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Dynamic mechanical analysis of injection-moulded discs of polypropylene and untreated and silane-treated talc-filled polypropylene composites

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Abstract

Dynamic mechanical analysis (DMA) is used to study the dynamic mechanical properties of injection-moulded discs of polypropylene, talc-filled polypropylene composites and silane-treated talc-filled polypropylene composites. Silane addition in a very low proportion improves the adhesion between the filler and the polymer and enables composites to be used where otherwise they could not. Therefore, on the one hand, the composites have different properties due to the filler and the coupling agent, and on the other hand the injection processing induces a structure, resulting in inhomogeneity and anisotropy for the moulded discs.

Scanning electron microscopy (SEM), differential scanning calorimetry (DSC) and wide-angle X-ray diffraction (WAXD) techniques are used to help state the microscopic structure and justify the macroscopic properties measured with DMA.

Keywords: Injection-moulded discs; PP–talc composites; DMA

1. Introduction

Nowadays, polypropylene (PP) is a polymer with a wide range of applications. The main reason for this choice is the possibility to modify its structure and properties depending on each particular application. One way of tailoring the properties of PP is by means of adding an appropriate filler. Talc is the most used filler in the market [1]. It began to be used because its addition made the composites cheaper to produce. However, some time later it was found that it introduced some peculiar properties to the final composites: a higher stiffness and deformation temperature; a bigger strength and flexion resistance; a higher thermal conductivity and a smaller thermal expansion [2]. In studying the complex structure and morphology of polymers modified by mineral fillers, some problems may arise concerning the character and extent of interaction at the polymer–filler interface, the homogeneity of filler distribution, the filler orientation in the case of filler anisometric particles, and the polymer–filler adhesion [3]. Because of that, a great number of coupling agents, such as silanes, have been developed in order to improve this adhesion [4].

Furthermore, the final product properties are highly dependent on the rheological conditions during the processing. One of the most used processing techniques is injection-moulding. The injection-moulded composites present a peculiar multilayer structure, in which the type, size and orientation of the amorphous and crystalline phase of the polymer change continuously from the skin to the core [5–7].

The dynamic mechanical properties of different materials can be characterised using dynamic mechanical analysis. This technique can also be used to follow main chain and side-groups motions, and in particular to study the glass transition of polymers. This fact turns it into a powerful technique for the characterisation of polymer structures, and provides a tool for studying the manufactured injection-moulded discs. It is worth mentioning that there are no previous known studies using the dynamical mechanical analysis (DMA) technique for the analysis of injection-moulded discs. However, it seems to be an appropriate method that could provide interesting results that are not available with other techniques.

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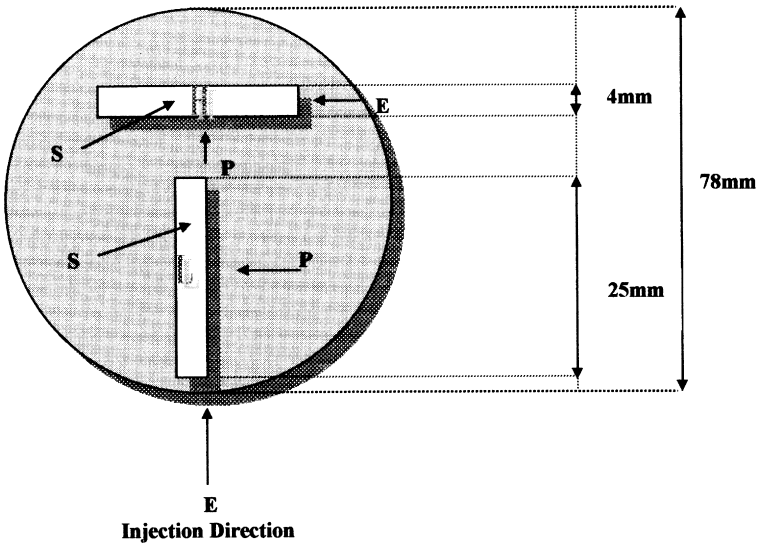

Fig. 1. Nomenclature and dimensions of the samples.

In the present work, DMA is used in order to study the dynamic mechanical properties of injection-moulded discs of PP, talc-filled PP and silane-treated talc-filled PP composites. The work focuses on two aspects: the characterisation of the different viscoelastic responses of the composites and the study of the homogeneity and anisotropy of injection-moulded discs. The discussion of DMA results is aided by the microscopic structure analysed by other techniques such as scanning electron microscopy (SEM), differential scanning calorimetry (DSC) and wide-angle X-ray diffraction (WAXD).

2. Materials and samples

Commercial isotactic polypropylene, Isplen-051 (Repsol Química S.A., Spain) was used as a matrix. Its molecular characteristics are as follows: $M_w = 248{,}297$, $M_w/M_n = 6.24$, isotacticity = 97%, MFI = 5.4 g/10 min (230°C and 2160 g). Talc LU-1445 (Luzenac, Toulouse, France) was used as a filler in our investigation. Its most important physicochemical properties are: laminar particles (97%), average platelet diameter (22 mm), specific surface (3.8 m^2/g) and density (2.7 g/cm^3).

A mixture of gamma-methacryloxypropyltrimethoxy silane and vinytrimethoxy silane (3:1 by volume) containing 1% dicumyl peroxide was used for surface treatment of the mineral to study the eventual improvements obtained in the composite properties [4]. For complete surface wetting, the following amounts were required per kilogram of talc: 240 cm^3 of methanol, 60 cm^3 of water and 20 cm^3 of silane mixture. After the wetting process, the mineral was dried in an oven for 24 h at 60°C.

Polypropylene was initially mixed with a nominal talc content of 40 wt%. Two different mixtures were prepared, one with untreated talc, the other with silane-treated talc; both extruded by a twin-screw APV-2030 equipment. By dilution of these materials with the original PP in a single screw extruder, composites were prepared with nominal talc contents of 20 wt%. The composite with untreated talc is denoted PPN, and the composite prepared with silane-treated talc PPF.

The PP and both composites were injection-moulded in discs (ASTM D-647) in a U-40/30 injection machine (Margarit S.L., Barcelona, Spain). The temperature profile of injection was 140, 190, 200 and 210°C, and the injection pressure was set at 20 MPa. Two different samples were cut from each injection-moulded disc: the sample near the injection point is called 'L sample' and the sample far away from this point is called 'H sample'. For each type of sample, s, p and e denote the three principal directions. The nomenclature, directions and dimensions used to characterise each sample are shown in Fig. 1.

3. Experimental

3.1. Thermogravimetric analysis (TGA)

The talc content of the samples was calculated by means of a Perkin Elmer TGA 7 thermogravimetric analyser, previously calibrated with two different ferromagnetic materials: nickel and iron. The weights of the samples were between 12 and 14 mg and the experiments were performed in the temperature range between 100°C and 900°C with a heating rate of 10°C/min.

3.2. Scanning electron microscopy (SEM)

A Jeol-820 scanning electron microscope was used for morphological observation of freeze-fractured samples after vacuum coating with gold.

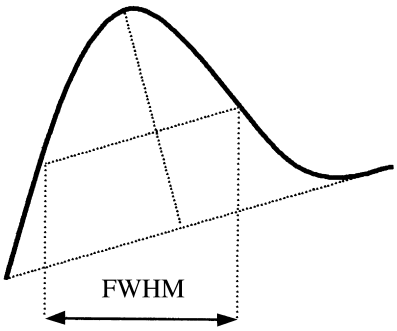

Fig. 2. Criteria of definition of the parameter FWHM.

3.3. Wide-angle X-ray diffraction (WAXD)

The experiments were performed using a Philips PW 1050/71 diffractometer. Radial scans of intensity (I) versus scattering angle (2θ) were recorded in the range 5.010–69.970° at 0.020/0.800 2θ/s with identical settings of the instrument by using filtered CuK_α radiation. The scans were taken from the s and p directions of each sample.

3.4. Differential scanning calorimetry (DSC)

A Mettler DSC-30 thermal analysis system was employed for the thermal characterisation. Indium, zinc and lead standards were used for temperature calibration, whereas the calibration for energy was done by using indium reference samples. Samples weighing about 5 mg were used. To investigate the influence of the injection processing, the samples were heated from −50°C to 220°C at 10°C/min. On the other hand, to study the activity of the filler and the effect of the coupling agent in the crystallisation behaviour of the composites, each sample was first heated at 200°C for 4 min in order to erase any previous thermal history. The calculation of the erasing optimal condition was carried out by applying the standard method described by Delvaux and Chambert [8]. The measurements were carried out from 200°C to 10°C at the following cooling rates: 10, 20, 30, 40, 50 and 80°C/min.

From DSC measurements, the crystallinity of the composites and the activity of the fillers were calculated. The melting enthalpies [ΔH_m (J/g)] are measured for the PP–talc samples, allowing us to estimate a crystallinity index (χ) by referral to the value of enthalpy of 100% crystalline polypropylene ($\Delta H_0 \approx 207.1$ J/g) [9].

$$\chi = \frac{\Delta H_m}{\Delta H_0} \tag{1}$$

To obtain the PP–talc composites crystallinity, it is necessary to introduce a linear correction factor because of the different weight of polymer in each sample:

$$\chi = \frac{\Delta H_m \dfrac{m_c}{m_p}}{\Delta H_0} \tag{2}$$

where m_c denotes the mass of the composite and m_p denotes the mass of the polymer (PP).

On the other hand, the non-isothermal crystallisation process is studied following the method developed by Dobreva and Gutzow [10,11] for the study of the crystallisation kinetics of molten polymers in the presence of nucleating agents. The relationship proposed by these authors is the following:

$$\log q \approx \text{const.} - \frac{B}{2.3\Delta T^2} \tag{3}$$

where q is the rate of crystallisation, ΔT is equal to $T_m - T_p$ [with T_m the melting temperature of PP in equilibrium ($T_m = 479$ K) and T_p the crystallisation temperature of the composites].

The activity of the filler is related to the parameter ϕ:

$$\phi = \frac{B^*}{B^0} \tag{4}$$

where B^* is the value of B when the polymer is filled and B^0 when it is unfilled.

Thus, from the experimental slopes in the representation $\log q$ versus $1/\Delta T^2$, according to Eq. (3), it is possible to obtain the B values. Finally, by using Eq. (4), the parameter ϕ can be estimated. The activity is related to the parameter ϕ so that a lower value of ϕ deals with a higher value of activity.

3.5. Dynamic mechanical analysis (DMA)

Dynamic mechanical testing has long been employed in the study of the viscoelastic response of polymers. The DMA equipment (Perkin Elmer DMA 7) was calibrated according to the manufacturer's recommended procedures. The storage modulus (E'), loss modulus (E''), and loss tangent ($\tan\delta$) were recorded in a three-point bending measurement system, a suitable configuration for the shape of the samples. These properties were measured at 1 Hz, in the temperature range between −40°C and 60°C with a heating rate of 5°C/min. Some previous experiments were done with lower heating rates (1°C/min and 0.04°C/min) to test the independence of the results with thermal inertia. The applied dynamic stress was 5×10^6 Pa and the static stress was as high as 6×10^6 Pa, due to the stiffness of the composites.

Three parameters were used to study the glass transition of each composite. The glass transition temperature (T_g) can be defined as the maximum of the transition in the loss modulus curve or in the loss tangent curve. The results presented in this work correspond to the latter definition [12]. The intensity of the transition (S) is a key factor in order to study the effect of the filler and the coupling agent as well as the injection-moulded discs homogeneity. This parameter is defined as:

$$S = \frac{E_a' - E_d'}{E_d'} \tag{5}$$

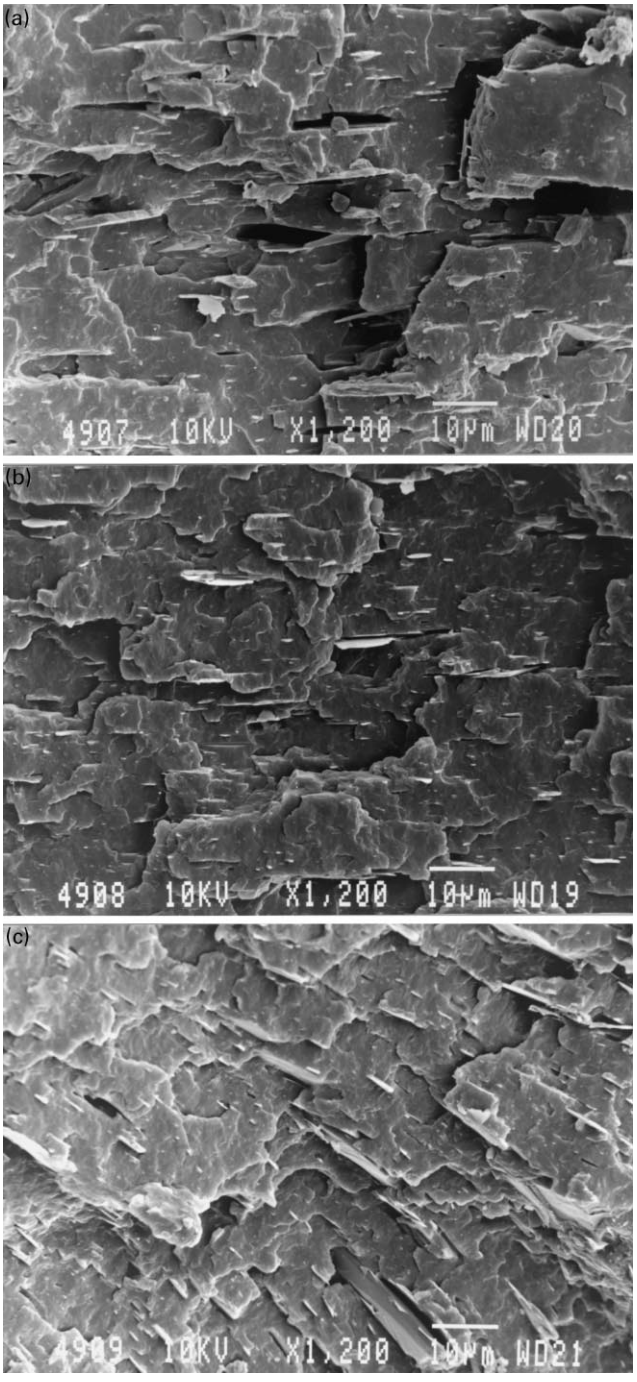

Fig. 3. Scanning electron micrographs of fractured samples: (a) PPNL; (b) PPFL; (c) PPFH.

where E_a' denotes the storage modulus before and E_d' that after the transition.

The parameter *S* refers to the mobility and content of the amorphous phase. A higher *S* value means a superior mobility or a higher content of the amorphous phase.

The full-width at half-maximum (FWHM) (see Fig. 2 for a scheme of the criteria used to define this parameter) refers to the homogeneity of the amorphous phase, so that a bigger FWHM value implies a higher inhomogeneity of the amorphous phase.

4. Results and discussion

4.1. Microscopic behaviour

4.1.1. Thermogravimetric analysis

This technique allows us to determine the talc content of our samples. The talc is a mineral with a very high sublimation point (over 900°C), so that at the final temperature of the TGA experiment (900°C) all the substances present in the disc have been decomposed, the talc being the only remaining component. The relationship between the mass at the end and at the beginning gives us the percentage of talc content of the composites. From the results obtained, there are no differences between L and H samples. Regardless of the area of the disc the samples came from, they have the same talc content. The PPN composites have higher talc content (19%) than the PPF composites (16%).

4.1.2. Scanning electron microscopy

Injection mouldings made from semicrystalline polymers possess a skin–core morphology. This technique makes it impossible to distinguish this structure in unfilled PP samples. Nevertheless, the presence of the talc made perfectly visible the skin–core structure in the composites.

Each sample has two zones of skin, where the talc particles lie parallel to the mould surface. Between these two zones a core zone, where the talc particles follow the flow lines describing ellipses, is appreciated.

Comparing samples with and without silane treatment [Fig. 3(a) and (b) show the same zone of PPNL and PPFL samples], it can be seen that the PPF samples show better adhesion between the polymer and the filler than the PPN samples, as expected. The PP–talc composites present more voids than the composites with coupling agents. In the fracturing process, due to the worse adhesion, the particles of talc jump, generating voids.

The skin is thicker in L samples than in H samples. An example of this behaviour can be observed in Fig. 3(b) and (c), where micrographs of PPFL and PPFH samples, taken at the same distance from the sample surface, are shown.

4.1.3. Wide-angle X-ray diffraction

The PP reveals an induced crystalline structure when it is mould-processed in the presence of mineral fillers such as talc, mica, calcite, etc. [3]. However, this effect is much more intense in the case of talc. The plates of talc particles lie parallel to the surface of the mould, and this organisation can induce a preferential orientation in the morphology of PP crystals placed near the filler.

In order to evaluate the degree of *b*-axis orientation of the PP unit cell, the ratio of the intensity between (040) to that of the (110) reflection was obtained [6,13–17]. Other ratios

Table 1
Intensity ratios: (a) polypropylene (PP); (b) polypropylene–talc (PPN); (c) polypropylene–talc–silane (PPF). The (040) and (110) planes correspond to PP, whereas the (020), (002) and (006) planes are associated with the talc particles

(a)	PPLs	PPLp	PPHs	PPHp
$I(0,4,0)/I(1,1,0)$	1.19	0.98	1.13	0.76
(b)	PPNLs	PPNLp	PPNHs	PPNHp
$I(0,4,0)/I(1,1,0)$	5.53	0.55	5.97	0.37
$I(0,0,2)/I(0,0,6)$	0.46	0.53	0.35	0.77
$I(0,2,0)/I(0,0,2)$	0.25	5.26	0.31	3.03
$I(0,2,0)/I(0,0,6)$	0.12	2.74	0.11	2.32
(c)	PPFLs	PPFLp	PPFHs	PPFHp
$I(0,4,0)/I(1,1,0)$	20.89	0.17	18.96	0.38
$I(0,0,2)/I(0,0,6)$	0.50	0.59	0.51	0.52
$I(0,2,0)/I(0,0,2)$	0.29	5.88	0.29	4.54
$I(0,2,0)/I(0,0,6)$	0.14	3.43	0.15	2.32

were also measured to study the orientation of the talc particles, and are shown in Table 1.

As deduced from the WAXD data, the plain polymer showed a non-isotropic crystalline structure: the ratio of the reflection intensities was about 1.19 [$I(040)/I(110) \approx$ 1.19 (PPLs)], whereas the reported value for the isotropic case was about 0.54 [18].

The analysis of the (110) and (040) PP diffraction peaks of the composites PP–talc in the s direction show that the intensity of the (040) reflection is strongly favoured in relation to the (110) reflection. This preferential orientation is much higher in the composites treated with silanes than in the untreated composites (Fig. 4).

Intensity studies in the diffraction patterns reveal that the plate of talc particles (001 exfoliation planes) and the *c*- and a^*-axes of the PP crystals randomly oriented are found parallel to the surface of the samples. Consequently, the *b*-axis of the PP unit cell is oriented perpendicular to this surface.

Despite the different orientations of the PP crystals in the composites PP–talc, treated and untreated with silane, there is no difference in the talc crystal orientation (Table 1). The analysis of the (002), (020) and (006) talc diffraction peaks shows that the treatment of the PP–talc composites with silane does not have an effect on the talc crystals orientation.

According to the diffraction patterns obtained, there is hardly any difference between the L samples (near the injection point) and the H samples. However, the analysis of the intensity ratio between (020) and (002) reflections in the p direction show that this ratio is bigger for the L samples. This means that in the L samples, the (020) plane is more parallel to the surface of the mould. So, it is possible to deduce that the orientation of the talc particles is bigger in the samples near the injection point, thus confirming the SEM results (L samples have a thicker skin than H samples).

4.1.4. Differential scanning calorimetry

According to the first type of experiments performed to characterise the influence of the injection processing, the crystallinity was studied in the different samples (Table 2). The crystallinity χ is almost unaffected by talc filling and considerably increased by the treatment with silanes [19]:

$$\chi(\text{PPF}) > \chi(\text{PP}) \geq \chi(\text{PPN})$$

In order to study the activity of the filler and the effect of the silanes, another kind of experiment was performed. Several authors have discussed the kinetics of non-isothermal overall crystallisation as well as the nucleating effect of

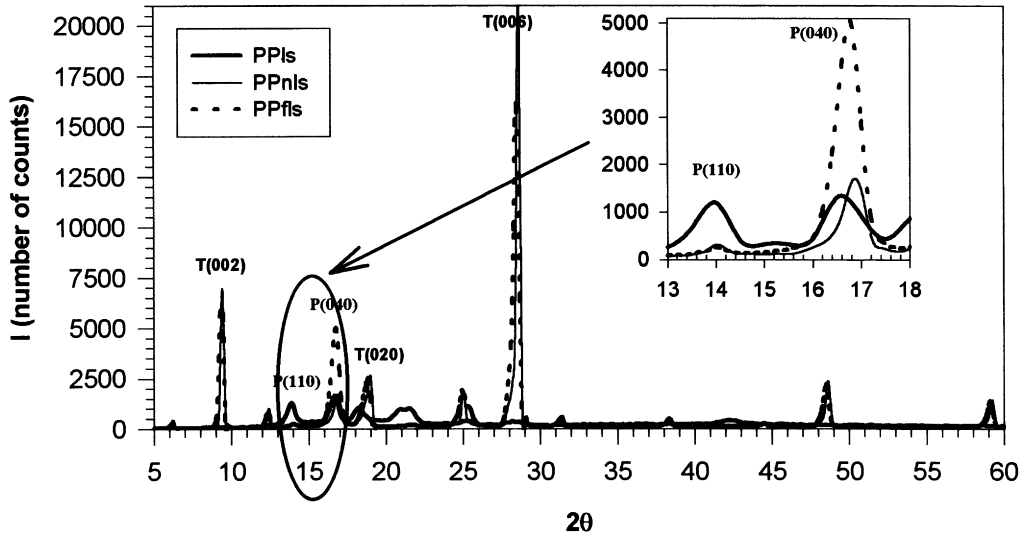

Fig. 4. 2θ scan diffraction curves of L samples in the s direction for all the composites: PP, PPN and PPF.

Table 2
Crystallinity (heating rate = 10°C/min)

Samples	χ (%)
PPL	50.6
PPH	49.9
PPNL	49.7
PPNH	49.9
PPFL	52.2
PPFH	53.8

the talc as a filler for the PP matrix [15,20]. We attempt to obtain the value of the nucleating activity of the talc treated and untreated with silane coupling agent.

According to the treatment schemed before, slopes and ϕ values were obtained for the studied composites. The analysis of the slopes in the curves log q versus $1/\Delta T^2$ allows us to say that there are almost no differences between L and H samples, whereas the behaviour of the composites PP, PP–talc and PP–talc with silanes differs considerably. From the results obtained in the study of the parameter ϕ (Table 3), it is possible to deduce that the activity of the filler is higher in the PPF composites, the nucleating effect increasing with the presence of silanes. In this sense, the treated talc is more active. Consequently, the interaction between the filler and the matrix will be higher or, in other words, the filler will influence the matrix structure to a greater extent. According to this result, the parameter activity seems to be very suitable to quantify the matrix–filler interaction.

Another characteristic of the PP thermograms obtained is the presence of two peaks around the melting point. The bigger one, at higher temperature (≈166°C), is related to the α phase (monoclinic) of the polymer, whereas the other peak, which is smaller and appears at lower temperature (≈135°C), is related to the β phase (hexagonal) of the polymer. Because of the injection-moulded processing, most of the polymer crystallises in the monoclinic system. Only a percentage of the 'core' zone crystallises in the hexagonal system. The small peak at ≈135°C disappears when the PP is filled with talc. The filler makes the matrix crystallise in the monoclinic system. On the one hand, the particles of talc crystallise in the monoclinic system as well. On the other hand, the filler makes the process of crystallisation faster, as we showed before, which causes the PP–talc composites to have only the α phase [21–26].

Table 3
Parameter ϕ for the different samples

Samples	Parameter ϕ	
	L samples	H samples
PP	1	1
PPN	0.48	0.47
PPF	0.34	0.31

Table 4
(a) Values of T_g, S and FWHM for all samples and directions. (b) Average values of T_g, S and FWHM for PP, PP–talc and PP–talc–silane

(a) Samples	T_g (°C)	S	FWHM
PPLs	14.2	0.67	21
PPLp	10.3	1.17	22
PPHs	14.3	0.82	22
PPHp	9.6	0.98	23
PPNLs	11.0	0.68	25
PPNLp	7.9	0.97	28.5
PPNHs	9.3	0.61	26
PPNHp	7.9	0.86	27
PPFLs	7.5	0.64	23
PPFLp	5.8	0.76	27
PPFHs	8.1	0.64	26
PPFHp	8.2	0.75	26
(b) Samples	T_g (°C)	S	FWHM
PP	12.1	0.91	22
PPN	9.0	0.78	26.6
PPF	7.4	0.69	25.5

4.2. Dynamic mechanical properties

4.2.1. Effect of the talc and the silane

Fig. 5 shows the evolution of the storage modulus with the temperature for the L samples in the s direction. It is important to say that the behaviour is analogous for all the samples. L and H samples, measured in the s and p directions, show that:

$$E'(\mathrm{PPF}) > E'(\mathrm{PPN}) > E'(\mathrm{PP})$$

The storage modulus always increases with adding the filler, because the talc is a stiffer material than the polypropylene. Besides, the presence of talc produces a more rigid interface in the PP matrix. If the talc is a treated one, the interface will be stiffer. So, the PPF composites, in spite of their lesser percentage of talc, have a higher storage modulus than the PPN composites.

Fig. 5 also shows the evolution of the loss tangent, which is always higher for the untreated talc PP composites (PPN). But what is really interesting is that the glass transition occurs at temperatures lower for the PP–talc composites than for the PP.

As has been pointed out above, and according to the results included in Table 4:

$$T_g(\mathrm{PP}) > T_g(\mathrm{PPN}) > T_g(\mathrm{PPF})$$

Despite previous studies on PP + filler composites, where the adding of a filler resulted in a higher temperature of glass transition [27], the experimental results prove that in the case of PP–talc composites the T_g occurs at lower temperatures. Some experiments were done at other heating rates in order to know if this result might be a consequence of a thermal delay due to the lower thermal conductivity of the

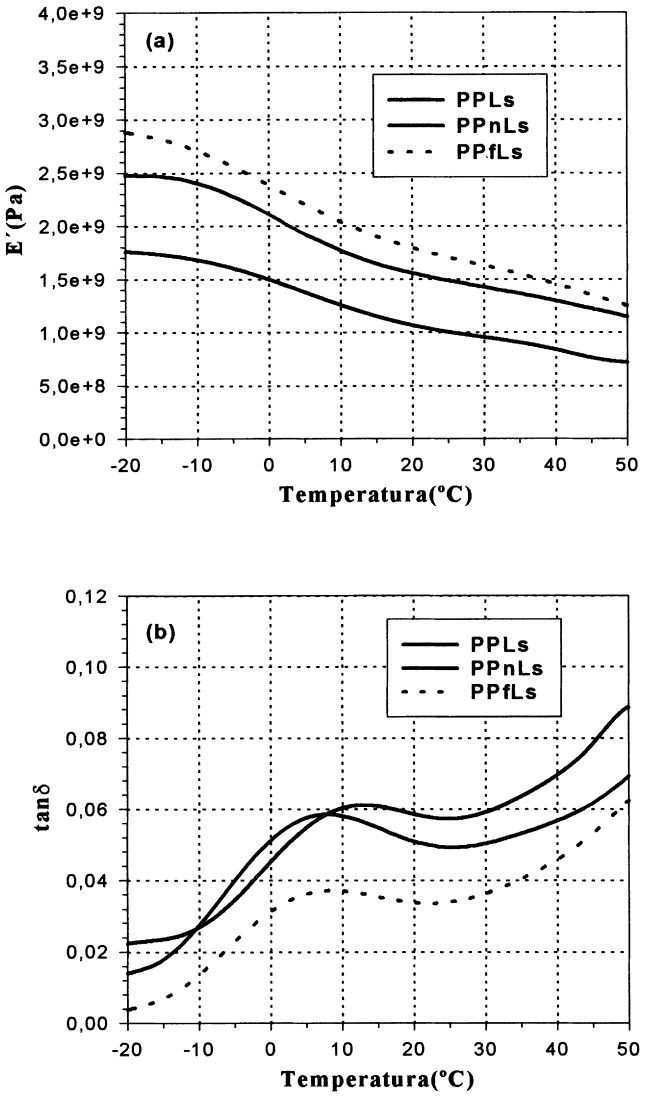

Fig. 5. (a) Storage modulus of the Ls samples (PP, PPN, PPF). (b) Loss tangent of the Ls samples (PP, PPN, PPF).

PP–talc composites. Nevertheless, the same tendency was observed.

This might be explained with the aid of the DSC results: the talc acts as a nucleating agent, and this effect of the talc leads to a faster crystallisation of the PP. This crystallisation speed-up causes an amorphous phase with bigger mobility in the PP–talc composites, which results in a lower T_g value. For analogous reasons, the PPF composites (with a higher nucleating effect) have the glass transition at lower temperatures than the PPN composites.

With respect to the intensity (S) of the transition, according to the results compiled in Table 4, the parameter S is bigger for the PP than for the PP–talc composites in the following manner:

$$S(\mathrm{PP}) > S(\mathrm{PPN}) > S(\mathrm{PPF})$$

There are two relevant reasons that justify this behaviour. Firstly, the glass transition occurs in the amorphous phase of the PP, so the greater the amount of polypropylene in the samples, the higher the intensity of the glass transition observed. Secondly, the addition of a rigid phase cuts down the mobility of the polymer chains, which results in a lower value of the transition intensity.

Regarding the glass transition measured FWHM values (Table 4); an increase for the PP–talc composites is observed as:

$$\mathrm{FWHM(PPF)} \approx \mathrm{FWHM(PPN)} > \mathrm{FWHM(PP)}$$

This can be explained considering the nucleating effect of the talc, which makes the crystallisation of the PP–talc composites faster, so the amorphous zone is more inhomogeneous than in the PP.

4.2.2. Homogeneity of the injection-moulded discs; L–H differences

According to the definition of L and H samples (Fig. 1), the homogeneity of the discs can be analysed from the different dynamic mechanical behaviour between samples near the injection point (L samples) and samples far away from this point (H samples).

There is a clear difference between the storage modulus of the L and H samples for the composites, however, there is no difference in the case of PP.

$$E'(\mathrm{PPL}) \approx E'(\mathrm{PPH})$$

$$E'(\mathrm{PPNL, PPFL}) > E'(\mathrm{PPNH, PPFH})$$

Fig. 6 includes the results measured in the p direction at room temperature. It is important to say that there seems to be a greater L–H difference in the s direction than in the p direction, which can be explained with the results obtained by means of SEM; the L samples have a thicker skin zone than the H samples. When the measurement is carried out in the s direction, the sample presents a skin zone + core zone + skin zone, whereas in the p direction it only presents a core zone.

Analysing the results obtained (Table 4), the T_g, S and FWHM values are almost the same for L samples and H samples. There seem to be no differences between these parameters for samples near to and far from the injection point. Taking into account the physical meaning of the parameters that characterise the glass transition, it is possible to conclude that L and H samples have no important differences in the amorphous phase:

$$T_g,\ S,\ \mathrm{FWHM(L)} \approx T_g,\ S,\ \mathrm{FWHM(H)}$$

4.2.3. Anisotropy of the injection-moulded discs; s–p differences

For all the samples (PP, PPN and PPF) the storage modulus is higher when it is measured in the s direction (Fig. 7).

$$E'(\mathrm{s}) > E'(\mathrm{p})$$

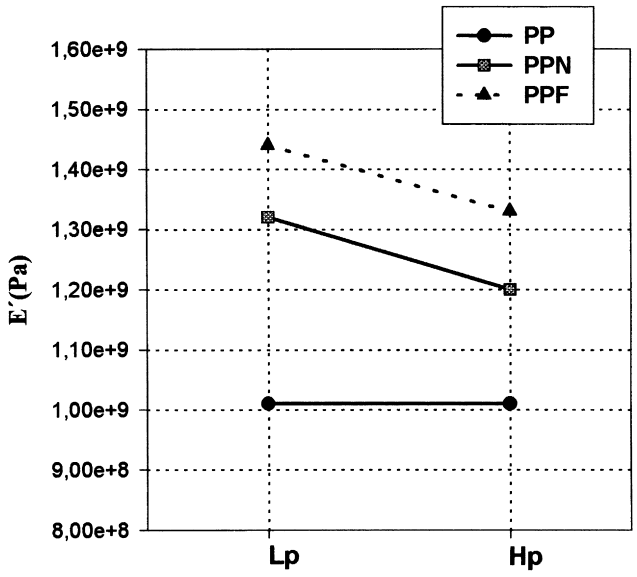

Fig. 6. Storage modulus; L–H differences (values at room temperature).

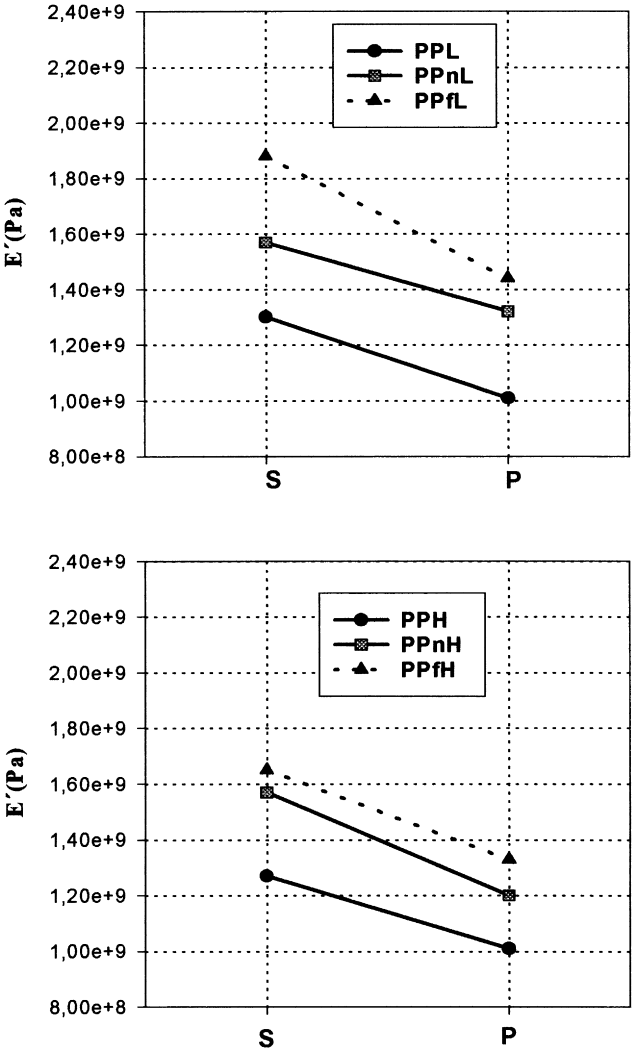

Fig. 7. Storage modulus; s–p differences (values at room temperature).

The s–p anisotropy appears in the PP as in the PP–talc composites. In the PP–talc composites it is very clear that the storage modulus must be bigger in the s direction because, as shown by means of SEM and WAXD, the particles of talc were situated parallel to the surface mould. Therefore, when the measurement is performed in the s direction, the particles of talc were perpendicular to the measuring direction, and because their stiffness (greater than the stiffness of the PP) makes difficult the process of deformation, so the storage modulus is higher. In the p direction the particles of talc are parallel to the measuring direction, so they do not offer difficulties to the deformation process, and consequently the storage modulus is smaller. In the PP samples, there are no talc particles, so the explanation must be based on other reasons. The injection process induces a peculiar skin–core structure in the unfilled PP. In the skin, the *b*-axes of the PP cells are oriented perpendicular to the surface, whereas in the core the *b*-axes are situated parallel to the surface [28]. Therefore, when the measurement is carried out in the s direction, there is a skin–core–skin structure, which results in two changes of *b*-axis orientation. On the contrary, when the measurement is made in the p direction, there is only a core zone, so there are no changes in the *b*-axis orientation. Based on this reasoning, it is logical that the s direction presents a higher storage modulus. On the other hand, the PP–talc composites do not have this change of orientation, i.e. the *b*-axis of PP is oriented perpendicular to the surface in the skin and in the core [28].

As shown in Table 4 for the PP–talc composites:

$$T_g(\mathrm{s}) > T_g(\mathrm{p})$$

$$S(\mathrm{p}) > S(\mathrm{s})$$

$$\mathrm{FWHM(p)} > \mathrm{FWHM(s)}$$

The distribution of the talc particles deals with a lesser mobility of the amorphous phase in the s direction, the glass transition occurs at lower temperatures when the measurement is carried out in the p direction. This direction presents smaller resistance to the transition (greater parameter *S*), and the amorphous phase seems to be more inhomogeneous (greater FWHM).

5. Conclusions

An investigation on dynamic mechanical properties of injection-moulded composites of polypropylene, polypropylene–talc and polypropylene–talc functionalised with silanes has been presented. Their microscopic characteristics have been determined by means of DSC, SEM and WAXD techniques, and helped in the interpretation of the macroscopic parameters measured with DMA. From the analysis of the experiments performed, the microscopic

structure has been characterised. Some of the most important results found were as follows:

The content of talc is distributed homogeneously in the different zones of the injection discs studied. The well-known skin + core + skin structure has been detected, the skin zone being thicker for the samples near the injection point. The adhesion between the polymer and the talc is improved when the talc is treated with silanes. The treated and untreated talc act as nucleating agents for the PP matrix, the effect of the treated talc is more intense. The adding of talc induces a preferential orientation of the PP crystals. This preferential orientation is much higher in the silane treated composites. The treatment with coupling agents has no effect on the orientation of the talc crystals.

5.1. Dynamic mechanical properties

The storage modulus always increases with adding talc because of the greater stiffness of the talc and because the talc generates a stiffer interface in the PP matrix. With the treated talc this interface is stiffer. The nucleating effect of the talc leads to a faster crystallisation of the polymer, which results in an amorphous phase with a bigger mobility (lower T_g). As the nucleating effect is stronger in the PPF composites, the T_g occurs at smaller temperatures. The intensity of the glass transition decreases with the talc and with treated talc, and furthermore the amorphous zone seems to be more inhomogeneous in the PP–talc composites.

The only difference between the samples taken from different zones of the discs studied is that the L samples present a higher storage modulus than the H samples, which can be explained by the greater orientation of the talc particles.

A large s–p anisotropy exists in all the samples (PP, PPN and PPF). The samples have a higher storage modulus in the s direction. The amorphous zone seems to have a minor mobility in the s direction, where the intensity of the transition is smaller. The amorphous phase seems to be more inhomogeneous in the p direction.

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