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Characterization of rigid polyurethane foams containing microencapsulated Rubitherm® RT27: catalyst effect. Part II

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Abstract Catalyst Tegoamin 33 has been used for the synthesis of rigid polyurethane (RPU) foams containing microencapsulated Rubitherm® RT27 and having a high mechanical resistance. These materials could be employed in buildings for thermal insulation and thermal energy storage (TES). The fillers content influence on the foaming process and also the foam properties was evaluated. It was observed that a foam containing up to a 18 wt% of microcapsules can be manufactured, improving the TES capacity while maintaining the mechanical properties of the neat foam. Besides, it was observed that the mechanical resistance of foams synthesized using catalyst Tegoamin 33 are higher than those obtained when catalyst Tegoamin BDE was employed, with the mechanical resistance of the foam containing 21 wt% being higher than those of foams synthesized with catalyst BDE containing only 11 wt% of fillers while maintaining the advantages of an improvement in TES capacity. A general model of reaction curve of *n* tank-in-series of a same time constant was used to fit the rising curves. This model allowed to predict the final volume of the synthesized foam. Finally, TES capacities and mechanical properties of the synthesized foams were in the range of those reported in literature. Moreover, foam densities satisfied the restriction established by the Spanish regulation for building applications.

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Introduction

Nowadays, the environmental impact of fossil fuels is undergoing an important attention due to their combustion releases to the atmosphere millions of tones of CO_2 per year, which is widely believed to be contributing to global warming [1]. According to the EU directive 2002/91/EC, close to 40% of the final energy consumption and also 36% of CO_2 emissions in the Europe Community in 2002 were due to the residential and tertiary sectors [2].

The energy demand in the residential sector can be reduced by taking advantage of the big external building surface exposed to solar energy that allows to consider this energy as one of the best alternatives to the combustion of fossil fuels for heating buildings. In fact, in the last years, new building concepts and designs are appearing with the aim of saving energy by means of using the solar energy. This new concept of construction has been called “Solar Passive Building Concept” [3].

The main problem of this natural and renewable energy resource is its intermittent availability, making storage a necessity. Energy storage by means of materials may be in the form of sensible and latent heat or even by chemical reactions. The latent heat allows storage of high quantities of energy per unit mass and the energy storage process occurs at approximately constant temperature with a negligible decrease of the material density. A large number of materials, commonly called phase change materials (PCMs), can be used to absorb or release the energy equivalent with their latent heat when the temperature of the material undergoes or overpasses the temperature of phase change. Between them, organic materials (paraffin waxes and eutectic materials) present some advantages with respect to inorganic ones (eutectic materials, salt mixtures and salt hydrates); they are supposed to have higher chemical and thermal stability, lack of segregation, supercooling or corrosion problems and also, an adjustable transition zone [3–5].

The incorporation of PCMs into the building materials can be carried out by direct or indirect way. Some authors have studied the direct incorporation of PCMs into construction materials and they have found that PCMs can be adsorbed in concrete pores improving the energy storage capacity of the wall [6–11]. However, this direct application can lead to leakage of the paraffin wax [12]. Therefore, paraffin waxes should be placed inside a container before being incorporated in buildings [13, 14]. Microencapsulation of PCMs with a polymeric shell is considered to be one of the best technical options to obtain PCM confinement. Besides, the microencapsulation allows the increase of the heat-transfer area and controls the volume change as the phase change occurs [15]. The most common methods described in literature for microencapsulation are interfacial polymerization, emulsion polymerization, in situ polymerization, suspension polymerization, coacervation and spray drying [16–21].

In previous works, PCMs were successfully encapsulated by a polymer cover (polystyrene) by means of a suspension like polymerization technique [19–21]. Furthermore, gypsum blocks were doped with synthesized microcapsules containing the paraffin wax Rubitherm® RT27, finding that the wall microcapsule content enhances the thermal energy storage (TES) capacity and the insulating effect of the wallboard [21]. In recent papers, it has been reported that the incorporation of PCM into rigid polyurethane (RPU) foams, which are materials widely employed to produce sandwich panels [22–24]. The leakage problem was observed by Sarier and Onder [22] when *n*-hexadecane was directly incorporated into the PU foam during its synthesis but with an increase in its TES capacity. On the other hand, different studies have been done using microcapsules containing PCMs, finding that the leakage problem is solved and also that a microcapsules content close to 21 wt% can be added in the foam recipe to give a PU foam with a TES capacity in the order of 18 J/g [24, 25].

In the Part I of this series we studied the effect of microencapsulated Rubitherm® RT27 in the foaming process and in the final properties of PU foams. The synthesized RPU foams rose properly, exhibiting good TES capacity, distribution of the microcapsules and density. However, it was observed that the foam mechanical strength decreased for microcapsules contents higher than 5 wt% [24]. According to Ligoure et al. [26], Campanella et al. [27] and Lubguban et al. [28], the foam structure and its properties such as density, compressive strength and thermal conductivity are dependent on the relative rates of the foaming reactions which could be controlled by the use of an adequate catalyst. Thus, the foaming process must be modified in order to obtain a RPU foam of higher mechanical strength.

The synthesis of RPU foams is basically based on the condensation reaction between diols or polyols and diisocyanates or polyisocianates in presence of a catalyst. Besides, blowing agents and surfactants are added to regulate the morphology of the foam cells [29]. This reactive process can be simplified considering the two main chemical reactions that take place in the foaming process: isocyanate reactions with polyol hydroxyl groups—crosslinking reaction—and with water to produce CO_2—blowing reaction. Different catalysts can be used to control the foaming process and depending on their characteristics they can be used to promote mainly one of the above reactions. In this sense, while the tertiary amines have been used to catalyze both reactions or mainly the blowing, organometallic catalysts can be used to enhance the foam structure, favouring mainly the crosslinking reaction [29, 30].

In Part I of this series a tertiary amine Tegoamin BDE, which catalyzes mainly the blowing reaction, was used [24]. In this work, a tertiary amine that catalyzes both blowing and crosslinking reactions was used in order to produce a RPU foam of higher mechanical strength.

Thus, the aim of this work is to synthesize RPU foams containing microencapsulated PCMs using catalyst Tegoamin 33 in order to improve the mechanical strength while maintaining the thermal properties reported in the previous paper. The foaming process and the physical and mechanical properties of the final product were analysed and a mathematical model was proposed for the final volume of the foam as a function of the filler content.

Experimental

Materials

Polyol used in this work was Alcupol R-458 from Repsol YPF S.A. As isocyanate, diphenylmethane-4,4′-diisocyanate (MDI) supplied by Merck Group, was used. Catalyst Tegoamin 33 and surfactant Tegostab B8404 were supplied by Evonik Degussa International AG. Deionized water was used as blowing agent. Spherical microcapsules containing Rubitherm® RT27 with a particle size ranging between 1 and 5 μm were obtained following the method described in the Patent EP2119498 [31].

PU foams synthesis

RPU foams were synthesized by weighting and mixing the desired masses of polyol, silicone, water, amine and microcapsules containing Rubitherm® RT27 and further stirring the mixture during 1 min. Then, the adequate quantity of isocyanate was added to the above mixture and the resulting solution was stirred for just 5 s, until the moment at which the foam started to grow up. Finally, the obtained foams were cured at room temperature. Table 1 shows the foam synthesis recipe.

Sample characterization

Rising process

The foams rising process was measured by an ultrasonic system sqs-01 recording the foam height with time by using the program Schaum SQS-01.

Density

Foam densities were calculated by weighting and measuring the volume of the samples taken out.

Average latent heat

The microcapsules presence and their content into the foam can be obtained by considering the peak observed at the melting transition of the encapsulated PCM. Differential scanning calorimetry (DSC) analyses of the samples were performed from 10 to 45 °C at a heating rate of 5 °C/min. In Part I of this series, DSC analysis was confirmed to be a suitable tool for obtaining the latent heat of foams containing microencapsulated PCMs due to the good agreement between the theoretical and experimental latent heat values. The average latent heat of each foam was obtained as a result of four different samples analyzed in the selected area.

SEM analysis

Synthesized RPU foams were depicted by SEM to visualize the possible changes in their cellular structure when different amounts of microcapsules were added and the foam cellular size distribution was determined by the Image Plus program.

Table 1 Weight percentage of raw materials for PU foams synthesis

Material	Weight percentage
Polyol	48.43
Water	1.21
Silicone	0.73
Amine	1.21
MDI	48.43

Compression test

Uniaxial compression tests were performed according to ASTM D1621 using the Model 5584 of an INSTRON universal testing instrument equipped with a 1 kN load cell. The compression tests were carried out at a cross-head speed of 2.5 mm/min and transversal to the rising direction. The tested foam specimens were 5.1 × 5.1 × 2.6 cm size and they were compressed to 13% of their original thickness. Each analysis was repeated in threefold. The foam compression strength (S) was obtained using the force required to get a specimen deformation of 10%. The compressive modulus (E) was calculated dividing the slope of the force–deformation curves obtained below the proportional limit by the area of the specimen and multiplying the result by the initial height.

Results

Foam growth

Figure 1 shows the rising curves of the PU foams containing different contents of microencapsulated PCMs and using catalyst Tegoaming 33. As expected, the foaming rate decreased with increasing microcapsules content, since a lower slope of the curve at the short time region was observed. Besides, a lower final foam height was obtained when the microcapsules content increased. The higher the microcapsules content, the higher the mass (general recipe plus particles) has to grow up with the same expansion force and therefore, the rising rate and final height are lower. These results agree well with those reported in Part I of this series [24], when the catalyst Tegoamin BDE was

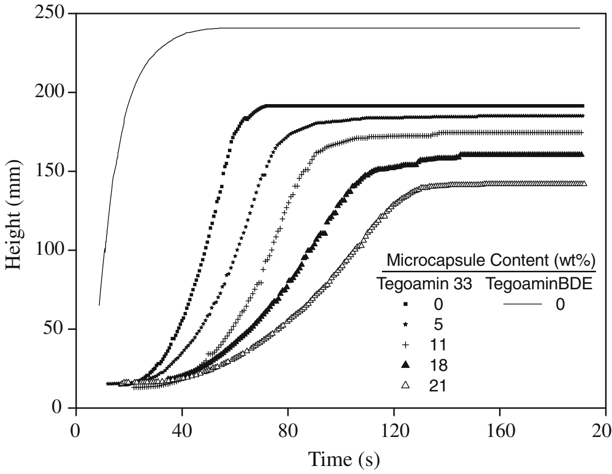

Fig. 1 Experimental rising curves of the RPU foams using catalyst Tegoamin 33 for different microcapsules contents and compared to the pure RPU foam obtained with Tegoamin BDE

used and with those reported by several authors [21, 26, 32]. Thus, this general influence of fillers on the foaming systems allows to consider this behaviour as a rule, in which the higher the filler content, the higher the viscosity of the foaming system and the higher the proportion of mass that has to be lifted by the same amount of expanding gas, which leads to a lower rising rate and a lower final height of the foam.

On the other hand, comparing the foam rising results of the RPU foams for both catalysts, RPU foams synthesized with Tegoamin BDE (in our previous paper [24]) had higher foaming rates diminishing the delay time and final heights higher than those obtained with catalyst—Tegoamin 33—used in this paper. This result can be explained taking into account the properties of catalyst Tegoamin 33 that catalyzes both the gelling—first step of crosslinking—and the blowing reactions, while Tegoamin BDE mainly promotes the CO_2 formation. Besides, these results agreed with those reported by Campanella et al. [27] who found lower foam heights and thus, smaller foam cell sizes when the curing rate was faster than the gas formation rate. They modified these reaction rates by changing the catalyst type and its proportion in the recipe.

As it is well known, the effects of microencapsulated PCMs on the characteristics of RPU foams are very important for further building applications because a specific foam thickness will be required to obtain a specific level of thermal insulation. In Part I of this series it was demonstrated that a model based on the reaction curve of first-order-with-dead-time, assuming that the addition of isocyanate can be represented as a step change of this process and registering the foam height as the output variable, can be a useful tool to describe the effect of filler content on the final volume of the foam. This kind of model avoids theoretical and complex models based on the physico-chemical phenomena usually used to describe the rising process of a PU foam [33–35]. Nevertheless, as can be seen in Fig. 1 the shape of the rising curves obtained with this new catalyst are convexes and thus, a model of reaction curve of first-order-with-dead-time does not describe it properly. Hence, the use of a new catalyst could change the type of the model that fits the rising curves of the RPU foams. Rodrigues [36] used a “tanks-in-series model” to reproduce the mass transfer by ion exchange that took place in a fixed bed column by dividing the column in several reactors with the same constant time. This model was quite advantageous because it allowed knowledge of the moment a zone of the column is exhausted.

The transfer function that represents the above mentioned model is shown in Eq. 1.

$$\frac{H(s)}{X(s)} = \frac{K \cdot e^{-\theta \cdot s}}{(\tau \cdot s + 1)^n} \tag{1}$$

where K is the maximum change of foaming height (h) for a unitary step change of the inlet variable x, τ is the constant time, θ is the dead time and n is the number of stirrer tank reactors.

Applying the inverse Laplace transform, Eq. 1 becomes:

$$h(t) = K \cdot \left[1 - e^{\frac{t-\theta}{\tau}} \cdot \sum_{j=1}^{n} \left(\frac{1}{(j-1)!} \cdot \left(\frac{t-\theta}{\tau} \right)^{(j-1)} \right) \right] \tag{2}$$

where j is a counter that represents each reactor of the tank-series.

This equation can be used to fit the rising curves of the RPU foams, using K, τ, θ and n as fitting parameters.

Figure 2 shows the influence of the value of n in the shape of the curve. As can be seen, when n is the unity, the shape of the curve is concave. Nevertheless, it becomes more convex with increasing n value. Thus, taking into account the convexity foam shape exhibited when the catalyst Tegoamin 33 is used, a value of n higher that 1 is required. Hence, the rising of the foam obtained using the catalyst Tegoamin 33 was fitted by assuming that the foam can be divided in a series of reactors.

On the other hand, in Part I of this series it was found that the three parameters (K, τ and θ) follow a linear relationship with respect to the weight percentage as shown in Eq. 3 [24].

$$y_i = a_i + b_i \cdot w_{\mathrm{PCM}} \tag{3}$$

where y is each of the three fitting parameters (K, τ and θ), a and b are constants of the linear equation, w_{PCM} is the content of PCM in the foam in percentage by weight and i is a counter that represents each fitting parameter.

Assuming that this linear relation continues to be valid, although the catalyst type is changed, Eq. 2 can be written in the form.

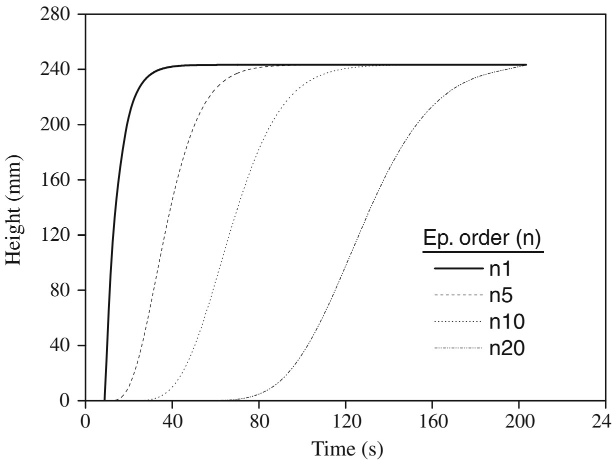

Fig. 2 Influence of the equation order of the reaction curve model, using the pure RPU foam parameters obtained with catalyst Tegoamin BDE: $\theta = 8.77$ s, $\tau = 5.99$ s, $K = 243.31$ mm

$$h(t)=(a_0+b_0\cdot w_{\mathrm{PCM}})\cdot\left[1-\sum_{j=1}^{n}\left(\frac{(t-(a_1+b_1\cdot w_{\mathrm{PCM}}))^{j-1}}{(j-1)\cdot(a_2+b_2\cdot w_{\mathrm{PCM}})^{(j-1)}}\cdot e^{\frac{t-(a_1+b_1\cdot w_{\mathrm{PCM}})}{(a_2+b_2\cdot w_{\mathrm{PCM}})}}\right)\right] \quad (4)$$

Thus, all the experimental rising curves were fitted together by non-linear regression to Eq. 4, in order to obtain reliable and meaningful values of the fitting parameters (a_0, a_1, a_2, b_0, b_1, b_2 and n). This fitting process was carried out using the *solver* tool in Microsoft excel and its accuracy was quantified by the statistical Student's t test assuming equal variances with 5% of significance level.

As observed in Table 2, a good fitting was obtained from a statistical point of view since the ratio t_c/t (t test) in all cases far exceeded the unity. Nevertheless, the higher value of ratio t_c/t (29.630) was found for a n value of 4, indicating that this foaming process can be reproduced by the reaction curve that represents a model of four tanks in series.

Figure 3 shows the experimental and predicting rising curves of the foams with the studied microcapsules contents. As can be seen, a good agreement between the experimental and theoretical data was obtained; except for the content of 21 wt%. In that case, the theoretical curve predicts a higher height. This behaviour can be explained because when a high amount of filler is added to the foam, these tend to agglomerate causing the cells to collapse leading to CO_2 losses [29]. Thus, it can be concluded that this equation is able to describe the rising process of foams synthesized with catalyst Tegoamin 33. Besides, the results shown in Fig. 4 indicate that the three parameters K, τ and θ of the reaction curve follow a linear relationship with the microcapsules content as it was reported in the previous manuscript [24].

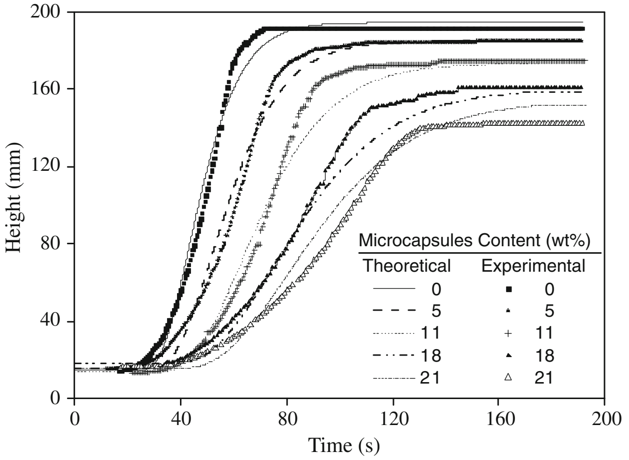

Fig. 3 Experimental and theoretical rising curves of the PU foams for different microcapsules contents

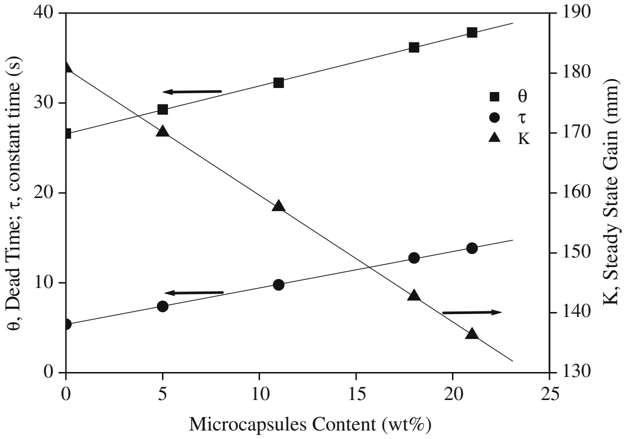

Fig. 4 Influence of the microcapsules content on the fitting parameters of the rising equation of the foams

Density

The density of the foam is an important parameter for building applications. Once the rising of the foam is quantified, it is possible to predict a theoretical density taking into account the effect of the fillers on the foaming process. The theoretical density was calculated by considering the mass added and the final volume of the foam.

Table 2 Parameters of equations and values of the ratio t_c/t obtained from the statistical Student's t test for different number of tank-in-series

Parameters	Number of tank-in-series (n)				
	1	3	4	5	10
a_0 (mm)	26.148	24.160	23.804	24.160	13.859
b_0 (mm/wt$_{\mathrm{PCM}}$%)	0.679	0.569	0.510	0.569	*−0.304*
a_1 (s)	25.461	8.565	6.469	8.565	3.547
b_1 (s/wt$_{\mathrm{PCM}}$%)	3.451	0.575	0.422	0.575	0.243
a_2 (s)	199.560	195.135	194.467	195.134	193.740
b_2 (s/wt$_{\mathrm{PCM}}$%)	0.009	−1.792	−1.918	−1.792	−2.002
t test statistical significance t_c/t	11.09	23.038	29.630	17.806	22.438

According to the geometry of the container used for synthesis purposes, the relationship between the foam volume and the instantaneous height can be expressed by Eq. 5, being the final foam volume obtained when the height is stabilized.

$$V(t) = \left(h(t) + 1.03 \times 10^{-2} \cdot h(t)^2 + 3.53 \times 10^{-5} \cdot h(t)^3\right) \cdot \pi \cdot R^2 \qquad (5)$$

where V is the instantaneous volume in mm^3, R is the maximum radius of the recipient where the foam was synthesized (60.5 mm in this work) and h is obtained by using the Eq. 4, both in mm.

The experimental and theoretical densities of the foams with different contents of microcapsules and their deviations are shown in Table 3. These results are in good agreement with those obtained in literature in which the experimental foam densities were always lower than the theoretical values due to the CO_2 mass losses produced during the formation of the foam structure [29, 35]. Besides, the maximum deviation of 7 wt% that was found for 5 and 18 wt% of microcapsules contents would indicate that the developed model can be used to predict the height of the foam.

On the other hand, it was observed that catalyst Tegoamin 33 promotes foams with higher densities than those of catalyst Tegoamin BDE [24]. These results can be explained taking into account that the foam density is mainly controlled by the amount of gas released by the blowing agent, being higher when catalyst Tegoamin BDE was used. Thus, the higher the gas formation rate, the lower the foam density and the larger and irregular the shape of the cells [27, 29, 37].

Nevertheless, it is important to point out that, regardless of the used catalyst, the foam density increased with the microcapsule content. This can be due to the large decrease in height caused by the collapse of the foam cells and the loss of CO_2 from the foam [29]. Moreover, all the synthesized foams satisfied the Spanish regulations for the use of foams in building applications (UNE 92120) that demands a minimum density of 30 kg/m^3.

Table 3 Experimental and theoretical density of the synthesized foams

Density (kg/m^3)	Microcapsules content (wt%)				
	0	5	11	18	21
Experimental	43.10	53.4	55.39	59.23	78.00
Theoretical	45.19	49.65	56.57	63.45	81.06
Exp. and theo. deviation (%)	4.85	7.02	2.14	7.12	3.92

Average latent heat of foams

Table 4 lists the latent heat values at different points of the foams obtained by DSC analysis. These results confirmed the presence of microcapsules in the foam because the analyzed samples showed a TES capacity higher than that of the pure foam. The theoretical average TES of the foams was calculated by means of Eq. 6.

$$\Delta H_{\mathrm{FPCMs_{Teo}}} = \frac{\Delta H_{\mathrm{mPCMs}} \cdot W}{\mathrm{FW}} \qquad (6)$$

where $\Delta H_{\mathrm{mPCMs}}$ is the microcapsule latent heat, $\Delta H_{\mathrm{FPCMs_{Teo}}}$ is the theoretical foam latent heat, W and FW are the microcapsules and foam weights, respectively.

The deviations between the experimental and theoretical average TES of the foams are quite high (up to 30% for the case of 5 wt% of microcapsules). The contrary effect was observed with catalyst Tegoamin BDE (Part I of this series) [24]. This high deviation can be due to the slower CO_2 formation that decreased the rising process and in this way the produced CO_2 was not enough to produce foams with a uniform microcapsules distribution.

On the other hand, the average TES capacity obtained for the foam containing a 21 wt% of microcapsules is 16 J/g, which is close to the TES capacity of 16.5 J/g reported by You et al. [25] for foams with a 22 wt% of microencapsulated *n*-octadecane and that of 18.5 J/g found in our previous paper [24] when catalyst Tegoamin BDE was used for the same amount and type of microcapsules.

SEM analyses

The structures of RPU foams obtained by SEM with magnification 50× are shown in Fig. 5. In the same way as when catalyst Tegoamin BDE was used, the foam cell sizes and its structures depended on the microcapsules content.

As can be seen in Fig. 5a–c, the foams presented polyhedral closed-cell structures with pentagonal or hexagonal faces containing nodes formed by the junction of four

Table 4 TES capacity of the foams with microencapsulated Rubitherm® RT27

Point of analysis	TES capacity (kJ/kg)			
	5%	11%	18%	21%
1	5.60	6.97	8.54	16.40
2	1.89	7.95	15.89	17.09
3	2.09	8.54	11.80	21.80
4	1.60	5.41	12.14	8.50
Average experimental TES (kJ/kg)	2.80	7.22	12.09	15.95
Average theoretical TES (kJ/kg)	4.00	8.80	14.40	16.80
Exp. and theo. deviation (%)	30.13	17.98	16.02	5.07

struts, three in the micrograph plane and the other out of the plane. Besides, it can be observed that the higher the microcapsules content, the lower the cell size to be promoted by the nucleating character of the added fillers [38–40]. On the other hand, Fig. 5d and e shows the rupture of struts, forming big holes into the foam. Thus, microcapsules contents higher than 11 wt% promote the agglomeration of microcapsules into the struts, weakening it and further driving its rupture. As commented in Part I of this series [24], these broken struts would explain the large reduction in the final foam height as a result of the carbon dioxide that escapes from these broken cells.

In order to know the effect of the microcapsules content on the internal characteristics of the foam, the Image plus program was used.

Figure 6 shows the cell-size distribution of all synthesized foams. As can be seen, the main cell-size of the foam without microcapsules had a size of 325 μm whereas the largest cell size of this synthesized foam was equal to 625 μm. On the other hand, the foams containing 5, 11 and 21 wt% presented a main cell size of 175 μm and that containing 18 wt% presents a main cell size of 125 μm, having also a high percentage at 175 μm. Besides, cells containing 18 and 21 wt% of microcapsules present a small

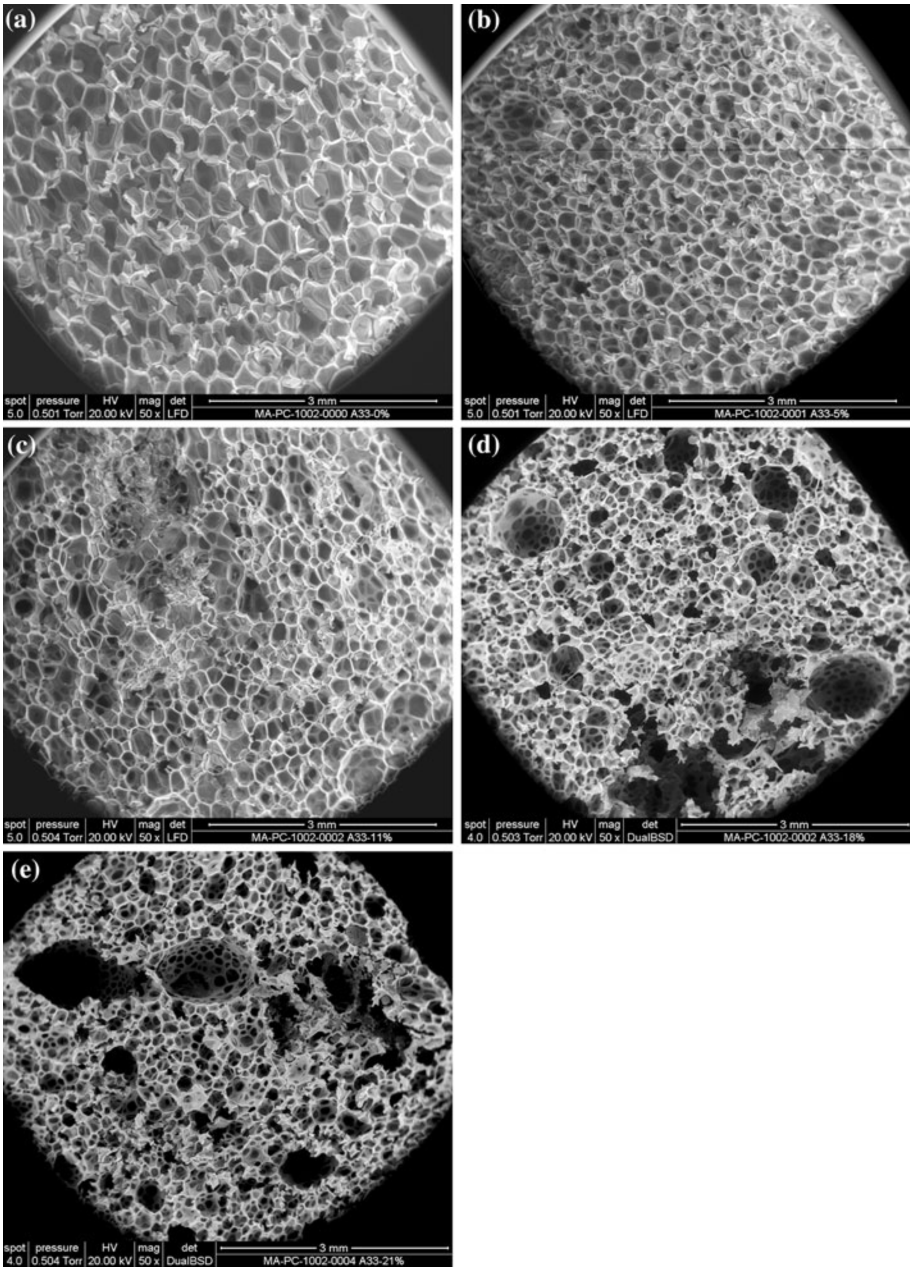

Fig. 5 SEM pictures with magnification ×50 of RPU foams with different microcapsules loadings **a** 0 wt%, **b** 5 wt%, c 11 wt%, **d** 18 wt%, **e** 21 wt%

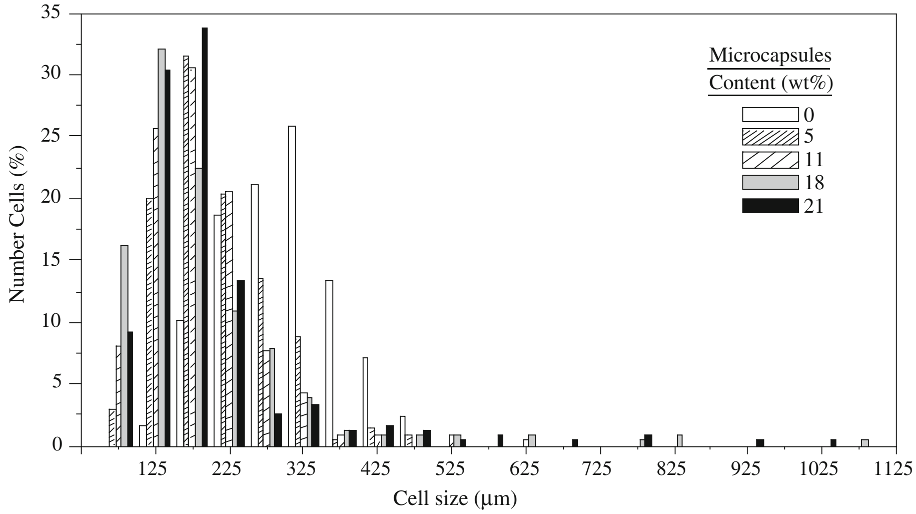

Fig. 6 Cell size distribution of the PU foams as a function of microcapsules content

number of cells quite much bigger than those observed in the rest of samples.

This lower cell-size exhibited by foams with microcapsules compared to that of the foam without fillers confirm the results obtained by other authors [38–40] who explained the diminishing in cell size by attending to the nucleating effect of the fillers during the cell formation.

Comparing cell sizes obtained with catalyst Tegoamin 33 with those produced by catalyst Tegoamin BDE in the Part I of this series (349.3, 157.2, 266.1 and 590 µm, for the 0, 11, 18 and 21 wt% respectively), a reduction in the cell size is obtained. These results would indicate that catalyst Tegoamin 33 produced foams of stronger structure.

Compression test

The specific or reduced compressive strength (RS) and modulus (RE) were calculated by dividing *S* and *E* by the sample density. Figure 7 shows the mechanical properties of the synthesized foams as a function of the fillers content.

As can be seen, both RS (Fig. 7a) and RE (Fig. 7b) decreased with the increase of microcapsules content but the reduction was really softer than when the catalyst Tegoamin BDE was used. Thus, although the particle-to-particle interactions appear at high filler contents promoting the particle agglomeration and increasing the number of collapsed cells, the stronger struts formed by using catalyst Tegoamin 33 increased the mechanical resistance of the foam. In this case, 66% of the RS and 84% of the RE of the foam without microcapsules were maintained even when 18 wt% of microcapsules was added. Only for 21 wt% of microcapsules, an abrupt reduction of 62 and 54% for the RS and RE, respectively, was obtained, but this

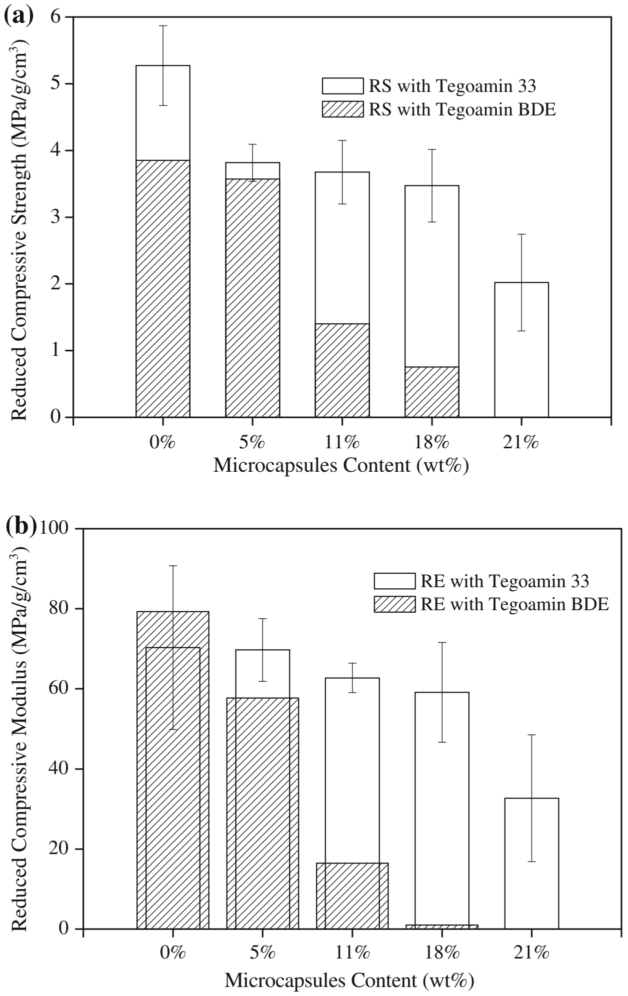

Fig. 7 Mechanical properties of RPU foams synthesized using Tegoamin BDE or Tegoamin 33 and incorporating different microcapsules contents: **a** reduced compressive strength, **b** reduced compressive modulus

reduction is even lower than that obtained for the foam containing an 11 wt% when catalyst Tegoamin BDE was used.

This reduction in the mechanical resistance was reported by Mahfuz et al. [41] when a low density filler such as SiC was used, observing that a high percentage of this filler decreased the mechanical properties of the foam.

It is also remarkable to point out that all the synthesized foams exhibited a RS and RE values similar as those reported in literature: from 0.2 to 8 and from 2 to 200 MPa/g/cm^3, respectively, using foams with densities in the range of the values required by the Spanish Regulation for building applications [38, 39, 42].

Hence, the higher mechanical properties of the synthesized foams using catalyst Tegoamin 33 would indicate that the initial crosslinking and the lower gas formation promoted the development of a foam with a higher mechanical resistance.

Conclusions

The use of Tegoamin 33 allowed to synthesize RPU foams containing microencapsulated Rubitherm® RT27 with proper TES capacities and better mechanical resistance and stiffness than those observed when catalyst Tegoamin BDE was employed. The different properties of catalyst Tegoamin 33, catalyzing both the gelling and the blowing reactions gave place to lower rising rates and heights than those for catalyst Tegoamin BDE. This way, a model of reaction curve of first order was not applicable, leading to the use of a more general model of reaction curve of *n* tank-in-series with the same time constant. The good agreement between the experimental rising data and the predicted values indicated that the model was appropriate. Besides, statistical analysis indicated that the fitting parameters obtained by non-linear regression were highly meaningful.

The decrease in the final height of the foam promoted an increase in the foam density compared with those of foams synthesized with catalyst Tegoamin BDE. It was observed that a foam containing up to a 18 wt% of microcapsules can be manufactured improving the TES capacity and holding the mechanical properties of the foam without fillers. Nevertheless, higher contents (21 wt%) resulted in a large reduction in mechanical properties but with RS and RE higher than those exhibited by the foams containing 11 wt% when catalyst Tegoamin BDE was used while maintaining the thermal properties. Finally, the TES capacities and the mechanical properties of the synthesized foams were in the range of those reported in literature. Besides, the foam densities satisfied the restriction established by the Spanish regulation for building applications.

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