

Characterization of Rigid Polyurethane Foams Containing Microencapsulated Phase Change Materials: Microcapsules Type Effect

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ABSTRACT: Rigid polyurethane (RPU) foams were synthesized incorporating up to 18 wt % of two different kinds of thermo-regulating microcapsules having a different shell material consisting of polystyrene or poly(methyl methacrylate), named as mSP-(PS-RT27) and Micronal[®]DS 5001X, respectively. The type of microcapsules and their content affected the final foam height, which decreased with the content and particle size. However, the foam rising curve shape was not dependent on the microcapsules type or content and was successfully predicted by means of a model of reaction curve of four tanks in series. Thermal energy storage (TES) capacity of PU foams was improved by incorporating both, mSP-(PS-RT27) or Micronal[®]DS 5001X, with the values close to those reported in the literature (16 J/g) for the highest content. Nevertheless, the highest particle size of the microcapsules from PS and the agglomeration of the microcapsules from poly(methyl methacrylate), promoted by their additive SiO₂, led to the strut rupture, damaging the final mechanical properties. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

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INTRODUCTION

According to the two last European Directives^{1,2} on the energy performance on buildings, the European Union considers the reduction of the energy consumption in buildings as a key point to achieve their climate and energy objectives. As the EU directive 2002/91/EC states,¹ in 2002 buildings were responsible for 40% of the final energy consumption and 36% of CO₂ emissions in the Europe Community. In the United States, where the energy consumption in buildings is about 30% of the total consumption, the situation is quite similar.³ This energy is mainly spent for buildings comfort and this consumption increases day by day with its subsequent contribution to global warming by the carbon dioxide emissions associated with it.

To achieve the development of more efficient buildings, new building concepts, designs, and materials are being studied.⁴ One of the most widely used insulating materials in buildings are RPU foams; owing to their low thermal conductivity, high mechanical and chemical stability, high durability, they are chlorofluorocarbon free and recyclable.^{5,6} However, their thermoregulation capacity could be enhanced by incorporating phase change materials (PCMs).⁷ PCMs are materials that can absorb or release the energy equivalent with their latent heat

when the temperature undergoes or overpasses the phase change temperature associated to them. Therefore, they can provide to the foams the ability to storage the solar energy that reaches the building surface to maintain the human comfort inside it.

In the previous studies, PCMs were successfully encapsulated by a polystyrene shell by means of a suspension-like polymerization (SP) technique.^{8–10} Furthermore, microcapsules of low-density polyethylene (LDPE) and ethylvinylacetate (EVA)-containing Rubitherm[®] RT27 were synthesized by Spray drying (SD) technique from LDPE and EVA copolymer following the method described in the Patent EP2119498.¹¹ Both types of microcapsules containing Rubitherm[®] RT27 were incorporated into gypsum blocks and PU foams, respectively, finding that the presence of the microcapsules into the wall enhanced the TES capacity and the insulating effect of wallboards.^{10,12,13} Thus, microcapsules containing PCMs can be used to modify the TES capacity of building materials, keeping the insulating properties.

The synthesis of PU foams is basically based on the condensation reaction between diols or polyols and diisocyanates or polyisocyanates in the presence of a catalyst. Blowing agents and surfactants are added to regulate the morphology of foam cells.¹⁴ It has been found in the literature that the incorporation

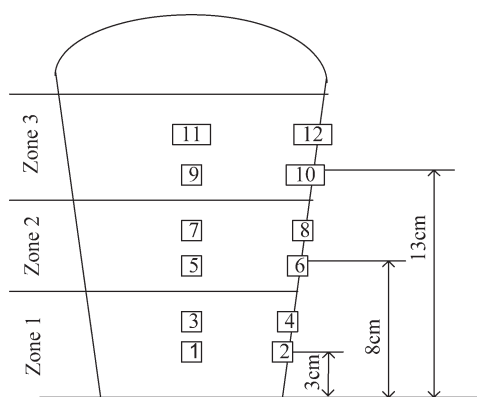


Figure 1. Distribution of the analyzed points and the considered zones of the PU foams for latent heat measurements.

of fillers into foams is effective to improve the properties and extend the applications of foams.¹⁵ Nevertheless, some problems have also been found with this addition. You et al.¹⁶ observed that microencapsulated *n*-octadecane using melamine-formaldehyde copolymers as shell material affected the surface tension and viscosity of the foaming system, having a negative effect on the physical properties of the foam. This negative influence was larger even at low concentration compared to the addition of other fillers such as CaCO₃, CaSO₄, or hollow glass microspheres. This behavior was justified on the basis of the chemical nature of the particle shell, as they have hydroxymethyl groups with a high ability for water absorption. These groups of the particle surface enter in competition with the hydroxyl groups of the polyol and affect the foaming system. In the same way, Ye et al.¹⁷ observed that a large size of the filler or a poor interfacial adhesion between the filler and the foam matrix can cause collapse of cells, reducing the mechanical properties of the RPU foams. Other authors have also found a reduction in the PU mechanical properties when carbon black, graphite nanosheets, expandable graphite, or SiC were incorporated as fillers.^{18–20} However, Zhang et al.²⁰ and Yu and Kim²¹ observed that the addition of expandable graphite-methyl methacrylate-acrylic acid copolymer particles and of multiwalled carbon nanotubes improved the mechanical properties of RPU foams and EVA copolymer foams, respectively, because these fillers had better compatibility with the foams matrix. Similar results were reported by Javni et al.²² who observed that Cloisite Na⁺ (natural montmorillonite) and nano-silica with a hydrophilic surface increased hardness and compression strength of the foams, whereas Cloisite_{10A} modified with a quaternary ammonium salt decreased the modulus, hardness, and compression strength. Additionally, results reported by Cao et al.,²³ König et al.,²⁴ and Harikrishnan et al.²⁵ indicated that other fillers such as organically modified montmorillonite, aluminum trihydroxide particles, or carbon nanofibers increased the compression strength of polyurethane foams.

In a previous article, it was found that the mechanical resistance of RPU foams is dependent on the catalyst used, being higher when catalyst Tegoamin 33 was used compared to those obtained using catalyst Tegoamin BDE.¹³ The aim of this work is to study the influence of the shell material of microcapsules containing PCMs added as fillers on the RPU foam properties

synthesized using catalyst Tegoamin 33. In this case, the materials to be tested were PS and poly(methyl methacrylate) (PMMA), whose influence on the foaming process and on the thermal and mechanical properties of PU foams has not been previously reported in the literature. The obtained results were compared to those previously published for PU foams containing mSD-(LDPE-EVA-RT27).

EXPERIMENTAL

Materials

Alcupol R-458 polyol from Repsol YPF S.A. is a polyether polyol of high functionality with a molecular weight of approximately 700 g/mol and a hydroxyl index of 380 mg KOH/g (according to the analysis following the Spanish regulation UNE 53985-1 : 2004). The used isocyanate was diphenylmethane-4,4'-diisocyanate supplied by Merck Group Catalyst Tegoamin 33 and surfactant Tegostab B8404 were supplied by Evonik Degussa International AG, (Barcelona, Spain). Deionized water was used as the blowing agent. Two new types of microcapsules containing PCMs were used in this study: mSP-(PS-T27) and Micronal[®]DS 5001X. mSP-(PS-RT27) were synthesized in our lab facilities by suspension polymerization technique using the paraffin wax Rubitherm[®]RT27 as core material, and, according to the LALLS analyses, their average particle size is 116.8 μm in number (dp_{n,0.5}) and 584.0 μm in volume (dp_{v,0.5}). Micronal[®]DS 5001X are commercial microcapsules supplied by BASF, (Ludwigshafen, Germany) and they were selected for this study as they have proven to be adequate for increasing the TES capacity of concrete.²⁶ Micronal[®]DS 5001X contains a paraffin wax of melting point close to 26°C as the core material and a shell from PMMA with dp_{n,0.5} and dp_{v,0.5} of 7.1 and 77.2 μm, respectively.

RPU Foam Synthesis

RPU foams were synthesized by weighting and mixing of desired masses of polyol, surfactant, water, catalyst, and microcapsules containing PCMs and further stirring during 1 min at room temperature in a plastic container having the geometry shown in Figure 1. Then, the adequate quantity of isocyanate was added to the mixture and the resulting solution was stirred for just 5 s until the moment the foam started to grow up. Finally, the obtained foams were cured at room temperature. Table I summarizes the foam synthesis recipe.

Sample Characterization

Scanning Electron Microscopy Analysis. Synthesized RPU foams were depicted by scanning electron microscopy (SEM) (FEI QUANTA 200, USA). The average cellular size of each foam was determined by the software Image Plus.

Table I. Weight Percentage of Raw Materials for PU Foams Synthesis

	Weight percentage
Polyol	48.43
Water	1.21
Surfactant	0.72
Catalyst	1.21
MDI	48.43

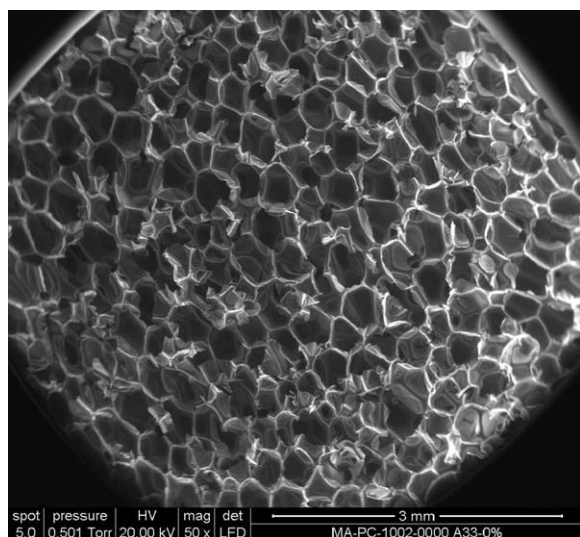


Figure 2. SEM images with 50× magnification of a PU foam without microcapsules.

Rising Process. The foams' rising process was measured by an ultrasonic system sqs-01 (Dr D. Wehrhahn, Germany) recording the foam height with time by using the program Schaum SQS-01.

Average Latent Heat. The microcapsules presence and their amount into the foam can be determined by measuring the area of the peak of the PCM melting transition. Differential scanning calorimetry (DSC, Q100 TA Instruments, USA) analyses of the samples were performed from -10 to 45°C at a heating rate of $5^{\circ}\text{C}/\text{min}$. The average latent heat of each one of the three foams zones shown in Figure 1 was calculated by the analyses of four different samples taken from them. The theoretical average TES of the foams was calculated by means of eq. (1):

$$\Delta H_{\text{FPCM}_{\text{ST}_{\text{teo}}}} = \frac{\Delta H_{\text{mPCM}_{\text{s}}} \cdot W}{F_W} \quad (1)$$

where $\Delta H_{\text{mPCM}_{\text{s}}}$ is the microcapsule latent heat, $\Delta H_{\text{FPCM}_{\text{ST}_{\text{teo}}}}$ is the theoretical foam latent heat, W and F_W are the microcapsules and foam weights, respectively.

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 crosshead speed of 2.5 mm/min and transversal to the rising direction. The tested foam specimens were $5.1 \times 5.1 \times 2.6$ cm size and they were compressed 13% of their original thickness. Each analysis was repeated three times. The foam compression strength (S) was obtained using the force required to get a 10% of specimen deformation and 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. The reduced compressive strength (RS) and modulus (RE) were calculated by dividing S and E by the sample density.

RESULTS AND DISCUSSION

SEM Analysis

The structure and cell size of a rigid PU foam without microcapsules is shown in Figure 2. It can be seen that presented are polyhedral closed-cell structures with pentagonal or hexagonal faces containing nodes formed by the junction of four struts, three in the micrograph plane, and other out the plane, with an average cell size value of $295.4 \mu\text{m}$.

SEM images of RPU foams with different amounts of mSP-(PS-RT27) are shown in Figure 3. Foam cell sizes and its structure seemed to be dependent on the microcapsules content. The cell size was reduced with the addition of 5 wt %, respect to the foam without fillers (from 295.4 to $238.8 \mu\text{m}$). So did, when mSD-(PEBD-EVA-RT27) was added.¹³ This reduction can be explained attending to both the behavior of the microcapsules as nucleation agents²⁷ and the increase in the foam viscosity.⁶ However, the cell size increased for the rest of microcapsules contents (405 and $535 \mu\text{m}$ for the 11 and 18 wt %, respectively) appearing big holes caused by struts rupture. The reason for this increase can be related to the average particle size of these microcapsules ($\text{dp}_{\text{n}_{0.5}} = 117 \mu\text{m}$ and $\text{dp}_{\text{v}_{0.5}} = 584 \mu\text{m}$), many of which were bigger than the average cell size of the PU foam free of fillers, leading to the rupture and the increase in size of the foam cells. Furthermore, it is observed a change in the wall morphology and appearance. Cells did not present polyhedral shape and the walls were not smooth but with some grains. These spherical shapes can be perfectly appreciated in the SEM images taken with $200\times$ magnifications (Figure 4). Instead of appearing microcapsules, it seemed that they were occupying a whole cell and were wrapped by the cell.

Figure 5 shows the SEM images of foams containing Micronal[®]DS 5001X. It can be observed that for microcapsule contents up to 11 wt %, the average foam cell size decreases (197 and 171 for microcapsules contents of 5 and 11 wt %, respectively), confirming that the fillers act as nucleating agents, whereas higher microcapsules content promotes cell rupture causing big holes.

Figure 6 shows that Micronal[®]DS 5001X presented a different behavior than those of mSD-(PEBD-EVA-RT27) or mSP-(PS-RT27), as they tended to form agglomerates that filled the foam cells causing some struts rupture (presence of big holes). This agglomeration of the PMMA microcapsules is opposite to the results presented by Ye et al.,¹⁷ who stated that the PMMA has a good compatibility with the PU foams matrix, being even used to improve the compatibility of fillers such as expandable graphite particles.

Figure 7 shows the TGA for the three studied microcapsules using N_2 as carrier gas. As it can be seen, independently on the microcapsules type, TGA curves present two steps of weight loss; the first one between 150 and 250°C , corresponding to the core material evaporation and the second step owing to the polymer degradation, between 370 and 480°C , that depends on the polymer network. Thus, these analyses confirm the temperature of degradability of the polymers as $\text{PMM} < \text{PS} < \text{LDPE-EVA}$ according to the values found in the literature.^{28,29} On the other hand, the weight loss owing to the core material

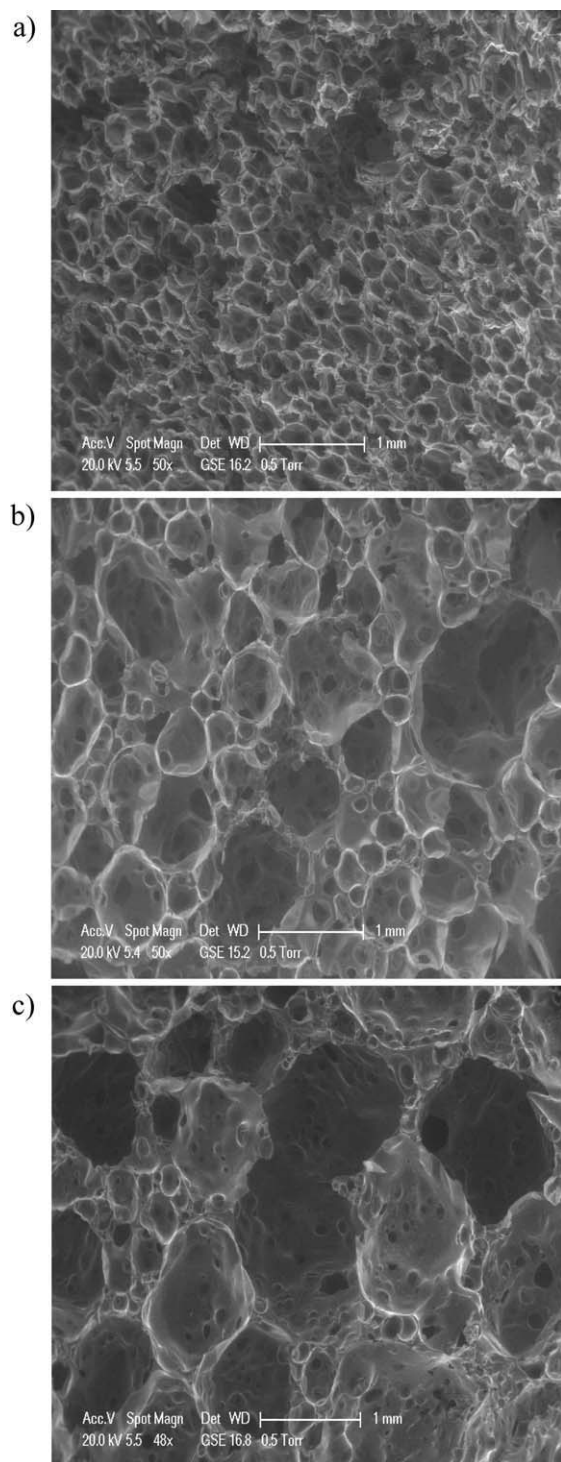


Figure 3. SEM images with 50 \times magnification of PU foams with different microcapsules loadings of mSP-(PS-RT27). (a) 5 wt %; (b) 11 wt %; (c) 18 wt %.

evaporation agrees with the paraffin weight percentage obtained from the latent heat of fusion by DSC (Table II). Besides, although the mSP-(PS-RT27) and mSD-(PEBD-EVA-RT27) were almost completely degraded for a temperature of 500 $^{\circ}$ C, indicating that they are fully formed by organic compounds; the PMMA analysis presented a residue of an 8.8 wt %, which

would correspond to inorganic additives added to these microcapsules. An X-ray fluorescence analysis of the Micronal[®]DS 5001X of this residue showed that it is mainly formed by SiO₂ (94.9%) and also Na₂O (4.03%), Al₂O₃ (0.32%), and traces of Cl and other metals. This presence of the SiO₂ justifies the agglomeration of the Micronal[®]DS 5001X, owing to its different polarities with respect to the foam matrix that hampers the microcapsules dispersion.³⁰

According to these results, the shell materials, their additives, and the particle size of the filler had a big influence on the way how these materials were incorporated into the foam. These SEM results confirmed that the rupture of the foam cells was promoted by big particles like those from mSP-(PS-RT27) or by low compatibility (Micronal[®]DS 5001X) with foam materials that caused the agglomeration and a worst distribution of fillers into the foam.

Foam Growth

Figure 8(a,b) shows the experimental rising curves of the PU foams containing different contents of mSP-(PS-T27) and Micronal[®]DS 5001X, respectively. The foaming rate decreased when increasing microcapsules content, as a lower slope of the curve at the short-time region was observed. Regardless the microcapsules type, the higher the microcapsules content, the lower the final foam height. Results agree well with those reported in the previous studies.^{12,13,24,31,32} This effect can be explained by the increase in the foaming system viscosity caused by the presence of the fillers,^{24,27} the nucleation effect of the microcapsules,²⁷ the higher amount of mass that has to be lifted by the same expanding gas generated during the foaming process, and by its less cell expansion.²⁴ The lower cell expansion is attributed to the absorption by the PCM of part of the heat released during the chemical reactions of this process what decreases the reaction temperature and reduces the expansion volume of the CO₂ cell gas.²⁴ On the other hand, Figure 9 shows a comparison of the final heights and foaming rates of

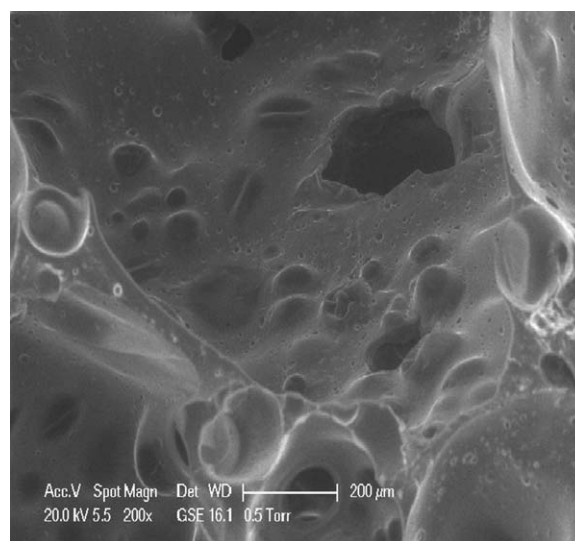


Figure 4. SEM images with 200 \times magnification of PU foams containing 18 wt % of mSP-(PS-RT27).

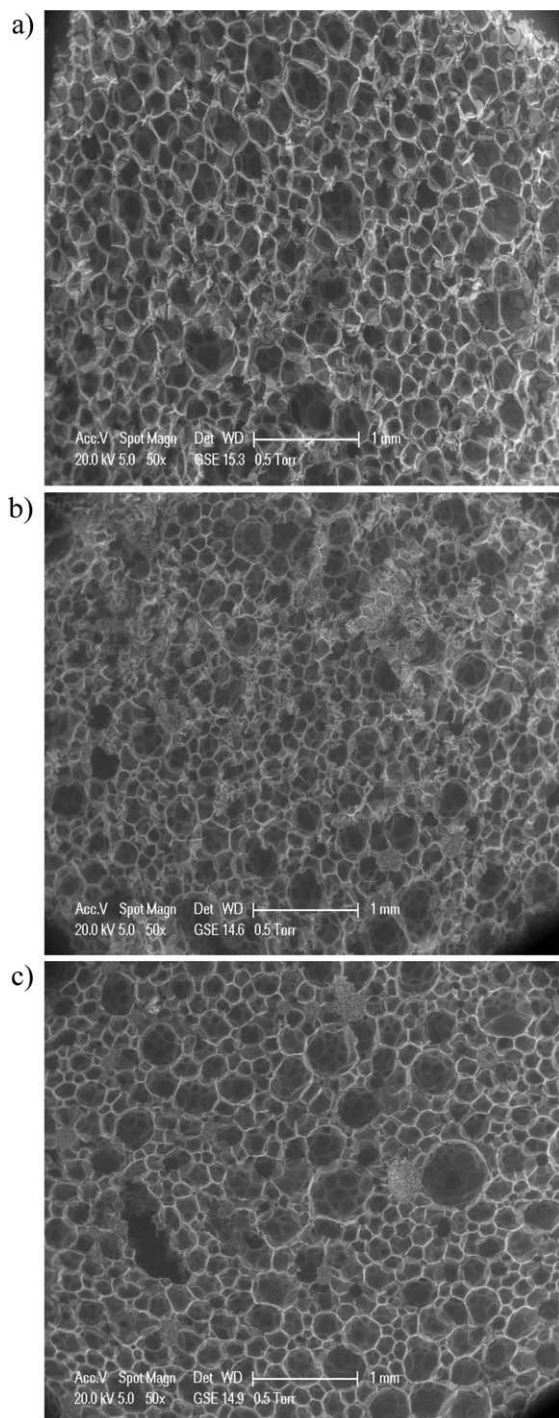


Figure 5. SEM images with 50× magnification of PU foams with different microcapsules loadings of Micronal®DS 5001X. (a) 5 wt %; (b) 11 wt %; (c) 18 wt %.

these two microcapsules types with those obtained with mSD-(LDPE-EVA-RT27). Figure 9 confirms the linear relationship between the final foam height and the amount of added filler that has been found in the previous studies.^{12,13} Equation (2) shows the linear relationship between the final foam height and the filler weight percentage:

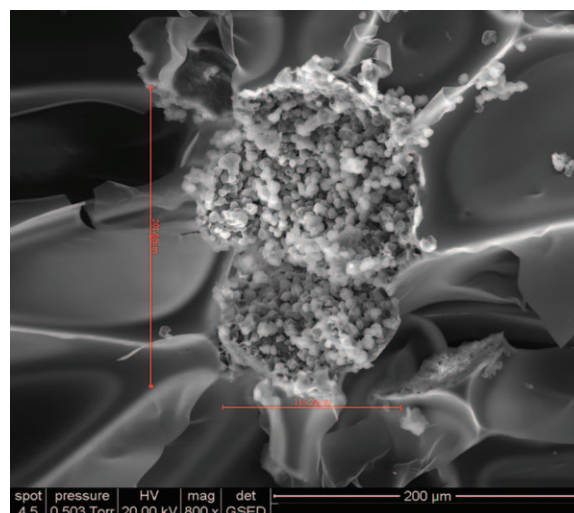


Figure 6. SEM images with 800× magnification of PU foams containing 18 wt % of Micronal®DS 5001X. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

$$h(t = \infty) = k = (a_0 + b_0 \cdot w_{PCM}) \quad (2)$$

where $h(t = \infty)$ and k are the final foam height, w_{PCM} is the foam content of microcapsules in percentage by weight and a_0 and b_0 are the fitting parameters.

The values of b_0 for all the studied fillers were obtained by linear regression fixing a_0 as the final height of the foam free of fillers (194.5 mm). The value of b_0 (the slopes of these curves) were -1.918 , -1.594 , and -0.922 for the mSD-(LDPE-EVA-RT27), mSP-(PS-RT27), and Micronal®DS 5001X, respectively, indicating that there was an increasing influence of the microcapsules on the final height as follows: mSD-(LDPE-EVA-RT27) > mSP-(PS-RT27) > Micronal®DS 5001X. According to the characteristics of the materials, these differences can be attributed to either the particle size or the shell material. The small particle size and good compatibility of mSD-(LDPE-EVA-RT27)

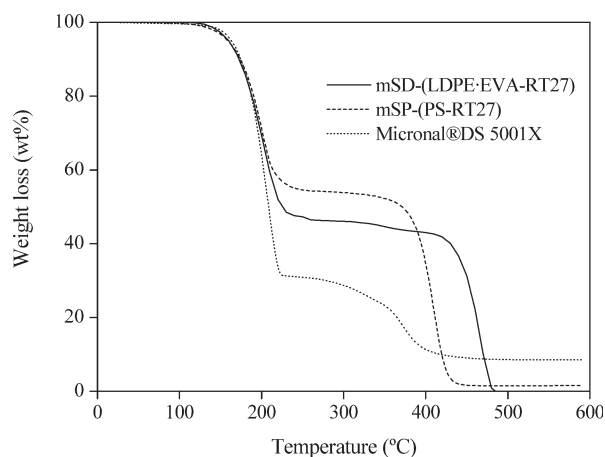


Figure 7. TGA analysis of the studied fillers.

Table II. DSC Analyses Results of the Three Microcapsules Types

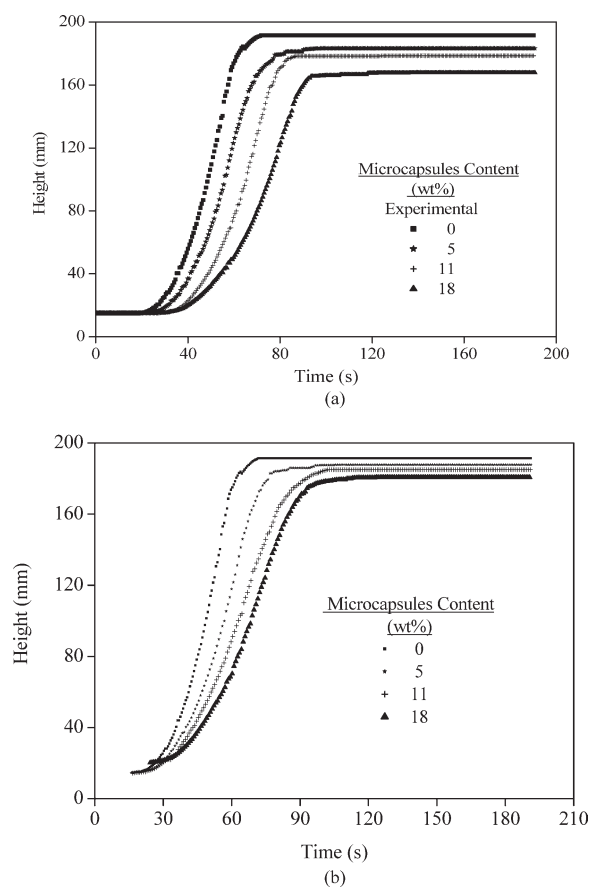
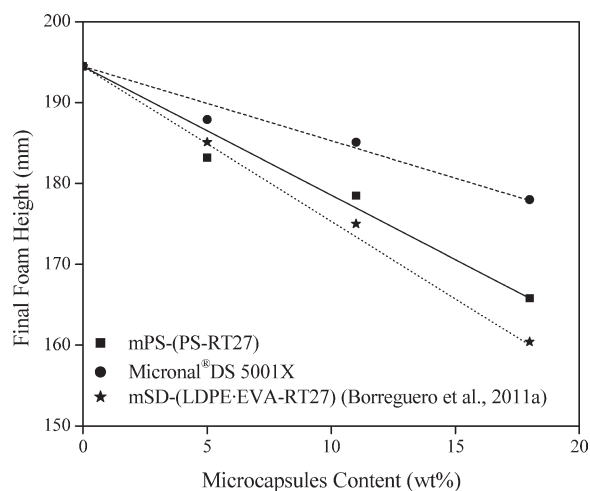
Microcapsules type	T_f (°C)	ΔH_f (J/g)	Core material content (wt %)
mSD-(LDPE-EVA-RT27)	28.40	98.14	49.32
mPS-(PS-RT27)	28.46	96.74	48.61
Micronal®DS 5001X	27.67	116.2	Unknown

promoted their good distribution into the PU foam. Borreguero et al.¹³ reported that they would act as nucleation agents, reducing the cell size and consequently the final height.

On the contrary, the reduction of the final height caused by the presence of mSP-(PS-RT27) must be attributed to their larger particle size that, as commented above, made more cells to collapse, losing the CO₂ responsible of the foams growth.¹⁴

In the case of the Micronal®DS 5001X, despite of presenting the lowest particle size, they suffered a heterogeneous distribution owing to their lower compatibility with foaming system, as observed in the SEM analyses. Thus, their effect on the foaming system viscosity, on the gas diffusion and on the final foam height, is lower than that caused by mSD-(LDPE-EVA-RT27).

On the other hand, the shapes of the rising curves are convexes and similar to those obtained using mSD-(LDPE-EVA-RT27) as

**Figure 8.** Experimental rising curves of PU foams containing different weight percentages of (a) mSP-(PS-RT27) and (b) Micronal®DS 5001X.**Figure 9.** Final heights of the PU foams depending on the filler type.

filler, which was related to the catalyst used. Thus, it is possible to apply the “tank-in-series model” (eq. (3)) developed previously¹³ to predict the foam growth as a function of the microcapsule content of mSP-(PS-RT27) or Micronal®DS 5001X. According to that study, the fitting parameters θ (dead time) and τ (constant time) have a linear relationship with the microcapsules content and the proper value of the number of tank in series is 4 when the catalyst Tegoamin 33 is used.

$$h(t) = k \cdot \left[1 - \sum_{j=1}^4 \left(\frac{(t - \theta)^{j-1}}{(j-1) \cdot \tau^{(j-1)}} \cdot e^{-\frac{t-\theta}{\tau}} \right) \right]$$

$$= k \cdot \left[1 - \sum_{j=1}^4 \left(\frac{(t - (a_1 + b_1 \cdot w_{PCM}))^{j-1}}{(j-1) \cdot (a_2 + b_2 \cdot w_{PCM})^{(j-1)}} \cdot e^{-\frac{t-(a_1+b_1 \cdot w_{PCM})}{(a_2+b_2 \cdot w_{PCM})}} \right) \right] \quad (3)$$

where h is the foam height, k is obtained from eq. (2), w_{PCM} is the foam content of microcapsules in percentage by weight, j is a counter that represents each reactor, t is time, and a_1 and b_1 (fillers effect on the cream time) and a_2 and b_2 (fillers effect on the foam rising rate) become fitting parameters related with θ and τ , respectively.

The fitting parameters a_1 and a_2 were obtained for the foam free of filler in a recent study¹³ and the unique unknown parameters, b_1 and b_2 , were calculated by nonlinear regression. Figure 10 shows a good agreement between the experimental data and the predicted ones for foams containing the 11 wt % of the three different types of microcapsules and the fitting parameters values are listed in Table III. b_1 -Values indicated that a large particle size of the filler increases the cream time as thus, mSP-(PS-RT27) damp the bubble formation, whereas mSD-(LDPE-EVA-RT27) and Micronal®DS 5001X presented similar effect. On the other hand, b_2 -values showed a similar rising rate for the foams containing mSP-(PS-RT27) and Micronal®DS 5001X, whereas the foaming reaction with mSD-(LDPE-EVA-RT27) was clearly slower, which could be associated to the increase of the foaming system viscosity and nucleating effect caused by this kind of filler that promotes a slower gas diffusion.^{33,34}

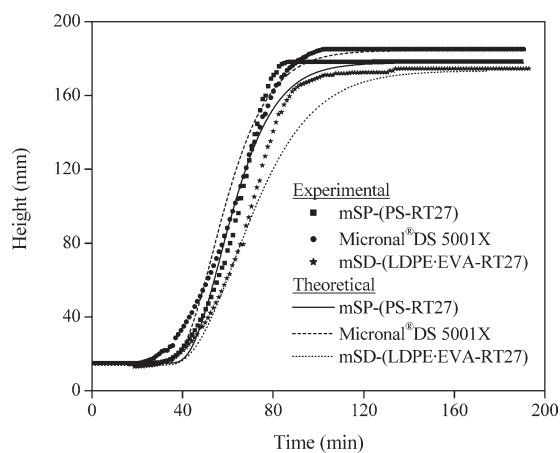


Figure 10. Experimental and theoretical rising curves of PU foams containing 11 wt % of the different fillers.

Average Latent Heat

Table IV lists the values of the latent heats of the foams obtained by DSC analysis at different zones shown in Figure 1. As expected, the higher the microcapsules content, the higher the latent heat. Thus, TES capacity of the PU foams can be improved by adding these microencapsulated PCMs. Besides, these results confirmed the presence of microcapsules in the whole foam because the TES capacities of all the analyzed samples were higher than that of the pure foam. However, there is a latent heat profile along the height of the foams, finding higher TES capacities at the bottoms of the foams (zone 1) than at the top, indicating that the produced CO₂ was not enough to drive up the microcapsules to the whole foam and achieve a uniform microcapsules distribution. This is also confirmed by the remarkable deviation between the experimental and the theoretical TES capacities. According to these results, mSP-(PS-RT27) were worst distributed inside the foam, justified by their larger particle size, whereas the Micronal®DS 5001X agglomeration had a less significant effect on their distribution.

Table IV. TES Capacity of the Foams Containing Microencapsulated PCMs

Microcapsules type	Content (wt %)	5	11	18	
mSP-(PS-T27)	Zone 1	2.24	10.74	10.68	
	Zone 2	1.43	7.94	8.69	
	Zone 3	0.95	4.86	8.52	
	Global	$\overline{\Delta H_f^{exp}}$ (kJ/kg)	1.54	7.84	9.30
		ΔH_f^{teo} (kJ/kg)	4.01	8.77	14.26
		Deviation between $\overline{\Delta H_f^{exp}}$ and ΔH_f^{teo} (%)	61.59	10.56	34.82
Micronal®DS 5001X	Zone 1	5.95	12.81	17.85	
	Zone 2	4.22	9.30	16.86	
	Zone 3	2.65	8.15	13.93	
	Global	$\overline{\Delta H_f^{exp}}$ (kJ/kg)	4.27	10.09	16.21
		ΔH_f^{teo} (kJ/kg)	5.52	11.50	17.69
		Deviation between $\overline{\Delta H_f^{exp}}$ and ΔH_f^{teo} (%)	22.62	12.24	8.36

Table III. Rising Curves Parameters

Parameters	Microcapsules type		
	mSD-(LDPE-EVA-RT27) ^a	mSP-(PS-RT27)	Micronal®DS 5001X
b₁ (s/W _{PCM})	0.510	0.746	0.467
b₂ (s/W _{PCM})	0.422	0.155	0.148

^aFrom Borreguero et al.¹⁰

On the other hand, the largest difference between the theoretical and the experimental values of storage energy capacity was observed for the foams containing a 5 wt % of microcapsules, which is owing to the small sample size required for the analyses.

It is important to point out that synthesized foams containing 18 wt % of Micronal®DS 5001X had a TES capacity close to 16 J/g. These values are similar to those reported by other authors (16.5 J/g), when microcapsules containing *n*-octadecane were added as the filler¹⁶ or those reported on previous articles when a 21 wt % of mSD-(LDPE-EVA-RT27) were incorporated into foams synthesized by using Tegoamin BDE or Tegoamin 33 as catalyst.^{12,13}

Compression Test

Figure 11 shows the mechanical properties of the synthesized foams depending on the filler type and content.

When adding up to an 11 wt % of mSP-(PS-RT27), the PU foams exhibit good mechanical properties, with a RE similar to those of PU foams containing mSD-(LDPE-EVA-RT27), but with a RS lower than 25%. However, their mechanical properties decreased abruptly when 18 wt % was added, compared to the properties of the foams with the mSD-(LDPE-EVA-RT27). This higher decrease in the mechanical properties was a consequence of the rupture of more struts owing to the higher particle of the microcapsules size, as mentioned above.

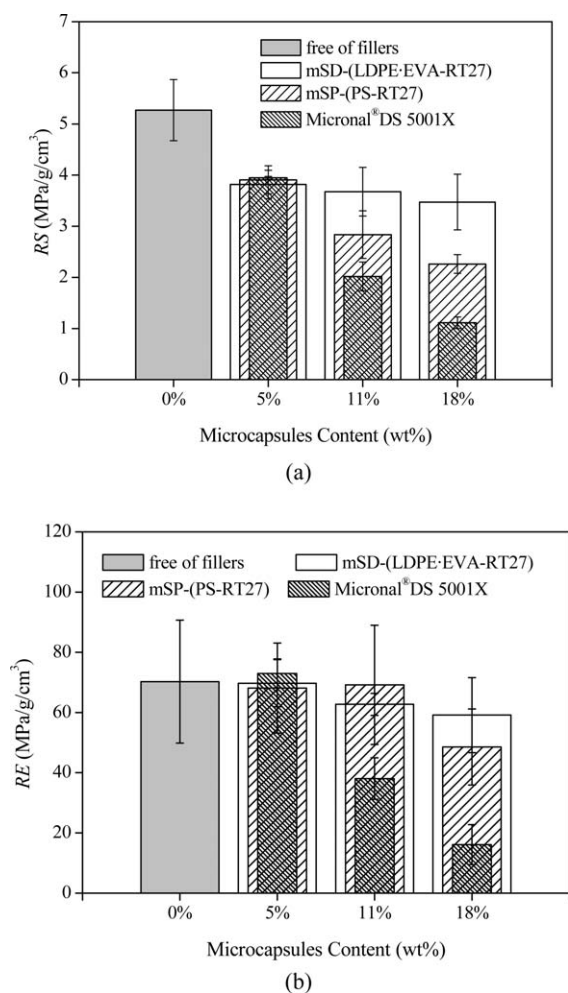


Figure 11. Mechanical properties of PU foams with different types and contents of microcapsules. (a) Reduced compressive strength (RS); (b) reduced compressive modulus (RE).

Despite of their lower average particle size, Micronal®DS 5001X led to a remarkable decrease in the RS and RE values (up to 78.9 and 77.1%, respectively, for a microcapsule content of 18 wt %) as a consequence of the previously mentioned agglomeration phenomenon that makes decreasing the strength of the composite foams.¹⁵ Furthermore, their lower cell size with higher final heights involves a reduction in the struts thickness and the consequent poorer mechanical resistance. From these results, it can be established the following sequence in terms of damaging the mechanical properties: mSD-(PEBD-EVA-RT27) < mSP-(PS-RT27) < Micronal®DS 5001X.

CONCLUSIONS

In this study, PU foams containing up to an 18 wt % of mSP-(PS-RT27) and Micronal®DS 5001X were prepared, showing a TES capacity similar to those reported in the literature. The final foam height variation resulted to be linear with the microcapsule content but it was also the function of the microcapsule type and the particle size. This way, the following order in the effect on this property was established: mSD-(LDPE-EVA-RT27) > mSP-(PS-RT27) > Micronal®DS 5001X. The rising process

was successfully predicted by means of a model consisting of four tanks in series, indicating that the foam rising curve shape was mainly dependent on the catalyst used. Contents of mSP-(PS-RT27) higher than 5 wt % promoted foams with higher average cell size. The three types of microcapsules exhibited similar mechanical properties for a content of 5 wt %. Higher contents led to a decrease in the values of these properties, being remarkable for foams containing Micronal®DS 5001. The Micronal®DS 5001 promoted a lower cell size than the mSD-(LDPE-EVA-RT27) with higher final heights, and therefore, their struts have to be considerably thinner and they showed poor mechanical resistance. This fact was associated to the agglomeration of microcapsules caused by the low compatibility between the SiO₂ contained in these microcapsules and the foam matrix. Microcapsules obtained from LDPE and EVA allowed to obtain foams with improved TES capacity (16 J/g) while minimizing the effect of the fillers on the mechanical properties of final foams.

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