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# Manufacturing of open-cell Mg foams by replication process and mechanical properties

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Open-cell pure Mg foams were produced by replication casting process. The preform used was manufactured with spherical particles of NaCl with sizes ranging from (A) 1 mm to (D) 2 mm. It was found that increasing the pore size, the relative density decreased, while the porosity increased, registering a minimum relative density of 0.22 and a maximum percentage porosity of 78% for sample (D) 2 mm. The mechanical properties and energy absorption characteristics were investigated by means of compression test. Under the present experiment conditions, the sample (A) 1 mm with the smaller pore size and the lower percent porosity 67%, showed the highest mechanical properties; Young's modulus, yield stress, and the high energy absorption capacity. The mechanical properties obtained and the large plateau region could be favorable for scaffold and energy absorbing applications.



## 1. Introduction

Metallic foams are a new class of materials used as impact absorbers, dust and fluid filters, heat exchangers, flame arresters, among others, because these show a combination of several properties such as high strength-to-weight ratio, high energy absorption capacity, large specific surface, high gas and liquid permeability, and low thermal conductivity [1,2]. But the prime advantage is their excellent combination of good mechanical properties and low weight [3]. These properties depend significantly on the pore structure (morphology, size, distribution) and metal matrix, their influence is even a topic of intense study.

The development of Mg and Mg-based alloys foams has been of great interest, because these show the same functionality of aluminum foams but with less weight [4,5]. Mg foams have been mainly studied for their functional properties; sound and energy absorption capacity, excellent vibration reduction capacity and recently have been recognized as promising biomaterial for bone implants [6]. Mg foam is an ideal scaffold for bone tissue regeneration, and has been reported to show suitable mechanical properties, including yield stress $\sigma_y{}^*$, Young's modulus $E$, plateau stress $\sigma_{pl}$ and ductility [7]. In addition these Mg foams are biocompatible with the human body, since the open-cell structure allows the ingrowths of the new bone tissue [8]; the material would even have the potential to break down *in vitro*, making it a resorbable implant allowing regrowth. Several methods have been used to fabricate pure Mg foams, the most common are powder metallurgical (PM), melt foaming method and vacuum foaming [1,9–11]. These methods have achieved to obtain porosities up to 72% with good mechanical properties and comparable energy absorption capacities of Al foams. However, studies on the mechanical properties and energy absorption capacities of open-cellular pure Mg foams are still limited.

The replication casting process offers the possibility to produce interconnected open-cellular Mg foams with a high degree of control over the porosity (size, shape, distribution, etc.) with the metal matrix free of foaming agents [12]. There are not reports on the fabrication neither the mechanical properties of open-cell pure Mg (~99.95%) foams manufactured through the replication casting method.

The current research was designed to fabricate open-cell pure Mg foams by the replication casting route with porosity of 67–78% and different pore sizes. The mechanical properties and energy absorption characteristics were investigated through a compression test. In addition, the fabricating process and the potential applications are described.

## 2. Experimental procedures

### 2.1. Fabrication of Mg foams

Open-cell magnesium foams were produced by a replication casting device with control of atmosphere, which is shown on Fig. 1. The apparatus is constituted by three parts; a cylindrical

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crucible of stainless steel, a valve system and an electric resistance furnace.

The porous preforms were produced by using spherical particles of pure NaCl supplied by Hydro soft, England. The NaCl particles were sieved and separated in four average sizes and classified as (A) 1 mm, (B) 1.4 mm, (C) 1.7 mm and (D) 2 mm. The NaCl particles were cold pressed within the crucible to produce porous preforms. Magnesium ingots of commercial purity (99.95%) were placed over the preform and then the crucible was sealed. The magnesium was subsequently melted at 750 °C under a low (Ar) gas pressure (0.1 MPa). Afterwards, the NaCl preform was infiltrated with molten metal using a (Ar) gas pressure of 0.4 MPa for 10 min.

When the formed Mg–NaCl composite was completely solidified, this was extracted from the crucible, and then machined to obtain cylindrical compression samples of dimensions: 25.4 mm in diameter and 20 mm in height. The NaCl particles were completely dissolved in a solution of NaOH + $H_2O$. Based on the potential-pH (Pourbaix) diagram [13], it was essential to use a pH = 13, in order to avoid corrosion by pitting in the magnesium matrix. The density of the magnesium foams was carried out calculating the volume of the cylindrical samples and measuring their mass. The porosity was characterized by optical images and calculated by means of the relative density $\rho_{Rel}$.

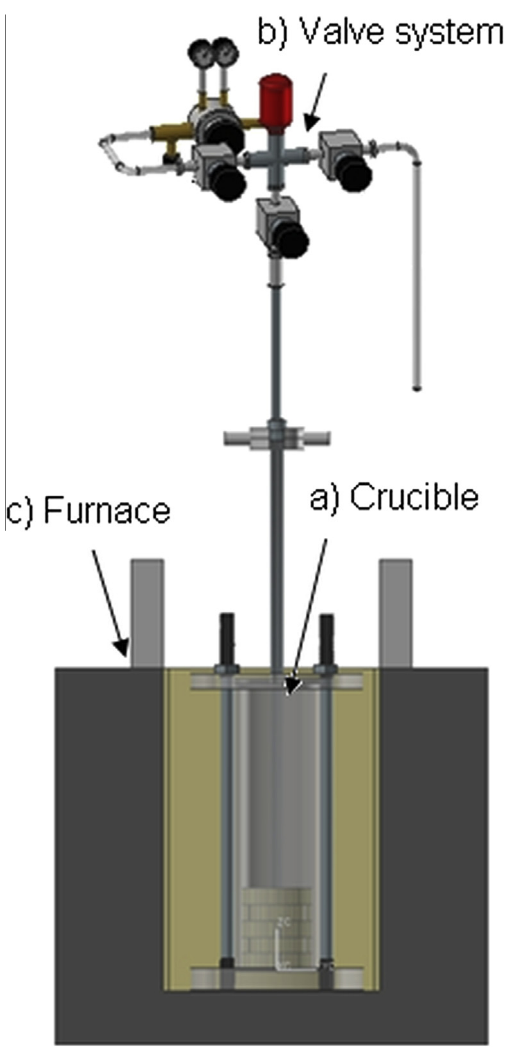


**Fig. 1.** Infiltration system: (a) steel crucible sealed with two covers, upper and lower (attached to a long steel pipe), (b) valve system and (c) heating furnace.

### 2.2. Compressive tests

The compression tests were conducted at room temperature using a universal testing machine (Instron 5500R) at a constant crosshead speed of 0.5 mm/s, corresponding to a nominal strain rate of $2.5 \times 10^{-2}$ $s^{-1}$. The stress–strain data are reported in terms of engineering stress and strain.

## 3. Results and discussion

### 3.1. Cell structure

Representative cylinder samples of the open-cell Mg foams with different pore size are shown in Fig. 2. In these images, it can be observed that all samples have a homogeneous distribution of the spherical pores with equivalent cell size to NaCl particles. The corresponding stereo micrographs of open-cell Mg foams are shown in Fig. 3, which demonstrates that all samples show open-cell structures with interconnected pores.

The percent porosity Pr(%) of the Mg foams was calculated using the relative density $\rho_{Rel}$ (defined as the density of the foams $\rho^*$ between the density of the metal matrix $\rho_{sMg}$ = 1738 kg/$m^3$) in the following expression:

$$\mathrm{Pr}(\%) = (1 - \rho^*/\rho_{sMg}) \times 100 \quad (1)$$

The density of the foams $\rho^*$ was calculated from their mass (M), and volume (V) by using the cylinder samples with volume = $1.013 \times 10^{-5}$ $m^3$. Table 1 summarizes the experimental parameters obtained of the open-cell Mg foams to calculate the percent porosity Pr(%). From this table, it can be observed that when the pore size decreases from sample (D) 2 mm to sample (A) 1 mm, both the density of the foams and the relative densities increased. On the other hand, the percent porosity Pr(%) increased with increasing pore size, reaching a value up to 77% for the sample (D) 2 mm.

### 3.2. Mechanical properties

The mechanical properties of the open-cell Mg foams were studied by compression test. The compressive stress–strain curves of open-cell magnesium foams with different pores sizes and relative densities are shown in Fig. 4.

In all cases the curves exhibit the characteristic open-cell foam behavior showing three distinct regimes: (1) an initial linear elastic region at very low strain (smaller than about 0.05) without the presence of peak stress, (2) an extended plateau region at a relative constant stress level where the stress increases slowly as the cells deform plastically. The obtained compression curves in the plateau regions are smooth, without the presence of oscillations or serrations, that are commonly observed in open-cell Mg foams manufactured by powder metallurgy (PM) [9] and (3) a densification region registered at around 0.5–0.6% strain where the collapsed cells are compacted together. The experimental mechanical properties obtained from the compressive stress–strain are summarized in Table 2.

From this table, it can be observed that all mechanical parameters; yield stress $\sigma_y{}^*$, plateau stress $\sigma_{pl}$ and Young's modulus $E$, showed the tendency of increasing with decrease in the percent porosity Pr(%) or when the relative density increases, as would be expected. However, the samples also change in pore size (with decreased pore size coinciding with improved properties). Although a pore size effect, with strength increases in smaller pore size foams has been reported for replicated aluminum foams [14] (caused by the generation of dislocation loops due to differential thermal contraction between the salt preform and the metal after

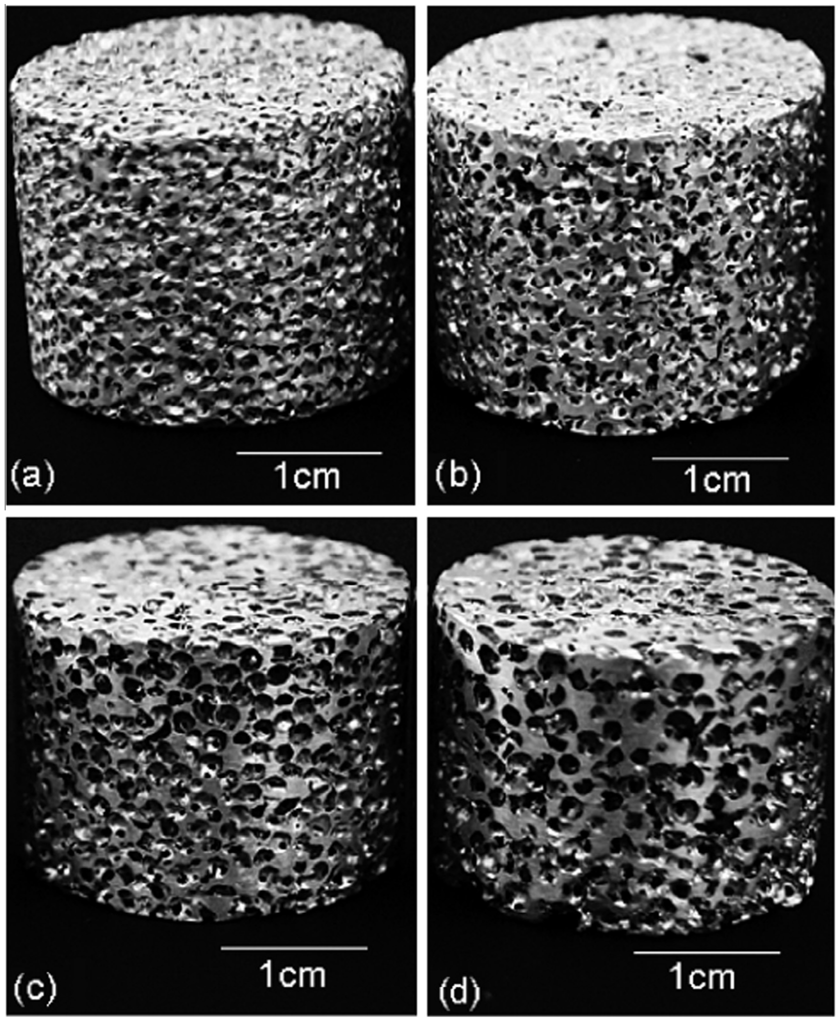


**Fig. 2.** Digital images showing the cell structures of the magnesium foams with different poros sizes: (a) (A) 1 mm, (b) (B) 1.4 mm, (c) (C) 1.7 mm) and (d) (D) 2 mm.

solidification) it is not generally to be anticipated that pore size would have an effect. Furthermore, the pore size range over which this is observed here is significantly larger than that where the effect was measured in aluminum. We therefore attribute these changes to the effect of density differences alone, and will return to this later in the discussion.

The yield stress $\sigma_y{}^*$ (the 0.2% offset criteria was used to identify the initial linear elastic behavior and the yield stress) increased from 1.2 MPa for porosity of 78% to 2.5 MPa for porosity of 67%. The low values of yield stress, $\sigma_y{}^*$, can be attributed to the high percentage porosity and the thin walls of the Mg foam cells.

Regarding the Young's modulus $E$, this increased from 0.61 GPa (sample (D) 2 mm with porosity of 78%) to 0.72 GPa (sample (A) 1 mm with porosity of 67%). The Gibson–Ashby model [15] is widely used to predict the mechanical properties of foams (principally $\sigma_{pl}$ and $E$) as a function of the relative density, $\rho_{Rel}$. From an analytical treatment of a simplified porous structure, the model proposes simple relations between the relative density, $\rho^*/\rho_{sMg}$, and the (2) Young's modulus $E$ and (3) plateau stress, $\sigma_{pl}$, as follows:

$$E/E_{sMg} = A(\rho^*/\rho_{sMg})^2 \tag{2}$$

$$\sigma_{pl}/\sigma_{ys} = C(\rho^*/\rho_{sMg})^{3/2} \tag{3}$$

In Eq. (2) $E_{sMg}$ is the Young's modulus of the solid cell edge material (taken as 40 GPa for magnesium) [16], $A$ is a constant related to the cell geometry with value = 1. In Eq. (3) $\sigma_{ys}$, is the yield stress of the solid cell edge material (21 MPa) [16]. Data of polyurethane foams and many cellular metals suggest that $C = 0.3$, although in practice the value of the constants $A$ and $C$ both vary over a wide range with different foam types.

The relation between $E$, $\sigma_{pl}$, and the relative density $\rho_{Rel}$, of the open-cell Mg-foams is plotted in Fig. 5a and b. Here, following the reasoning discussed earlier we make the assumption that the pore size does not affect the mechanical property results. It can be observed that the experimental Young's modulus $E$ results are not close to the predictions, Fig. 5a. However, as noted above, the constant $A$ takes different values in different foam types, and so the trend with density is a more important comparison to make. It is seen that the slope of a straight line fit to the data in Fig. 5a (the exponent in an equation of the form of Eq (2) as the plot is logarithmic) would be close to 0.35, while the slope of the Gibson–Ashby prediction is 2. This does not seem like a good prediction, and could be due to effects from the structural change brought about by the different pore sizes.

The experimental plateau stress, $\sigma_{pl}$, values are high compared to the predictions of the Gibson–Ashby model, Fig. 5b, but the same factor of the variable value of $C$ apply in this case. Once again the important observation is the exponent, and it is observed that

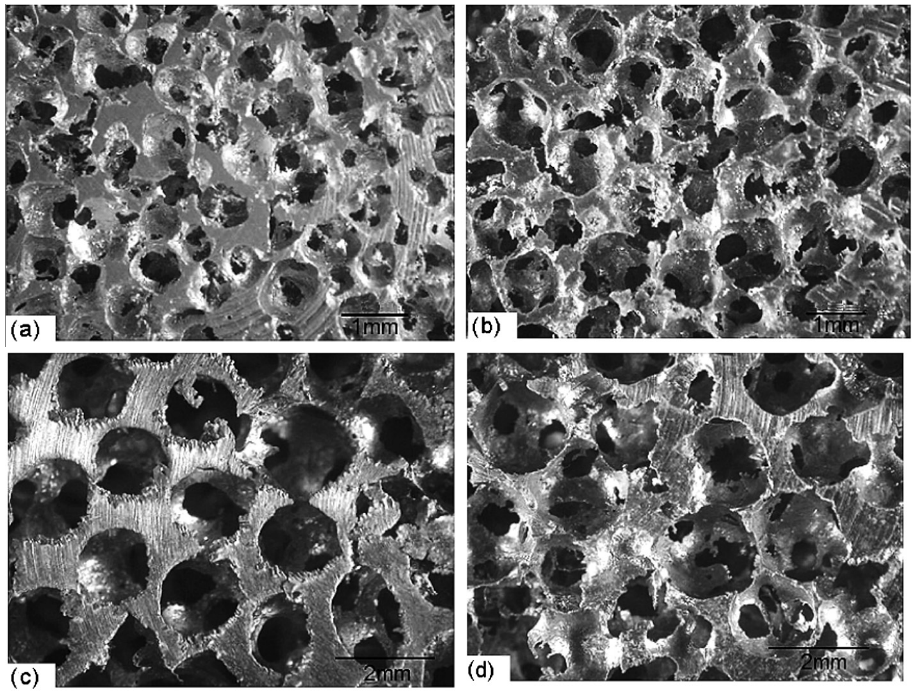


**Fig. 3.** Stereo micrographs of open-cellular Mg foams: (a) (A) 1 mm, (b) (B) 1.4 mm, (c) (C) 1.7 mm and (d) (D) 2 mm.

**Table 1**
Experimental densities and percent porosity (%) of the open-cell Mg foams.

| Sample | Density of foams, $\rho^*$ (kg/m$^3$) | Relative density $\rho^*/\rho_{sMg}$ | Pr (%) |
|---|---|---|---|
| (A) 1 mm | 572.5 | 0.33 | 67 |
| (B) 1.4 mm | 463.4 | 0.27 | 73 |
| (C) 1.7 mm | 414.6 | 0.24 | 76 |
| (D) 2 mm | 385.1 | 0.22 | 78 |

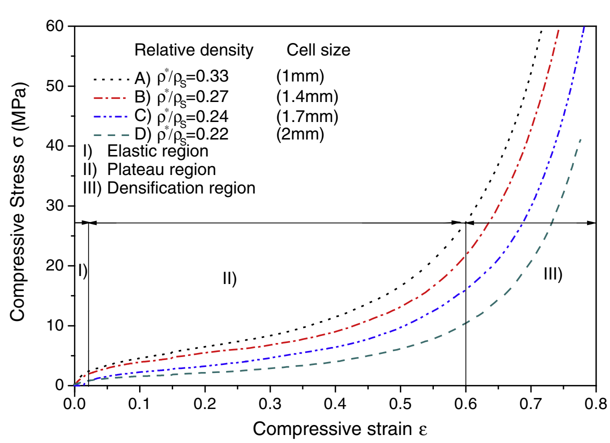


**Fig. 4.** Compressive stress–strain curves of magnesium foams of different pore sizes.

there is a good match in the trend in the experimental data and the prediction of the equation.

The experimental results are also similar to those obtained in closed-cell pure Mg foams; manufactured by a melt-foaming process, for equivalent relative densities, ranging from 0.22 to 0.33 [17]. When the percentage of porosity in the open-pore and foams is high (70–80%), the walls that produce the structure of such foams tend to be rather thin, therefore, it is expected that the mechanical properties could be similar to those of foams with closed pores.

From a practical point of view, the yield stress $\sigma_{ys}$ values obtained in the present study are similar to that of trabecular bone, whose resistance ranges from 1.5 MPa to 5.3 MPa [18]. Furthermore, the Young's modulus $E$ of the foams produced here are similar to the values of cancellous bone (from 0.01 to 2 GPa) [19,20]. Thus, it can be concluded that the replication casting technique could be useful for the manufacture of open-cell pure Mg foams for scaffolds, with the possibility of control the mechanical properties by means of the pore size, porosity and relative density $\rho_{Rel}$. If better properties were required than those shown by the best performing foam tested here (Young's modulus $E$ = 0.72 GPa, and yield stress $\sigma_{ys}$ = 2.5 MPa for the sample (A) 1 mm) then these could be achieved at the expense of higher density.

The plateau stress, $\sigma_{pl}$, (calculated by averaging the stress values obtained between the elastic region and the densification region) increased from 5.2 MPa (sample (D) 2 mm) to 8.1 MPa (sample (A) 1 mm).

**Table 2**
Experimental mechanical properties and energy absorption capacity of the open-cell Mg foams.

| Sample | Yield stress $\sigma_y^*$ (MPa) | Young's modulus $E$ (GPa) | Average plateau stress $\sigma_{pl}$ (MPa) | Energy absorption $W$ (MJ/m$^3$) |
|---|---|---|---|---|
| (A) 1 mm | 2.5 | 0.72 | 8.1 | 5.5 |
| (B) 1.4 mm | 1.9 | 0.69 | 7.5 | 4.6 |
| (C) 1.7 mm | 1.5 | 0.65 | 6.4 | 3.1 |
| (D) 2 mm | 1.2 | 0.61 | 5.2 | 1.9 |

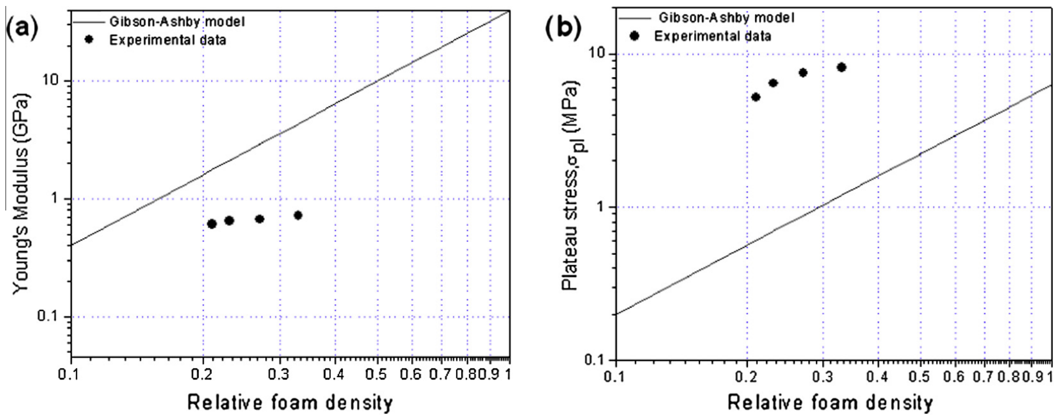


**Fig. 5.** Relation between the relative density, $\rho_{Rel}$, and: (a) Young's modulus $E$ and (b) plateau stress, $\sigma_{pl}$.

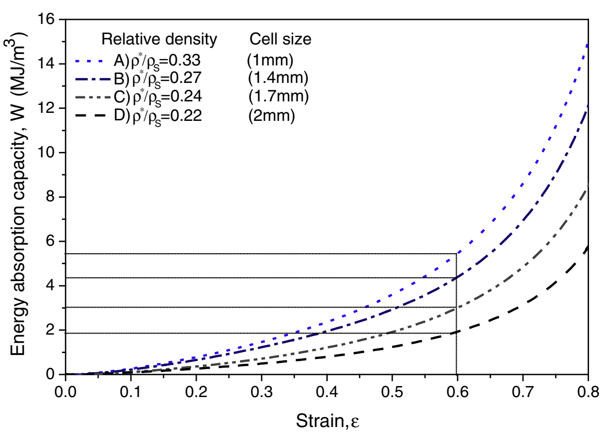


**Fig. 6.** Energy absorption capacity of open cell Mg foams.

The absorption energy capacity, $W$, was obtained from the stress–strain curves, calculating the area under plateau region (also defined as the total kinetic energy absorbed by the foam during the compression test) prior to the onset of densification, according with the expression [21]:

$$W = \int_0^{\varepsilon} \sigma d\epsilon \quad (4)$$

where $\sigma$ and $\varepsilon$ are the compressive stress and strain, respectively. The energy absorption capacity of the open-cell Mg foams with different pore sizes was calculated in the strain range from 0.05 to 0.6 and the results are shown in Fig. 6 and Table 2.

From Table 2, it can be seen that the absorption energy increased with decreasing the pore size and the percent porosity, Pr(%). As mentioned above, the yield stress, $\sigma_{ys}$, increased with the decrease of pore size, therefore, the area under the stress–strain curve also increased.

Similar results were reported in a previous study on the open-cell ZA22 alloy foams carried out by Sirong Yu et al. [22]. They found that foams with higher relative density can dissipate more energy than those with lower density during the compression test, which is consistent with the results shown in Fig. 6.

Although the closed-cell foams have higher energy absorption capacity than the open-cell foams, the experimental values obtained are comparable with the results reported for closed-cell foams at the same levels of porosity (67–78%) [17].

## 4. Conclusions

Open-cell Mg foams with pore size ranging from (A) 1 mm to (D) 2 mm and homogeneous structure were successfully fabricated by the replication casting route using preforms constituted by spherical particles of NaCl. The effect of pore size and relative density on the mechanical properties and energy absorption capacity were investigated.

Under the present experiment conditions, the sample (A) 1 mm with the smaller pore size and the lower percent porosity 67%, showed the highest mechanical properties; Young's modulus (0.72 GPa), yield stress (2.5 MPa), and the high energy absorption capacity (5.5 MJ/m$^3$).

From a practical point of view, the mechanical properties obtained in the present study are enough to fulfill the required mechanical response of some scaffold materials, energy absorbers and those of other functional applications such as filters, catalyst supports, and $CO_2$ capture media.

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