CHARACTERIZATION OF COMPOST POLYPROPYLENE WITH BEE WAX.

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To evaluate the influence of the bee wax in the mechanical, rheological and processing properties in the propylene-ethylene copolymer, three formulations had been prepared contend different amounts of bee wax. The mixture was made in a twin screw extruder. Sample contends 1% w/w of bee wax was the one that presented the best values for the mechanical properties, fact evidenced for the tensile test. The samples contend 3% and 6% w/w of wax doesn't presented significant changes in the mechanical properties, due the exudation wax excess, fact corroborate by scanning electron microscopy (SEM). The plasticizer effect of the bee wax was observed in dynamic mechanical analyze (DMA), that it evidenced a decrease in the glass transition temperature for all samples.

Introduction

The search for new materials with the aim of minimize the environmental impact produced by the increasing consumption of raw material didn't renew. Problem so argued in the last times, what it has been conducting the scientists to develop new products that use the maximum of biodegradable resources in its composition. Diverse types of polymeric composite using natural products, like coconut fiber, banana fiber, wood dust and carnauba wax, already are used currently in the industry of the plastic as lubricant, plasticizer and reinforcements agents[1-3]. These products, beyond presenting an excellent balance between final properties and costing, with the advantage to possess less amounts of plastic material, when compared with the conventional products.
It has much time that bee wax lubricant properties are known by the man and explored by the industry. Its applications go since cosmetics until the preparation of industrial lubricant agent [4]. However, studies to evaluate the influence of this product in thermoplastic resin, evidencing the possible modifications in the mechanical, thermal properties and of processing don't exist.
The random copolymer of propylene and ethylene (PP random) have excellent characteristics of processability and mechanical resistance at room temperature, allied to a relatively low price when compared with the other copolymers that could be used in the same applications[4]. However, for applications where the mechanical resistance at low temperatures is demanded, the material cannot be used, therefore its glass transition temperature is not so low to allow the use in these applications.
If a plasticizer agent is going added to copolymer PP random, a sensible reduction in Tg can be obtained[2-4], becoming the adequate material for applications where low temperature is one of the limiting factors.
The aim of this work was to evaluate the influence of different amounts of bee wax in the PP random to study its possible use as lubricant or plasticizer agent. Replacing the plasticizers more used that in its majority are derivate from inorganics products or the oil.

Experimental

Material

The copolymer of propylene-ethylene with melt index of 1.50g/10min was used to evaluate the influence of different bee wax concentration. This material was supplied by Braskem. The wax was supplied in an only solid block, which was minced to be incorporate in PP random.

Sample Preparation

The samples were processed in the chamber of twin screw rheometer from Haake, with temperatures zone off 175, 180, 185 and 190°C, and the screw speed maintained constant in 40 RPM for all samples. These conditions were chosen by providing better homogenization of the mixtures without the risk of material degradation due the retention time in the extruder [2]. Three samples were prepared, contend 1%, 3% and 6% w/w of bee wax, these samples was called in agreement show table 1.

Table 1 – Mixture of wax concentration.

Sample A	Pure copolymer
Sample B	Copolymer with 1% of wax
Sample C	Copolymer with 3% of wax
Sample D	Copolymer with 6% of wax

Tensile Test

The tensile test samples were of type IV complying ASTM D 638-03[6] standard obtained by thermopressing. The samples were obtained by thermopressing at 190°C in a Hidralmac thermopress during 15 min, being 10 min for melting and 5 min with pressure of 80 Bar.

An EMIC DL 300 universal tensile testing machine equipped with a data acquisition system was utilized to carry out the tensile tests. The speed velocity of testing was 50 mm/min and the tests carried out at a temperature of 23°C. At least five specimens were tested from each condition.

Melt flow index

The melt flow index of the samples were measured in a Melt flow equipment of CEAST in which the samples were flowed through an orifice of 2.00 mm diameter during 1.0 min under loading of 2.16 Kg at 230°C (ASTM D 1238-04c).

Scanning electron microscopy

The Scanning electron microscopy (SEM) was made using a microscope EDAX PHILIPS XL 30. The photos were obtained in the surface of the samples with previous gold covering.

Rheological analyses

The characterization in shear flow was performed at a temperature of 200°C using rotational Physica rheometer (MCR 300) with parallel-plate geometry and diameter of 25mm. The amplitude oscillatory was performed in the frequency range 0.01 – 100 1/s with a strain of 5% and a gap of 1.0mm. Samples with 1.2 mm of thickness and 25 mm of diameter were prepared by thermopressing with the same conditions used for the preparation of the samples for mechanical test.

Dynamic mechanical analyse

The glass transition temperature (Tg) was determined in an equipment DMA of the Netzsch using the three point bending mode. The range of temperature -40°C to 40°C, with heating rate of 2°C/min. The frequencies used were 1, 2 and 5Hz and the amplitude of measure of 60 µm. The samples dimensions were 50x8x2mm^3 and had been prepared by thermopressing under the same conditions used in the preparation of the samples to mechanical test.

Results and Discussion

The obtained results had evidenced the influence of the bee wax in the material characteristics. In the tensile test, the variation of the values of the mechanical properties with different amounts of wax can be observed. Sample B, with 1% of wax, was the one that presented the better values for all the properties, except in the elongation at the yield, see table 2. On the other hand, samples C and D were not so significant values of mechanical properties, for example the elastic modulus and strength at rupture, when compared the sample A. These effects must, probably, the exudation of the bee wax in the samples with superior amount of 1%. These results show's that 1% concentration, the wax acted as reinforcement, increasing the values of mechanical properties.

Table 2 – Results of mechanical properties at yield rupture

Sample	Yield strength (MPa)	Elongation at yield (%)	Strength at rupture (MPa)	Elongation at the rupture (%)	Elastic modulus (MPa)
A	21.78	8.02	15.92	392.86	845.48
B	25.10	9.39	16.94	408.08	906.84
C	21.75	9.02	9.85	399.00	711.14
D	20.31	9.68	10.83	351.46	725.66

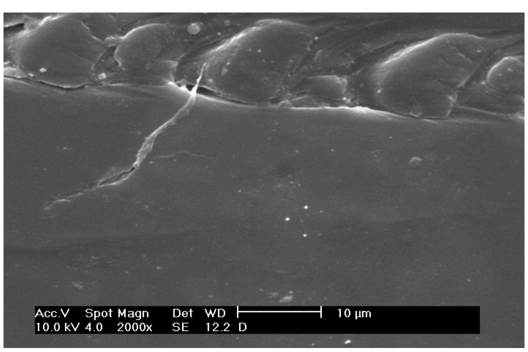
The melt flow index of the samples were also modified by increase concentration of bee wax add, as can be see in table 3.

Table 3 – Melt flow index of the samples.

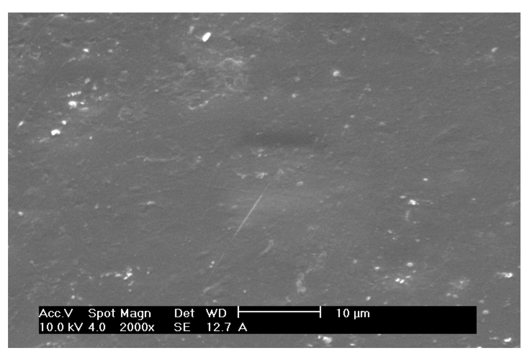
Sample	Melt flow index (g/10min)
A	1.50
B	1.97
C	2.34
D	3.07

For sample B, the increase in melt flow index was not significant when compared to the sample A. However, for samples C and D growth significantly, probably due to the exudation of wax in the respective samples. As the melting temperature of the wax is relatively low, around 65°C, the material that was not incorporated to the polymeric matrix migrates to the surface, acting as external lubricant.

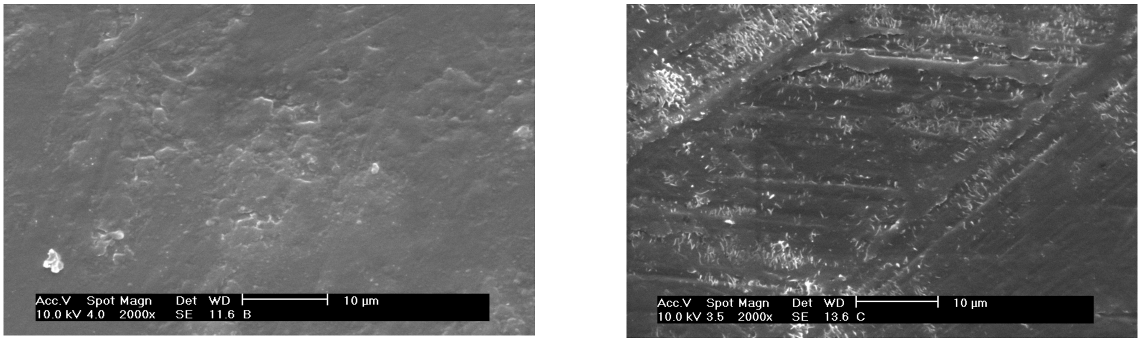
The exudation behavior of the bee wax in samples C and D can be observed in the micrographics obtained by scanning electron microscopy (SEM).



(a)



(b)



(c) (d)

Figure 1 – Micrographs of the four samples, 2000 X: (a) pure; (b) 1%; (c) 3% and (d) 6%.

Comparing the micrographs of samples C and D, is possible to observe the irregularity onto surface for both samples. In the case of sample D the wax particle excess is clearer. These evidence showed in figure 1(c) and 1(d) prove the lubricant effect[3-4] of the wax in concentrations above of 1%.

If the argument is true, as mentioned, the viscosity of modified copolymer will be lower than pure resin, as can be verified in figure 2, to complex viscosity.

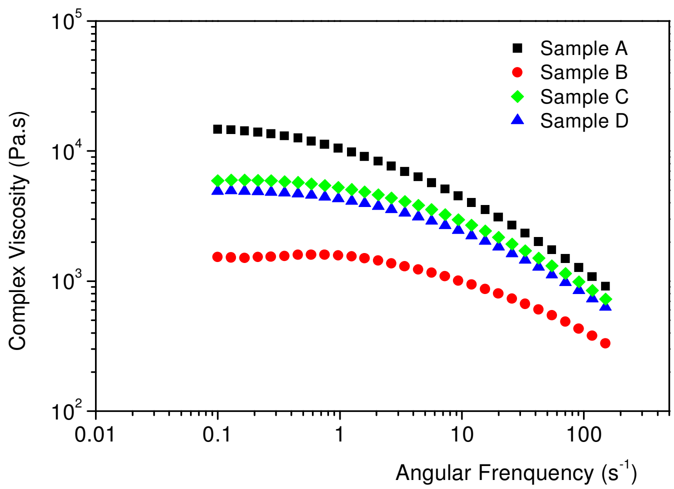


Figure 2 – Complex viscosity as a function of angular frequency.

A significant drop in the complex viscosity occurred in the sample B when compared with all samples. However, samples C and D, presented intermediated values of η^* in relation to the sample A and B. This behavior can be attributed to the plasticizer effect that the 1% concentration exerted in the material.

The glass transition temperature (Tg) of the samples was moved to low temperature with the bee wax addition, as can be observed in figure 3. In this way the increase of wax concentration improve the chain mobility and decreasing the Tg. These results were obtained after the subtraction of exponential background from each curve.

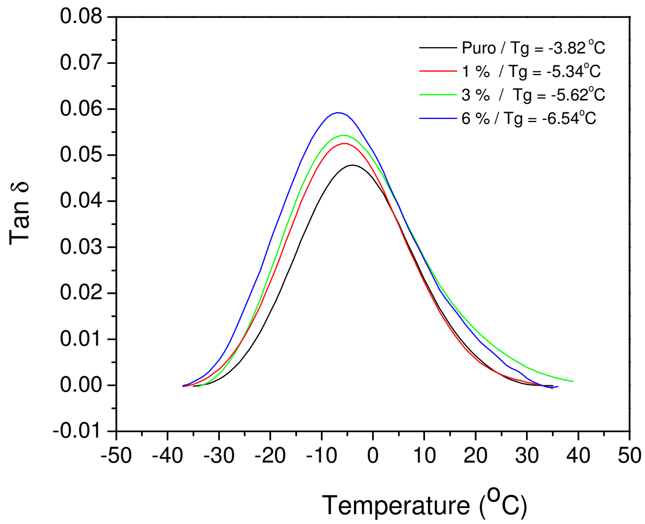


Figure 3 – Tan δ curves as a function of wax concentration.

Conclusion

The incorporation of the bee wax in the PP random influenced its mechanical properties at low concentration. The sample B containing 1% of wax was the one that improved mechanical properties as elastic modulus and elongation at rupture. Rheological properties were also affected by the wax addition, the melt flow index decreased and the complex viscosity reduced with 1% and increases for the 3 and 6%, but with values lower than the pure one.
The glass transition temperature decrease with concentration which denotes a chain mobility improvement. The SEM results show that the wax exuded to the copolymer surface in the samples with higher concentration, actuating as external lubricant.

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