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# Ultra-porous and lightweight microwave absorber based on epoxy foam loaded with long carbon fibers

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**Abstract:** A simple methodology to fabricate ultra-porous and lightweight epoxy foam composites loaded with carbon fibers is presented. These materials are proposed for **microwave absorbing application**. **The elaborated composite density** can be controlled via proper proportioning of carbon fiber/solvent/surfactant/epoxy components constituting the absorber composites. The influence of the weight percentage of **these** different components, on the composite structure, is studied. **The density of the composites can be reduced to  $0.05\text{g}\cdot\text{cm}^{-3}$  while the density of the neat epoxy foam is about  $0.12\text{g}\cdot\text{cm}^{-3}$** . Dielectric characterization of composites elaborated with low weight percentages of carbon fibers (from 0.0125wt.% to 0.075wt.%) is conducted using free space method. The simulation of a multilayer absorber is conducted and predicts a promising absorption performance (reflection coefficient  $<-10\text{dB}$ ) for the wide studied frequency band; this result was confirmed by the measurement of a multilayer absorber prototype. **Finally**, these performances are compared to commercial absorber materials having same dimensions.

**Keywords:** *lightweight epoxy foam; carbon fibers; composite absorber; dielectric properties; microwave absorber.*

## 1. Introduction

Recently, a great progress has been reached in the development of new microwave absorber materials that meet the new requirements of different applications from military domain (for example stealth) to civil domains (interference shielding in electronic devices, anechoic chambers...) [1]. Whatever the application for which the absorber is intended, the challenge is to develop new thin and/or lightweight and broadband microwave absorber presenting high absorption performance. The composite materials, and specifically the porous composites, are among the top absorber materials that gained much interest in the EM domain thanks to their light weight, low cost, corrosion resistance and broad absorption bandwidth [2–6]. For example, these composite absorber materials are used in the anechoic chambers; **the** latter are covered by lightweight multilayers **or** pyramidal **absorbers**. **These** two types of absorbers present first, a large dimensions (several tens of centimeters in height) to insure absorption in large frequency wide range, and second, a composition or geometry gradient **that** allows **a high absorption performance, thanks** to a perfect impedance **matching** [7]. Frequently, **lightweight multilayers and pyramidal absorbers** are made of polyurethane foams which bring low density thanks to their porous structures. They are loaded with nanometric conductive carbon black particles to provide the absorption property. However, these absorbers present also some disadvantages, on one hand, their high flexibility limits the reproducibility machining of the absorber geometry, **leading to** a non-reproducibility absorption performance, **on the** other hand, the loss of the volatile nanometric charges located inside the open porosities leads to a bad ageing of the absorbers and risks to human health. In this regard, the study of a new absorber material based on a rigid matrix (such as epoxy foam) associated with a non-volatile absorbing load (such as long carbon fibers) appears as a promising alternative to replace these commercial absorbers based on

PU foam loaded with carbon nanoparticles [8]. Indeed, the higher mechanical property of epoxy foam [9] compensates the flexibility of PU foam [10], **further it** allows the addition of the filler to the resin before the foaming process **as well as making** it possible to trap the filler inside it [11]. However, the epoxy foams show a higher density than that of PU foam composites [12–14] which further limits their use in anechoic chambers, where voluminous absorbers are used to provide absorption at low frequencies (typically < 2 GHz). **Several studies have focused on reducing the density of epoxy foam [15] and epoxy resin [12,16]** based composites. Indeed, several foaming methods of composite materials are studied [17] **and** porous composites can be prepared from resins using mechanical [18–20], physical [21] and chemical blowing agents [22,23] or by processing techniques like foaming by injection or extrusion molding [24,25]. For example, microcellular epoxy foam was prepared using the CO<sub>2</sub> foaming injection of **the epoxy resin** and the density was reduced from 1.18 to 0.19 g·cm<sup>-3</sup> [12]. In another example, **the content** of the chemical foaming agent **was changed and** the density of the epoxy foam was reduced from 0.321 to 0.151 g·cm<sup>-3</sup>, as shown by Chang *et al.* [15]. However, this density remains higher than the density of polyurethane foams, usually used in anechoic chambers, which is around 0.07 g·cm<sup>-3</sup> [26].

Otherwise, in order to ensure maximum absorption of the electromagnetic waves inside an absorber, the reflection of these waves at the interface of this absorber must be minimized [27–29]; in other words, the waves must be introduced into the absorber before it can be subsequently attenuated inside it. For this **reason**, an impedance transition, also called impedance matching, between the material and air is achieved by adjusting either the design of the material (**by pyramidal geometry for example**) or its composition (gradient of composition) [6,30]. When a planar absorber is needed, the multilayer absorbers, with a gradient of composition, can be used. For this multilayer, the impedance **matching** should be **maximized** at the air/absorber interface

and also between the different layers which allowed the wave to propagate deeper into the material, in which, it will be gradually dissipated, and so, absorbed [31]. Therefore, a low real part of the permittivity (as close as possible to that of the air,  $\epsilon' = 1$ ) is needed for the first layer of absorber in order to lower the reflection at the absorber-air interface, and gradual dielectric properties are needed for the other layers [32]. One can note that the low real part of permittivity of foam composite based absorbers is mainly due to the presence of porosities (therefore air). **So, the increase of the porosities in these materials decreases their densities, as well as, their dielectric properties. Consequently, those provide** a light-weight material with a better impedance matching at the air/absorber interface.

An EM absorber made from epoxy foam filled with carbon fibers is developed by our team to be used in the anechoic chamber [8]. Previous works have shown a very interesting absorption performance of pyramidal absorber based on this novel material, compared to PU based commercial absorber with **the** same geometry and dimensions [11]. Recently, in [33], we have shown that the use of long carbon fibers makes it possible to increase the dielectric losses of these composites while keeping low permittivities. The use of these long fibers is therefore promising for the production of planar multilayer absorbers for which the impedance matching can be obtained by the use of low permittivities, close to that of air. However, the dispersion of the long carbon fibers **seems** to be difficult, which negatively affects the homogeneity of the material, and thus **the** reproducibility of absorption properties. It is shown that this inhomogeneity increases when weight percentage of carbon fibers increases [8,33]. Different studies have been conducted to optimize the dispersion of these long CFs [33–38], only the sonification method and the use of chemical surfactants have shown a relatively good dispersion without damaging of the CFs length [33,34]. Other studies showed an interest in the chemical dispersants; they compared the

performance of multiple surfactants on the carbonaceous particle dispersion [39,40], for example in the study of Z. Sun *et al.* [39] the SDS (Sodium dodecyl sulfate) show a better dispersion compared with other surfactants such as LDS (lithium dodecyl sulfate), SDBS (sodium dodecylbenzene sulfonate), TTAB (tetradecyl trimethyl ammonium bromide) and SC (sodium cholate). In our case, the use of SDS and ultrasound [33] made it possible to disperse the long fibers without altering their length.

The purpose of this article is to jointly benefit from long carbon **fibers and** the reduction in density of epoxy foam, in order to guarantee low permittivity associated with high losses of these composites, with the aim of their use for elaboration of planar multilayer absorbers.

In this work, we present a simple process to fabricate ultra-porous **epoxy** foam filled with low percentages of long carbon fibers (12 mm) which can be used for the achievement of a multilayer absorber. First, the elaboration method, using acetone as solvent and SDS as a chemical dispersant, is described. Then, the influence of acetone, CFs and surfactant (SDS) on the morphology, the density and mechanical property is studied. Furthermore, dielectric characterization of epoxy foams filled with different CFs concentrations is conducted for the purpose of their use for a planar multilayer absorber **prototype**. The simulated and measured absorption performances of this prototype are finally presented; they are compared to the one of commercially available absorber.

## **2. Materials and Methods**

### *2.1. Light-weight absorber composite elaboration*

The CFs filled epoxy foam composites were prepared using PB170 epoxy resin and DM02 hardener (foaming agent) purchased from Sicomin. Carbon fibers, of 7  $\mu\text{m}$  diameter and 12 mm

length from Apply Carbon, were used as the absorbing filler. SDS surfactant (from SIGMA-ALDRICH) and Ultrasounds (Sonics Materials VCX-750-220) were used for the dispersion of carbon fibers in acetone. Different weight percentages of acetone (between 0 and 20 wt.%), of carbon fibers (from 0 to 0.8 wt.% for the structure study and from 0.0125 to 0.075 wt.% for the dielectric study) and of SDS (respecting SDS : CFs ratio of 0.5 : 1) were used in this work. Fig. 1 shows the different steps of the elaboration method of the composites. The chosen weight percentages of CFs and SDS were firstly dispersed in 60 ml of acetone with the aid of ultrasounds for 30 minutes. The used parameters for the sonication were: 10sec/10sec of On/Off pulses of 300 Watts power. Then, this CFs/SDS/acetone solution was added to the epoxy resin (10 g) and placed in the oven (50°C) in order to evaporate the acetone excess and so leave only the desired acetone percentage. The quantity of acetone was controlled thanks to the weighing of the resin/CFs/SDS/acetone mixture at different times during the evaporation step. Finally, the hardener (3.6 g) was added to the mixture with a 10 : 3.6 epoxy resin : hardener ratio. The final mixture remains at ambient temperature within 6 hours for the foaming and the polymerization steps and then it was cured at 60°C for at least 6 hours to achieve the polymerization of the epoxy foam composites. After curing, samples were cut to desired dimensions needed for the different characterizations.

## *2.2. Structural characterizations*

The structure of the samples was observed by an optical microscope Leica DM 2500M and a scanning electronic microscope (SEM) JEOL 5600. The composite densities were deduced from the weight and volume of samples (of 3x3x3 cm<sup>3</sup> dimensions), according to the following

formula:  $\rho = \frac{m}{V}$ . A measurement uncertainty of  $\pm 0.01 \text{ g}\cdot\text{cm}^{-3}$  was estimated and taken into account for the presented results.

### *2.3. Viscosity measurements*

The viscosity of the epoxy/acetone/SDS mixtures was measured by means of a Brookfield viscometer (DVII+ PRO from Labomat), at 23°C. A standard spindle LV3 model and a rotation speed between 10 and 100 rpm were used. The spindle model and the rotation speed were adapted according to the range of the measured viscosity, by respecting a percentage of torque between 10 and 100 %. A measurement uncertainty of 10 % is taken into account for this measurement technique.

### *2.4. Mechanical characterization*

The **flexural** modulus of epoxy foam composites was determined, at room temperature, according to ISO 178 **standard**. Foams were cut into rectangular test specimens with dimension 80x10x5 mm<sup>3</sup>. The bending test support span using in the 3-point bending test was 64 mm and the crosshead displacement speed was 2 mm/min. The tests were conducted using the universal testing machine (MTS system). At least 4 specimens were tested for each type of composite and the averages values were calculated.

### *2.5. Dielectric characterization*

The dielectric characterization of the composites was carried out, **in an anechoic chamber**, using a quasi-monostatic configuration with two horn antennas operating in the 2-18 GHz range. The target (samples of 15x15x6 cm<sup>3</sup>) **was placed at a 2.6 m** distance from the antennas, ensuring the far field condition, as explained in the work of *Pometcu et al.* [41]. The complex permittivity



( $\epsilon^* = \epsilon' - j\epsilon''$ ) of **the material** is extracted from the measured reflection coefficients using *Fenner et al.* method [42]; dielectric losses, which indicate the overall microwave attenuation inside the composite material, are defined by  $\tan \delta = \frac{\epsilon''}{\epsilon'}$ .

## 2.6. Electromagnetic absorption performance: Simulation and measurement

In the case of multilayer absorbers consisted of  $i$  layers, the reflection **coefficient** calculated according to the transmission-line theory [43], is given by:

$$R_L = 20 \lg \left| \frac{Z_i - \eta_0}{Z_i + \eta_0} \right| \quad (1)$$

where the input impedance to each  $i$ -th layer is described by:

$$Z_i = \eta_i \frac{Z_{i-1} + \eta_i \tanh(\gamma_i d_i)}{\eta_i + Z_{i-1} \tanh(\gamma_i d_i)} \quad (2)$$

with  $Z_i$  is the input impedance at the interface of the  $i$ th layer,  $\eta_i = \eta_0 \sqrt{\frac{\mu_i}{\epsilon_i}}$ ,  $\gamma_i = i2\pi f \sqrt{\mu_i \epsilon_i / c}$ ,  $\eta_0$  is the characteristic impedance of the free space (equal to 377 Ohms); and  $\epsilon_i$  and  $\mu_i$  are the relative complex permittivity and permeability of  $i$ th layer. In our case, the permeability is considered **equal to 1 because of the pure dielectric behavior of our materials.**

**The simulation** of reflection coefficient of the multilayer absorber was **performed** using CST Microwave Studio software. **The measured dielectric properties (between 2 and 18 GHz) and dimensions of commercial absorber, APM12 from Siepel [26], were used. The absorber** was backed by a perfect electric conductor (PEC) **as resumed** in the work of *Lamri et al.* [44]. The measurement of the absorption performance of the achieved prototype was done in the anechoic chamber using the same configuration as that used for the dielectric characterization [8].

## 3. Results and discussions

### 3.1. Morphology and density of epoxy foam composites

#### 3.1.1. Influence of acetone amount

In order to observe the influence of acetone on the density of composites, several unloaded epoxy foams (without carbon fibers) were elaborated using different weight percentages (between 0 and 20 wt.%) of acetone. Fig. 2 shows the optical micrographs and scanning electron microscopy (SEM) images of the elaborated epoxy foam samples. These figures show that the size of cells increases significantly with the **increase of the** acetone content. For example, the average cell size increases from 0.5 mm for the epoxy foam without acetone (Fig. 2.a) to larger than 6 mm for the sample with 15 **wt.%** of acetone (Fig. 2.f).

Fig. S1 presents the photos of the whole elaborated samples. These photos show an increase of the sample volume, as a function of the acetone content, which is consistent with optical and SEM images (Fig. 2); this means a decrease of sample densities. The density of the different samples was measured **as well**, the values are summarized in the Table 1, and their evolution, as a function of the acetone concentration, is represented in Fig. S2. The neat epoxy foam shows a density of 0.12 **g·cm<sup>-3</sup>**; **the** addition of acetone decreases this density from 0.12 **g·cm<sup>-3</sup>** to 0.05 **g·cm<sup>-3</sup>** when the amount of acetone increases from 0 wt.% to 15 wt.%, respectively. This is due, firstly, to the evaporation of acetone, during the curing step under 60°C, **that generates** an increase of the amount of gas during the foaming process, and so, a decrease of epoxy foam density. **Secondly**, the addition of acetone to the epoxy resin **probably** induces a decrease of its viscosity [45]; this less viscous resin expands faster, exhibiting a larger cells **and so, a lower foam** density [15].

**The viscosity of the different epoxy/acetone mixtures was measured (Fig. S3) at room temperature (23°C); the neat epoxy resin (without any acetone addition) shows a viscosity**

value around 9220 mpa.s. One can note that the addition of small amount of acetone can significantly decrease the viscosity of the resin as shown in Fig. S3. This viscosity decreases by up to 55 % (4030 mpa.s) and up to 92 % (745 mpa.s) when 2 wt.% and 8 wt.% of acetone are added to resin, respectively. Thus, it seems that when the viscosity of the resin mixture decreases, the cell size of the final sample increases. The relation between the viscosity of polymeric foam and their cell size, related to the expansion of the micro air bubbles inside the resin, was explained according to the Rayleigh–Plesset equation (3) and was done in [46]:

$$\rho_l \left( R\ddot{R} + \frac{3}{2}\dot{R}^2 \right) = -\frac{2\sigma}{R} + \frac{P_i R_i^3}{R^3} - 4\mu \frac{\dot{R}}{R} \quad (3)$$

where  $R$  is the radius of the bubble,  $\rho_l$  is the density of the resin,  $\mu$  is its viscosity,  $\sigma$  is the surface tension,  $P_i$  and  $R_i$  are the initial pressure of the gas inside the bubble and the initial radius of the bubble, respectively.

However, it's worth noting that when the amount of acetone exceeds 15 wt.%, the density increases as it can be observed on sample made with 20 wt.% of acetone which presents a density of 0.23 g·cm<sup>-3</sup> (Table 1). This increase was induced by the deterioration of the epoxy foam structure (Fig. S1 and S4) which was probably related to the very low viscosity of this acetone/epoxy resin mixture. Indeed, Fig. S3 shows that after the addition of 20 wt.% of acetone, the viscosity decreases to 214 mpa.s (see inset zoom in Fig. S3); it corresponds to only 2 % of the initial epoxy resin viscosity. This excessive addition of acetone to the resin surely weakens the inter-chain interaction (cross linking process) in the polymeric structure as shown by [45,47].

Finally, considering the appearance of samples with different amounts of acetone (Fig. 2 and Fig. S1), and their density values (Table 1), we conclude that 8 wt.% addition of acetone presents

a better compromise between homogeneity and low density ( $0.07 \text{ g}\cdot\text{cm}^{-3}$ ) of samples. This chosen density is **identical to** the one of a commercial absorber based on polyurethane foam currently used in anechoic chambers. Consequently, **for the next parts of this article, 8 wt.%** of acetone is adopted as the best fixed amount to be added to make lightweight epoxy foams filled with different carbon fibers concentrations.

### *3.1.2. Influence of CFs rate*

For the targeted absorption application, CFs are used to insert dielectric losses to epoxy foams. In this part, the influence of adding CFs to the lightweight epoxy foam and the effect of their amount on the structure and the density of foam composites is presented. Samples with different CFs fillers (0, 0.1, 0.2, 0.4, 0.6 and 0.8 wt. %) were prepared. Here, relative high CFs loads were used to emphasize their influence on the foam density. The SDS content, used for the CFs dispersion in each sample, was calculated by respecting the **SDS : CFs ratio of 0.5 : 1**. A fixed amount of acetone of 8 wt.% was used for the elaboration of all these samples. Fig. 3 presents the photos of the different elaborated composites.

According to Fig. 3, it can be noticed that when the CFs concentration increases the foaming volume decreases, suggesting that the density of foams increases. The measured densities presented in Table 2 confirm **this density increase**. The addition of long CFs weighs down the mixture thus restricts the foaming process. This is probably due to the fact that long fibers, especially fiber agglomerates, prevent the expansion of gas bubbles. Indeed, fibers agglomerates are often observed when high **rates (wt.%)** of long fibers are used [8,33]. Moreover, the addition of fibers certainly increases the **apparent** viscosity of the fibers/resin mixture, which goes against the expansion of foaming resin.

Optical micrographs and SEM images of samples elaborated using different amounts of CFs are shown on Fig. 4. From **these images, one can note** that beyond 0.4 wt.% of CFs the shape of the cells is clearly distorted leading to inhomogeneous aspect of the sample by comparison with loads below 0.4 wt.% of CFs. This deterioration is clearer on sample filled with 0.8 wt.% of CFs (Fig. S5) on which the cells were deformed, and their shapes were flattened. Moreover, these **images (Fig 4)** confirm that the addition of fibers goes against the expansion of foaming resin and therefore the size of cells. This effect appears clearly on the sample loaded with 0.2 wt.% of CFs, the size of cells decreases slightly in comparison with the ones of neat epoxy foam. However, if we compare the epoxy foam filled with 0.1 wt.% of CFs with the neat epoxy foam (0 wt.%) in Fig. 3, we noticed that the foaming volume of the epoxy foam with 0.1 wt.% of CFs is slightly higher than the one of neat epoxy foam. The same appearance was noticed on the morphological images (Fig. 4); as we can see, the size of cells increases slightly by adding 0.1 wt.% of CFs compared to the neat resin epoxy. This appearance is in contradiction with our previous argument: when we add CFs the foaming volume decreases. In this case, the assumption of another effect **that** counterbalances the effect of adding fibers is **suggested**, such as the SDS used for the dispersion of these fibers. The influence of SDS **addition** will be investigated in the next part.

### *3.1.3. Influence of SDS*

The chemical surfactant SDS (sodium dodecyl sulfate) was used in this work for the CFs dispersion. **Fig. S6 shows photos of CFs/epoxy resin mixtures with 8 wt.% of acetone after spatula dispersion. CFs used for these mixtures were dispersed without SDS (Fig. S6.a) or with SDS (Fig. S6.b), during ultrasound dispersion step (see elaboration steps in Fig. 1). A more homogenous mixture was obtained when the SDS surfactant was used (Fig. S6.b).**

In the previous paragraph, we assumed that SDS particles, which remain on the dispersed CFs, may exhibit an effect on the epoxy foam structure. Here, the effect of SDS on the morphology and the density of epoxy foam is investigated; **to emphasize the influence of SDS, samples** with 0.005 g and 0.15 g of SDS were prepared. The 0.005 g content of SDS is the quantity of SDS relative to our standard elaboration of 0.075 wt.% of CFs loaded composite (this CFs amount is the higher one used for the elaboration of absorber composite). The second content of SDS (0.15 g) was used **in order to observe the effects** when very high amount of SDS is used. Two categories of samples were elaborated with **two** different acetone contents (0 and 8 wt.%) to investigate the influence of SDS in presence and absence of acetone in the **samples**; **Fig. 5** presents the pictures of the elaborated samples. These pictures show clearly the difference in volume between samples elaborated with and without acetone. **Nevertheless**, when we compare samples elaborated with the same acetone content, the difference in volume is very insignificant, even non-existent. Indeed, samples elaborated without acetone present practically the same aspect and volume, whatever the **amount of SDS**. Samples elaborated with 8 wt.% of acetone show a slight increase of the volume, as a function of SDS contents, predicting a decrease of the sample densities. The measured density values are summarized in Table 3. These results, and considering the measurement uncertainly, don't show any density modification due to SDS. Indeed, density values of 0.12  $\text{g}\cdot\text{cm}^{-3}$  and 0.07  $\text{g}\cdot\text{cm}^{-3}$  were obtained for all samples elaborated without and with acetone, respectively, whatever the SDS content.

**The effect of SDS addition on the viscosity of the epoxy/acetone/SDS mixture is also investigated. For this purpose, the viscosity of epoxy/acetone mixtures with various acetone percentage (from 0 to 20 %) and with 0.005 g or 0.15 g of SDS is measured. Fig. S7 present the viscosity values of the different mixtures at 23°C; these values are compared to the ones of epoxy/acetone mixtures (0 g SDS). It can be noticed that the addition of SDS don't have a**

**significant effect on the viscosity of the epoxy/acetone/SDS mixtures; only a small variation in viscosity of mixtures without acetone is observed. This variation is probably due to the measurement uncertainty of the viscometer (that is about 10 %).**

The morphological images of the different elaborated samples are presented in Fig. 6. Note that the two categories of images (Fig. 6.a1 – 6.c1 and Fig. 6.a2 – 6.c2) have two different scales. When the amount of SDS increases from 0 to 0.15 g, samples without acetone (Fig. 6.a1 – 6.c1) show no difference in cells size. However, samples with 8 wt.% of acetone (Fig. 6.a2 – 6.c2) show that the cells of epoxy foam containing 0.005 g of SDS are slightly **larger compared** to the ones without SDS. This increase of porosity size is more noticeable on samples made with 8 wt.% of acetone and 0.15 g of SDS. Indeed, the surfactant can facilitate the formation of cells through lowering the surface tension of the mixture as proposed in [15]. Moreover, it can be noticed that when the content of SDS increases in samples, large inhomogeneous cells were observed, simultaneously at the sides (Fig. S8.a – S8.b) and inside of samples (Fig. S8.c). This observation can be explained by the fact that the inhomogeneous distribution of SDS particles in the resin can increase locally the cells size during the foaming process, and so the coalescence phenomena.

In summary, our results show that the SDS can slightly alter the foaming process and the homogeneity (cells size distribution) of epoxy foams loaded CFs, especially when a large amount of SDS is used. **Accordingly**, it is recommended to minimize the amount of this surfactant in the epoxy foam composites.

### *3.2. Mechanical properties*

Samples with or without acetone, CFs and SDS are elaborated and compared in order to consider the effect of these different components on the mechanical properties of the achieved

composites. For this purpose, epoxy foams loaded with three different amounts of 12mm-CFs (0, 0.075 and 0.4 wt.%) are prepared with (8 wt.%) and without (0 wt.%) acetone. To investigate the influence of SDS, two samples of neat epoxy containing 0.15 g of SDS with and without 8 wt.% of acetone are also prepared. Table 4 summarizes the elaborated samples for this part of **study**; Fig. S9 shows photos of the samples cut for the mechanical characterization.

Fig. 7 shows the flexural modulus of different epoxy foam composites. The flexural modulus shows a noticeable decrease for epoxy foams elaborated with 8 wt.% of acetone compared to samples without acetone, owing to the decrease in densities of samples (Table 4). Samples without acetone show a flexural modulus around 35 MPa. Taking into consideration the measurement uncertainty, we didn't notice an important influence of SDS or CFs contents on the mechanical properties of epoxy foams. The flexural modulus decreased to around 4 MPa for samples made with 8 wt.% of acetone. If we compare the epoxy foams containing 8 wt.% of acetone, we don't notice an important effect of SDS even of the low CF loads (0.075 wt.%) on the mechanical properties. However, we notice that the high loads of CFs (0.4 wt.%) can reinforce the mechanical properties of the low weight epoxy foam to around 12 MPa.

It should be noted here that these mechanical properties seem low but are completely consistent with the densities of the presented materials. To our knowledge, our low densities are the lowest presented to date for rigid epoxy foams in literature [15]. Furthermore, the presented mechanical properties remain higher than those of commercial materials used for the anechoic chambers (flexible PU) [10]. **In** fact, the high mechanical properties are not required for the application in the anechoic chamber; **nevertheless**, it is important to take into account this mechanical property decrease if the material has to be mechanically solicited.

### *3.3. Dielectric properties*



In the next paragraphs, CFs loaded ultra-porous and lightweight epoxy foam composites are elaborated and characterized for an application as multilayer absorber material. Our previous works have shown that long fibers (CFs length > 3 mm) present interesting dielectric properties (high dielectric losses) needed for absorbing application [33]. **Additionally**, and as explained above, for a multilayer absorber, on the one hand, the use of a very lightly loaded first layer is necessary in order to achieve an impedance matching between the air and the absorber material, and thus, **reduce** the reflection at its interface. On the other hand, the use of successive layers with gradient compositions is useful in order to ensure a gradual transition of the EM waves, and thus, their attenuation inside **the multilayer** [32].

**In order** to verify that the dielectric losses of our composites, which brings absorption performance to these composites, are not affected by the low density of the samples, **two composites** loaded with 0.075 wt.% of CFs was elaborated **with and** without using acetone (**8 wt.%**). The measured density values are 0.12  $\text{g}\cdot\text{cm}^{-3}$  and 0.06  $\text{g}\cdot\text{cm}^{-3}$  for **these** samples elaborated without and with 8 wt.% of acetone, respectively. Fig. 8 **presents** the photos **of these elaborated** composites (Fig. 8.a) and their dielectric properties (real part of permittivity  $\epsilon'$  and dielectric losses  $\tan\delta$ ) (Fig. 8.b) **as** a function of frequency. Fig. 8.a shows that composite elaborated with acetone presents a better homogeneity; this is **probably** due to the lower viscosity of the mixture which allows to its easier mixing. **Chuang et al. have shown that the viscosity changes the rheological properties of CFs aqueous solution and affect their dispersion** [48]. **Fig. S10 shows this influence of viscosity (acetone addition) on the dispersion of CFs in the epoxy resin. It can be noticed that the solution with 8 wt.% of acetone presents the best dispersion of CFs (Fig. S10.b). Indeed, it seems that the very high viscosity of epoxy resin (0 wt.% of acetone) blocks some aggregates of CFs inside the resin,**

**thus prevents their dispersion (Fig. S10.a). On the other hand, the positive shear effect of the viscosity is lost when 15 wt.% of acetone is used; so, a bad dispersion of CFs is obtained (Fig. S10.c). Consequently, a moderate viscosity of epoxy resin, obtained in our case with 8 wt.% of acetone (730 mpa.s), appears as an optimum dilution condition to help the CFs dispersion, due to the shear viscosity effect induced by the mixture [49].**

Furthermore, Fig. 8.b shows that the dielectric losses of composites elaborated with and without acetone are similar, even identical. In fact, for these long carbon fibers, the losses brought by long fibers are quite high that the impact of the density (and therefore of air) is neglected. On the other hand, the low density induced a reduction in the real part of the permittivity; it decreases (at 4 GHz) from 1.86 to 1.34 for dense and lightweight samples, respectively, thanks to the air contribution ( $\epsilon'_{\text{air}} = 1$ ). It must be remembered here that we are looking for composites with low permittivity and high dielectric losses in order **to bring a low reflection, and so high absorption performance. To verify this aspect, the measured reflection coefficients of samples presented in Fig 8.a (made with 0.075 wt.% of CFs and with or without acetone) are presented in Fig. S11. This figure shows a better (lower) reflection coefficient for a sample made with 8 wt.% than the one without acetone. This confirms that the increase of cells size provides a better impedance matching at the air/sample interface. Consequently, The EM wave will be less reflected on the surface of sample and so, can be introduced into the material to be absorbed inside it.**

So, these results **show** that thanks to the new proposed elaboration process, we are able to prepare low weight and more homogeneous samples presenting more interesting dielectric properties, with regard to the intended application.

Afterwards, the chosen weight percentages of 12mm-CF loads, used for the elaboration of composites for an application as multilayer absorber, are ranging from 0.0125 to 0.075 wt.%. It should be noted that for these low CFs rates, a very low amounts of SDS were used (up to 0.005 g of SDS / 10 g of resin) to provide a homogeneous structure for the elaborated composites; 8 wt.% of acetone was used for the elaboration of all these composites. Fig. S12 presents the pictures and the densities of the achieved composites.

Fig. 9 shows the frequency dependence of the real part of the permittivity  $\epsilon'$  (Fig. 9.a) and dielectric losses  $\tan\delta$  (Fig. 9.b) of the achieved composites. These figures show, as expected, an increase in the dielectric properties with the percentage of CFs load. For these composites, the dielectric losses, at 4 GHz for example, increase from 0.08 to 0.61 when the CFs percentage increases from 0.0125 to 0.075 wt.%, respectively. This increase in properties has already been reported by several studies using CFs load with different matrix [8,44,50]. One can note that this low CF loads present high dielectric losses compared to losses of epoxy foams with higher carbon fiber loads obtained in other works [41]. For example, at 4 GHz, the epoxy foam loaded with 0.25 wt.% of 3mm-CFs shows loss tangent value ( $\tan\delta$ ) around 0.30 in the work of *Pometcu et al.* [41] while our epoxy foam loaded with **only** 0.075 wt.% of 12mm-CFs shows  $\tan\delta$  of 0.61 (two times higher); a maximum of  $\tan\delta$  of 0.66 is obtained at 2.2 GHz with the most loaded sample. These high dielectric losses are obtained thanks to the high length of the used fibers (12 mm). Indeed, it was shown that the length of the fibers directly influences the dielectric loss values: the longer the fibers, the higher the losses are, as shown in our previous works [33] and other works using CFs [51]. This could be explained; on the one hand, by the percolation threshold which must be lower for long fibers and **that** induces high conductivities, and therefore high losses. **Indeed**, the latter are directly related **to the conductivity** by the known formula

$\tan \delta = \frac{\varepsilon''}{\varepsilon'} = \frac{\sigma}{\omega \varepsilon_0 \varepsilon'}$ , with  $\sigma$  the electric conductivity of the composite,  $\varepsilon_0$  the vacuum permittivity and  $\omega$  the wave pulsation. On the other hand, this increase of dielectric losses can be explained by a decrease of the depolarization field with the increase of the fiber length, as explained in [51].

Furthermore, one can note that the low used CF percentages, associated with the low density of elaborated composites, permit to **keep very** low permittivity values. Above 6 GHz all samples show a permittivity ( $\varepsilon'$ ) between 1.04 and 1.1, very close to the one of the air ( $\varepsilon' = 1$ ). These low permittivity values will ensure a smooth EM wave penetration; **and the gradual** increase in the  $\tan \delta$  (with increasing CFs concentration) **will ensure** a gradual EM wave **attenuation, promising a good** absorption performance.

#### *3.4. Absorption performance of a multilayer prototype: Simulation and measurement*

In this section, a multilayer absorber composed of 5 layers (Fig. 10.a) is first simulated using the measured dielectric properties (Fig. 9) of epoxy foams filled with 0.0125, 0.01875, 0.025, 0.05 and 0.075 wt.% of 12 mm-CFs. The order of layers was chosen according to the gradient dielectric losses of the material (Fig. 9.b) [32]. The thickness of each layer was 25 mm with a total thickness of 125 mm. This prototype (Fig. 10) has the same dimensions as the commercially available PU foam based multilayer absorber used in anechoic chamber for the same frequency band [26].

The simulation and the measured reflection coefficient of the achieved multilayer prototype were compared in Fig. 11. It should be noted here that in order to consider the material as an absorber, a minimum of a reflection coefficient of -10 dB is needed in the absorbing bandwidth, **this indicates an absorption greater than 90%. Fig. 11** shows a good matching between the simulation and measured coefficients with a slight difference. This is probably due to the

difference between simulated absorber, with infinite dimensions, contrary to the measured prototype with finite dimensions (length and width) of 15 x 15 cm<sup>2</sup>. Moreover, results show that the performance of our prototype is very close to commercial absorber performance in the frequency band 2-18 GHz. The reflection coefficient can reach values below -10 dB over the entire frequency band and underneath -20 dB between 2.6 and 18 GHz.

Even with very low CFs loads, our material presents a good absorption performance compared with the commercial one. The long carbon fibers can ensure a low percolation threshold, especially in foamed matrix; therefore, very small quantities of filler provide a good absorption in the epoxy foam. These results confirm the potential of the proposed material, based on light-weight epoxy foams loaded with low percentages (between 0.0125 and 0.075 wt.%) of long carbon fibers (12 mm), to replace a commercial absorber based on PU foam loaded with volatile carbon nanoparticles.

#### **4. Conclusions**

**In this work, a simple method for the fabrication of very light-weight epoxy foam composites, using the acetone, is proposed. The effect of the solvent (acetone), the filler (CFs) and the surfactant (SDS) contents, on the epoxy foam structure, density and mechanical property is investigated. Results show that the density of the unloaded epoxy foam decreases from 0.12 g·cm<sup>-3</sup> to 0.05 g·cm<sup>-3</sup> when the amount of acetone increases from 0 wt.% to 15 wt.%; however, beyond 15 wt.% of acetone the structure of the foam is deformed.**

The mechanical properties (flexural modulus) of **the epoxy** foams are altered when their density is reduced. **The addition of 8 wt.% of acetone reduced the density from 0.12 to 0.07 g·cm<sup>-3</sup> and the flexural modulus from 35 MPa to 4 MPa.** Therefore, a compromise must be

made between mechanical properties and density if these materials have to undergo mechanical solicitation. The addition of CFs increased the density of the epoxy foam and reinforced the mechanical property of composites containing acetone, especially for high CFs percentages (**0.4 wt.%**). **The presence of SDS** has no effect on the density and the mechanical property of the epoxy foam but it can alter the homogeneity of the epoxy **structure if high content is used**.

**The dielectric** characterization of very low CFs ( $\leq$  **0.075 wt.%**) loaded light-weight epoxy foam in **the** anechoic chamber shows very interesting properties according to the absorbing targeted application. **Permittivity values** lower than 1.6 in all the studied frequency range associated to high dielectric losses (up to 0.66) **are obtained**. Furthermore, a multilayer absorber **composed of 5 layers (125 mm)** has been realized and characterized. Results show a good performance, with a reflection coefficient **lower than** -10 dB, in the entire studied frequency band (**2-18 GHz**) and below -20 dB between 2.6 and 18 GHz; **these results** are in accordance with the simulation **results**. Our material based on very low load and weight composite can achieve an absorption performance very close to the one of the commercial absorber over the entire frequency band 2-18 GHz.

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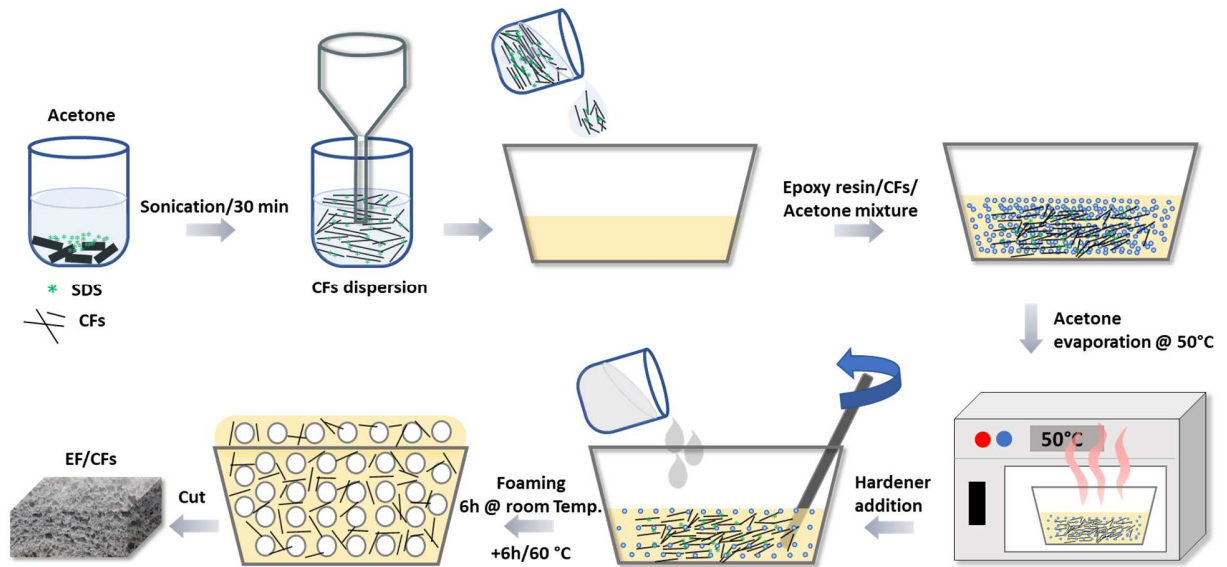
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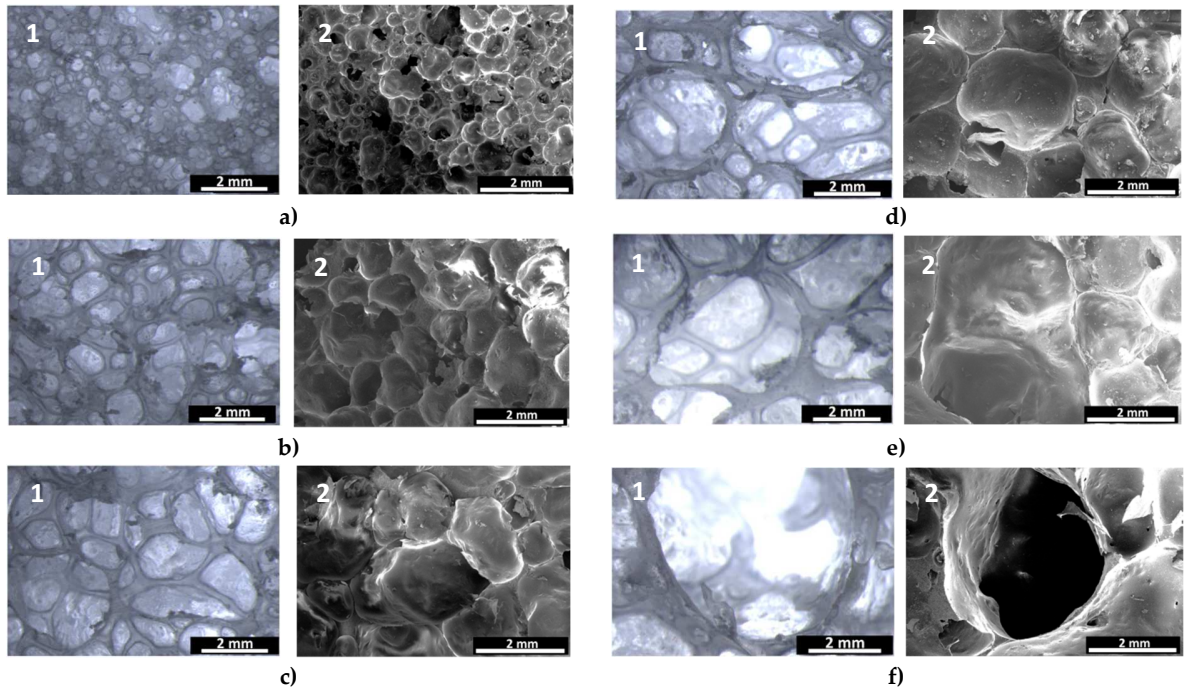
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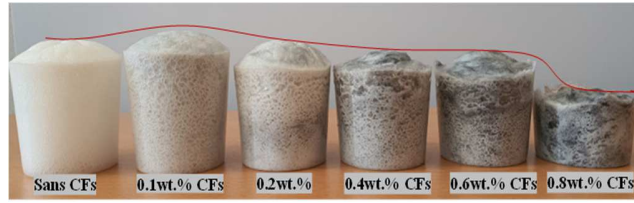


**Fig. 1.** Schematic diagram for the elaboration method of the light-weight CFs loaded epoxy foam.

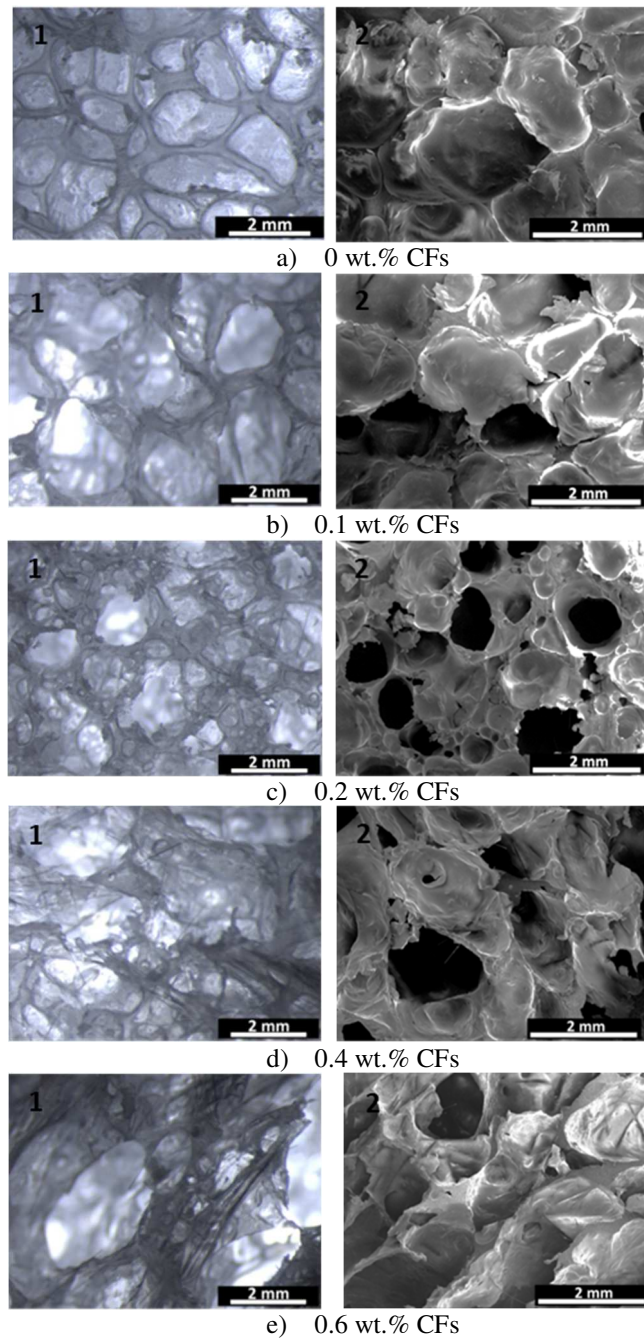


**Fig. 2.** Optical micrographs (a.1 - f.1) and **SEM** images (a.2 - f.2) of epoxy foams containing 0, 6, 8, 10, 12, 15 wt.% of acetone respectively. The scale bar is 2 mm.

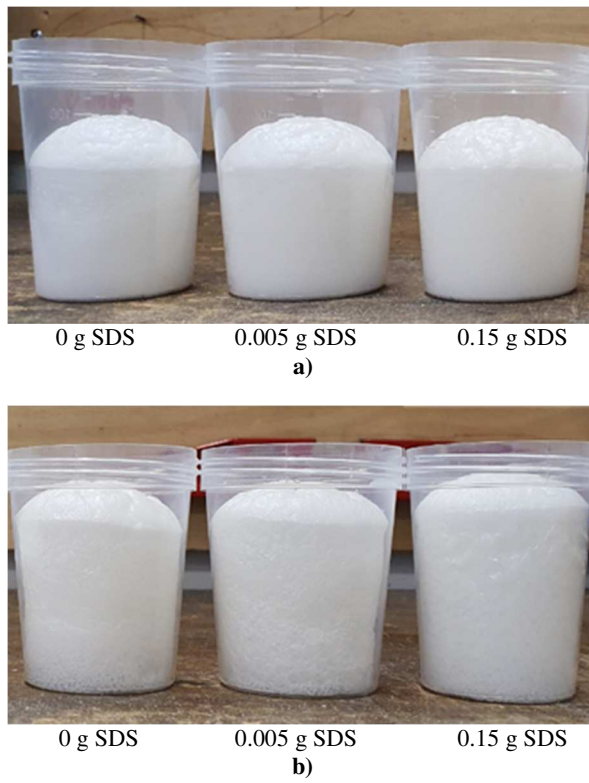




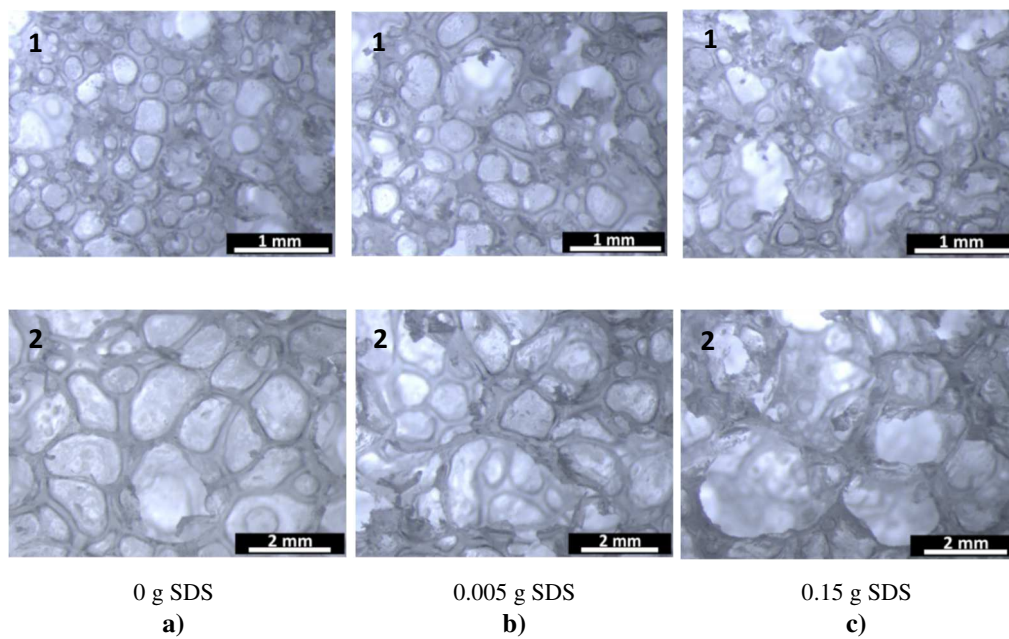
**Fig. 3.** Evolution of the CFs loaded composite foaming, as function of CFs content, with 8 wt.% of acetone.



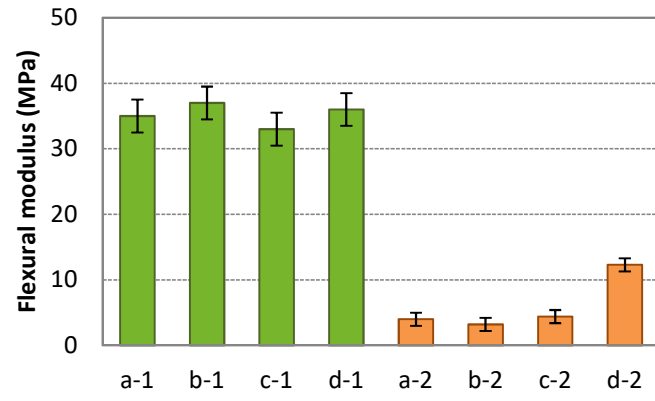
**Fig. 4.** Optical micrographs (a.1 - e.1) and SEM images (a.2 - e.2) of epoxy foams elaborated with 8 wt.% of acetone and with different contents of CFs.



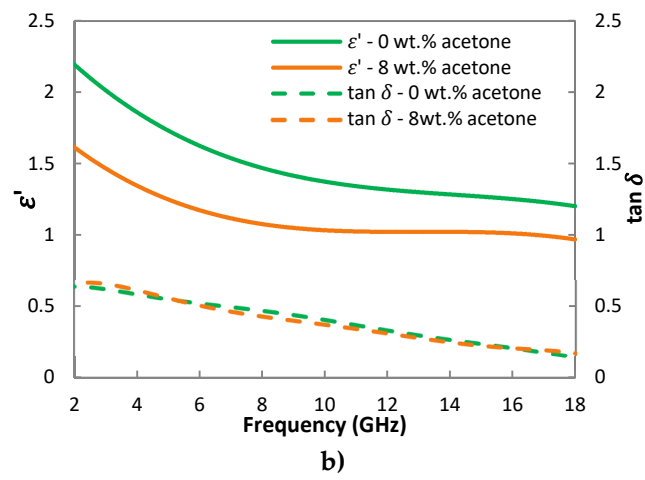
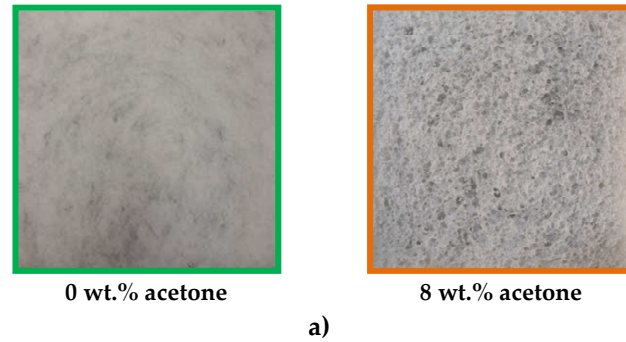
**Fig. 5.** Evolution of epoxy foams as function of SDS content a) without acetone and b) with 8 wt.% of acetone.



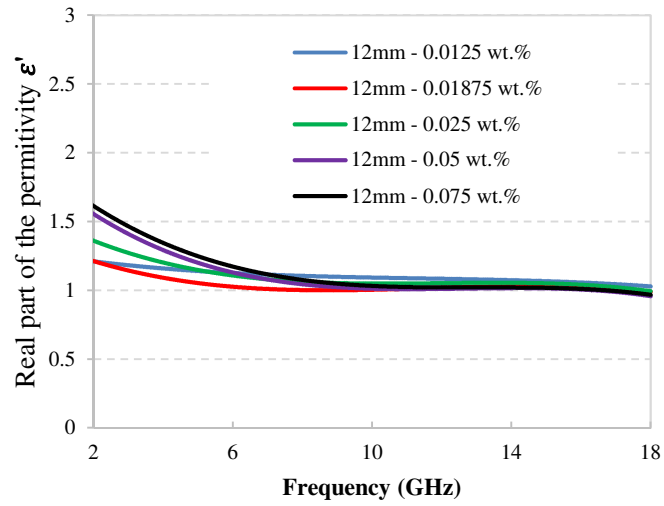
**Fig. 6.** Optical micrographs of epoxy foams elaborated with different contents of SDS: (a.1 – c.1) without acetone and (a.2 - c.2) with 8 wt.% of acetone.



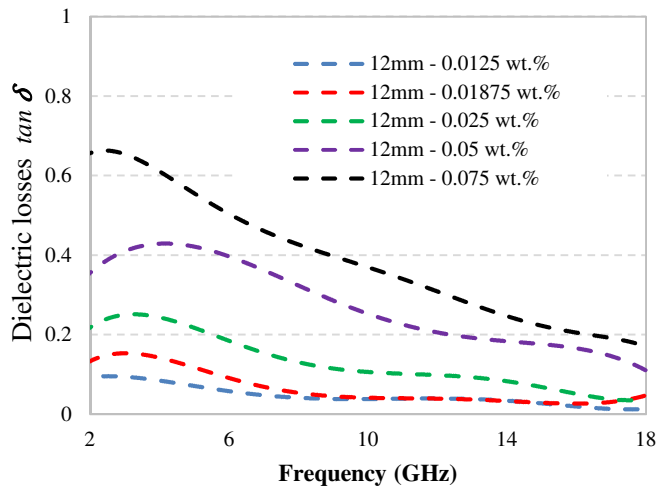
**Fig. 7.** Flexural modulus of different samples of epoxy foams with different contents of acetone, CFs and SDS.



**Fig. 8.** a) Photos and b) dielectric properties of 0.075 wt.% - CFs loaded composites elaborated with and without acetone.

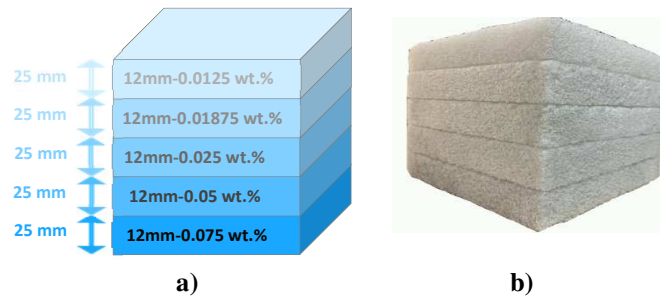


a)



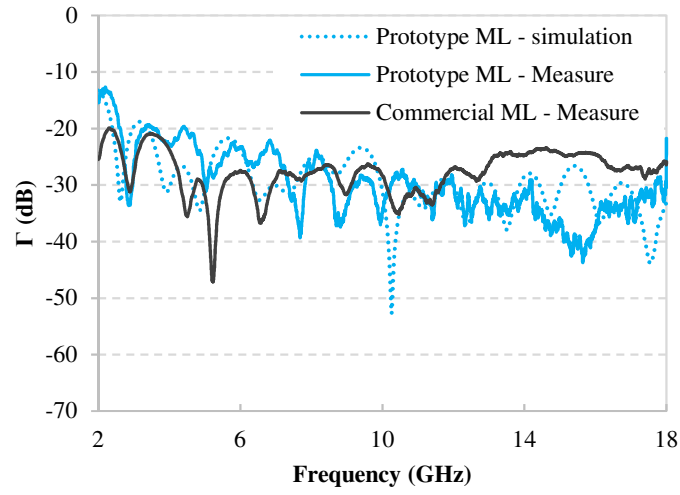
b)

**Fig. 9.** Measurement of a) real permittivity  $\epsilon'$  and b) dielectric losses  $\tan \delta$  for epoxy foams filled with different contents of CFs of 12 mm length.



**Fig. 10.** a) Detailed diagram of 5 layers loaded with different concentrations of 12mm-CFs and b) photo of achieved multilayer absorber prototype composed of 5 layers of epoxy foam loaded with 12mm-CFs.





**Fig. 11.** The simulation and measurement of reflection coefficients of the multilayer absorber prototype based on 12mm-CFs loaded lightweight epoxy foam compared to commercial absorber measurement.

Acetone content (wt.%)	0	6	8	10	12	15	20
Density (g·cm <sup>-3</sup> )	0.12	0.07	0.07	0.06	0.06	0.05	0.23

**Table 1.** Density of epoxy foams as a function of acetone content.

<b>CFs content (wt.%)</b>	0	0.1	0.2	0.4	0.6
<b>Density (g·cm<sup>-3</sup>)</b>	0.07	0.07	0.08	0.09	0.11

**Table 1.** Density of epoxy foams elaborated with 8 wt.% of acetone and with different contents of CFs.

<b>Acetone (wt.%)</b>	0	0	0	8	8	8
<b>SDS (g)</b>	0	0.005	0.15	0	0.005	0.15
<b>Density (g·cm<sup>-3</sup>)</b>	0.12	0.12	0.12	0.07	0.07	0.07

**Table 1.** Density of epoxy foams elaborated with different contents of SDS for two quantities (0 and 8 wt.%) of acetone.

<b>Sample</b>	<b>a-1</b>	<b>a-2</b>	<b>b-1</b>	<b>b-2</b>	<b>c-1</b>	<b>c-2</b>	<b>d-1</b>	<b>d-2</b>
<b>Acetone (wt.%)</b>	0	8	0	8	0	8	0	8
<b>CFs (wt.%)</b>	0	0	0	0	0.075	0.075	0.4	0.4
<b>SDS (g)</b>	0	0	0.15	0.15	0.005	0.005	0.027	0.027
<b>Density (g·cm<sup>-3</sup>)</b>	0.12	0.07	0.12	0.07	0.13	0.06	0.11	0.07

**Table 1.** Contents of acetone, CFs and SDS used for the sample elaboration for the mechanical characterization.

