

# Effect of the filler on the nanomechanical properties of polypropylene in contact with paraffinic phase change material

J. Giro-Paloma <sup>a,1</sup>, E. Rayón <sup>b,2</sup>, J.J. Roa <sup>c,3</sup>, M. Martínez <sup>a,1</sup>, A.I. Fernández <sup>a,1,†</sup>

<sup>a</sup> *Universitat de Barcelona, Faculty of Chemistry, Department of Materials Science and Metallurgical Engineering, C/Martí i Franquès, 1, 08028 Barcelona, Spain*

<sup>b</sup> *Universitat Politècnica de Valencia, Materials Science Institute, Camí de Vera s/n, E-46022 Valencia, Spain*

<sup>c</sup> *CIEFMA, Departament de Ciència dels Materials i Enginyeria Metal·lúrgica, ETSEIB, Universitat Politècnica de Catalunya, Avda. Diagonal, 647, 08028 Barcelona, Spain*

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## abstract

As the changes on the mechanical properties with depth of indentation provide valuable information that may be suitable for design purposes, nanoindentation is an adequate technique for investigating the nanomechanical changes in the surface and in the inner polymers. This research focuses on the study of the nanomechanical properties of two grades of polypropylene (PP) after the long term exposure to an organic fluid, such as paraffin wax used as a phase change materials (PCM). PCM are used for thermal energy storage (TES) in buildings applications for passive systems or heating and cooling usages. In any of these uses PCM must be encapsulated and PP is a possibility within the container materials used. Four different samples of the polypropylene were evaluated: PP, filled polypropylene with 60% Mg(OH)<sub>2</sub> (PP-60Mg), PP-60Mg with PCM (RT-25), and PP-60Mg with PCM (RT-42). It was studied the thermal stability by Thermogravimetric analysis of these samples, and also it was evaluated the Hardness (*H*), Elastic modulus (*E*) and Loss modulus (*E*<sub>loss</sub>) for the unfilled PP and PP-60Mg in contact with two different PCM at different temperatures (30 °C, 45 °C and 60 °C for RT-25 and 45 °C for RT-42) for 32 days. Results show that the mechanical properties *H*, *E*, and *E*<sub>loss</sub> are higher for PP-60Mg than for PP. Nevertheless, these properties decrease significantly when the PP and the PP-60Mg are in contact with PCM, because it acts as a plasticizer, softening the polymer. Besides, the higher PCM's melting point, the lowest mechanical properties were observed.

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## 1. Introduction

Phase change materials (PCM) have the principal advantage of high capacity energy storage in form of latent heat [1–4]. When a solid PCM is heated up and reaches its melting point, it goes through a phase change, from solid to

liquid absorbing heat, known as melting enthalpy while the temperature remains constant. Equally, when the phase change process is reversed, that is from liquid to solid, the stored latent heat is released, again at a nearly constant temperature [5]. Materials studied for this application are salt hydrates, paraffin waxes, fatty acids, and eutectics of organic and non-organic compounds [6]. Paraffin waxes are one of the organic PCM most employed [7,8] due to their latent heat, thermal energy storage (TES) capacity, abundance, low cost, large number of applications, and because their stability after several charging/discharging cycles [9]. Paraffin waxes with a melting point between 30 °C and 90 °C have chains with the number of

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† Corresponding author. Tel.: +34 934021298; fax: +34 934035438.

E-mail addresses: [jessicagiro@ub.edu](mailto:jessicagiro@ub.edu) (J. Giro-Paloma), [emraen@upvnet.upv.es](mailto:emraen@upvnet.upv.es) (E. Rayón), [joan.josep.roa@upc.edu](mailto:joan.josep.roa@upc.edu) (J.J. Roa), [monicamartinez@ub.edu](mailto:monicamartinez@ub.edu) (M. Martínez), [ana\\_inesfernandez@ub.edu](mailto:ana_inesfernandez@ub.edu) (A.I. Fernández).

<sup>1</sup> Tel.: +34 934021298; fax: +34 934035438.

<sup>2</sup> Tel.: +34 963879625.

<sup>3</sup> Tel.: +34 93 401 07 12.

carbons atoms in the range between 18 and 50. The longer the chain of the paraffin waxes, the higher the melting point [10]. Depending on the service temperature, it will be chosen one PCM or another, so the temperature of the system is really important when the PCM has to be selected [11].

PCM can be used in passive systems such as floors [12] and walls, or in active systems like in domestic applications as heating [13] and hot water [14,15]. Otherwise, they must be encapsulated or contained to avoid leakage when phase change occurs. This encapsulation may be in the microscale [16], leading to microencapsulated PCM (MPCM), or in the macroscale where the use of common thermoplastics as container materials for PCM macroencapsulation is used in commercial applications [17], being the polypropylene (PP) suitable for active systems in domestic hot water applications (DHW). It has been also tested as good container for PCM in microscale, as a PCM shell [18]. Krupa et al. [19] reported the use of PP as matrix to create a shape stabilize PCM, a polymer blend obtained by mixing isotactic PP and paraffin. They report a plasticizer effect of the paraffin on the viscoelastic properties of the PP. Besides, long exposure to organic substances may be responsible of the premature mechanical failure of containers made of polypropylene [20]. ESC mechanism is a physical interaction connecting highly localised plasticisation via stress enhanced fluid absorption, which does not carry molecular degradation of the plastic nor chemical variations. The absorption of organic PCM (such as paraffin waxes) plasticises the polymer and also reduces its yield strength. This reduction is directly associated with the concentration of absorbed fluid [1]. For this reason, it was decided to evaluate filled PP as it is well known that inorganic fillers increase the mechanical resistance and stiffness of thermoplastics. Moreover, fillers may reduce the degradation effect avoiding the premature mechanical failure, and improve properties against fire, which is relevant in this study because the paraffin used as PCM are flammable materials. A filler that is commonly used for PP is  $Mg(OH)_2$  [21,22] because of its flame retardant property [23], which also improves the mechanical response [24–26]. Therefore, the filler used in this work has been  $Mg(OH)_2$ , and the nanomechanical properties of filled PP compared with PP unfilled.

The nanoindentation technique was used to measure the nanomechanical properties of selected materials containing PCM. Nanoindentation technique is perhaps the most commonly applied means of testing the mechanical properties of materials at micrometric and nanometric scale. The ability to measure the microscopic regions responses is a key to understand the mechanical behavior of technological material systems [27].

The probe in nanoindentation technique is forced into the surface at a programmed rate and to a selected maximum force or depth. By means of special transducers the load and penetration depth are registered during the experiment. The area of contact between indenter and sample is then estimated using the known geometry of the indenter. For a Berkovich indenter, which is used in this work, the relationship between the projected area  $A$  of indentation and the indentation depth  $h$  beneath the

contact is  $A = 24.5h^2$ . Consequently, Hardness ( $H$ ) and Elastic modulus ( $E$ ) can be calculated by the stiffness obtained by the known equations reported elsewhere [27] without the necessity to observe the indentation marks. This procedure has the advantage that very low loads can be used avoiding the material damage and makes possible to analyze thin films and small volume of material.

There are a great number of studies using the nanoindentation technique in polymers showing that is a simple but effective mechanical testing method. Nanoindentation has been successfully used to study the hardness and elastic properties of several polymers and nanocomposites. To give some examples of these studies on several polymers under different experimental strategies, Lee et al. [28], studied the  $H$  and  $E$  of a single cellulose fiber and PP matrix in a cellulose fiber-reinforced PP composite using the continuous stiffness measurement technique. Besides, Fang and Chang [29] studied the nanomechanical characteristics of polycarbonate polymer films under different applied loads, hold times, and loading rates. Moreover, Hu et al. [30] used it to investigate mechanical properties of Nylon 11 (PA11) and its nanocomposites with different clay loading.

Nanoindentation has been successfully compared with other techniques normally used to study the mechanical properties of polymers such as the Atomic Force Microscopy (AFM) and the Dynamic Mechanical Analysis (DMA) instruments; Griepentrog et al. [31] compared the nanoindentation technique and the Atomic Force Microscopy (AFM) methodology for the determination of mechanical properties of poly(methyl methacrylate) (PMMA) and polycarbonate (PC) polymers. Besides, Jee and Lee [32] studied AFM through both the force-indentation and area-depth curves for different polymers, concluding that the two methods give almost identical results with self-consistency. In a previous work, Giro-Paloma et al. [27] published a comparison study between mechanical data extracted from nanoindentation measurements and from classical dynamic mechanical analysis of several amorphous and crystalline polymers. In that study, we demonstrated that the elastic modulus obtained by nanoindentation can be well correlated with that obtained by DMA.

In this study were used two different types of paraffin waxes provided by Rubitherm® with different melting temperatures ( $T_m$ ): RT-25 ( $T_m = 25$  °C) and RT-42 ( $T_m = 41$  °C).

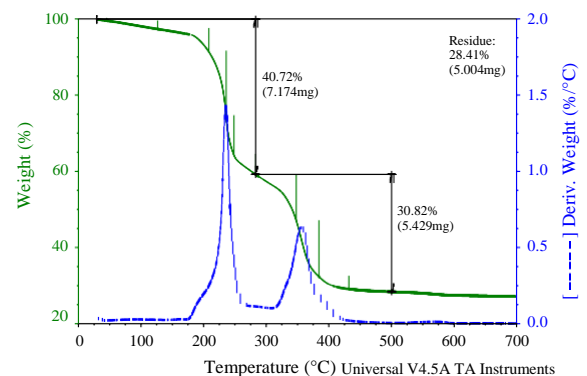


Fig. 1. Thermogravimetric analysis of PP-60Mg.

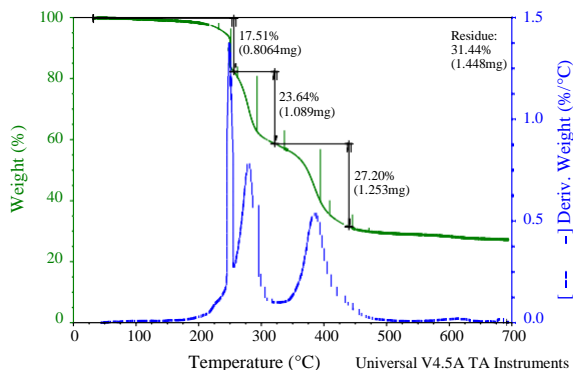


Fig. 2. Thermogravimetric analysis of PP-60Mg with PCM.

These paraffin waxes were melted in separate vessels and two different polymeric materials were submerged in each paraffin sample for 32 days. Polypropylene (PP) was chosen as case study as it has been evaluated as container material of PCM [1] and a filled PP sample with a filler content of 60%  $\text{Mg}(\text{OH})_2$  (PP-60Mg) was also evaluated to compare the effect of the filler in the decrease of mechanical properties after long exposure to an organic fluid.

## 2. Experimental procedure

### 2.1. Materials

Polymeric samples were prepared at the Centre Català del Plastic (CCP). Two kinds of polypropylene samples were used, PP homopolymer and PP filled with 60%  $\text{Mg}(\text{OH})_2$  named PP and PP-60Mg, respectively. They were obtained by melt compounding with a co-rotating twin screw extruder. PP (Sabic PP 513A) was used and the PP-60Mg sample prepared by mixing PP with 60% magnesium hydroxide powder (Magnifin H-5 kV from Martinswerk). The obtained pellets were compressed moulded with a hot press plate, and different specimens were machined from the discs.

Paraffin waxes used were commercial products from Rubitherm (RT-25 and RT-42), having melting temperatures around 25 °C and 41 °C for RT-25 and RT-42, respectively.

### 2.2. Procedure

Polymeric specimens of 1 cm×1 cm×0.4 cm were submerged during 32 days in two different melted PCM and the temperature was kept constant. The temperatures tested were 30 °C, 45 °C and 60 °C for the RT-25 and 45 °C for the RT-42.

### 2.3. Characterization

#### 2.3.1. Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was used to study the thermal stability of the materials [33]. Thermal stability of the samples under study was evaluated with a TA Instruments, Simultaneous SDT Q600 under 100 ml min<sup>-1</sup> N<sub>2</sub> atmosphere. The procedure of the TGA analysis was a scanning rate of 10 °C min<sup>-1</sup> in the temperature range between 30 °C and 700 °C.

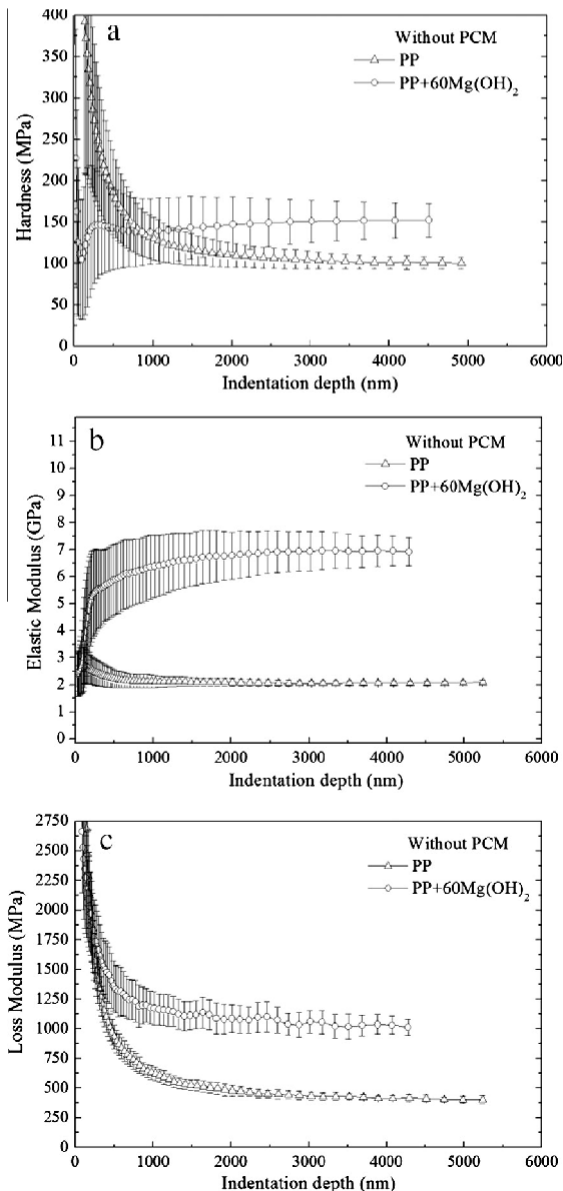


Fig. 3. Comparison of filled and unfilled PP without PCM. (a) Hardness,  $H$ ; (b) Elastic modulus,  $E$ ; and (c) Loss modulus,  $E_{\text{loss}}$ .

#### 2.3.2. Nanoindentation technique

It was performed nanoindentation assays to evaluate the mechanical response in an indentation analysis with the purpose to calculate Hardness ( $H$ ), Elastic modulus ( $E$ ), and Loss modulus ( $E_{\text{loss}}$ ) on the microscopic length scale and to estimate the changes produced by the contact with the organic fluid. Nanoindentation measurements were performed on samples PP and PP-60Mg at room temperature before and after being immersed during 32 days in two different types of PCM (RT-25 and RT-42) at the experimented temperatures.

A Nanoindenter G-200 (Agilent Technologies) was employed using a diamond three-sided Berkovich indenter geometry. This was calibrated with a silica standard specimen with a known Young's modulus. The stiffness

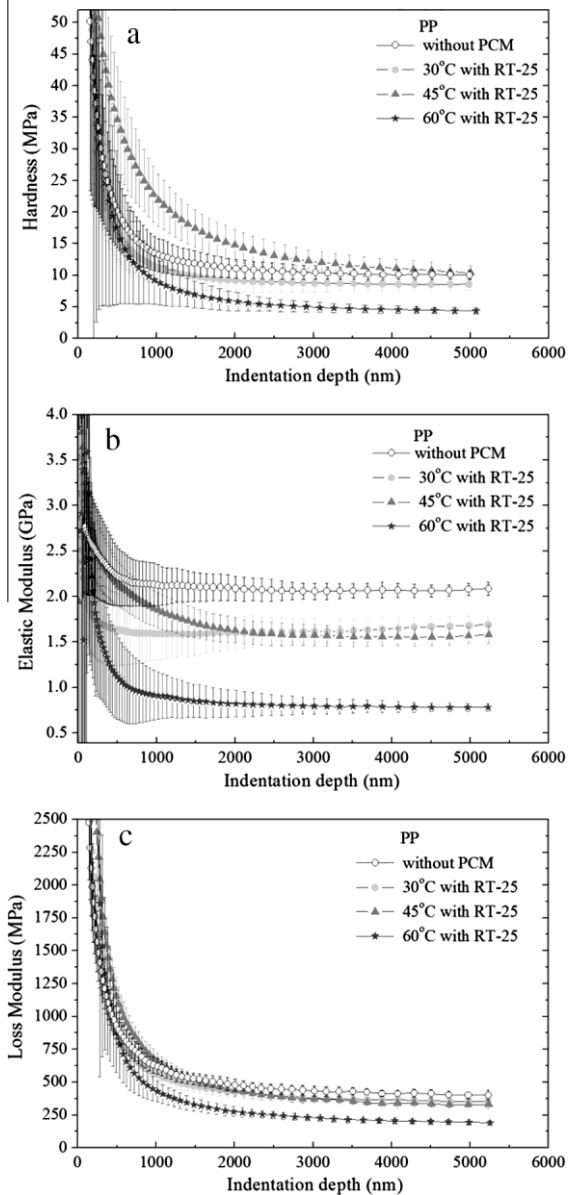


Fig. 4. Comparison of PP: without PCM, and in contact with RT-25 at 30 °C, 45 °C and 60 °C of (a) Hardness,  $H$ ; (b) Elastic modulus,  $E$ ; and (c) Loss modulus,  $E_{loss}$ .

was acquired under the continuous stiffness measurement (CSM) at an oscillating frequency of 75 Hz and at 10 nm harmonic amplitude.

An array of 100 nanoindentation imprints was performed at 600 mN maximum load and each nanoindentation imprint was separated a constant distance of 500  $\mu$ m. Several considerations were carefully observed. Due to the viscoelastic characteristic behavior of these materials, the typical hold segments in the unload curve were avoided by setting the hold time segment to zero. In order to calculate the thermal drift without constant load segments, a double  $P$ - $h$  curve was executed for each test. Thermal drift is then considered well corrected by overlapping both unloading curves. Besides, a strain rate

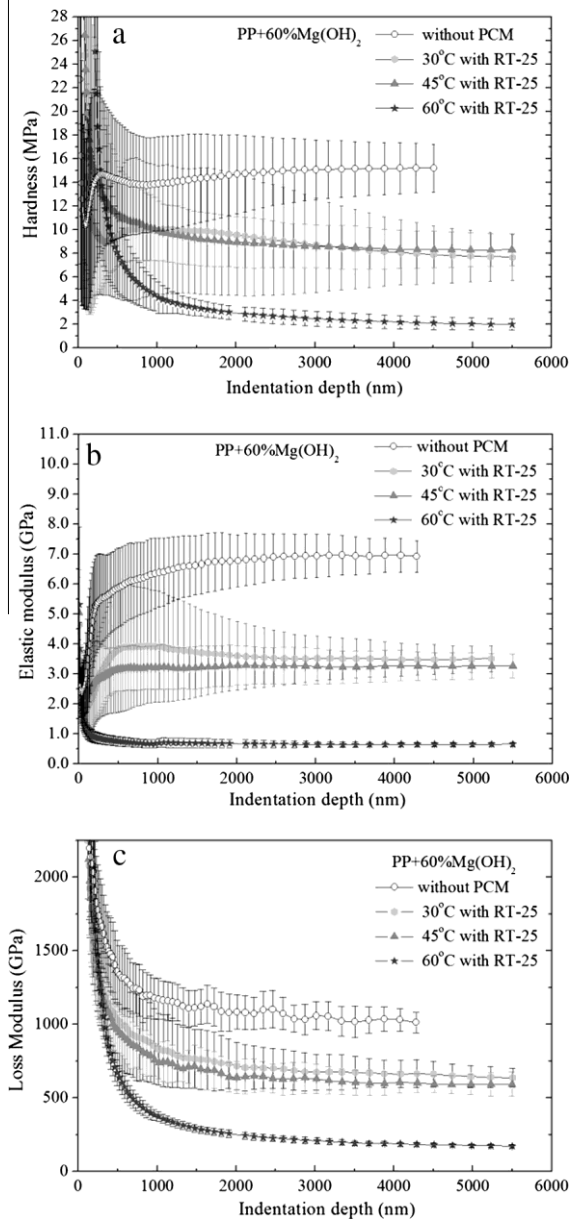


Fig. 5. Comparison of (a) Hardness,  $H$ ; (b) Elastic modulus,  $E$ ; and (c) Loss modulus,  $E_{loss}$  of PP and filled PP that was in contact with PCM RT-25, at different temperatures.

of 0.2  $s^{-1}$  was chosen to assure that this parameter is not sensitive to the mechanical properties, following the study of Giro-Paloma et al. [27].

### 3. Results and discussion

#### 3.1. Thermogravimetical analysis

The thermal oxidative degradation of PP-60Mg was complete at temperatures up to 450 °C, with 28% of residue remaining, as it is shown in Fig. 1, revealing two decomposition steps. The first step corresponds to  $Mg(OH)_2$  decomposition around 250 °C and the second step is attributed to

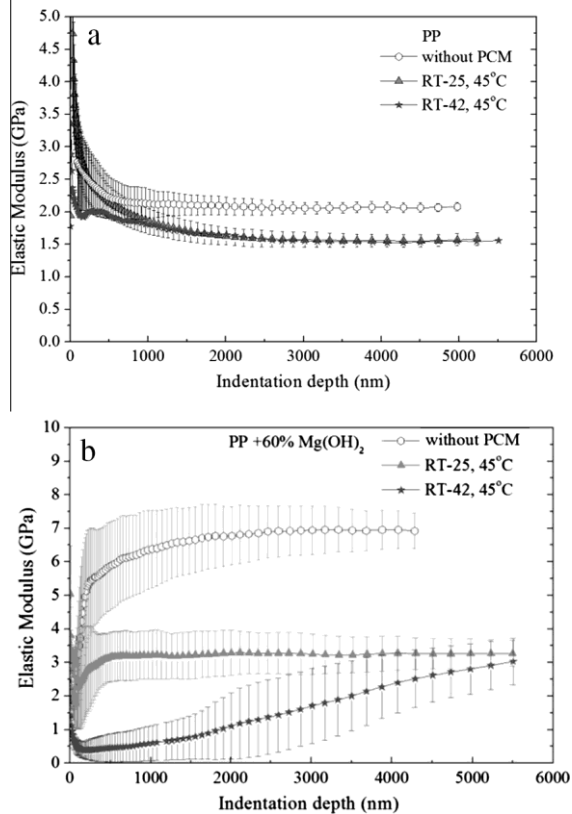


Fig. 6. Elastic modulus comparison at 45 °C for RT-25 and RT-42 (a) unfilled PP, and (b) filled PP.

the thermo oxidative decomposition of polypropylene matrix which is in accordance with literature [34].

The thermal degradation curve of the PP-60Mg, after being submerged in PCM RT-25 during 32 days, shows three decomposition steps, as it is shown in Fig. 2. Thermal analysis of polymer immersed in organic PCM, such as paraffin wax, show a PCM absorption by the plastic in agreement to the observations made by Castellón et al. [1].

The first degradation is because of  $Mg(OH)_2$  decomposition being around 17%. Then, the degradation of the PCM, around 24% of the sample takes place between 250 °C and 325 °C. Finally, the third step PP degradation finishes around 450 °C [35]. The 31% of residue corresponds to the  $MgO$ .

### 3.2. Nanoindentation technique

#### 3.2.1. Mechanical properties of filled PP and unfilled PP

The filler effect on mechanical properties can be observed in Fig. 3. As expected, the sample of PP-60Mg presents higher  $H$ ,  $E$ , and  $E_{loss}$  than PP ones, due to the reinforcement effect of filler.

#### 3.2.2. Filled PP and unfilled PP in contact with an organic fluid (PCM)

A prolonged contact of PP with PCM softens the material. The results for PP samples in contact with RT-25

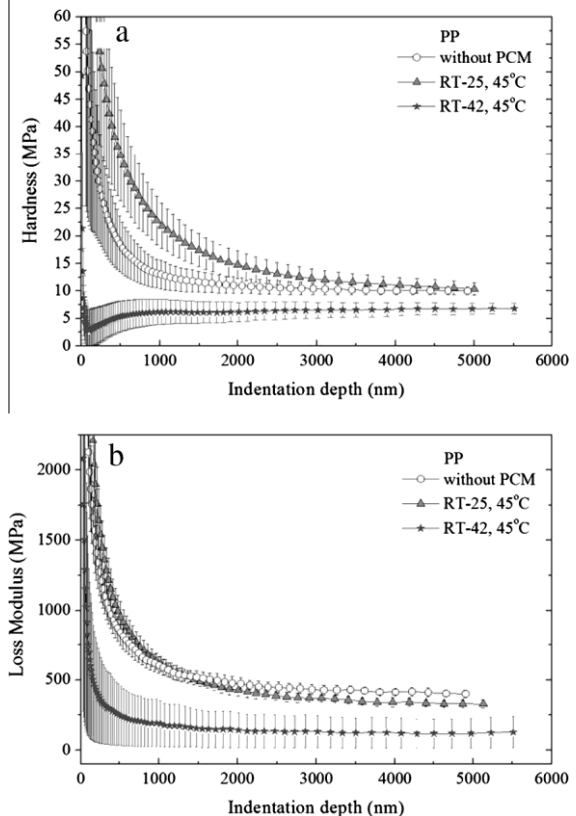


Fig. 7. Comparison of PP: without PCM, and in contact with RT-25 and RT-42 at 45 °C of (a) Hardness,  $H$ ; and (b) Loss modulus,  $E_{loss}$ .

during 32 days at different temperatures (30 °C, 45 °C and 60 °C) are shown in Fig. 4. It is seen that the plasticizer effect of the paraffin increases with the temperature of exposition to the organic fluid. Also, it is observed a strong correlation between the temperature and the drop of mechanical properties when the temperature increases.

For this reason, after 32 days at 60 °C, the  $E$ ,  $H$  and  $E_{loss}$  decrease significantly, whereas the mechanical properties after these 32 days in contact with the paraffin at 35 °C and 45 °C do not differ significantly between them and are slightly lower than those for the reference sample.

Notably, when it is used the same paraffin (RT-25) in contact with the filled PP, it is observed that the studied mechanical properties decrease in the same way as the temperature increase after 32 days of exposure as it can be observed in Fig. 5.

If the properties of Figs. 4 and 5 are compared, it can be observed the effect of the filler on the mechanical properties. As expected,  $E$  and  $E_{loss}$  values for the PP-60Mg are higher than the unfilled PP as the filler contributes making the composite stiffer and reducing the viscoelastic behavior. The presence of the filler counteracts the softening effect produced by continuous contact with PCM, except for those samples exposed to PCM at 60 °C, temperature in which the softening effect caused by the diffusion of paraffin in the polymer is highly favored. This effect is not so relevant in  $H$  values, which do not differ significantly of



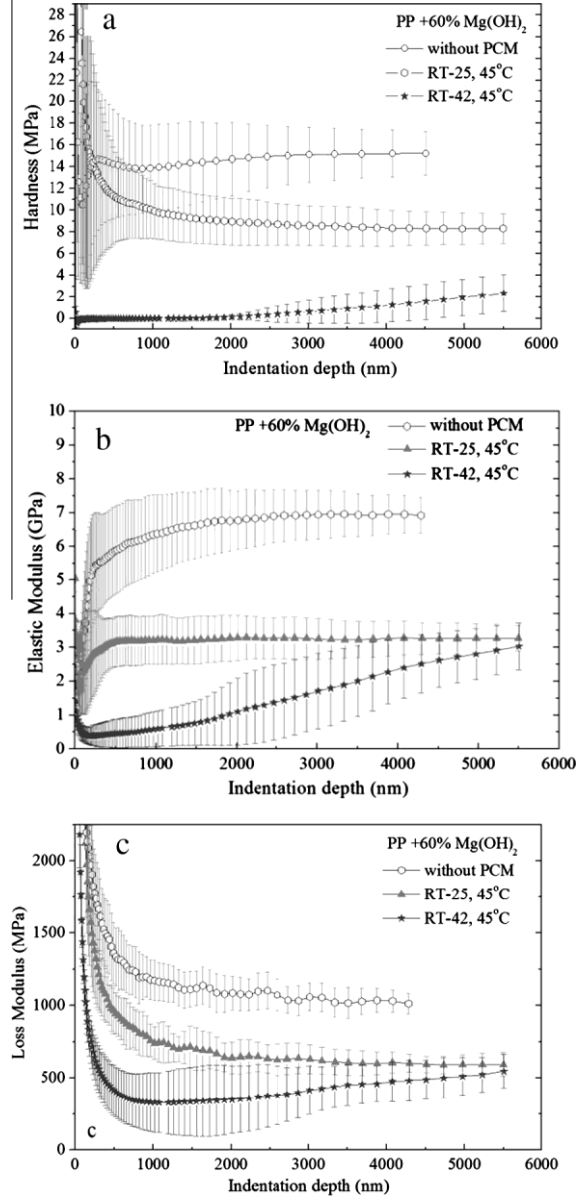


Fig. 8. Comparison of filled PP: without PCM, and in contact with RT-25 at 45 °C and RT-42 at 45 °C of (a) Hardness,  $H$ ; (b) Elastic modulus,  $E$ ; and (c) Loss modulus,  $E_{loss}$ .

Table 1  
Comparison of the nanoindentation results for the filled and unfilled studied samples.

Sample	PCM	Working temperature of PCM (°C)	$H$ (MPa)	$E$ (GPa)	$E_{loss}$ (MPa)
PP	Without	–	$105.0 \pm 10.0$	$2.0 \pm 0.1$	$437 \pm 10$
	RT-25	30	$8.7 \pm 0.9$	$1.7 \pm 0.1$	$365 \pm 0.0$
		45	$10.4 \pm 1.0$	$1.2 \pm 0.1$	$345 \pm 0.0$
		60	$5.0 \pm 0.7$	$0.8 \pm 0.0$	$205 \pm 0.0$
		45	$6.2 \pm 1.1$	$1.5 \pm 0.1$	$126 \pm 60$
PP-60Mg	Without	–	$150 \pm 20$	$6.8 \pm 0.3$	$1095 \pm 90$
	RT-25	30	$8.5 \pm 1.9$	$3.5 \pm 0.2$	$675 \pm 80$
		45	$8.5 \pm 3.5$	$3.2 \pm 0.1$	$620 \pm 85$
		60	$2.3 \pm 0.4$	$0.6 \pm 0.0$	$205 \pm 15$
		45	$1.2 \pm 1.2$	$2.5 \pm 1.0$	$350 \pm 110$

the unfilled PP. Both samples follow the same tendency after being in contact with PCM, showing lower  $H$  values, even lower for the samples subjected to higher temperatures, as PCM has a tendency to infiltrate and soften some plastics [36].

When a paraffin of a higher melting point as RT-42 is used as organic fluid, no significant differences were observed in the measured elastic modulus of unfilled PP respect to the sample submerged in RT-25, showing both samples a modulus reduction around 35% over the measured modulus for reference PP sample, as is shown in Fig. 6a. For the PP-60Mg sample the reduction of this parameter is almost the 60% of the initial value (Fig. 6b).

Another effect observed between samples immersed in different paraffin waxes is the low value of elastic modulus at penetrations lower than 2000 nm, which is attributed to the presence of a residual paraffin coating that diminishes while the tip penetrates in the sample.

Otherwise, there are some differences in the  $H$  and  $E_{loss}$  between PP unfilled samples in contact with RT-25 and RT-42 PCM at 45 °C, as Fig. 7 shows. As mentioned before, curves obtained for PP sample tested with RT-42, show anomalous behavior at indentation depths lower than 2000 nm, which are attributed to a residual paraffin coating on the sample. There is a stabilization of the curve after this indentation depth and for  $H$  and  $E_{loss}$  it is seen that the softening effect and the viscoelastic behavior are higher when RT-42 was used as organic fluid. Thus, this softening effect needs to be considered when choosing unfilled PP as a polymeric container of paraffinic PCM. The container wall will soften during thermal cycles, and the effect will be more noticeable for paraffin with higher melting points.

However, when a filled PP is considered, the results are quite different. In Fig. 8 can be observed that the presence of inorganic filler in the PP-60Mg partially counteracts the softening effect, as the PCM cannot be soaked in this sample as much as in unfilled PP. For the sample that was submerged in RT-25, even though there is a reduction in mechanical properties compared with the reference PP-60Mg, the results show that PP-60Mg is a better option than unfilled PP (especially at indentation depths higher than 3000 nm). Nevertheless, for the sample that was submerged in RT-42 there is a severe reduction in mechanical properties that is not counteracted by the filler content.

Table 1 summarizes the results of  $E$ ,  $H$  and  $E_{loss}$  for a maximum penetration depth of 5000 nm.

#### 4. Conclusions

Nanoindentation technique is an adequate tool to evaluate nanomechanical properties for polymers. The usage of the fire retardant inorganic filler  $Mg(OH)_2$ , in a polypropylene (PP) matrix, improves significantly the Hardness ( $H$ ), Elastic modulus ( $E$ ) and Loss modulus ( $E_{loss}$ ). PP is used as material container for paraffinic PCM in TES systems, and in this study it is demonstrated that there is a severe reduction in the mechanical properties after thermal cycles and a long exposure to paraffin. Moreover, for a given PCM as contact fluid, the lowest mechanical properties were observed for the higher temperature tested. Even though the softening effect of the paraffin is counteracted using the filled PP sample with paraffin RT-25, this improvement was not observed testing the paraffin RT-42, with a higher melting point. The main conclusion is that is better to use a filled PP instead of unfilled PP as a container material for paraffin in TES systems. In the case of paraffin with melting point above 25 °C, the decrease of mechanical properties because of softening effect must be considered in the design of the containers as it can compromise their mechanical performance.

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