

Mechanical and rheological properties of polypropylene/bentonite composites with stearic acid as an interface modifier

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ABSTRACT: In this study, we present a study of polypropylene/bentonite composites where stearic acid was used as both a surface and interface modifier during the compounding of composites. The concentration of bentonite was 1.5, 2.5, 5.0, and 10 parts per hundred. The composites were characterized by impact resistance and tensile tests, rheological analysis, the dispersion state of the filler observed by optical microscopy, and interaction between bentonite and stearic acid, as analyzed by Fourier transform infrared spectroscopy. No chemical interaction was found between bentonite and stearic acid. Composites with modified bentonite and stearic acid used as interface modifiers increased the elongation at break; these samples also showed better dispersion of the filler in comparison with the other compounds. In addition, stearic acid acted as a lubricant, favoring the interaction of the polymer with the filler and decreasing the viscosity of the compounds. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 2015, 132, 42264.

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INTRODUCTION

Polypropylene (PP) is used in a wide array of disposable and durable products, such as food containers, disposable diapers, children toys, and automotive parts. Its wide use in a variety of applications is due to the characteristic strength of the resin, the high melting temperature compared to other polymers, and the ease with which it can be processed.1 However, on an industrial scale, the use of micrometer-scale fillers is a common practice for reducing costs or improving other factors, such as processing conditions, density, and flame-retardant properties and many times for enhancing the mechanical properties.^{2–7} Several investigations have shown that considerable improvements in the mechanical properties of PP have been achieved through the use of clay minerals as fillers.³⁻⁵ Among these fillers is bentonite, a layered aluminum silicate with exchangeable cations and reactive hydroxyl groups on the surface, primarily constituted from montmorillonite.^{5,8} This clay has been widely studied and used

in diverse applications because it exhibits a variety of attractive properties, such as a high specific surface area, high porosity, and surface activity.8 Furthermore, bentonite is an interesting filler because of its large worldwide availability (including Mexico) and ability to form layered microcomposites. Nevertheless, the hydrophilic nature of this clay makes modification with organic surfactants a required treatment. Fatty acids, such as oleic acid, palm oil, and stearic acid, are frequently used to provide a hydrophobic surface on clay to increase the compatibility with certain polymer molecules. 4,5,9 Stearic acid has been used as a modifier in other particulate systems because of advantages, such as its low cost and ease of processing; additionally, modification with this fatty acid reduces water absorption, prevents interaction between filler particles, and lowers the surface energy to make them more compatible with PP and easily dispersible in this matrix. 10 Several studies have shown that fatty acids have provided a variety of benefits to the mechanical properties, mainly the resulting strength and stiffness of the composites. 4,5,11 Kamal

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et al.¹² conducted a study of the effects of surface treatment with oleic acid and stearic acid for the PP/CaCO₃ system; the results indicate the superior properties of the composites obtained from the filler modified with stearic acid.

Normally, the modifier of the surface is added in a previous mixing process to the compounding step of the PP composites. In this investigation, stearic acid was used to modify the bentonite surface; it was also added in small amounts to the hopper during the injection process as an interface modifier. The surface treatment of bentonite with stearic acid was based on that reported by Li *et al.*⁶ and Tabtiang and Venables.⁷ This fatty acid has weak interactions with clays, and its hydrocarbon tail is compatible with PP.¹³

Rheological properties play an important role in the processing conditions, so a good interaction between the polymer and filler results in better rheological behavior. 14 As reported, the use of clay-modified fillers increased the mechanical properties of the plastic and influenced its rheological properties. 15 Fillers, including calcium carbonate and talc, have been used to investigate the rheological effects on PP. 16,17 It has been found that composites with untreated fillers have an increased viscosity; therefore, an interface agent is necessary to reduce the effect of the viscosity augmentation. 16-18 In this regard, fatty acids can also be used to serve as lubricants, facilitating the processing of composites by improving the flow properties and, hence, the production rate, energy consumption, and machinery wear.^{4,5} The viscosity of polymers at an industrial level is an important factor to consider, whereas a high viscosity is related to high machinery process costs and long processing cycles. Therefore, it is relevant for researchers to study the behavior of compounds with lower viscosities without reducing the mechanical performance.

To our knowledge, the use of stearic acid as an interface modifier for a PP/bentonite system has not been reported yet. In this article, we highlight the importance of this approach for favoring the mechanical properties of the composites, and this approach also contributes to a decrease in the processing costs because of a reduction in the viscosity of the compounds. Thus, the influence of a stearic acid treatment in the PP/bentonite system on the mechanical properties, dispersion, and rheological properties is addressed in this research. In addition, we found evidence of how the use of stearic acid improved the elongation at break for composites containing micrometer-scale bentonite particles. We finally broke the effect of the stearic acid when we used it as a lubricant; this decreased the viscosity of the compounds and allowed a better melt flow, which is an important characteristic when processing conditions must be optimized.

EXPERIMENTAL

Materials and Sample Preparations

The polymer matrix used was isotactic PP (Valtec, HS005M) supplied by Indelpro (Mexico). Its melt flow index was 2.2 dg/min, as calculated according to the ASTM D 1238. The filler was a bentonite mineral provided by BARMEX Mexico. The mean diameter of the filler was 5 μ m, and the specific surface

Table I. Nomenclature and Composition of the Prepared Specimens

		Material			
Number of blocks	ID	Unmodified bentonite (pph)	Modified bentonite (pph)	Stearic acid (in hopper of the extruder; pph)	
0	PP	_	_	_	
1	PP_B_1.5	1.5	_	_	
	PP_B_2.5	2.5	_	_	
	PP_B_5.0	5.0	_	_	
	PP_B_10	10.0	_	_	
2	PP_BM_1.5	_	1.5	_	
	PP_BM_2.5	_	2.5	_	
	PP_BM_5.0	_	5.0	_	
	PP_BM_10	_	10.0	_	
3	PP_BMT_1.5	_	1.5	2.0	
	PP_BMT_2.5	_	2.5	2.0	
	PP_BMT_5.0	_	5.0	2.0	
	PP_BMT_10	_	10.0	2.0	
4	PP_SA_2			2.0	
	PP_SA_4			4.0	

area obtained from the Brunauer–Emmett–Teller analysis was $12 \text{ m}^2/\text{g}$.

The modified filler was prepared with a procedure proposed by Li *et al.*⁶ and Tabtiang and Venables.⁷ The dried bentonite was loaded into the chamber of a Henschel blender with the stearic acid at 1.5 parts per hundred (pph) bentonite. The mixing was initiated at low speed (1400 rpm) for 5 min and then increased up to 2800 rpm for 15 min; at this speed, the temperature of the blend reached 45°C. Then, the chamber was cooled down to 40°C. Finally, the coated filler was stored at room temperature in a desiccator.

A Beutelspacher single-screw extruder was used to compound the samples. The screw speed was 50 rpm, and the temperature profile was from 190°C in the feed section to 210°C at the die. Each sample was extruded twice to ensure proper mixing of the components. Three types of PP/bentonite compounds were prepared with 1.5, 2.5, 5.0, and 10.0 pph PP as follows:

- 1. Unmodified bentonite and PP, roughly mixed in a plastic bag and introduced into the hopper of the extruder (block 1 of the compounds).
- 2. Modified bentonite with stearic acid and PP, roughly mixed in a plastic bag and introduced into the hopper of the extruder (block 2 of the compounds).
- 3. Modified bentonite with stearic acid, PP, and stearic acid as an interface modifier, roughly mixed in a plastic bag and introduced into the hopper of the extruder (block 3 of the compounds).

Compounds with PP/stearic acid were also prepared for rheological tests with concentrations of stearic acid of 2 and 4 pph.



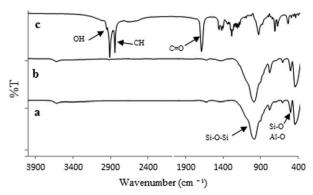


Figure 1. IR spectra of the (a) unmodified bentonite, (b) modified bentonite, and (c) stearic acid.

Table I shows the nomenclature and composition of the compounds.

Characterization of the Samples

A PerkinElmer IR spectrometer coupled with a diamond attenuated total reflectance cell (Fourier transform infrared; Spectrum 100 model) was used to observe the chemical changes in the stearic acid modified bentonite. A wave-number range from 4000 to 400 cm⁻¹ was used. We obtained the Young's modulus, tensile strength, and elongation at break according to the ASTM D 638 method by testing samples in a Shimadzu AG-20kN universal machine. Izod impact tests were evaluated according the ASTM D 256 method. The dispersion of the filler was observed with a JENCO optical microscope by means of thin films approximately 50 µm thick with compression molding of the PP/bentonite compounds at 200°C. Ten fields of view at low magnifications were randomly taken for each sample. An estimated number of 1000 particles were detected in the 10 fields of view at low magnification. The volume mean diameter (D_{ν}) was used to evaluate the filler dispersion in this study. 19 The rheological properties were studied with a PaarPhysica MCR300 rheometer, and the experiments were carried out at 200°C. The rheometer was equipped with a concentric disk geometry, and the strain was 0.5%. The diameter of the plates was 25 mm, and the gap about 1.2 mm. The measurements were carried out under a dry nitrogen atmosphere.

RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopy

Figure 1 shows the spectra of stearic acid and unmodified and modified bentonite. In the spectrum of stearic acid [Figure 1(c)], the strong peak located at 1700 cm⁻¹ was due to the stretching of C=O, and the peak at 1300 cm⁻¹ was due stretching of C-O; both characteristic peaks account for the existence of a -COOH functional group. The peak at 1470 cm⁻¹ is a feature of the scissoring group of -CH₂. The unmodified and modified bentonite almost had the same spectra; this indicates that there was no chemical reaction between stearic acid and bentonite. The strong signal at 1000 cm⁻¹ belonged to the stretching vibrations of the Si-O-Si bonds, and signals around 600 cm⁻¹ were attributed to Si-O vibrations and Al-O stretching. Mihajlović *et al.*⁹ explained that monocarboxylic acids with an aliphatic hydrocarbon chain (also known as fatty

acids and their salts) are most frequently used as surfactants; these fatty acids were used as surface modifiers and applied by dry coating and from an acid solution. According to their research, the adsorption of stearic acid onto calcite was of mixed nature, depending on the modification method used; this resulted in chemical and physical adsorption.

On the other hand, Li *et al.*⁶ and Meng and Duo² reported a chemical reaction between some clays (wollastonite) and fatty acids. However, in this investigation, a chemical reaction was not detected between the bentonite and the stearic acid. In our results, the interactions of the components during mixing were mainly physical. The very weak peaks located at about 2800 and 2900 cm⁻¹ observed after bentonite modification [Figure 1(b)] were due to the stretching vibrations of the C—H bond of stearic acid, as shown in Figure 1(a). According to Demirbas *et al.*,⁸ this was evidence of the adsorption of stearic acid onto bentonite.

Tensile Tests

Table II shows the results of mechanical properties of the composites. The Young's modulus increased slightly for the compounds of block 1 when a filler concentration above 5 pph was used, reaching an enhancement of up to 10% for the PP_BM_10 composite. This behavior was expected because the addition of the filler conferred rigidity to the matrix. Furthermore, the modification of bentonite provided better dispersion and interfacial interaction between the filler and matrix; therefore, this resulted in a superior load transfer when the Young's modulus was tested. This was clearly seen in the improved stiffness response of block 2; in this set of compounds, the elastic modulus was 16% higher than the pure polymer when the highest modified filler concentration was used. On the other hand, the results obtained in the tensile modulus of the composites of block 3 were slightly lower or very similar to that of the pure polymer. In these composites, an extra amount of stearic acid was added to the PP/modified bentonite system. This approach was done with the purpose of using the stearic acid as an interface modifier; however, according to these results, it can also be said that it acted as a lubricant, as explained in detail in the rheology section. In this regard, this lubricity effect reduced the intermolecular forces in the polymer and allowed a better flow of the polymer chains; this arose from the decreased viscosity and, therefore, eased the processability, but this also reduced viscosity was reflected in the higher values of elasticity of this set of compounds.

The elongation at break was clearly improved as a result of the approach used in block 3, where this property was increased by 29, 22, 50, and 12% for each of the compounds of this set, and the sample PP_BMT_5.0 was the one with the best elongation at break (1140%). These results are very important because in most of the composites filled with clays, the polymer elongation at break was dramatically reduced even at low concentrations of filler.^{20–22} It is important to notice that the rise in elongation was not detrimental for the Young's modulus response. Blocks 1 and 2 of compounds did not show significant changes in the elongation at break.

The tensile strength results did not show considerable changes in most of the prepared samples. This property is also sensitive to the degree of dispersion and coating of clays. 4,6,20



Table II. Results of the Mechanical Behavior

Number of blocks	ID	Young's modulus (MPa)	Tensile strength (MPa)	Elongation at break (%)	Impact strength (kJ/m²)
0	PP	1162.10 ± 27	43 ± 1.18	758 ± 24.67	1.22 ± 0.09
1	PP_B_1.5	1140.04 ± 41	43 ± 2.89	709 ± 15.44	1.12 ± 0.045
	PP_B_2.5	1117.46 ± 35	43 ± 2.50	728 ± 11.87	1.28 ± 0.057
	PP_B_5.0	1250.70 ± 46	43 ± 2.89	814 ± 9.13	0.94 ± 0.07
	PP_B_10	1287.42 ± 43	45 ± 1.92	802 ± 32.84	0.99 ± 0.054
2	PP_BM_1.5	1256.91 ± 48	44 ± 2.10	750 ± 77.17	1.37 ± 0.064
	PP_BM_2.5	1245.19 ± 51	42 ± 1.27	726 ± 30.16	1.08 ± 0.053
	PP_BM_5.0	1254.84 ± 50	43 ± 0.48	727 ± 16.69	1.59 ± 0.048
	PP_BM_10	1356.19 ± 40	41 ± 0.83	801 ± 25.55	1.20 ± 0.031
3	PP_BMT_1.5	1014.21 ± 22	48 ± 0.83	982 ± 15.63	1.28 ± 0.07
	PP_BMT_2.5	1022.49 ± 20	47 ± 0.96	930 ± 10.00	1.26 ± 0.053
	PP_BMT_5.0	887.87 ± 31	50 ± 0.48	1140 ± 30.89	1.20 ± 0.048
	PP_BMT_10	1198.99 ± 46	41 ± 1.18	853 ± 47.95	1.23 ± 0.078

The second value indicates the standard deviation.

Impact Tests

The impact strength results are shown in Table II. According to these results, there was no significant change in this property in most of the samples. As observed in block 1, despite the fact that untreated bentonite has no good interaction with the polymer matrix, and bad dispersion was found. The impact resistance values remained close to those found in the pure polymer when filler contents up to 5 wt % were used. However, at the highest loading used in this study (10 wt %), this property was diminished by 23% as compared to the sample containing only PP. This suggest that the agglomerates found in this sample favored the formation of microvoids because of the bad interaction of the untreated bentonite and the polymer, which resulted in a more significant decrease in this property. This finding has also been found in other systems of clay composites, as mentioned in the review by Pavlidou and Papaspyrides.²⁰ On the other hand, the use of stearic acid as a bentonite modifier (block 2) and stearic acid as an interface modifier (block 3) kept the impact strength similar to that found in the samples containing only PP even at a high content of the filler. These results are due to the good surface interaction of the mineral filler when it was mixed with the polymer; this resulted from the modification with stearic acid. Furthermore, the stearic acid added to the hopper provided a favoring effect on the interactions between the filler and the PP. No further improvements on the impact strength of block 3 were related to the lubricity behavior imparted by the extra addition of stearic acid to this set of samples, where a decrease in the chain entanglement promoted easier chain slippage during the impact tests.

Dispersion State

In Table III, the D_{ν} values of the agglomerates in the compounds are reported. Figures 2-4 show optical micrographs of the PP/bentonite composites at concentrations of 1.5, 2.5, 5.0, and 10 pph with different types of compounding. As shown in Figure 2, the micrographs show that as the concentration increased, the filler tended to agglomerate (black points in the

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micrographs) because of the difficulties for dispersing the filler in the polymer matrix; hence, a treatment of the filler was required. The D_vs were the largest for block 1 of the compounds (see Table III). Figure 3 shows the dispersing effect of the stearic acid; the observed size (D_{ν}) of the agglomerates and their amounts were lower in the composites of this block when compared to the previous set of samples where no modification was conducted. However, the compounds with modified bentonite and stearic acid in the hopper of the extruder showed the best dispersion state (Figure 4). This block of compounds (block 3) gave the lowest values of D_{ν} of the agglomerates. This means that the surface treatment with stearic acid significantly reduced the filler surface free energy and particle-particle interaction; this led to better dispersion of the particles in the polymer matrix and reduced the filler agglomerates.²³ The extra addition of this fatty acid as an additive to the formulation improved the interactions between the polymer and the filler

Table III. D_v Values of the Agglomerates in the Compounds

Number of blocks	ID	$D_{\rm v}$ (μ m)
0	PP	
1	PP_B_1.5	30.12
	PP_B_2.5	33.37
	PP_B_5.0	36.72
	PP_B_10	40.12
2	PP_BM_1.5	19.58
	PP_BM_2.5	20.14
	PP_BM_5.0	22.36
	PP_BM_10	28.12
3	PP_BMT_1.5	17.39
	PP_BMT_2.5	17.74
	PP_BMT_5.0	18.86
	PP_BMT_10	24.75

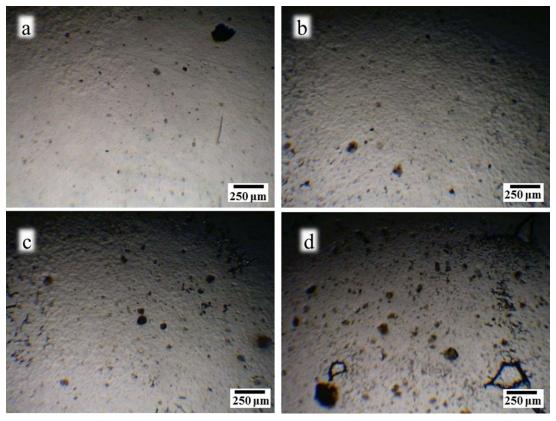


Figure 2. Micrographs of compounds of block 1: (a) PP_B_1.5 pph, (b) PP_B_2.5 pph, (c) PP_B_5.0 pph, and (d) PP_B_10 pph. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

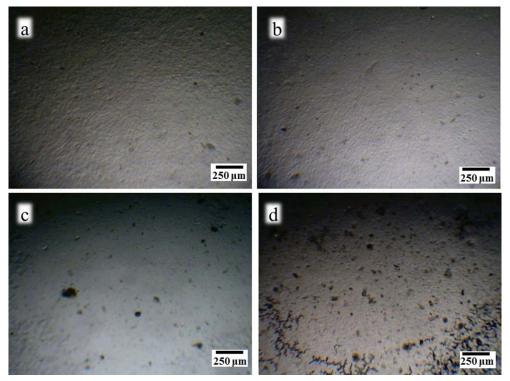


Figure 3. Micrographs of compounds of block 2: (a) PP_BM_1.5 pph, (b) PP_BM_2.5 pph, (c) PP_BM_5.0 pph, and (d) PP_BM_10 pph. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

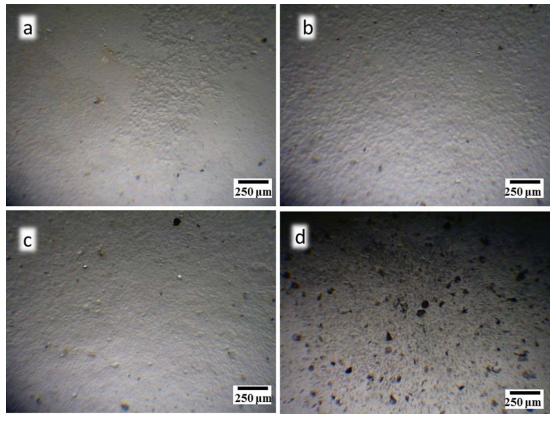


Figure 4. Micrographs of compounds of block 3: (a) PP_BMT_1.5 pph, (b) PP_BMT_2.5 pph, (c) PP_BMT_5.0 pph, and (d) PP_BMT_10 pph. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

even more because of interfacial modifications. As a result, a better dispersion state promoted better mechanical behavior, as was previously demonstrated in this study. Large agglomerates, on the other hand, were responsible for failures in the plastic materials (especially at the interfaces). Although it is a common practice to add stearic acid to the filler in a step previous to the extrusion process, our results show that stearic acid used as an interface modifier added to the hopper of the extruder led to a better wetting of the mineral particles by the polymer because of interactions of stearic acid with bentonite and the compatibility of this acid with PP. Generally speaking, this better wetting produced a better dispersion state, which is evidenced in the micrographs of Figure 4.

Rheological Properties

Stearic acid also influences the rheological behavior of the composites. Figures 5 and 6 show the behavior of the complex viscosity for compounds with filler concentrations of 2.5 and 5 pph and diverse treatments, respectively. Both figures show that the viscosity for the samples containing the untreated filler was higher than that of PP (HS005M); as shown in the micrographs, the hydrophilic nature of the untreated filler and the hydrophobic behavior of PP made them incompatible. Additionally to these features, the high viscosity caused the bentonite particles to agglomerate. On the other hand, as explained earlier, stearic acid acted as a lubricant; this behavior was also found with the addition of the surface-modified bentonite into

PP; thus, this reduced the viscosity. Kamal *et al.*¹² showed that fatty acids promoted flow. The improvement of lubricity during the processing provided good dispersion of the modified filler, and it was also in agreement with the decrease in size and amount of agglomerates showed by the optical microscope where few agglomerations were observed.

The treatment of the PP/modified bentonite composite with extra stearic acid added into the hopper of the extruder showed the highest reduction of the complex melt viscosity even when compared to that of pure PP. This was in agreement with

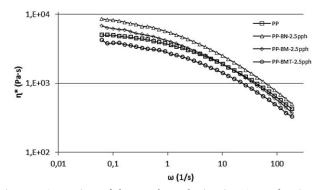


Figure 5. Comparison of the complex melt viscosity η^* as a function of the frequency ω for compounds of PP/bentonite at 2.5 pph with different treatments.



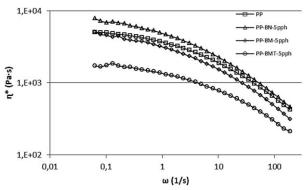


Figure 6. Comparison of the complex melt viscosity as a function of the frequency for the PP/bentonite compounds with concentration of 5 pph with different treatments.

Banerjee *et al.*,¹⁴ who recently studied the dispersion of clays in PP and compatibilized PP systems. They found that the lower melt viscosity due to the compatibilizer used promoted good dispersion and intercalation of polymer chains into the clay galleries. In this study, for the composites with a concentration of 2.5 pph of the modified bentonite (see Figure 5), the viscosity decreased slightly in comparison to that of pure PP. For the composites containing 5 pph filler, the complex melt viscosity diminished to a higher degree, as shown in Figure 6. This behavior can be explained in terms of the lubricant effect on these samples, where the extra amount of stearic acid reduced the intermolecular forces in the polymer chains; this, in turn, diminished the resistance to their flow, and this was reflected in the reduction of the viscosity of these compounds.

The dispersion was also affected by the concentration of filler because the increasing concentration led to a higher number of agglomerates. For the composite with 5 pph filler, the attained elongation at break was the highest. This may have been due to the good dispersion of the filler and the interactions between the polymer matrix and filler surface because of its modification. In addition, this could have been related to the interface modifier effect but also the lubricity obtained from this processing approach, which decreased the viscosity and had a plastificant behavior. Other reports^{16–18} have also shown similar results, where the stearic acid induced plasticizing action

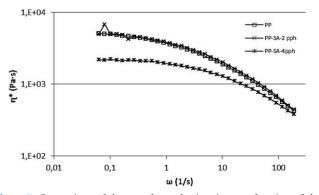


Figure 7. Comparison of the complex melt viscosity as a function of the frequency for the PP/stearic acid compounds at different concentrations.

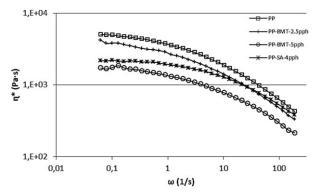


Figure 8. Comparison of the complex melt viscosity as a function of the frequency for the PP/bentonite and PP/stearic acid compounds.

decreased the viscosity values because of a better molecular motion between the polymer and filler. ¹⁶

Figure 7 confirms the behavior of the stearic acid as a lubricant when mixed with PP. The comparison of the complex viscosity of the compounds of PP with stearic acid at 2 and 4 pph with that of the pure PP showed that the viscosity did not change in any range of frequency at the low concentration, but for that of 4 pph, the viscosity was clearly reduced at low frequencies and tended to reduce the viscosity difference as the frequency increased. This behavior showed that stearic acid not only enhanced the chain movement of the polymer matrix when it acted as an interface modifier of a filler, but it also acted as a lubricant when used with the pure PP; this allowed the melt resin to flow faster because the polymeric chains could slide each other more loosely.

A comparison between the best results in decreasing the viscosity obtained for the PP/bentonite compounds and the PP/stearic acid compounds is depicted in Figure 8. We noticed that stearic acid decreased the viscosity of the PP/bentonite composites in the whole range of frequency, in contrast with the PP_SA_4 pph compound, where the complex melt viscosity increased after a certain frequency. We also observed that the best results were observed for the PP_BMT_5 pph compound; the decrease in the viscosity was not only greater but also uniform in the whole range of frequency. These results give feasible evidence of the stearic acid acting as an interface modifier and lubricant. These effects produced good mechanical behavior because of the appropriate dispersion of the filler into the polymer matrix, which reduced the agglomerates and improved the elongation at break of the composite. During processing of the composites, the stearic acid also reduced the viscosity of the melt compound because of the free movement of the polymer chains and the good interfacial interaction between the polymer and the filler.

CONCLUSIONS

The effects of stearic acid on the mechanical properties, filler dispersion, and rheological behavior of PP/bentonite composites was investigated. The modification of the filler with stearic acid did not show any chemical changes on its structure; consequently, the interactions between the filler and the stearic acid were exclusively physical. The composites based on the modified



bentonite showed better properties than those found in pure PP; this was in contrast to the results obtained from the addition of untreated bentonite to PP. In this set of composites, the mechanical properties and filler dispersion were poor, and this caused agglomerates in the polymer matrix. Compounds prepared with modified bentonite and an extra steric acid amount used as an interface modifier showed the best results in the elongation at break, a better dispersion, and the lowest complex viscosity values. Thus, this research demonstrated that stearic acid can be used in the PP/bentonite system and highlights its function as both an interface modifier and lubricant to increase the mechanical properties without compromising the flowing properties.

ACKNOWLEDGMENTS

The authors contributions are described as follows. Cesar Del Angel performed the experiments regarding the compounding of the composites and interpreted the results. Saul Sanchez-Valdes and Guillermo Martinez-Colunga conducted some of the mechanical tests (tensile and impact properties). Sergio Alonso and Roberto Zitzumbo executed the rheological tests. Eduardo Ramirez-Vargas obtained the optical micrographs. Ana B. Morales, Fabiola Navarro-Pardo, Tomas Lozano, and Pierre G Lafleur made the discussion of the results and wrote the manuscript.

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