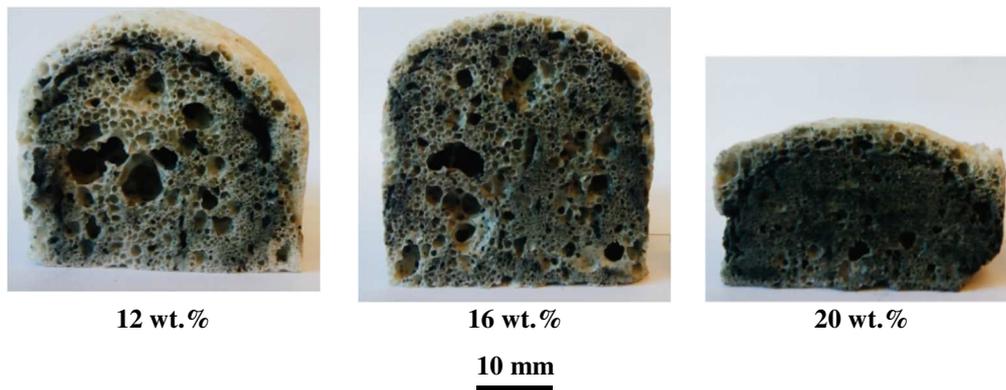


Fig. 1. Visual aspects of glass foams with different aggregate contents of tire waste.

For the most loaded sample (20 wt.%), a higher density (0.43 g.cm^{-3}) can be observed. This is probably due to excessive outgassing during the heat treatment induced by the high percentage of the tire load. This leads to the collapse of the gas bubbles produced during the heating process, as already observed in the literature when an excessive amount of foaming agent is used for the preparation of the foam [6,32].

Fig. 2.a shows the SEM micrograph of a glass foam prepared with 12 wt.% of tire granulates. As revealed by its density value (0.27 g.cm^{-3}), this sample presents a significant porosity with pores diameter ranging from a few tens of microns to a few millimeters. One can see thick cell walls, that limit the millimetric pores interconnectivity, and which contain micro-pores. Fig. 2.b shows an area where the tire load can be detected in the glass foam structure. This observation is confirmed by a localized EDS analysis (Supplementary Data 2) which showed that this spot composition is 99 at.% of carbon, whereas the analysis of the unloaded zone is consistent of that of a soda-lime glass.

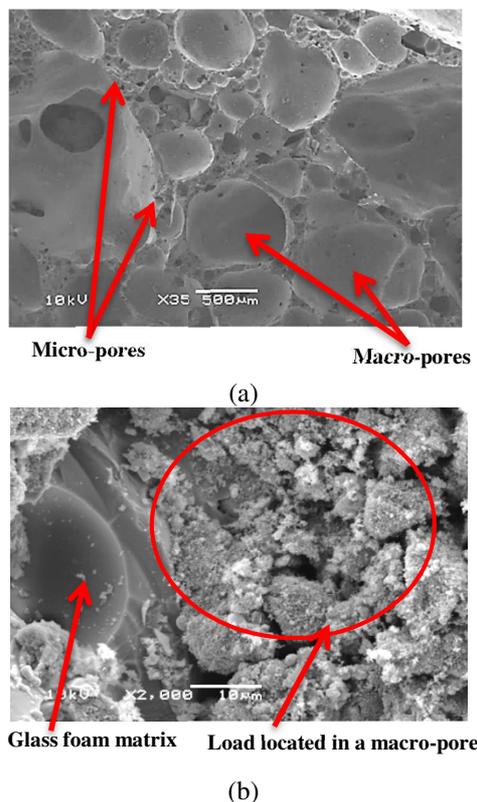


Fig. 2. Scanning electron microscope images at different magnification of (a) a foam matrix and (b) tire load in foam composite loaded with 12 wt.% of tires.

The results of the TGA analysis of tire aggregates, made under nitrogen and air, between room temperature and 800°C are shown in Fig. 3. These results show a significant weight loss in both tested atmospheres (N₂ and O₂) starting at 200°C. Tires analyzed under nitrogen or oxidative atmosphere present a main decomposition step in the range [300°C-450°C] associated with a weight percent decomposition around 60%. Tires analyzed under air atmosphere (Fig. 3) show a second decomposition step at higher temperature [450°C-600°C] leading to an almost total weight loss of 96 wt.% up to 800°C. The thermo-oxidative degradation behavior of such waste tires has already been studied by [49] and they showed that the increase in temperature causes the breaking of the sulfur-sulfur (S-S) and carbon-sulfur (C-S) bonds. This leads to a three-step decomposition process, the first weak weight loss refers to the decomposition of the oil and other organic polymeric additives (from 200°C to 300°C), a second weight loss, and the most important one in our case, corresponds to the

decomposition of rubber (inflection point at 390°C) and the last one (inflection point at 530°C), observed only under oxidizing atmosphere (Fig. 3), is relative to the oxidation of the carbon black which constitutes the main additive used in the tire formulation. That way, the final mass loss difference observed between the samples treated under dinitrogen or air is in perfect accordance with the carbon black content in the tires which varies between 15 wt.% and 40 wt.%, depending on the nature of the tires [50,51].

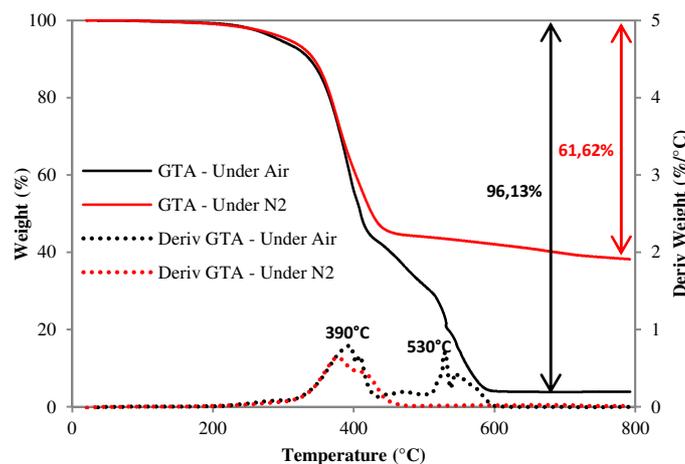


Fig. 3. Gravimetric thermal analysis (GTA) of tire aggregates under atmospheres of nitrogen and air.

As a consequence, the decomposition of the tire granulates induces a release of gas during the foaming process (concurrent with the calcium carbonate decomposition) which could explain the observed inhomogeneity in structure of the tire granulates / glass foam composites (Fig. 1). One can observe that, although prepared at 800°C under air for 30 minutes, the main part of the carbon black load resists oxidation. The vitreous glass matrix appears to prevent the C-containing load to react with atmosphere. Indeed, a white airtight millimeter-thick top skin can especially be observed on the most loaded tire granulates / glass foam composites (Fig. 1).

Fig. 4(a) presents the visual aspect of the foam loaded with 12 wt.% of tires coated with cement. It shows first that, and as expected, the foaming process has not been altered by

the degradation of the tire granulates. Tire granulates, embedded in the refractory ceramic paste, have clearly been protected leading to an homogeneous structure.

Fig. 4.b and Fig. 4.c show, respectively, the picture and the microscope photo of foam loaded with 12 wt.% of pre-calcined tires. This calcined tire based composite has an homogeneous structure **in the center** with a well dispersed carbon load Fig. 4.c. The pre-calcination step at 500°C **under air atmosphere**, according to the TGA results, converts the tire granulate into carbon black powder. Cleaned from this rubber fraction, the C-containing load does not interfere with the foaming process anymore. Moreover, the composite foam skin presents a bleach color corresponding to the glass, which confirms that the top vitreous matrix protects the inner carbon load from oxidation.

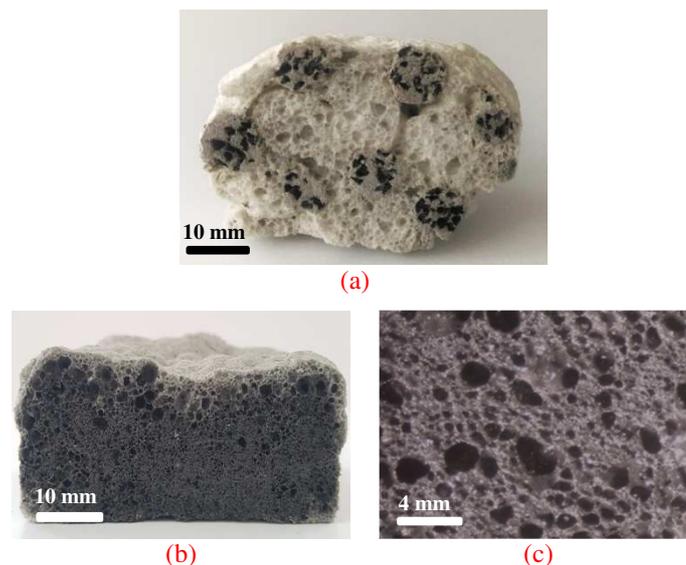
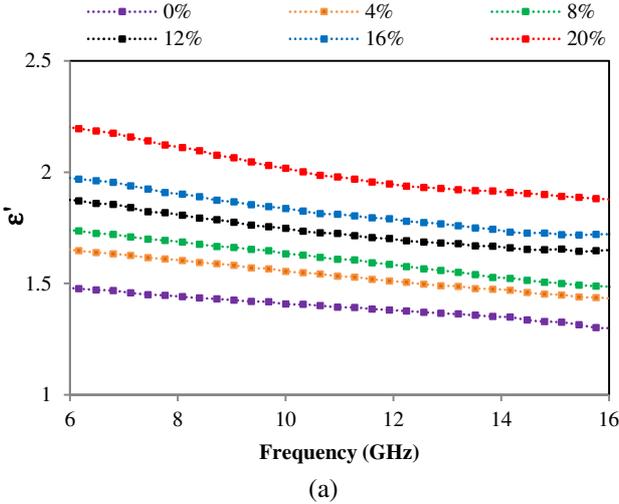


Fig. 4. (a) Picture of foam glass loaded with coated tire granulates (b) picture and (c) microscope photo of foam loaded with 12 wt.% of calcined tires (4.8 wt.% of carbon).

3.2. Dielectric characterization

It should be recalled that the necessary dielectric properties needed for microwave absorbers are, above all, a low **real part of the permittivity** (as close as possible to that of the air) in order to lower the reflection at the absorber-air interface [52] and high dielectric losses, higher than 0.2, in order to absorb EM wave inside the material [53].

Fig. 5 shows the variation of the real part of the permittivity ϵ' (Fig. 5.a) and the dielectric losses $\tan\delta$ (Fig. 5.b) of our glass foam samples loaded with different amounts of tire granulates as a function of frequency. These figures clearly show an increase in the dielectric properties with the percentage of tire load. For these composites, the real part of the permittivity, at 10 GHz, is 1.6 and 2.0 for 8 wt.% and 20 wt.% of tire loads, respectively. The dielectric losses are 0.14 and 0.17, respectively, for the same composites. These properties are higher than those of the unloaded glass sample which has an ϵ' and $\tan\delta$ of 1.4 and 0.11, respectively. Supplementary Data 3 presents the evolution of the dielectric permittivity and loss, at 10 GHz, as a function of the weight percentage of tire granulates. These figures show a quasi linear evolution of the dielectric properties as a function of load amount, as can be observed with other carbonaceous loaded absorber composites [54].



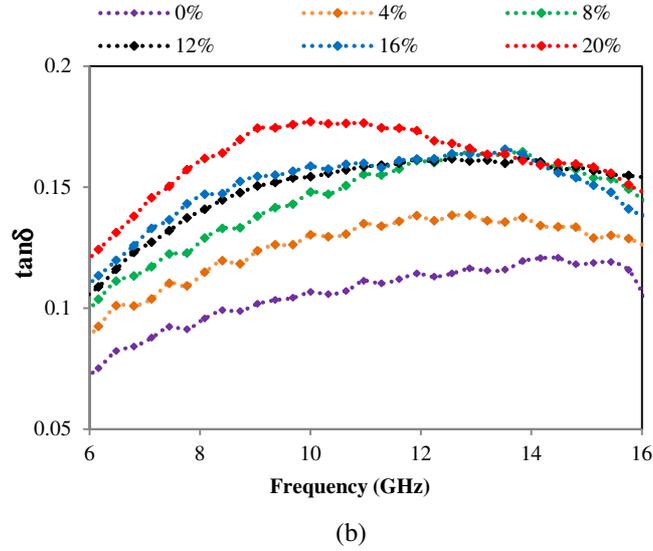


Fig. 5. (a) Real part of the permittivity ϵ' and (b) dielectric losses $\tan\delta$ of the glass foams loaded with tire granulates as a function of frequency.

As presented before, our loaded foam glasses present different apparent densities. In order to study the effect of the density on the dielectric properties of our samples, Fig. 6 shows the evolution of the real part of the permittivity ϵ' and the dielectric losses $\tan\delta$, at 10 GHz, as a function of density of all loaded foams. A quasi-linear increase can be observed on the curves; this confirms the relationship between dielectric properties and density as observed by Laur *et al.* [32]. Here, the values of the real part of the permittivity ϵ' of the foams vary between 1.4 and 2.0 and the dielectric loss $\tan\delta$ between 0.11 and 0.17 for densities of 0.27 and 0.43, respectively.

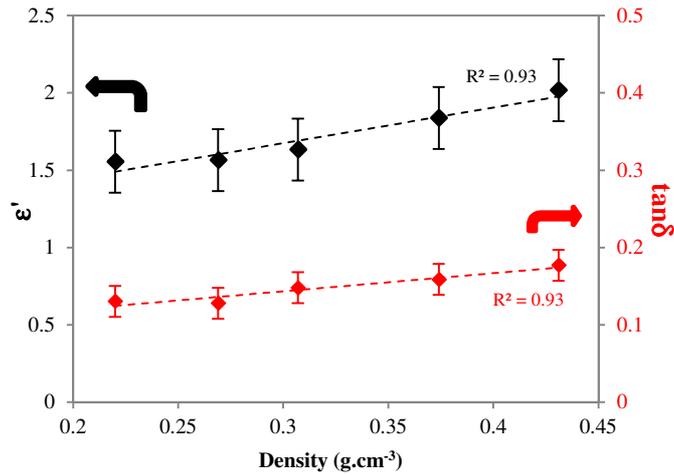
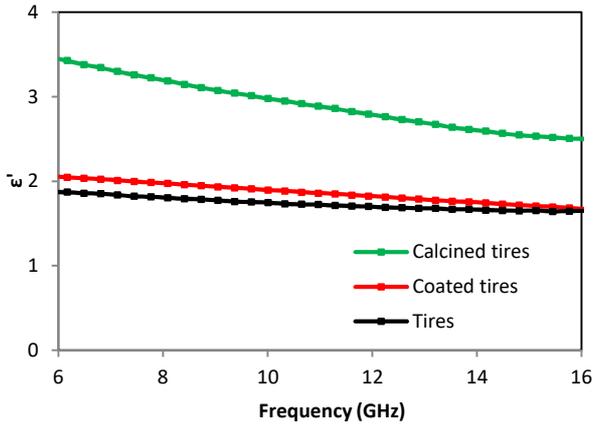


Fig. 6. Evolution of **real part of the permittivity ϵ'** and dielectric losses $\tan\delta$, at 10 GHz, of the foams loaded with tire granulates as a function of sample density.

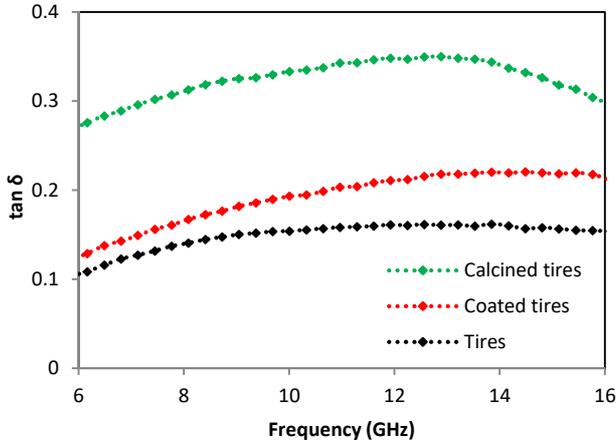
The obtained dielectric losses remain acceptable when compared with literature [34]. The later presents glass foams loaded with different weight percentages of carbon fibers. Here, samples present negative dielectric loss values; this is due to an artifact of the measurement of samples which present a very low losses. In [2], authors present glass foams loaded with graphite and zinc. **Sample produced with carbon graphite (2 wt.%) as the sole absorbing additive shows a dielectric loss ($\tan\delta$) close to 0.14 (@ 10 GHz). When 17 wt.% of zinc are added to the composite, dielectric loss increases to 0.38 (@ 10 GHz) but leads to a foam density of 1.77 g.cm⁻³ (not comparable with our highest density of 0.43 g.cm⁻³).**

Fig. 7 shows the variation of the **real part of the permittivity ϵ'** (**Fig. 7.a**) and the dielectric losses $\tan\delta$ (**Fig. 7.b**) of the foams loaded with 12 wt.% of tires (coated tires and calcined tires) as a function of frequency. These results are compared to those obtained previously with 12 wt.% of tire granulates (**Fig. 5.a** and **Fig. 5.b**). The percentage of 12 wt.% was chosen because it exhibited high losses while maintaining a relatively low permittivity (**Fig. 5**) and low density (Table. 2). The permittivity and the dielectric losses are slightly higher for the foam loaded with coated tires than in the foam loaded with tire granulates, whereas the percentage of charge is identical for the two composites. This difference can be

explained by the higher density of the foam made with coated granulates. The latter presents a density of 0.43 g.cm^{-3} , while for the same percentage of tires, the foam filled with uncoated granulates has a density of 0.27 g.cm^{-3} . This difference can be explained by the addition of the cement, which is a material with a high density (2 g.cm^{-3}) and also by less emission of gas due to the fact that granulates are encapsulated, and thus, protected by their cement coating.



(a)



(b)

Fig. 7. (a) Real part of the permittivity ϵ' and (b) dielectric losses $\tan\delta$ of the foams charged with 12 wt.% of tires, coated tires and calcined tires as a function of frequency.

The dielectric properties of the sample loaded with calcined tires are higher than those obtained for the glass foams loaded with tires and coated tires (Fig. 7). Dielectric losses reached a value of 0.33, at 10 GHz, more than twice the loss of the sample loaded with of tires. Even if a relatively high density of this composite (0.63 g.cm^{-3}) must be partly in the

origin of these high dielectric losses, these latter are high enough to leave us to think that the carbon resulting from the calcination of the tire granulates intervenes in the improvement of the dielectric properties and leads to losses needed for the absorbent application.

One can note here that the measurement technique (coaxial probe) used for dielectric characterization underestimates the dielectric properties (especially dielectric losses) of solid materials if they do not have a perfectly flat surface, which is more impacting in the case of foam materials [55]. However, our composite shows better dielectric loss/density compromise than most absorber materials made of loaded glass foams presented in literature [2,6]. The next part of this article is devoted to the simulation of the absorption performance of the calcined tires based composite. The latter presents the best homogeneity (structure and load distribution) than the two other studied composites (based on tire granulates or cement-coated tire granulates). Moreover, the chosen composite presents the higher dielectrics loss, compared to the two other composites; the dielectric losses are higher than the value of 0.2, between 6 and 16 GHz, loss needed for microwave absorption application [53].

3.3. Microwave absorption performance

In order to evaluate the absorption performance of the last composite (glass foam loaded with calcined tire granulates), simulations of reflectivity (called also reflection loss), are done, using CST Microwave Studio software and measured dielectric properties. Here, and in order to compare our results with those existing in the literature (for absorber foam materials), only properties in the X band frequency range were considered. Fig. 8 shows simulation results of the reflection coefficients, as a function of frequency, obtained with different absorber thickness (between 12 and 15 mm) of foam composite. A minimum reflectivity value (-29.62 dB) is obtained at 11.90 GHz with absorber thickness of 12 mm. Minimum reflectivity of -29.49 dB (@ 10.85 GHz), -28.59 dB (@ 9.98 GHz) and -27.41 dB (@ 9.22 GHz) are obtained with material thickness of 13 mm, 14 mm and 15 mm,

respectively. These reflectivities are accompanied by a large absorption band-width ($R < -10$ dB) in X-band frequency range of 2.02 GHz, 2.99 GHz, 2.90 GHz and 2.58 GHz for the same thickness. If we consider frequencies beyond the X-band range, a larger absorption bandwidth of 3.62 GHz can be obtained for the 12 mm thick sample (as shown in Supplementary Data 4).

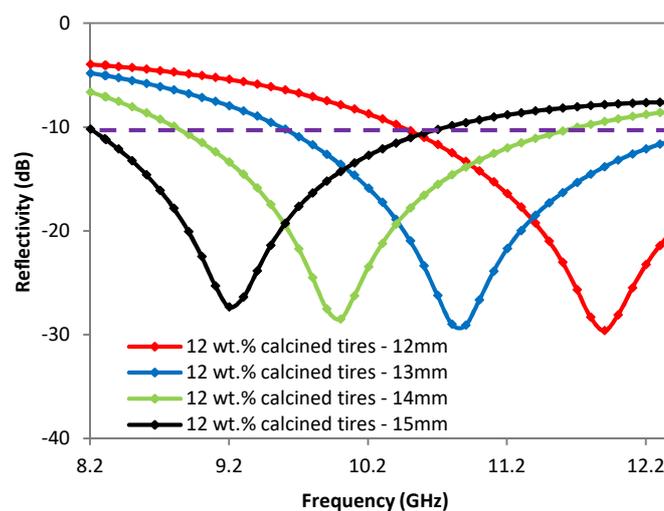


Fig. 8. Simulation of reflectivity, as a function of frequency, for calcined tire loaded composites with different thicknesses.

The skin depth, related to the glass foam sample loaded with 4.8 wt.% of carbon (obtained from calcined tire granulates), has been calculated using the measured dielectric properties (real part of the permittivity and dielectric losses) of the sample (Fig.7) and the calculation method [56] resumed in Supplementary Data 5. It should be remembered here that the simulation of the reflectivity involves the use of a conductor (metallic plate) on the back of the absorber, unlike the skin depth calculation.

The calculated skin depths range from 44.5 mm to 15.3 mm for frequency between 6 GHz and 16 GHz, respectively (Supplementary Data 5). Skin depth values associated with the minimum reflectivity (observed on Fig. 8) are 25.0 mm, 23.0 mm, 20.5 mm and 18.7 mm at frequencies of 9.22 GHz, 9.98 GHz, 10.85 GHz and 11.9 GHz, respectively. These values are in concordance with simulated values of reflectivity (Fig. 8) which present optimum

thicknesses, between 12 and 15 mm, for absorption in the X-band range. It should be noted that the skin depth and optimum thickness for low reflectivity cannot be compared directly. In fact, without the presence of the metallic plate (which induces a reflection of the wave and then, a re-absorption of the latter during its return trajectory) the absorption depth is inevitably higher than the simulated optimum thickness.

4. Discussion

Glass foam composites made from glass waste and used tire's waste have been studied for absorbent applications. Different microstructures, as a function of the carbon load (type and weight percentage), have been obtained associated with densities ranging from 0.20 to 0.63 g.cm⁻³. These values are in the range of the densities (between 0.1 and 2.6 g.cm⁻³) that can be found on silicate glass foam materials [57]. Several values of mechanical or thermal properties of these materials are available in the literature. For example, the compressive strength can vary between 1 and 4 MPa for foams with densities of 0.27 g.cm⁻³ and 0.876 g.cm⁻³, respectively [58,59]. In addition, excellent thermal and acoustic insulation are often associated with these glass foam related to their porous structure. As expected, the thermal conductivity increases as the foam density increases; for example, thermal conductivity value shifts from 50 to 80 mW.m⁻¹.K⁻¹ when the glass foam density increases from 0.21 to 0.38 g.cm⁻³ [60]. Concerning acoustic properties, D'Amore *et al* [30] show that glass foam presenting a density around 0.2 g.cm⁻³ presents sound absorption coefficients higher to that of rock wool. It is for these different (mechanical, thermal and acoustic) properties that these materials are good candidates for insulation material in building applications. Our glass foam composites made from glass waste and used tire's waste have been studied for absorbent applications to add electromagnetic protection to their potential scope keeping in mind that

their density should stay in the range [0.1 – 0.7 g.cm⁻³] to keep light weight and insulating properties.

Dielectric characterization shows that the targeted high dielectric loss ($\tan\delta > 0.2$ @ 10 GHz) can be obtained using low weight percentage of calcined crumb rubber. There are very few studies dealing with microwave absorption properties of C-containing inorganic foams. For example, Chen et al. [2] reported on glass foam reinforced with 2 wt.% of commercial graphite and, possibly, a variable amount of metallic Zinc powder as a co-absorber. Without Zinc, their resulting C-reinforced foam presents a density of 0.41 g.cm⁻³ and dielectric loss close to 0.14 (@ 10 GHz). The presence of Zinc (up to 17 wt.%) helps to increase the dielectric loss to 0.38 (@ 10 GHz) but drastically reduces the foaming process leading to high density glass foams (1.77 g.cm⁻³). It should be noted here that our low density (0.63 g.cm⁻³) and low cost composite, only containing 4.8 wt.% of carbon (no metallic co-additive load) resulting from tire calcination, presents efficient dielectric losses ($\tan\delta = 0.33$ @ 10 GHz). As shown in Fig. 6 and in our previous studies [32,33], foam density greatly influences the dielectric losses and a trade-off between high losses and lightness must be considered.

In the X band frequency range, our electromagnetic simulations, based on the data extracted from the previous dielectric results, reveal that the absorption performance of our calcined tire loaded foam glass met expectations. Literature data on C-reinforced foam glass [2] reports a reflectivity higher than -1 dB (in the X Band) with composite of 2.2 mm thickness, loaded with 2 wt.% of graphite and presenting a density of 0.41 g.cm⁻³. As previously explained, the use of a metallic co-additive could help to improve the absorption performance at the expense of the density. This way, a reflectivity of -15.6 dB (@ 12 GHz) has been obtained, on a dense foam glass composite (1.77 g.cm⁻³) prepared with a dual load (2 wt.% of graphite and 17 wt.% of Zinc) and with the same thickness (2.2 mm).

Moreover, comparable results can be found on glass foams loaded with carbon fibers having a fiber length of 3 mm [61]. In this study, absorbers with compositions between 2 to 10 wt.% of fibers are produced and show densities ranging from 0.21 to 0.52 g.cm⁻³. The lowest reflectivity (highest absorption) of -28 dB has been estimated on a 15 mm thick absorber loaded with 2 wt.% of C-fiber. This performance is directly comparable with our result obtained on a 12 mm thick foam glass prepared with calcined tire's waste.

To our knowledge of glass foam absorber literature, the only result which presents lower reflectivity than that presented here for our material is that given by Laur *et al.* [32]. In the latter, low reflection loss values, between -30 dB and -70 dB in the X-band frequency range, are shown for foam absorbers with 70 mm length. This performance is induced by the specific length and the pyramidal geometry of this absorber, which reduce the impedance mismatch at air / material interface and thus, the reflection loss of this absorber [32].

Furthermore, the porous structure of these materials can provide additional absorption of electromagnetic waves, especially in high frequencies, than that estimated from their dielectric properties. Indeed, the size of the porosities observed on our materials, and also on glass foam materials of the literature [2,6,32,33], is of the order of magnitude of a millimeter, or even several millimeters (here maximum 5 mm). This last dimension corresponds to the quarter-wavelength for frequencies above 9 GHz. In this case, and as the properties of material are different than those of air (constituting the porosities), a multi-reflection of the waves can occur at this high frequencies as proposed in [62,63]. It should be noted here that the quarter-wavelength decreases when the frequency increases and here reaches the value of 3 mm for the maximum studied frequency of 16 GHz, as shown in supplementary Data 6.

As a conclusion, our work shows that the use of an elastomeric material (tires) that contains a lot of carbon, as load in a dielectric matrix, is not sufficient to increase the dielectric losses of the composite. This is especially true if tires are used as granulates;

therefore, they do not make it possible to absorb the EM waves. When the foam composites are loaded with calcined tires, the dielectric loss of composites is then increased and effective absorber can be obtained, as showed by the simulation of reflection coefficients of 12 mm composite absorber which presents a minimum of reflectivity of -29.62 dB at 11.90 GHz.

5. Conclusion

Organic-inorganic composite foam glasses are manufactured by combining solid industrial wastes (glass cullet and crumb rubber from scrap tires) and an abundant raw material (limestone). The original goal of this study is to produce a low-cost microwave (MW) absorbing material that could be used in building industry. Carbon-containing crumb rubber extracted from scrap tires has been used as microwave absorber in powder or granular form (raw, calcined or embedded in a refractory aggregate).

Glass foams loaded with different percentages of tire granulates (between 0 and 20 wt.%) were elaborated and characterized. These composites showed a linear evolution of the dielectric properties (permittivity and dielectric losses) as a function of the percentage of charge and foam density. Nevertheless, the dielectric losses of these composites remain lower (ex: 0.12 at 10 GHz for a foam loaded with 12 wt.% that has a density of 0.27 g.cm^{-3}) than that of absorbents in the literature. These composites have also shown an inhomogeneity of the structure (porosity) due to degassing (degradation) of the rubber charge during the heat treatment necessary for the preparation of these composites. In order to avoid this degassing, and hence the inhomogeneity of the foams, a process of encapsulating the load in a refractory structure, supporting temperatures higher than that of the heat treatment, has been attempted. The obtained sample showed better structure's homogeneity but a relatively high density. The latter is probably responsible for the increase of the dielectric properties of this material (dielectric losses of 0.19 at 10 GHz associated with a density of 0.43 g.cm^{-3}). Finally,

carbonization (by heat treatment at 500°C **under air atmosphere**) of the load (tire's waste) was tested; the carbonaceous powder resulting from this treatment was used as a load to produce a composite based on glass foam. By this method, the produced absorbent composite, with the equivalent of 12 wt.% of tire's load, has a very good homogeneity in structure (porosity distribution) and also in load distribution. This sample showed an increase in dielectric losses ($\tan \delta = 0.33$ at 10 GHz) compared to that of the 12 wt.% non-carbonized tires sample, but presents also a high density (0.63 g.cm^{-3}). However, our composites show better dielectric loss/density compromise than most absorber materials made of loaded glass foams presented in the literature. The simulated reflectivity of absorbers, with thickness between 12 mm and 15 mm, shows very promising results compared to the reported works. Here, a low reflection of -29.62 dB (at 11.90 GHz) and a large absorption bandwidth (3.62 GHz) are obtained for calcined tire's loaded foam composite with 12 mm thickness. By this way, absorber material composed of waste glass and waste of used tires can be achieved.

Acknowledgments

The authors would like to thank F. Benhaoua for her technical assistance. This work is supported by the Algerian Ministry of Higher Education and Scientific Research (MESRS) and the Union European through the European Regional Development Fund (ERDF), the Ministry of Higher Education and Research, the Brittany Region, the Department of Côtes d'Armor and Saint-Brieuc Armor Agglomération, through the CPER 2015-2020 project "MATECOM".

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