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Functionalized polypropylenes as efficient dispersing agents for carbon nanotubes in a polypropylene matrix; application to electromagnetic interference (EMI) absorber materials

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ABSTRACT

Carbon nanotubes (CNTs) have been dispersed within polypropylene with the purpose to prepare electromagnetic interference (EMI) absorbers. In order to limit the reflectivity of the electromagnetic waves at the interface of the materials while achieving good absorbing properties, the CNTs concentration must be kept low ($<$3 wt%) which means that a perfect dispersion must be ensured. Since CNTs do not disperse well within apolar polymer matrices such as polypropylene, two compatibilizers bearing aromatic moieties, i.e. pyrene and pyridine, able to develop π-π interactions with the CNTs have been synthesized starting from polypropylene grafted by maleic anhydride (PP-g-MA). A masterbatch is first prepared by dispersion of CNTs within the compatibilizers by melt-mixing and coprecipitation followed by further dispersion within the PP matrix. Rheological and electromagnetic characterizations of the nanocomposites have demonstrated the efficiency of these compatibilizers to promote the dispersion of CNTs in PP and the good EMI shielding effectiveness of the PP matrix at a low CNTs concentration (2 wt%).

1. Introduction

Carbon nanotubes (CNTs) are receiving steadily increasing attention from material scientists. CNTs are rolled sheets with diameter ranging from a few angstroms to several tens of nanometers. They possess very good mechanical properties because the two-dimensional arrangement of carbon atoms in a graphene sheet allows large out-of-plane distortions, while the strength of carbon–carbon in-plane bonds keeps the graphene sheet exceptionally strong against any in-plane distortion or fracture [1]. Moreover, CNTs are also known to impart electrical conductivity properties to composite even at low filler content thanks to their high aspect ratio. Recently, CNTs have been used to impart electromagnetic interference (EMI) shielding properties to polystyrene [2], polymethylmethacrylate [3], polycarbonate [4] and polycaprolactone [5,6]. Indeed, these undesirable EMI signals could cause (i) noise signal and even malfunction of the electronic appliances and (ii) radiative damage to the human body [7]. However, even if good dispersion has been achieved in specific polymer matrices by melt-blending [8,9], coprecipitation [10,11] or in-situ polymerization [12–14] without CNTs modification, CNTs are generally poorly dispersed within polymers, particularly within polypropylene [15–19], resulting in poor improvement of conductive and mechanical properties. Non convalent and covalent modifications by organic molecules, including polymer chains, are generally used to improve CNTs dispersion. “Grafting from” and “grafting to” methods have been reported for the covalent bonding of polymers onto CNTs. The “grafting from” technique relies on the immobilization of initiators onto the tubes followed by surface-initiated polymerization [20–23] while the “grafting to” technique uses polymers bearing functionality capable to react with CNTs such as azide [24] and radicals [25,26] or with complementary functional groups previously anchored on CNTs [27,28]. On its side, non covalent functionalization of CNTs relies on the use of polymers bearing aromatic molecules able to interact by π-π stacking with the graphitic surface of CNTs [29–34]. This technique has the advantage to keep the nanotube structure and electronic properties unmodified. Recently, polypropylene-graft-maleic anhydride has been used as a compatibilizer to improve the CNTs dispersion within polymer [35,36]. Although this modified polypropylene was efficient for that purpose, no significant improvement of the electrical conductivity of the polymer matrix was observed [35]. Imparting electrical conductivity to PP by the efficient dispersion of low amounts of CNTs is still very challenging [15,17,37].

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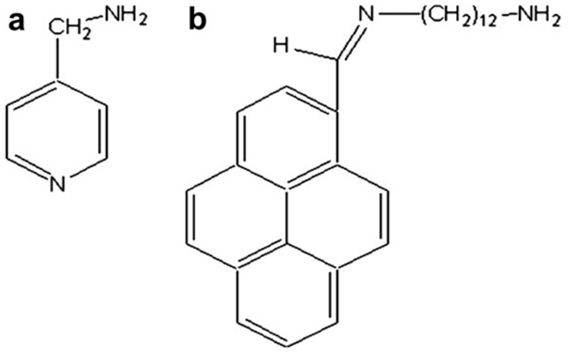

Fig. 1. Chemical structure of the molecules used to react with PP-g-MA (a) aminomethylpyridine and (b) amino-pyrene derivative.

In this paper, we report the synthesis of polypropylene grafted with aromatic moieties able to interact by π-π stacking with the surface of CNTs in order to improve their dispersion in this polyolefin. Reaction of polypropylene-graft-maleic anhydride with aminomethylpyridine (AMP) or amino-pyrene derivative (Py-NH_2) is therefore considered. Dispersion within polypropylene has been characterized by rheological and mechanical measurements, and the electromagnetic properties of the nanocomposites have been considered.

2. Experimental part

2.1. Materials

Commercially available MWNTs thin (average outer diameter: 10 nm, purity higher than 95 wt%) were supplied by “Nanocyl S.A.” Belgium and produced by Catalytic Carbon Vapour Deposition (CCVD). Polypropylene (BP GA 110 12) (PP) was received as a gift from BP Solvay and Polypropylene-graft-maleic anhydride (PP-g-MA, Priex, 20 097, 0.5 wt% MA content) was received from Solvay. Amino-functionnalized pyrene (Py-NH_2) has been synthesized by reaction between 1-pyrenecarbaldehyde (Aldrich) with 5 eq of diaminododecane in ethanol at room temperature during 16 h. The formed precipitated has been filtered, washed by ethanol and dried under vacuum. ^{1}H NMR ($CDCl_3$): d (ppm) 9.29 (1H, CHPy); 8.9 (1H, CHPy); 8.53 (1H, CHPy); 8.20–7.96 (7H, CHPy and HC=N); 3.84 (2H, CH2–N=); 2.67 (2H, CH2NH2) 1.85 (2H, CH2CH2–N=); 1.49–1.1 (20H, CH2 and NH2).

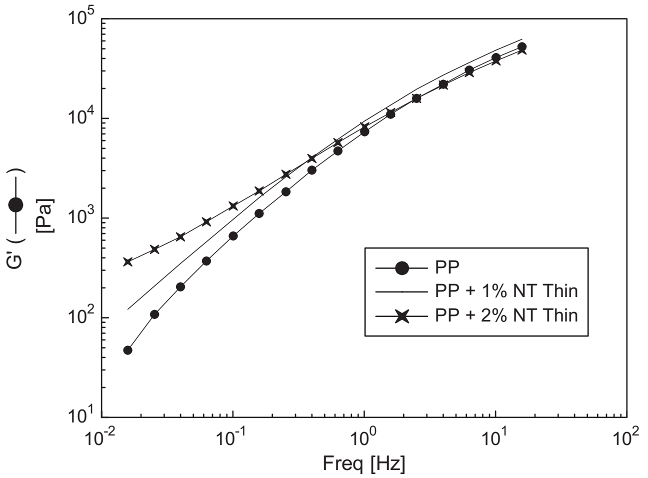

Fig. 3. Storage modulus of PP as a function of frequency for different CNT contents.

2.2. Preparation of polypropylene-graft-pyrene (PP-g-Py) and polypropylene-graft-aminomethylpyridine (PP-g-AMP)

Polypropylene-graft-pyrene (PP-g-Py) and polypropylene-graft-aminomethylpyridine (PP-g-AMP) were synthesized in a 5-cm^3 DSM microextruder by adding 5 eq (compared to maleic anhydride functions) of Py-NH_2 or aminomethyl pyridine within PP-g-MA at 160 °C under nitrogen during 10 min. The resulting polymer was solubilized in p-xylene at 120 °C and precipitated three times in methanol in order to remove the excess of the amine bearing molecules.

2.3. Dispersion of CNTs in PP-g-MA, PP-g-Py and PP-g-AMP, followed by dispersion in PP

CNTs were dispersed within PP-g-MA, PP-g-Py and PP-g-AMP by two techniques in order to prepared masterbatches to be redispersed in polypropylene. Two CNTs concentrations were used (10 wt% and 20 wt%) in order to obtain two CNTs contents in the

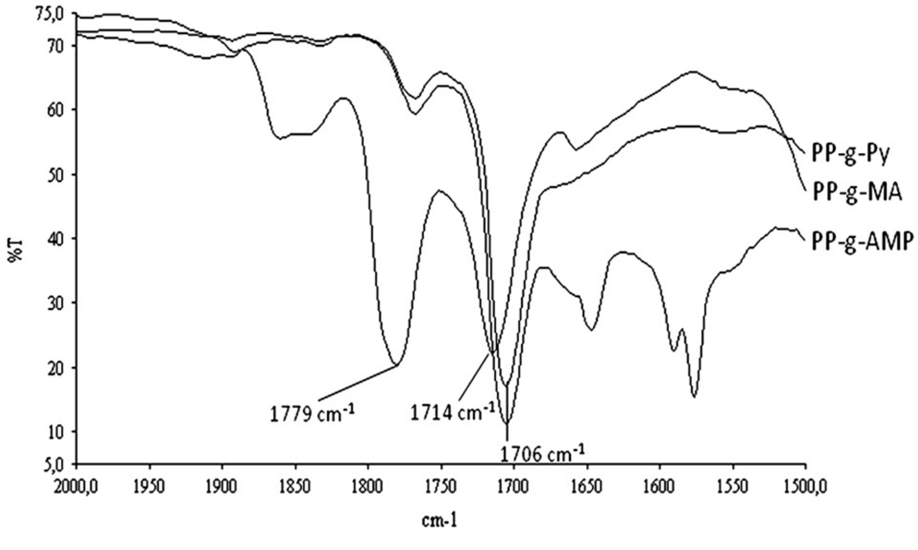

Fig. 2. FTIR spectra of PP-g-MA, the product of the reaction between PP-g-MA and the amino-pyrene derivative (PP-g-Py) and the product of the reaction between PP-g-MA and aminomethylpyridine (PP-g-AMP).

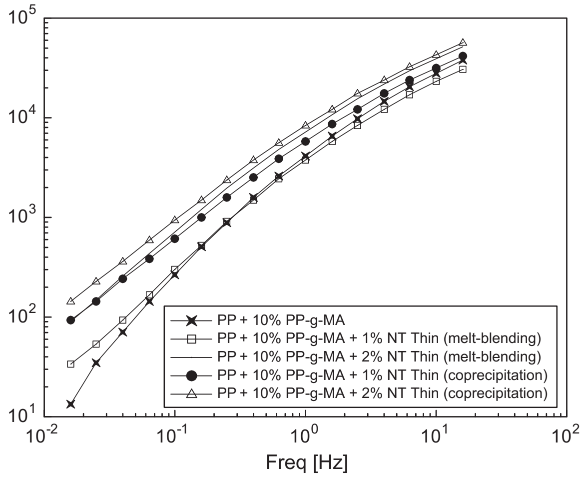

Fig. 4. Storage modulus of PP/PP-g-MA blends as a function of frequency for different CNT contents.

final PP nanocomposites (1 wt% and 2 wt%) while keeping the amount of compatibilizer constant. The first technique (melt-blending technique) consists in melt-mixing the polymer with the required amount of MWNT at 160 °C in a 5-cm^3 DSM micro-compounder under nitrogen at 200 rpm for 10 min. In the second technique (the coprecipitation technique), PP-g-MA, PP-g-Py or PP-g-AMP were dissolved in *p*-xylene at 120 °C and the required amount of CNT was added to the solution. After 30 min of an ultrasonic treatment (35 kHz, 320 W, Sonorex RK 255H) to dissociate the bundles while limiting the carbon nanotubes cutting [5], the solution was precipitated in methanol. Finally the resulting masterbatches prepared by the melt-blending and coprecipitation technique were redispersed within polypropylene by melt-mixing in a 5-cm^3 DSM microcompounder at 180 °C for 10 min. CNTs (or masterbatches) and Polypropylene (pellets) were not pre-mixed before the injection in the microcompounder but added separately.

2.4. Characterizations

MWNTs dispersions were observed with a transmission electron microscope PHILIPS M100 at an accelerating voltage of 100 kV. Thin sections (90 nm) were prepared by ultramicrotomy (ULTRACUT E from REICHERT-JUNG) at −130 °C. Micrographs were analyzed by using the megaview GII (Olympus) software.

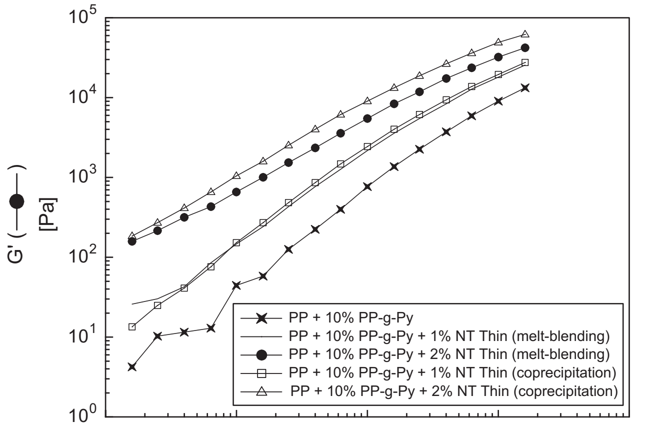

Fig. 5. Storage modulus of PP/PP-g-Py blends as a function of frequency for different CNT contents.

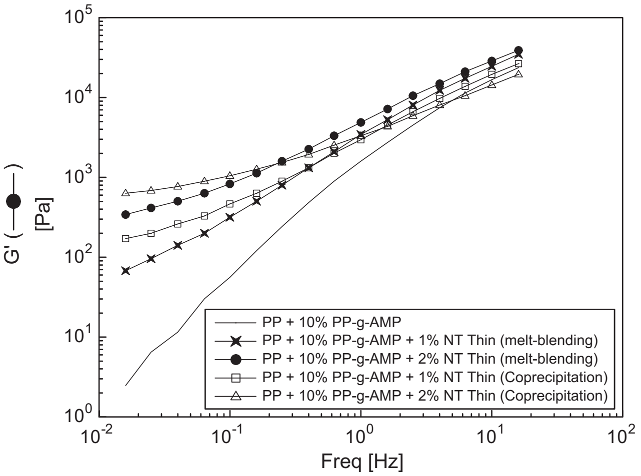

Fig. 6. Storage modulus of PP/PP-g-AMP blends as a function of frequency for different CNT contents.

Stress–strain curves were recorded at room temperature with an INSTRON tester (model DY24) at 20 mm/min tensile speed. Dog-bone samples (Thickness: 1.2 mm, working area: 40 × 5 mm) were prepared by injection molding using a 5.5 ml injection molding machine from DSM (Barrel temperature = 180 °C, mould temperature = 80 °C). For each sample, the measurements were repeated at least 3 times. The relative error was found to be smaller than 5%. The tensile modulus was calculated from the slope in the linear regime at 1% deformation.

Infrared spectra were recorded with a Perkin–Elmer FTIR 1720X. Dynamic rheological measurements were carried out with an “advanced rheometric expansion system” (ARES) from Rheometrics. Samples (diameter 25 mm, thickness 2 mm) were run at 180 °C with a strain of 1%.

Electrical measurements were performed with a Vector Network Analyser Model Wiltron 360B operating over the frequency range 40 MHz–40 GHz. Each nanocomposite sample to be characterized consists of a thin plate of same thickness (1 mm) and surface (4 × 4 mm^2) prepared by compression moulding (180 °C, 80 bars, 5 min), and is used as a microwave substrate on which a ground plane and a microstrip line are deposited [38]. This ensures broadband guided propagation of the signal inside the nanocomposite substrate. A standard calibration of the VNA is performed before measuring the microstrip configuration, in order to remove any influence of the connecting cables and of their transitions to the sample. Measurements provide the so-called vector *S*-parameters S_{11} and S_{21}. Parameter S_{11}, measured when a short-circuit reflective termination is placed at the output of the sample, corresponds to the reflectivity, $R = 20\log_{10}|S_{11}|$, i.e. the reflected power measured at input interface of the substrate, normalized to input power. Parameter S_{21} corresponds to normalized transmission through nanocomposite. Hence the shielding effectiveness SE is simply equal to $-20\log_{10}|S_{21}|$. From those S_{ij} measurements the conductivity of each nanocomposite is also extracted.

3. Results and discussion

Novel polymeric compatibilizers able to improve the CNT dispersion within polypropylene (PP) were prepared by reaction in a 5 cm^3 mini-extruder at 160 °C between polypropylene-graft-

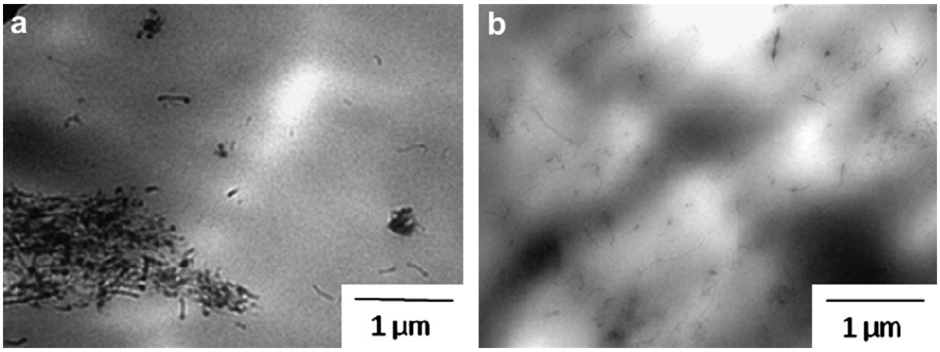

Fig. 7. TEM micrographs of (a) PP + 1% NT Thin and (b) PP + 10% PP-g-AMP + 1% NT thin (coprecipitation).

maleic anhydride (PP-g-MA) and two different molecules: aminomethylpyridine (AMP) and an amino-pyrene derivative ($PyNH_2$) (Fig. 1). These molecules were chosen because (i) they interact by π-π stacking with CNTs, and (ii) their amine group serves to anchor the molecules to PP by reaction with maleic anhydride functions of PP-g-MA. The extent of the reaction between the amino groups and the anhydride functions was assessed by FTIR with the disappearance of the characteristic peaks of anhydride (1779 cm^{-1}) and carboxylic acid (1714 cm^{-1}) and the appearance of the characteristic peak of amide functionality (1706 cm^{-1}) (Fig. 2).

CNTs were then dispersed within these two compatibilizers by melt-blending and coprecipitation in order to prepare "masterbatches" that could be further dispersed within polypropylene. Masterbatches based on PP-g-MA have also been prepared as a reference in order to attribute the eventual beneficial impact to the presence of compatibilizing function and not to the addition of low viscous polymer. Since the contrast between CNT and the semicrystalline polypropylene in transmission electron microscopy (TEM) is relatively poor, CNT dispersions were mainly characterized by rheological and conductivity measurement. Indeed, the frequency dependence of the storage modulus (G') of the base polymer is sensitive to the percolation of nanofillers in the low frequency range [12–14,35]. Beyond the percolation threshold, a dramatic increase of the storage modulus is generally observed at low frequency which is indicative of transition from a liquid-like to a solid-like viscoelastic behavior. Figs. 3–6 show the evolution of the storage modulus as a function of frequency for PP/CNT nanocomposite without compatibilizer and in the presence of PP-g-MA, PP-g-Py and PP-g-AMP, respectively. When CNTs are directly dispersed in PP by melt-blending (Fig. 3), no significant effect of the CNT loading is observed at low frequency on the resulting composite, a small increase of the storage modulus starts to be detected only at 2 wt% CNTs. The influence of CNT on the rheological behavior is also very small when they are dispersed in PP in the presence of 10 wt% PP-g-MA by melt-blending suggesting that the dispersion is also very poor (Fig. 4). When dispersed by the coprecipitation technique, a small improvement is observed but the transition from liquid-like to solid-like viscoelastic behavior is not achieved yet. In the presence of PP-g-Py as compatibilizer (10 wt%), the effect of the CNTs loading on the storage modulus is more pronounced, suggesting a better CNTs dispersion in PP. This increase is however similar in the whole frequency range and no plateau is observed at low frequencies which indicates that the percolation threshold is not reached yet. No major difference exists between the nanocomposite prepared by melt-blending and coprecipitation. On the other hand, the dispersion of CNTs in the presence of PP-g-AMP has a drastic effect on the rheological behavior of PP even at loading as low as 1 wt%. A plateau is clearly observed at low frequencies, suggesting that the percolation threshold is reached. This effect is even more pronounced when the CNTs are dispersed within the compatibilizer by coprecipitation, which indicates that the CNTs dispersion is improved by coprecipitation compared to melt-blending. This latter technique is very suitable for the CNTs dispersion since it allows the use of a short ultrasonic treatment recognized as a useful tool for the disentanglement of CNTs bundles. TEM micrographs confirm the efficiency of the PP-g-AMP masterbatch prepared by coprecipitation. Indeed, when CNT nanotubes are directly dispersed within polypropylene, large agglomerates are observed (>1 µm) with only few CNTs

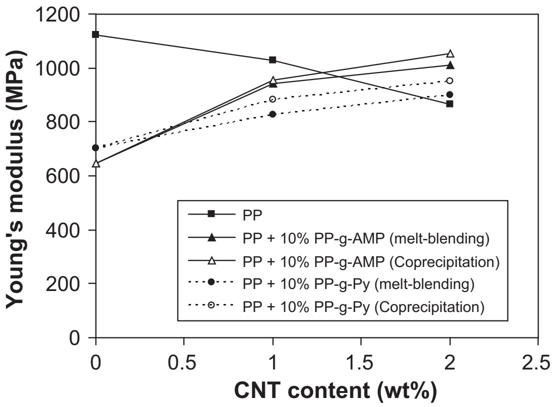

Fig. 8. Young's modulus for PP filled with CNTs (standard deviation = ±5%).

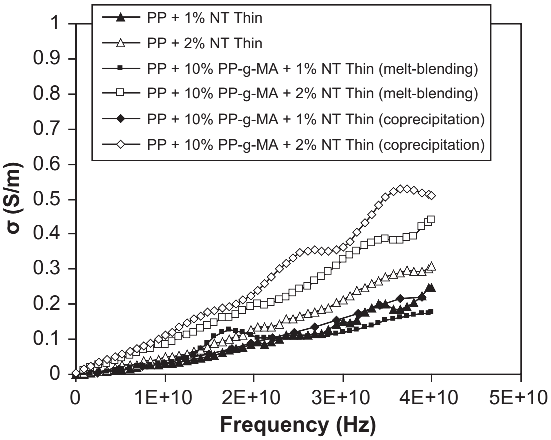

Fig. 9. Electrical conductivity as a function of frequency for PP/MWNT nanocomposites in the presence of PP-g-MA.

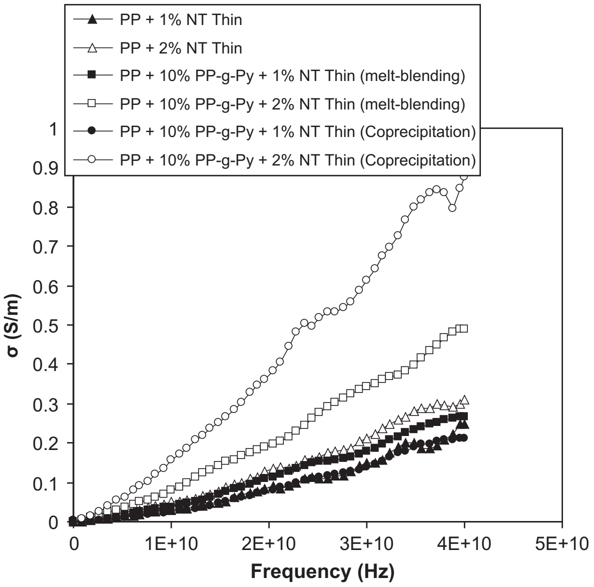

Fig. 10. Electrical conductivity as a function of frequency for PP/MWNT nanocomposites in the presence of PP-g-Py.

isolated while a great amount of CNT are isolated when the masterbatch route is used (Fig. 7).

Fig. 8 illustrates the effect of CNTs on the Young's modulus of PP in the presence or not of the compatibilizers. The addition of CNTs in polymers is known to reinforce them, provided that the dispersion is good enough. On the other hand, when the dispersion is poor, CNTs bundles form stress concentration similar to those caused by voids in composite system which results in a decrease of the Young's modulus [39]. This behavior is observed when CNTs are dispersed in neat PP which confirms the poor dispersion of the nanofiller in this case. The addition of the compatibilizers (PP-g-Py and PP-g-AMP; 10 wt%) to PP matrix is however detrimental to the Young's modulus of PP that decreases significantly (Fig. 8). This effect is generally observed when PP-g-MA is added to PP [40] and can be explained by the low molecular weight of the modified polymer (PP-g-MA) caused by the shortening of the chains during the functionalization of PP by maleic anhydride. Importantly, the addition of CNTs within PP in the presence of the compatibilizers increases significantly the Young's modulus. In both the cases, the effect is more important when the coprecipitation technique is used in direct relation with a better CNTs dispersion. The Young's modulus increase is more pronounced with PP-g-AMP (60% at 2 wt%) as the compatibilizer which confirms its higher efficiency to disperse CNTs in PP, in total agreement with the rheological observations. At this stage, it is important to note that, for all these experiments, two different CNT concentrations in the masterbatches have been used in order to keep the concentration in compatibilizer in the final composite constant (10 wt%). Indeed, the addition of low viscous polymer can have an influence on the quality of dispersion and on the mechanical properties of the final nanocomposite [17]. It is therefore important to compare composites with the same amount of compatibilizer.

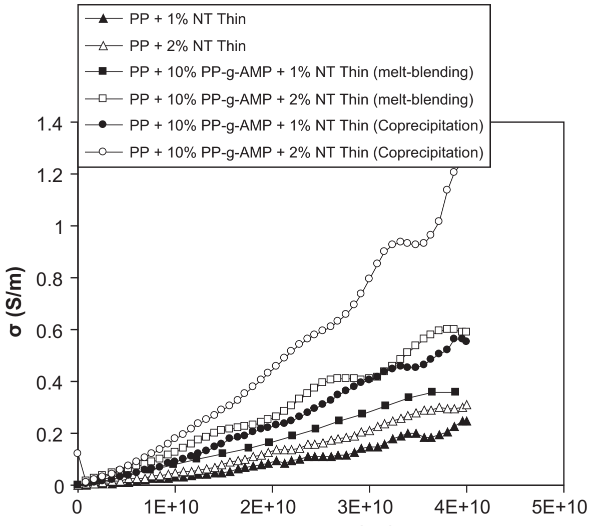

Fig. 11. Electrical conductivity as a function of frequency for PP/MWNT nanocomposites in the presence of PP-g-AMP.

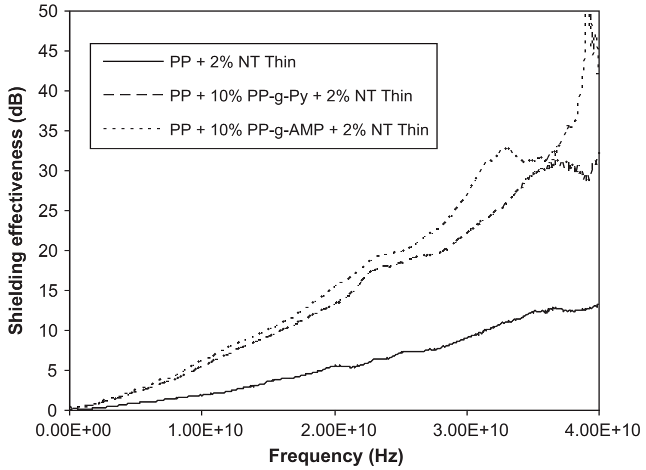

Fig. 12. Shielding Effectiveness (SE) as a function of frequency for PP/MWNT nanocomposites in the presence or not of PP-g-Py or PP-g-AMP.

The electrical conductivity of the CNTs/PP nanocomposites was then measured in the microwave frequency range (40 MHz–40 GHz) using microstrip transmission lines. Electrical conductivity is of the utmost importance for electromagnetic interference (EMI) performances, because it expresses the intrinsic ability of the material to dissipate electromagnetic waves [38]. As a rule, a good EMI shield must exhibit a conductivity of about 1 S/m. Results shown in Figs. 9–11 are in qualitative agreement with the

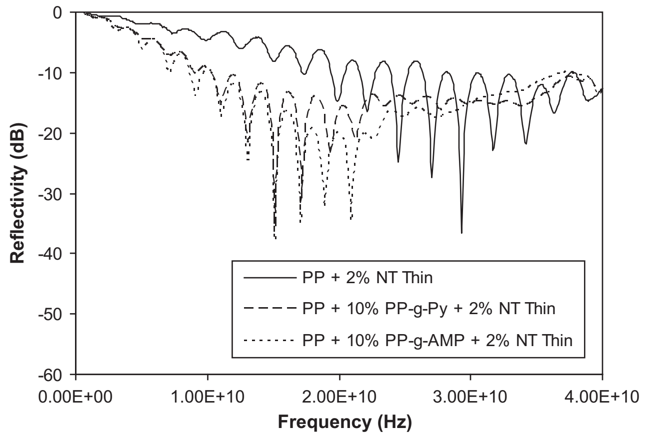

Fig. 13. Reflectivity as a function of frequency for PP/MWNT nanocomposites in the presence or not of PP-g-Py or PP-g-AMP.

rheological behavior of the nanocomposites. Indeed, over the whole frequency range, the measured electrical conductivities remain low (<0.3 S/m at 1 wt% and <0.5 S/m at 2 wt%) for CNTs/PP nanocomposites and CNTs/PP-g-MA/PP nanocomposites in line with the poor dispersion of the nanofiller (Fig. 9). Addition of PP-g-Py only allows a small increase of the electrical conductivity at 1 wt% CNTs which becomes however important at 2 wt% when the coprecipitation is used to disperse CNTs in the compatibilizer (Fig. 10). On the other hand, the use of PP-g-AMP as compatibilizer is able to increase significantly the electrical conductivity over the whole frequency range. A two-times increase (as compared to dispersion of CNTs in neat PP) in case of melt-blending and a three times increase in case of coprecipitation are observed at 1 wt% content (Fig. 11). This compatibilizer is thus efficient for dispersing CNTs in PP since it allows the targeted value for good electromagnetic absorber to be reached at a 2 wt% content when the coprecipitation method is used.

The electromagnetic absorption performances are illustrated in Fig. 12 for three samples with 2% CNT concentration: CNTs/PP, CNT/PP-g-AMP/PP, and CNTs/PP-g-Py/PP. For each sample, the measured Shielding Effectiveness (SE, defined as $SE = 10 \log (P_{in}/P_{out})$ where P_{out} is the power transmitted through the material and P_{in} is incident power) is in direct correlation with its conductivity. Both compatibilizers are efficient to greatly improve the shielding effectiveness of PP/CNT nanocomposites with a higher effect with PP-g-AMP. The SE continuously increases with the frequency. The target value of 20 dB (1% of the incident power is transmitted) is reached at 20 GHz with both compatibilizers while it is never achieved for PP/CNT nanocomposites. At lower frequencies, the SE is also interesting since compatibilized nanocomposites exhibit an SE of 8 dB (15% of the incident power is transmitted) at 10 GHz. It has to be noted that our nanocomposites (with low CNT content (<2 wt%)) function mainly as absorbers which is quite different to most of the systems published in the literature (with high content of CNT (>10 wt%)) [2–4] where the contribution to SE mainly comes from reflection at input interface. Indeed, reflectivity measurements at Fig. 13 show that when a sufficient conductivity is reached, the reflectivity is strongly decreased, meaning that signal is attenuated twice inside the material: over the forward path, from the input to the reflective termination used for the measurement of reflectivity, and over the reverse path, from the termination back to the input. Other said the nanocomposite material exhibits sufficient absorption capability to cancel any reflection from targets. The higher the conductivity, the wider the frequency range where the composite behaves as a good EMI absorber. Indeed composites with compatibilizers exhibit a reflectivity lower than −10 dB (10% of the incident power is reflected) starting from 10 GHz, while it is only the case above 30 GHz for neat PP.

4. Conclusions

New compatibilizers for the efficient dispersion of CNTs within PP were prepared by reactions of aminomethylpyridine and an amino-pyrene derivative with commercially available polypropylene-graft-maleic anhydride in the melt. The coprecipitation and melt-blending techniques were used to disperse the nanofillers within the compatibilizers, before dispersion in PP by melt-blending. The coprecipitation appeared to be the most efficient technique most likely because it allows the application of an ultrasonic treatment. In contrast to unmodified PP-g-MA, the two compatibilizers were able to significantly increase the conductivity of PP/CNTs nanocomposites. The best results were obtained when using polypropylene grafted by aminomethylpyridine as the CNTs compatibilizer. Indeed, the electrical conductivity is three times increased compared to the dispersion of CNTs in neat PP. The target value for good electromagnetic absorbers is then achieved at 2 wt% CNTs content while the mechanical properties of the polymer matrix are improved.

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