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ROLE OF A POLYPROPYLENE BASED INTERFACIAL MODIFIER IN A PRISTINE POLYPROPYLENE AND A POLYPROPYLENE/TALC COMPOSITE AS EVIDENCED BY DYNAMIC MECHANICAL ANALYSIS

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Abstract

Present paper is devoted to a comprehensive interpretation of the correlation existing between the dynamic mechanical thermal parameters and the molecular motions taking place in the polymeric phase in heterogeneous materials based on polypropylene. Consequently, it deals with the influence of a polypropylene grafted succinic anhydride interfacial modifier (iPP-SA) on the ultimate behaviour of these materials. This work has been performed by using four different samples consisting in isotactic polypropylene (iPP) and a 75/25 iPP/talc composite; both of them with and without added the interfacial modifier. The study has been tackled by evaluating the five different

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temperature dependent relaxation phenomena occurring in the polymer matrix. The proven capability of dynamic mechanical analysis (DMA) to follow transitions, structural, and morphological changes in polymer based materials has been used in the present work to determine the interfacial phenomena caused by the presence of an interfacial modifier. Samples studied here were obtained by compounding the materials followed by compression moulding. This moulding technique was used in order to minimize additional effects on the final properties of the sample apart from the interfacial ones. Additionally, some thermal properties obtained under dynamical conditions were measured and correlated to the reorganization possibilities of the material through the amorphous-crystal interphase in the polymer. The effect of the solely interfacial agent on the neat polymer matrix behaviour has been shown to be a very important item to be considered when designing composite materials (this is a fact too often dismissed in composite based studies). Otherwise, it has been proved that the presence of the interfacial agent in the composite can be detected (even at the very little concentration used here) not only by the changes in properties but also by the fact that exhibit their own α transition apart from that of the iPP matrix.

1. Introduction

One important aspect to consider in the study of polypropylene/mineral systems is the low affinity between the components because of the very different polarities they exhibit. Consequently, the need to enhance the interfacial interaction between the components of the composite must be achieved [1-3].

Hence, the way these heterogeneous materials have been obtained appears as the key. In this sense, it is important to define the fundamentals of the experimental strategy mainly concerning with the mixing and moulding operations. In fact, this looks to pass the polymeric material into the solid state by minimizing the effects caused by the distribution and orientation degree of the solid particles present into the polymer bulk. The latter is done in order to maximize as far as possible the solely interfacial modification purchased [1, 2]. It is noteworthy to remark that the reinforcement (talc particles) are aggregates of smaller primary silicate platelets consisting of thin lamellae of ultra thin dimensions [4] (a few nanometers). This is just the scale where the interfacial phenomena take place. Assuming the latter, the study of the interfacial interaction efficiency by different characterization techniques [2, 5] appears as a promised way to understand and to control the performance of such kind of advanced materials.

Present paper intends to follow a top-down approach [6], such as assumed by authors in their early studies performed onto heterogeneous materials based on organic polymers [2]. So, based on mechanical and thermal properties of the composites measured at the macroscopic scale, the goal would be to find correlations between the different hierarchical response domains of the material up to the nano-scale level at the interphase. Hence, dynamic mechanical analysis (DMA) is considered here to follow the changes in behaviour of a set of compounds incorporating or not succinic anhydride grafted isotactic polypropylene (iPP-SA) as the interfacial modifier (from the matrix side). Thus, likely than in a recent work concerning to modifications but from the mineral side [1], DMA has been used here to establish a proper comprehensive interpretation of the interfacial changes caused by iPP-SA in the polymer based compound.

2. Experimental

The polypropylene (Isplen 050) and the laminar talc (20M00S) used at present work were supplied by Repsol Química (Spain) and Luzenac (France), respectively. Some physical properties of these have been compiled at Tables 1 and 2. As the interfacial modifier from the matrix side, a maleic anhydride grafted isotactic polypropylene (iPP-SA), commercial grade, Hercoprime G, was chosen because of the very high (the highest commercially available) grafting content. Some of their relevant properties have been compiled at Table 1.

Table 1. Physical properties of the polypropylenes used at this work as received

Parameter	Matrix (iPP)	Interfacial Agent (iPP-SA)
$\mathbf{M_w}$	3.3×10^5	6.9×10^4
$\mathbf{M_n}$	5.9×10^4	3.9×10^3
HI $(\mathbf{M_w/M_n})$	5.6	17.7
ρ $(\mathbf{kg/m^3})$	901	900
Melt index (5kg/190°C) (g/10min)	5.8	–
HDT (°C)	58.5	–
$\mathbf{T_m}$ **(°C)**	164.0	159.0
$\mathbf{T_c}$ **(°C)**	117.5	98.5
$\lambda_\mathbf{m}$ **(%)**	41.8	40.5
$\lambda_\mathbf{c}$ **(%)**	41.9	42.5

T_m and T_c are the temperature obtained from the melting and the crystallization peaks, respectively.

λ_m and λ_c are the crystallinity obtained from the melting and the crystallization curves, respectively.

Table 2. Physical properties of the talc mineral as received

Density $(\mathbf{kg/m^3})$	**Specific Surface (BET)** $(\mathbf{m^2/g})$	**Mean Particle Size** (μm)
2750	5.3	10.1

Present study has been performed over four different samples in order to ascertain the different behaviour of the neat PP and the effect on it of the solely reinforcement, the solely interfacial agent (iPP-SA) and both of them. So, the compounds studied were a pristine PP (iPP); PP incorporating iPP-SA (named as iPP/iPP-SA); and two composites (without and with the interfacial agent, respectively) compounded at

75/25w/w PP/talc ratio, and resulting in composites named as iPP/talc and iPP/talc/iPP-SA, respectively. The interfacial agent (iPP-SA) was incorporated by replacing 1.5% of PP in the composite [7]. The amount of both iPP-SA and talc were chosen because these were identified as the optimal in improving ultimate properties and in maximizing interfacial variations in previous works by authors [8-10]. The materials (components) were first dry blended and then compounded by feeding the mixture into a counter-rotating twin-screw extruder Collin ZK-50 working at 85rpm with a temperature profile from 200 up to 220ºC [1]. The pelletized compounds obtained (after conditioned as described elsewhere [1]) were compression moulded in a Dr. Collin press at 180 and 3.3MPa two step applied and quick cooling at constant pressure [1]. The compound quality was checked by ash content and optical microscopy analysis of the samples as described elsewhere [11]. The absence of any kind of moulding defects such as inner bubbles or holes, warpage, etc., in the finished plies was always verified. After a conditioning period at room temperature and 50% R. H., of at least 48 hours, the plies were ready to be cut and shaped into prismatic specimens (21.2 × 10.4 × 4mm) according the specification of dimensions imposed by the DMA measurement mode.

Dynamic-mechanical measurements were performed under reverse loading (torsion) mode in a TA-983 Dynamical Spectrometer. The test frequency and oscillation amplitude were fixed at 1Hz and 0.2mm, respectively, with a clamping distance of 14.5mm. Meanwhile, the thermal scanning was from – 30 up to 150ºC at a heating rate of 2ºC/min.

Additionally, and in order to check the accurate processing history of the samples studied, thermal properties measured under dynamical conditions were obtained by operating a differential scanning calorimeter DSC-7 (Perkin-Elmer) previously calibrated with indium standards. All the samples were scanned at heating/cooling rates of 10ºC/min from 40ºC up to 200ºC under nitrogen atmosphere. After the first heating step, each sample was left in the molten state (softening state) for 5 minutes. The cooling scan was then begun, followed by the second heating step. Enthalpy calculations were performed by using the DSC-7 software. Each compound (approx. 10mg) was tested for three times in order to ensure reproducibility of data. The crystalline content was determined by considering 209J/g as the standard enthalpy for a hypothetical 100% crystalline polypropylene [12]. In all the cases, the enthalpy values were corrected by considering the real amount of polymer in the sample. Table 3 compiles some thermal properties of these samples.

3. Results and Discussion

Dynamic Mechanical Analysis (DMA) it is a very versatile technique that can provide, if adequately used, more information than any other single characterization method. The study of polymer based materials under cyclic mechanical and thermal conditions (DMA) allow the determination of both their plastic and viscoelastic behaviours separately. It can be said that DMA provides a direct link between the chemical make-up of a material and its mechanical behaviour. However, it is well assumed that the difficulty of this technique lies in the understanding of macroscopic measurements in terms of their microscopic origin [13].

This work has been purchased by distinguishing five different zones or intervals in the dynamic-mechanical spectra wherein a different (typical) mechanical behaviour of the material can be appreciated. These are dependant of the kind of relaxation phenomena taking place. Thus, these can be clearly appreciated when plotting any dynamic-mechanical parameter (G', G'', and $\tan\delta$) versus temperature.

Otherwise, it must be considered that the presence of mineral particles and/or interfacial agent is expected to cause higher crystalline content due probably to nucleation effects, as it can be observed from values for crystalline content (λ) listed in Table 3. Also, it is well worth to use a parameter $\Delta\lambda$ (as defined and listed in Table 3) [1, 14-17] as a proper method to estimate the processing-properties relationship by the thermal and un-sheared reorganization possibilities through the amorphous-crystalline inter-phase. Hence, values for $\Delta\lambda$ close to zero would be indicating a well-balanced processing step [1, 14-17]. The latter implies that when a polymer based material is characterized, both the polymer and the way the morphologies are generated therein (processing steps) are jointly characterized.

Table 3. Thermal properties of the samples studied at present work

Sample	**T_m (°C)**	**T_c (°C)**	**λ_m (%)**	**λ_c (%)**	**$\Delta\lambda^*$ (%)**
Neat iPP	164.2	123.0	27.0	24.8	8.87
iPP/Talc	164.0	125.5	42.2	43.8	0.90
iPP/iPP-SA	163.8	122.8	40.0	41.2	– 2.91
iPP/Talc/iPP-SA	164.1	133.3	48.0	47.8	0.42

$^*\Delta\lambda = [(\lambda_m - \lambda_c)/\lambda_c] \times 100$,

where λ_m and λ_c are the crystalline content obtained from the melting and the crystallization curves, respectively.

Figure 1 shows the evolution of storage modulus (G′), loss modulus (G″), and loss factor (tan δ) with temperature for the neat polypropylene (iPP) and an unmodified iPP/talc composite (iPP/talc). It is including at present paper to give continuity to the overall characterization studies by DMA of the PP/talc system by authors looking for the processing-properties relationship and has been fully discussed in a previous work [1] by defining five study zones in the spectra. So, and in order to make easier the understanding of the further discussion, they are included here:

(1) Interval located between – 20°C and – 10°C, is that wherein the dissipation capability is the lowest possible, being this due to mere atomic vibration motions. Consequently, in this zone, the variation of G′, G″, and tan δ for each one of the measured samples must be very little.

(2) A second zone, placed between – 10ºC and 40ºC, is distinguished by the presence of the β relaxation peak or glassy transition zone, wherein (besides the former motions described) short range diffusion motions at a chain segment level occur.

(3) Interval between 40ºC up to 80ºC. This one is characterized by the so called rubbery to elastic plateau transition associated to rapid short range diffusion motions that are sharply dependent on the molecular mass and the chain entanglement density.

(4) A fourth interval is the placed above 80ºC till 140ºC, wherein the polymer macromolecules improve greatly their capability to flow and, consequently, easily participate in dissipative phenomena through long range configuration changes.

(5) Above 140ºC. This one is distinguished for being the interval wherein the material is passing from the softening state to the liquid state and, consequently, the curves tend to converge each other [1].

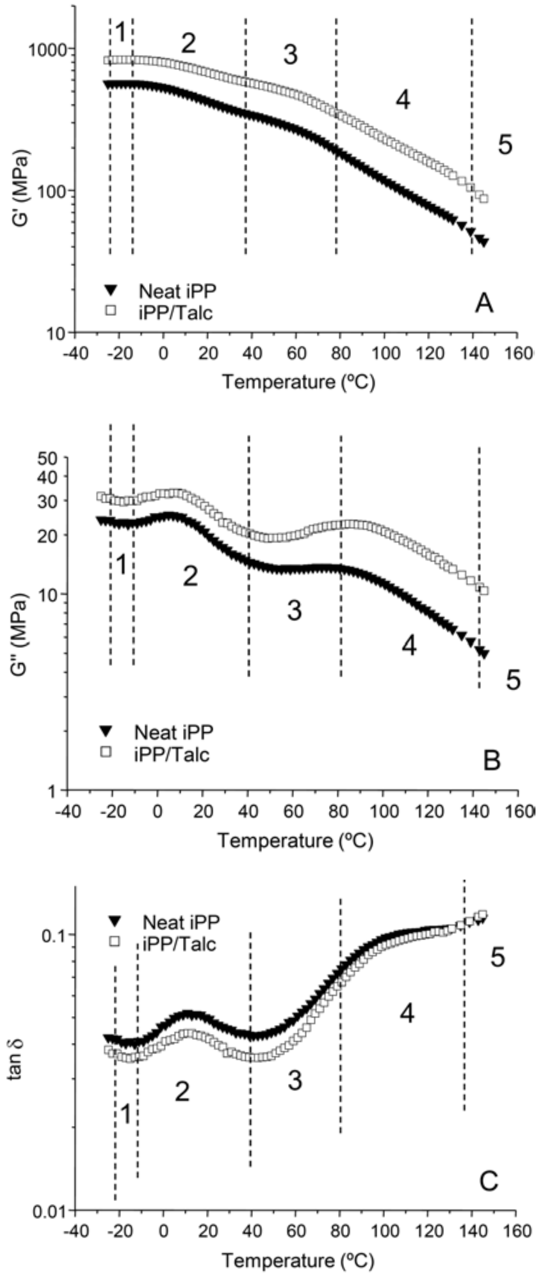

Figure 1. Evolution of storage modulus (A); loss modulus (B); and loss factor (C) with temperature for the neat iPP and the unmodified iPP/talc composite indicating the five different zones considered in the study (1 to 5).

3.1. iPP and iPP/Talc behaviour in absence of interfacial agent

As mentioned before, in a previous work [1], the interested reader can find a full discussion on the dymanic-mechanical parameters for the neat iPP and the iPP/talc system in the absence of any interfacial agent as plotted in the (Figure 1). At the effects of the present paper, mainly it is worth to note that the talc particles act as disturbing component in the iPP matrix causing a decrease in its viscous response, a decrease in the phase out between G' and G" and, in consequence; a much more elastic behaviour of iPP/talc respecting iPP. The latter was in agree with the higher crystalline content of the iPP matrix in the composite than in the neat iPP and with a lower reorganization possibilities through the amorphous-crystal inter-phase on the composite, as deduced from the value for the PP/talc $(\Delta\lambda = 0.9)$ and neat PP $(\Delta\lambda = 8.87)$ such as listed in Table 3. Further, if as it was discussed elsewhere [1, 2, 17-21], values for $\Delta\lambda$ close to zero implies a well balanced processing step, it must be mentioned here that the processing conditions for the composite are closer to the optimum $(\Delta\lambda = 0.9)$ than those for the neat PP $(\Delta\lambda = 8.87)$. It must be noted (and for the purposes of this investigation) that the processing conditions were chosen in order to optimize the mechanical performance of the PP/talc system and were remained constant to process the neat PP in order to further comparisons. In fact, if we compare the crystalline level of the iPP matrix as received (Table 1), it shows crystalline contents similar to those in the composites, Table 3, which otherwise would be in agree with an optimized processing window to obtain the PP pellets, showing a well amorphous-crystal interface $(\Delta\lambda = -0.24)$. The latter implies the processing steps play prime role that is often dismissed in plenty of works in literature [1, 2]. As said above for a full discussion on the evolution of G', G'', and tan δ parameters as plotted in the Figure 1, the interested reader is kindly submitted to the previous work by authors [1]. This work is devoted to the specific DMA transitions observed on either the PP/talc composite as well on the neat PP incorporating a 1.5 percent of an interfacial agent based on a commercial chemical modified maleic anhydride isotactic polypropylene, and assigned to the isotactic nature of this interfacial modifier.

3.2. iPP and iPP/Talc behaviour: Influence of the interfacial agent

Figure 2 compiles the plots showing the evolution of G', G'', and $\tan\ \delta$ with temperature for the neat iPP and iPP/talc (mentioned up these lines and fully discussed by authors [1]) as well as for those corresponding to the behaviour of an iPP and a composite incorporating 1.5% of the interfacial modifier from the matrix side (iPP-SA) defined in the experimental section.

The changes in the dynamic thermo-mechanical parameters caused by the presence of, on one hand, 25% of talc in the polypropylene bulk (iPP/talc), and on the other, of a mere 1.5w/w percent of iPP-SA in the composite (iPP/talc/iPP-SA) or as just as an additive into the neat polypropylene matrix (iPP/iPP-SA) are clearly observed in Figure 2. Besides, it is perhaps noteworthy to mention here the fact that the presence in the material of other substance rather than the mere PP matrix implies changes not only in the crystalline content but in the reorganization possibilities through the amorphous-crystal interphase. This can be observed in Table 3, where values for iPP/talc $(\Delta\lambda = 0.9)$, iPP/iPP-SA $(\Delta\lambda = -2.91)$, and iPP/talc/iPP-SA $(\Delta\lambda = 0.42)$ are closer to zero than neat PP $(\Delta\lambda = 8.87)$. It must be said that (under this criterion) the presence of the interfacial agent in the iPP/iPP-SA compound implies a displacement of the thermo-dynamical equilibrium towards negative values that implies higher values for crystalline content in the cooling scan than in the heating scan [21], and just the opposite than all the others (Table 3). The latter implies that this parameter should exhibit a value closer to the equilibrium in the case of the iPP/talc/iPP-SA composite $(\Delta\lambda = 0.42)$ respecting the other compounds investigated.

The fact that the values for G′ and G″ in the case of iPP/iPP-SA increases respecting to those in the unmodified neat iPP compound indicates that iPP-SA appears as a disturbing agent for the polypropylene matrix (Figure 2A and 2B).

As aforementioned, it is possible to observe that both the modified composite (iPP/talc/iPP-SA) and the neat PP incorporating iPP-SA (iPP/iPP-SA) do not exhibit differences in their dynamic mechanical thermal behaviour below – 10ºC (where the dissipation mechanisms are restricted to atomic motions), if compared to those of the unmodified systems (iPP and iPP/talc). Indeed, the presence of the interfacial modifier in the composite (iPP/talc/iPP-SA) makes that G′, G″, and tan δ values tend to approach to those of the neat PP, and results very different to that of the unmodified iPP/talc system. So, an increase in the affinity between the talc particles and the polypropylene matrix by the presence of iPP-SA can be concluded. Moreover, the same values for G″ are obtained in absence of talc but presence of interfacial agent (iPP/iPP-SA). This let assign the dissipation capabilities at this level to the succinic anhydride groups in iPP-SA disturbing the polypropylene matrix (Figure 2B).

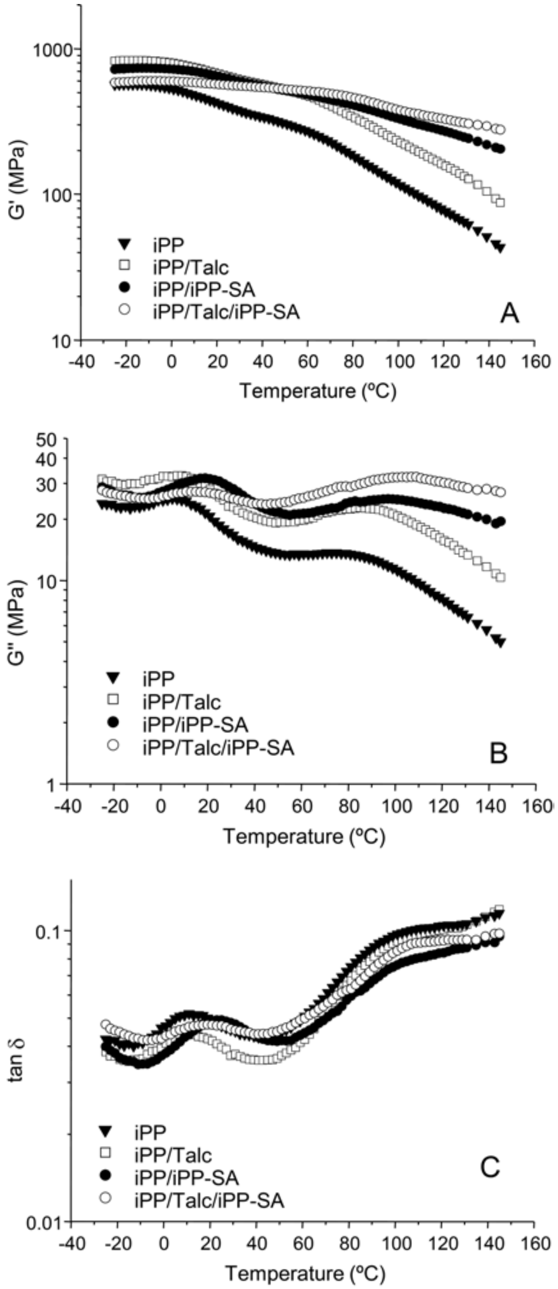

Figure 2. Evolution of storage modulus (A); loss modulus (B); and loss factor (C) with temperature for the indicated samples.

In the second zone, above – 10ºC and up to 40ºC, we can observe the β relaxation peak typical of the glass transition occurring in the "free" amorphous phase of the polypropylene matrix. Both the decrease in $\tan\delta$ as well as the widening and even asymmetry of the β relaxation peak indicates the decrease in the undisturbed/disturbed amorphous ratio in a semi-crystalline polymer [23]. So, and according to data plotted in Figure 3, while the unmodified iPP/talc system exhibits lower $\tan\delta$ values and a slightly lower peak area than the neat iPP (Table 4) with a temperature maximum placed at 10ºC, the iPP/talc/iPP-SA system exhibits a widening and a decrease in the peak area caused by the decrease in the free amorphous phase able to participate in the cooperative segmental motions that characterizes the glass transition. Such features are coherent with the role played by the succinic anhydride groups grafted in the interfacial agent (iPP-SA). They are able to increase the affinity between the surface of the talc particles and the polypropylene matrix as it can be concluded from the increase in the asymmetry and from the up-displacement of the maximum temperature of the β relaxation peak for PP/i-PP-SA too. So, and due to the absence of talc particles, the iPP/iPP-SA compound is showing a peak area, (i.e., amount of "free" amorphous phase) almost three times higher (0.61) than the corresponding to either the neat iPP (0.27) or the iPP/talc (0.24) as listed in Table 4.

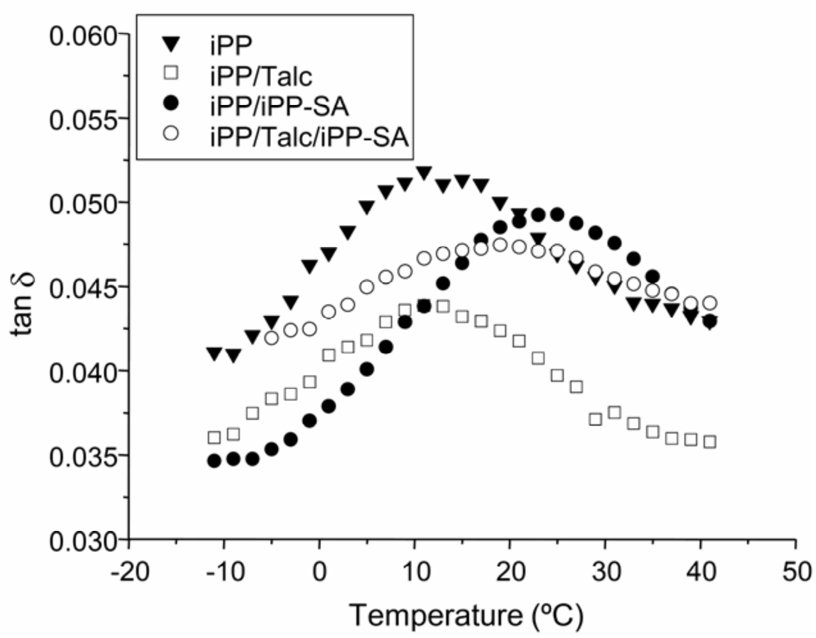

Figure 3. Evolution of the loss factor (tan δ) with temperature for the indicated samples in the interval between – 10°C and 40°C (second or β transition zone).

Table 4. Parameters associated to the β transition for the indicated samples

Sample	**Area (a.u)**	**T_{max} (°C)**	**tan δ**
iPP	0.27	10	5.15
iPP/Talc	0.24	10	4.30
iPP/iPP-SA	0.61	24	4.90
iPP/Talc/iPP-SA	0.38	20	4.70

According to the above discussed, Figure 4 shows the G” evolution of the rubbery plateau located between 40°C and 80°C. It is perhaps noteworthy to remember that the latter is dependent of the molecular mass and the chain entanglement density of the polypropylene matrix. So, we can observe that only the neat iPP exhibits such rubbery plateau, while above 60°C, all the other three materials (iPP/talc, iPP/iPP-SA, and iPP/talc/iPP-SA) increase G″ values and so the α relaxation peak appears. Moreover, the plots corresponding to both PP/talc/iPP-SA and iPP/iPPSA are showing a shoulder (or α′ peak) located close to 80ºC (76.4ºC and 81.7ºC, respectively) that agrees with the molecular weight dependant relaxation phenomena occurring up close 80ºC. This α′ peak can be assigned to any kind of contribution to the α relaxation peak due to the interfacial agent (iPP-SA), and related to the isotactic and semicrystalline nature (equally than iPP) but with higher heterogeneity index (HI) and lower molecular weight than iPP (Table 1). Also, the interfacial agent can be said to be highly constrained at the interfacial matrix/particle interfaces where it is preferably allocated [1, 18-21]. The latter is in excellent agreement with the secondary crystallization phenomena occurring on isotactic polypropylenes grafted with polar monomers as discussed by authors elsewhere [15] and observed in findings by others [22, 23].

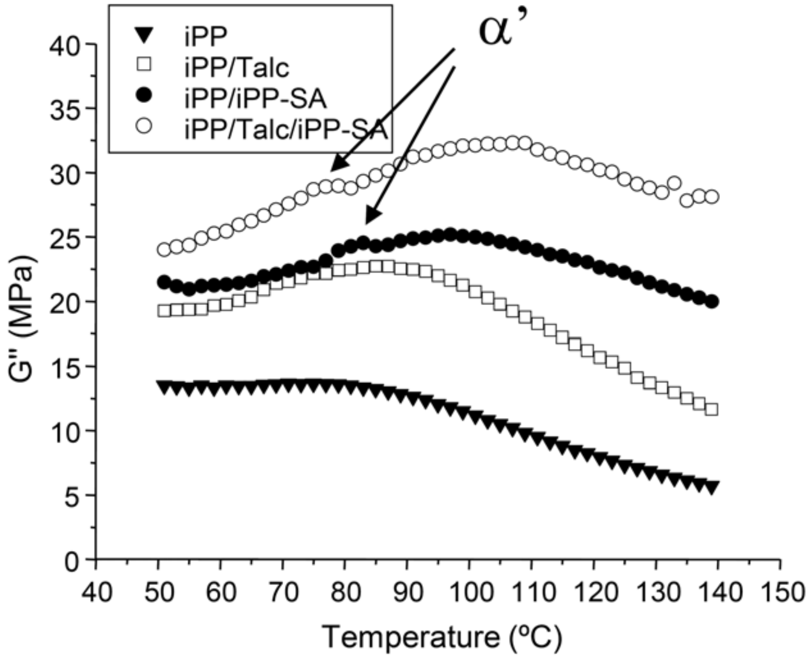

Figure 4. Evolution of loss modulus (G″) with temperature for the indicated samples in the interval between 50ºC and 140ºC.

Finally, the high stabilization in the G' values with temperature observed for the PP/talc/iPP-SA compound (Figure 2A) exhibiting an almost constant value up of the 80ºC (where the maximum at the α relaxation peak is placed) clearly shows the enhancement in the mechanical behaviour of the modified composite further of being coherent with the interpretation given to the response mechanisms of the different materials here discussed.

4. Conclusion

Dynamic-mechanical analysis (DMA) has been used here in the understanding of the macroscopic responses and measurements of the polypropylene/talc system in terms of their microscopic, meso, and nano origin. As expected, the incompatibility between the inorganic talc particles and the organic isotactic polypropylene matrix has been diminished by the presence of an isotactic succinic anhydride grafted polypropylene (iPP-SA) acting as interfacial modifier from the matrix side in the polypropylene/talc composite system.

Hence, the five different thermo-mechanical response zones of the materials has been clearly identified and used to ascertain the efficiency of the interfacial modification performed. The latter has been undertaken by following the evolution of the main relaxation phenomena of the polypropylene matrix (i.e., the β relaxation peak as governed by the glass transition on the "free" amorphous phase and the α relaxation peak assigned to the crystalline phases) recorded on those compounds with disturbing component (talc and/or iPP-SA) for the morphology of the iPP matrix.

The fact that a little amount of solely interfacial agent affects greatly the matrix behaviour has been also confirmed by this technique. The presence of the interfacial agent, in spite of the little amount incorporated, has been showed to be detected by their own α peak (α') in the samples containing it (iPP/iPP-SA and iPP/talc/iPP-SA). Hence, it can be concluded that the ultimate properties of the materials studied are the result of the coupled effects of the reinforcement and/or the interfacial agent with the polypropylene matrix. Finally, thermal parameters determined by DSC agree with the expected from the processing steps.

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