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Polypropylene/Polystyrene and Clay Nanocomposites: Elaboration, Morphological and Thermal Characterizations

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Abstract: This study focuses on the preparation and characterization of polymer blends-clay nanocomposites based on thermoplastic polymers and nanometric clay, with the aim of investigating the influence of compatibilizing agents namely grafted polymers. The polypropylene (PP)/polystyrene (PS)/ montmorillonite (CA) clay blends were prepared by melt processing using PP-grafted silane (PP-g-VTEOS) as compatibilizer. At fixed content of clay (5 wt%), addition of PP-g-VTEOS was found to create, firstly, PS droplet well dispersed and distributed with reduced size proved by Scanning Electronic Microscopy (SEM) images and at the same time showed the improvement of the dispersion and distribution of clay. X-ray Diffraction(XRD) spectra of nanocomposites revealed that intercalated/partial exfoliated structures were obtained. Thermogravimetric analysis showed that the degree of dispersion and distribution of clay within polymer matrix plays a vital role in property improvement. This study thus highlights the crucial role of compatibilizing agents in the development of high-performance polymer nanocomposites, paving the way for new formulations aimed at high value-added applications.

Keywords: Immiscible PP/PS Blends, Clay, Silane Grafting, Nanocomposites, Thermal Characterisation

1. Introduction

Poor compatibility between thermodynamically immiscible polymer blends leads to weak interfacial adhesion and unstable morphology. In order to enhance miscibility some interfacial stabilizers, known as compatibilizers are often used. Such a compatibilizer may be a homopolymer [1], a graft, block or star copolymer and their choice depends on the nature of polymers and their characteristics [2-4]. The components of good polymer blends are selected in such a way that the primary advantages of the first one would compensate the deficiencies of the second and vice versa considering their cost and the objectives [5]. The use of nanocharge was fund as one of ways to improve compatibility of polymer blends and leads to obtain high-performance polymeric materials [6]. Polypropylene (PP), polystyrene (PS) and their nanocomposites have found many applications in industry due to their low cost, easy processing and recyclability but PP/PS blends have poor compatibility due to the large differences in their polarities, chemical nature, and high interfacial tension. Generally, most immiscible polymer blends consist of droplets of a dispersed phase in a continuous phase, and these droplets may approach each other and finally coalesce [7]. Many researches were carried out focusing on compatibilization of PP/PS blends using different copolymers, reactive polymers, or nanoparticles as compatibilizers [8-11]. The most used compatibilizer polymer based is MA grafted polypropylene (PP-g-MA) derivative [12, 13]. It consists of modifying polymer with a wide variety of polar groups leading to many outcomes such as enhancing compatibilizing effect or creating covalent bonds between two phases leading to better dispersion/distribution and favorable specific interactions. As nanoparticles, Ting Wu et al. used a nanocharge like montmorillonite clay to improve the interfacial adhesion and the strategy was found to reduce the PS droplet size and improve the compatibility of PP/PS [6]. To improve the interfacial adhesion of PP/PS blends the use of silane grafted PP (PP-g-Si) was not done yet.

Present work aims at preparing nanocomposites based on PP/PS/clay and compatibilized with vinyl triethoxysilane (VTEOS) by melt processing in twin-screw extruder. VTEOS has the advantage to play the role of compatibilizer. To the best of our knowledge, this is the first report on the use of silane to compatibilize the PP/PS blends. The aim of this study is therefore to investigate the effect of modified polymer on structural, morphological and thermal properties of the obtained nanocomposites and their relationship. The strategy of the preparation of the nanocomposites are presented in figure 1.

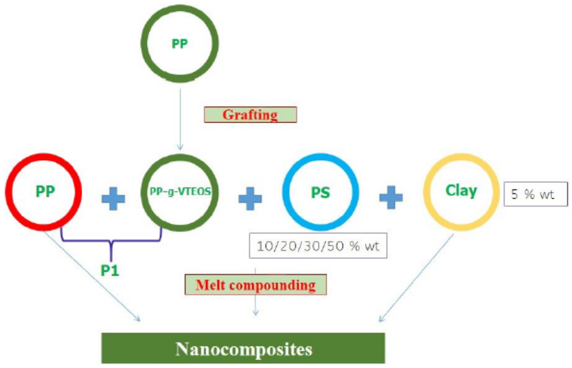


Figure 1. *Strategy for preparing bio(nano)composite films.*

2. Materials and Methods

Organoclay, Cloiste 20 A, was obtained from Sothern Clay Products. The PP used throughout this work was a homopolymer, PP 5032E1, commercialized by Exxon Mobil chemical. Dicumyl peroxide (DCP, 98%), vinyl triethoxysilane (VTEOS, 97%) were from Sigma Aldrich. The dibutyltin dilaurate (DBTDL, 98%) was from Alfa Aesar. And the polystyrene (PS) used in this study was polystyrol 143 E purchased from D-BASF the chemical.

2.1. Grafting Procedure and Nanocomposites Preparation

PP powder (50g) was tumble mixed with a solution of DCP (0,2 wt%) in C (5 wt%) for 20 min. The grafting reaction was carried out in Haake Polylab rheometer equipped with Rheomix mixing chamber and roller rotor. The mixture was reacted for 5min at 180 °Cat a rotation speed of 60 rpm, giving PP-g-VTEOS. The characterization of functionalized PP is detailled in Risite et al.[13]. Melt mixing of PP/PP-g-VTEOS/C20A and PP/PP-g-VTEOS/PS/C20A composites were carried out using contra-rotating twin screw micro extruder Minilab II. PP, PP-g-VTEOS, PS and nanoclay were tumble mixed and introduced in the hopper. 500 ppm of irganox-1010, 1000 ppm of Irgafos-168 and 600 ppm of calcium stearate were added after 5 min of mixing time for 2 min to stabilize PP-g-VTEOS for nanocomposites samples. The temperature, screw speed and residence time were fixed at 180 °C, 60 rpm and 5 min respectively. The composition of PP/PP-g-VTEOS is kept at 90 and 10 wt % for all blends and the clay concentrations was fixed at 5 wt %. The notation used the following for defining the blend composition is P1 for PP/PP-g-VTEOS, PS and C20A, expressed in weight fraction. The various compounds with their respective proportion are reported in Table 1.

Table 1. *Material designation and composition.*

Sample	Composition wt%
PP	100
PS	100
P1PS10	(90/10)/10
P1PS20	(90/10)/20
P1PS30	(90/10)/30
P1PS50	(90/10)/50
PP-PS10CA5	90/10/5
PP-PS20CA5	80/20/5
PP-PS30CA5	70/30/5
PP-PS50CA5	50/50/5
P1PS10CA5	[(90/10)/10]/5
P1PS20CA5	[(90/10)/20]/5
P1PS30CA5	[(90/10)/30]/5
P1PS50CA5	[(90/10)/50]/5

2.2. Structure and Morphological Characterization

The template is used to format your paper and style the text. All margins, column The XRD experiments were performed using X-ray scattering with a Bruker D8 AXS X-ray diffractometer. The angular range (2θ) was scanned from 1 to 10° at a step size of 0.05°, and the interlayer spacing (d_{200}) is calculated using the Bragg equation: $\lambda = 2d\sin\theta$, where λ is the wavelength of radiation (Cu-Kα radiation of 1.542 Å). SEM observations were made on ultrathin sections of nanocomposites prepared with a Leica EM UC7 ultramicrotome at room temperature, and then analyzed with a Philips CM200 TEM microscope operating at 200 kV, coupled with an EDS microprobe analyzer EDAX DX-4.

2.3. Thermal Characterization

Thermogravimetric analysis, TGA, was performed on a Q500 TA instrument thermoanalyzer. The experiments were conducted at a temperature ramping of 10 °C/min from room temperature to 1000 °C, under air flow.

3. Results and Discussions

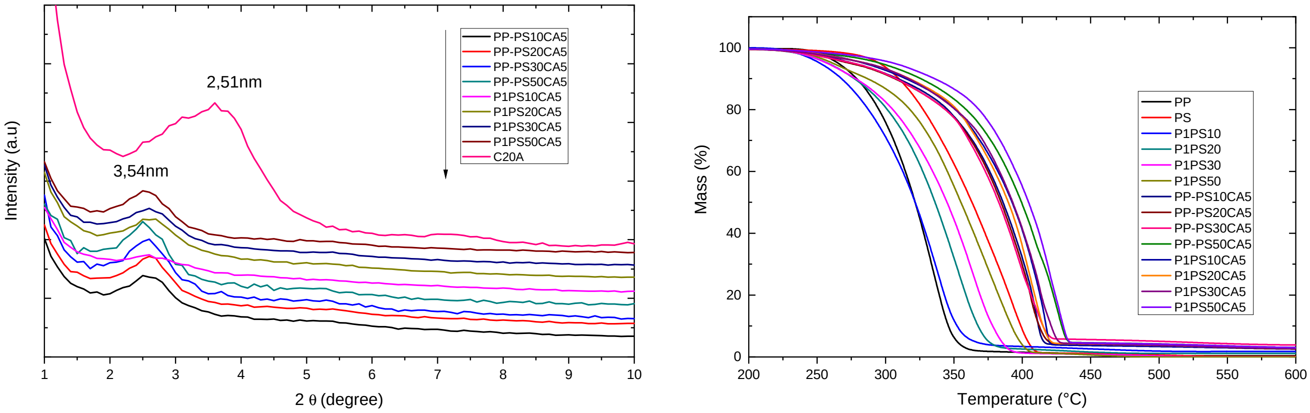
The nanocomposites were elaborated by melt compounding method thanks to that method is closed to industrial production. The obtainend nanocomposites were subjected to morphological studies by X-Ray diffraction, scanning Electronic Microscopy and thermal stability by thermogravimetric analysis. The figure 2a presents the X-Ray diffraction patterns of the nanocomposites at different compositions of PS and compatibilizer. The virgin clay pattern showed a curve with a peak at 2θ of 3,6 equivalent to interplanar distance of 2,51 nm. However, all nanocomposites without and with compatibilizer at different rate of PS showed curves with peaks around 2θ of 2,4 equivalent to 3,54 nm. This shift is attributed to the intercalation of the polymeric chains into the clay cavity leading to the augmentation of the distance interfoliar. The results corresponds to the literature [12, 14]. Remarkably, for nanocomposite P1PS10CA5, XRD spectra revealed a decrease in peak intensity which informed about a reduction of stacks intercalated structure leading to a higher degree of delamination. It means that the best degree of delamination is obtained at the low percentage of PS.

The thermal stability of nanocomposites by TGA or thermogravimetric analysis showed in 2b. The figure presents the mass loss of every composite in fonction of temperature. The studies showed that the nanocomposites with silane have better maximal decomposition temperature compared to those of without silane and the maximum is at the 50 % in PS about 430°C, an augmentation of about 100°C. The detailed data corresponding to the decomposition temperatures at 5% (T5%) and Tmax are presented in Table 2. The better dispersion and distribution of clay in the nanocomposites as told in morphological studies is responsible of these augmentation in terms of temperature. The clays acte like flame retardant which lead the decomposition temperature shifting to the higher [15, 16].

The last characterization studied in this work is a morphological properties with SEM technics and presented in the figure 3 with selected samples. The SEM images of the lower content of PS without and with compatibilizer are compared at different magnifications. The images showed 3 significant points. First, it can be seen that PP and PS are immiscible. Secondly, PS droplet well dispersed and distributed with reduced size were obtained with the addition of compatibilizer showing that its use affected the morphology of nanocomposites. Finally, the nanocomposites without compatibilizer showed disordered and random of PS phase and clay and it can be seen that the continuous phase doesn't contain clays. The presence of compatibilizer is important in morphological improvement properties.

Table 2. *TGA data of pure PP and PS, PP/PP-g-VTEOS/PS and all nanocomposites.*

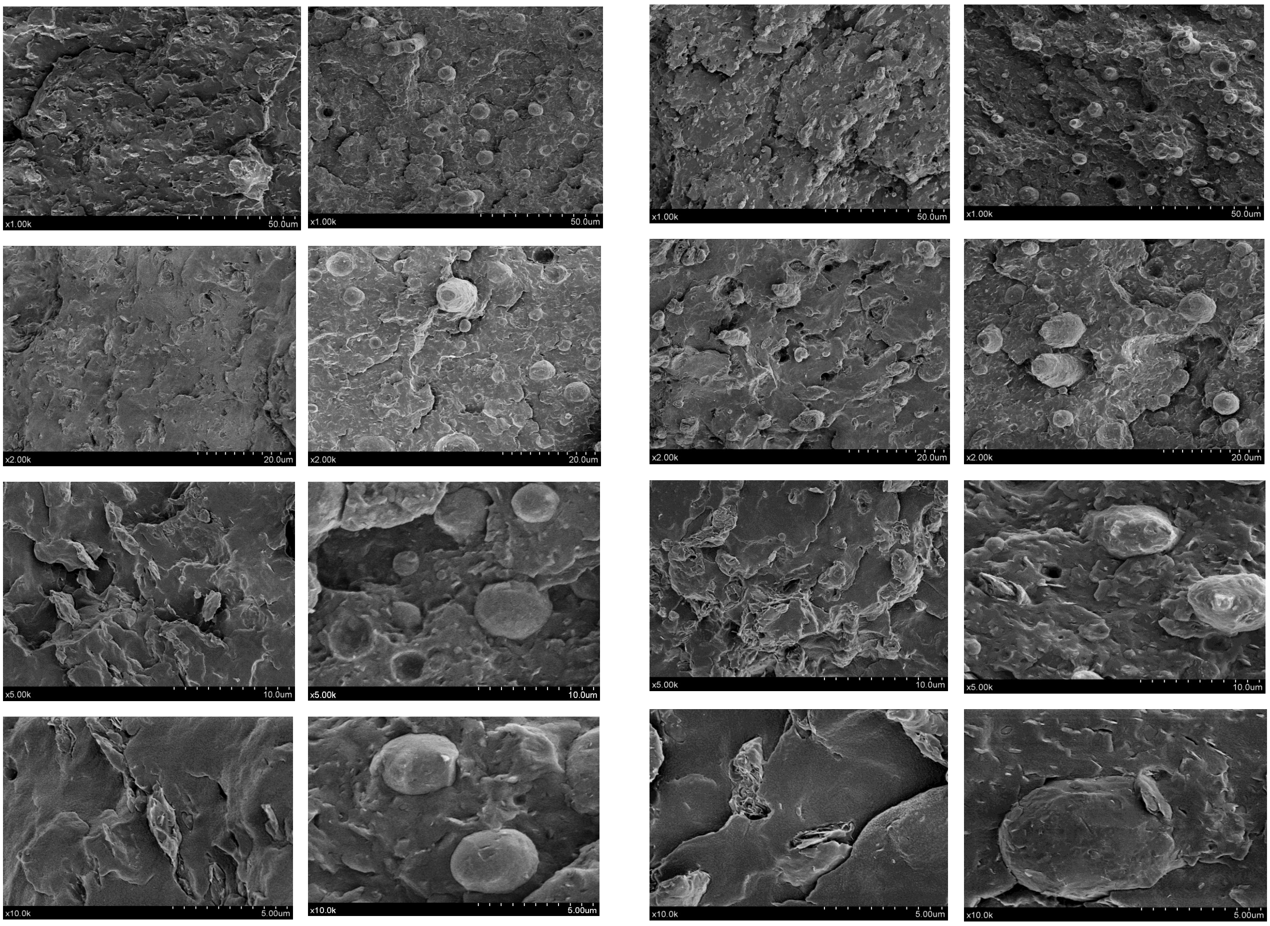
Sample	$\mathbf{T_{5\%}}$(°C)	T_{max} (°C)
PP	265	334
PS	295	393
P1PS10	252	337
P1PS20	257	353
P1PS30	257	362
P1PS50	261	375
PP-PS10CA5	276	407
PP-PS20CA5	274	406
PP-PS30CA5	276	409
PP-PS50CA5	290	425
P1PS10CA5	284	414
P1PS20CA5	285	422
P1PS30CA5	290	425
P1PS50CA5	306	430



(a) *XRD patterns for the clays and PP/PS nanocomposites at different contents of PP/PS.*

(b) *TGA curves of pure PP, PS, and all nanocomposites at different contents of PP/PS.*

Figure 2. *Comparative XRD and TGA images of nanocomposites with different PP/PS content, at various magnifications(PPPS10CA5 vs P1PS10CA5) and (PPPS50CA5 vs P1PS50CA5).*



(a) PPPS10CA5 vs P1PS10CA5. *(b) PPPS50CA5 vs P1PS50CA5.*

Figure 3. *Comparison SEM images of nanocomposites at different contents of PP/PS at different magnifications. (PPPS10CA5 vs P1PS10CA5) and (PPPS50CA5 vs P1PS50CA5).*

4. Conclusion

PP/PS clay based nanocomposites using PP-g-VTEOS were prepared by melt compounding. The use of PP-g-VTEOS promoted the improvement of propeties of nanocomposites by the change in morphological structures. Despite the state of intercalation of clay nanocomposites, a noticeable increase was seen in the thermal properties, due to the better dispersion and distribution of clay and the creation of PS droplet with the reduced size. It can thus be expected that nanocomposites would have better properties only if the clay platelets were in the state of exfoliation.

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Abbreviations

TGA	Thermogravimetric Analysis
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
PP-g-VTEOS	Polypropylene grafted silane
CA	Montmorillonite
PP	Polypropylene
PS	Polystyrene

Author Contributions

Rabearison Mizea Miller : Writing - original draft, Writing - review & editing

Oudinot Bricharles Tsarambita: Software, Visualization

Heriarivelo Risite: Project administration, Funding acquisition, Supervision

Joyeux Heriniaina Theophyle: Conceptualization, Investigation, Formal Analysis, Methodology

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Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper. No financial or personal relationships with other people or organizations have inappropriately influenced this work.

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