



# Development of smart gypsum composites by incorporating thermoregulating microcapsules



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## ABSTRACT

Smart gypsum composites were manufactured by adding different kinds of microcapsules containing phase change materials (PCMs) in order to develop building materials with high thermal energy storage (TES) capacity useful for being applied in high comfort constructive systems. The physical, thermal and mechanical properties of these composites such as density, porosity, thermal stability, thermal conductivity ( $k$ ), equivalent heat capacity ( $c_p$ ), the accumulated heat power ( $q_{acc}$ ) and the maximum compressive strength were studied. Results showed that the higher the microcapsules content, the lower the density and  $k$  and the higher the  $c_p$  and  $q_{acc}$ , due to the PCM action. Besides, the addition of 15 wt% of microcapsules respect to the hemihydrate would allow to save 4.5 kWh of energy per operating cycle in a standard room covered with 1 m<sup>3</sup> of this gypsum. This energy is equivalent to the energy spent by three incandescent light bulbs of 60-W kept on all the day and a reduction of 1.395 kg of CO<sub>2</sub> emissions to the atmosphere. The addition of these thermoregulating materials to gypsum decreases their compressive strength but all the developed materials satisfied the Spanish mechanical regulations for gypsum as building material, being possible to increase the total amount of added microcapsules.

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## 1. Introduction

The social concern about the consequences of global warming is increasing the regulation requirements for the energy consumption in most of the developed countries, promoting the reduction of the fossil fuels use. It is known that buildings are responsible of close to 40% of the final energy consumption and also the 36% of CO<sub>2</sub> emissions in the Europe Community in 2002 [1] and the EU directive 2010/31/UE [2] focus its environmental objectives in the reduction of the total emission of the greenhouse gases in at least a 20% in 2020 with respect to that of the year 1990. A similar situation is found in the USA, since there buildings consume the 30% of the total energy consumption [3]. The energy consumption is well related with the CO<sub>2</sub> emissions and according to the Energy National Center of Spain (CNE), 0.31 kg of CO<sub>2</sub> are emitted to the atmosphere per kWh of produced energy [4].

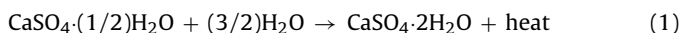
The fossil fuel consumption in buildings could be minimized by the application of the solar energy but this energy is intermittent and its exploitation requires the development of proper technologies to storage it. A noteworthy alternative for storage this energy in buildings is the use of thermoregulating materials, whose inversion is more than recovered considering their economic and environmental benefits. The final properties of the building envelopes containing these materials must be analyzed in order to evaluate the suitability and real gains of the constructive system. Several authors have proposed the incorporation of phase change materials (PCMs) in order to regulate the inside building temperatures by storing or releasing the solar energy proportional to their latent heat of fusion depending on the external temperature. This way, a PCM stores energy at temperatures higher than its melting point but when the temperature outside the building goes down to the PCM melting temperature, it releases the previously stored energy [5–7]. One of the building materials used for the PCM incorporation is gypsum; which is environmental friendly, fire resistant, esthetics, presents low prices and can be applied in situ or as precast slabs. Besides, this material can be used for interior wall, ceiling or exterior if proper hydrophobization treatment of the wall

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**Table 1**  
Microcapsules data.

Product	$dp_{n0.5}$ ( $\mu\text{m}$ )	$dp_{v0.5}$ ( $\mu\text{m}$ )	$T_f$ ( $^{\circ}\text{C}$ )	$\Delta h_f$ (J/g)
mSD-(LDPE-EVA-RT27)	$3.9 \pm 0.11$	$10.0 \pm 0.42$	$28.40 \pm 0.90$	$98.14 \pm 2.17$
mSP-(PS-RT27)	$116.8 \pm 2.84$	$584.0 \pm 17.05$	$28.46 \pm 2.74$	$96.74 \pm 1.95$
Micronal <sup>®</sup> DS 5001X	$7.1 \pm 2.32$	$77.2 \pm 21.06$	$27.67 \pm 0.94$	$116.2 \pm 4.11$
mSD-CNFs	–	–	$27.6 \pm 1.55$	$95.64 \pm 5.73$

is accomplished [8]. From the chemical point of view, gypsum is calcium sulfate dihydrate obtained by adding water to the calcium sulfate hemihydrate (hemihydrate) in powder according to the stoichiometric reaction (Eq. (1)) [9].



Different authors have studied the addition of PCMs directly into the building materials and they have observed that PCMs are easily adsorbed into porous concrete or in the polyurethane foams matrix, enhancing the thermal energy storage (TES) capacity of the facade and reducing the consumed energy [10–17]. Unfortunately, in the direct application the PCMs can interact with the rest of materials and leak when remaining melted [18]. These problems can be solved by the PCMs encapsulation, it is putting them into containers, before their incorporation in the construction materials [19,20]. Microencapsulation of PCMs with polymeric shells stands out as one of the best encapsulation options for this particular application [21]. Additionally, the microencapsulation allows to increase the heat-transfer area, control the variations of volume during the change of phase and avoid deleterious effect on the mechanical properties of building materials [22]. Therefore, the development of a gypsum block with high TES capacity by means of the addition of microencapsulated PCMs into the hydrating hemihydrates could lead to a reduction of the energy demand in the residential and tertiary sectors [23].

A commercial product with this technology has been manufactured and distributed by the chemical company BASF. This gypsum wallboard is named Knauf Gips KG's PCM SmartBoard<sup>®</sup> and contains 3 kg of Micronal<sup>®</sup>PCM per square meter and 15 mm of thickness [24]. Toppi and Mazzarella [9] synthesized gypsum wallboards containing PCMs by using the commercial product of BASF DS 5000 which consists of a dispersion of Micronal<sup>®</sup>PCM having a solid content of 42 wt%. They found that even a very small amount of microcapsules presents a big effect on the gypsum viscosity and provides crystallization nuclei, promoting the gypsum solidification and modifying the composite material properties. The microcapsules addition increases the porosity and reduces the thermal conductivity. Nevertheless, the effects of thermoregulating microcapsules having different shell materials on the thermal and mechanical properties of the lightweight gypsums have not been previously reported in literature.

In previous works, microencapsulated PCMs with a polystyrene shell were successfully synthesized by means of a suspension like polymerization (SP) technique and called mSP-(PS-RT27) [25,26,6]. Furthermore, the microcapsules mSD-(LDPE-EVA-RT27), with shell from low density polyethylene (LDPE) and Ethyl vinyl acetate (EVA) and with Rubitherm<sup>®</sup>RT27 as core material, were prepared by Spray drying (SD) technique following the process described in the Patent EP2119498 [27]. Both types of microcapsules containing Rubitherm<sup>®</sup>RT27 were incorporated into rigid polyurethane foams, finding that the presence of the microcapsules into the foam enhanced the TES capacity and the insulating effect of wallboards [28–30]. Thus, these microencapsulated PCMs can be used to increase the TES capacity of building materials while keeping the insulating properties [28–30].

The aim of this paper is to develop smart gypsum blocks by adding three different kinds of microcapsules containing PCMs at a

mass ratio of microcapsules/hemihydrate in percentage from 7.5 to 15.0%. The physical, thermal and mechanical properties such as porosity, density, thermal conductivity, the equivalent specific heat and the maximum compressive strength must be determined. Besides, in order to know the effect of increasing the thermal conductivity of microcapsules on the thermal properties of the gypsum block, mSD-(LDPE-EVA-RT27) containing 2 wt% of carbon nanofibers (CNFs) as filler were incorporated and named mSD-CNFs.

## 2. Materials and methods

### 2.1. Materials

Four types of microcapsules containing PCMs were used in this work: mSP-(PSt-RT27), mSD-(LDPE-EVA-RT27) with and without CNFs and Micronal<sup>®</sup>DS 5001X. mSP-(PSt-RT27) were synthesized with polystyrene shell and paraffin wax Rubitherm<sup>®</sup>RT27 by suspension polymerization technique in our lab facilities. mSD-(LDPE-EVA-RT27) with and without CNFs are also made in our laboratory from a polymeric shell of low density polyethylene (LDPE) and Ethylvinylacetate (EVA) copolymer and paraffin wax Rubitherm<sup>®</sup>RT27 as core material, following the process described in the European Patent EP2119498 [31]. The incorporated CNFs were synthesized following the method developed by Jiménez et al. [32]. Finally, the commercial microcapsules Micronal<sup>®</sup>DS 5001X supplied by BASF were selected since they have exhibited a large TES capacity in concrete blocks [33]. Micronal<sup>®</sup>DS 5001X contains n-heptadecane as the core material whose melting point is 26  $^{\circ}\text{C}$  and a shell from Polymethylmethacrylate (PMMA). The average particle size in number ( $dp_{n0.5}$ ) and in volume ( $dp_{v0.5}$ ) of the microcapsules were obtained by low angle laser light scattering (LALLS) and their melting point ( $T_f$ ) and latent heat of fusion ( $\Delta h_f$ ) were determined by differential scanning calorimetry (DSC) analyses. These data of the four microcapsules types are gathered in Table 1. Black gypsum was supplied by Yesos Juarez S.A. (Spain) and demineralized water with a conductivity value lower than 5  $\mu\text{S}/\text{cm}$  were used for the gypsum blocks.

### 2.2. Gypsum blocks manufacturing

Gypsum blocks were synthesized by weighting and mixing the masses of calcium sulfate hemihydrate, water and microcapsules according to the proportions shown in Table 2. The hemihydrate and microcapsules were mixed first and then the mixture was added to the water mixing constantly under vigorous agitation

**Table 2**  
Weight of raw material for the studied gypsum blocks.

Microcapsules/ hemihydrate (wt%)	Component		
	Water (g)	Hemihydrate (g)	Microcapsules (g)
0.0	110	231	0
7.5	110	231	17.3
15.0	115	231	34.7

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