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# STUDIES ON POLYPROPYLENE/CELLULOSE MICROFIBER COMPOSITES



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In this work composites of Polypropylene homopolymer (PPh), Polypropylene copolymer (PPc), and microfibers of cellulose were produced with and without compatibilizing agent. The interfacial behaviour of these composites was studied using torque rheometry, tensile tests and scanning electron microscopy (SEM). Torque values indicated that the maleic anhydride grafted polypropylene (PP-MAH), used as compatibilizing agent, indicated a different interaction with each matrix studied. Addition of cellulose microfibers leads to a slight increase in the tensile strength and modulus for the copolymer composites, whereas for the homopolymer a decrease was observed. In the latter one an improvement on this behaviour could be obtained only by using PP-MAH. The elongation was decreased significantly in all composites.SEM showed that PP-MAH seems to improve adhesion for some systems, consistent with torque rheometry results.

#### Introduction

Polypropylene (PP) as one of the most popular versatile thermoplastic polymer provides many advantages such as low cost, recycle ability, and high thermal stability, allowing the production of many kinds of composites [1]. The combination of properties of polypropylene and cellulose has been studied by several authors, with different kinds of cellulose, like highly crystalline cellulose [1], nanoscale microfibrils of cellulose [2], spun cellulose fibers from the viscose, lyocell and carbamate processes [3], PP hybrid composites [4], and microcrystalline cellulose and sulfite fiber [5].

Addition of cellulose in PP/cellulose composites reduces the tensile strength when a compatibilizing agent it is not used. This property can be increased when 10% wt of maleic anhydride grafted polypropylene (PP-MAH) is added [1].

The aim of this work was to compare different compositions using PP homopolymer, PP copolymer, and cellulose microfiber, using torque rheometry, tensile tests, and scanning electron microscopy.

### Experimental

#### Materials

Polypropylene homopolymer (PPh), grade KM6100, (melting flow index of 3.15g/10 min, 230°C/2.16kg),

from Polibrasil and a random polypropylene copolymer from propylene and ethylene (PPc) grade RP347, (melting flow index of 10g/10min, 230°C/2.16kg), and from Braskem were both used as matrix, as indicated. Maleic anhydride grafted polypropylene (PP-MAH) (Orevac®) supplied by Arkema was used as interfacial compatibilizing agent. CF11, 50-350µm in length. about 20 µm in diameter; crystallinity 93% (cellulose type I) was supplied by Whatman Int. Ltd.).

# Processing and characterization

The compositions containing PPh, PPc, Cellulose, and PP-MAH (Table 1) were blended in a Rheomix 600 mixer connected to a HAAKE torque rheometer at 200°C and 50rpm for 10 minutes. All materials were previously dried during 15h at 60°C under vacuum prior to processing. Films with dimensions of 80mmx80mm, and 0.4mm of thickness were obtained for all compositions (Table 1). To obtain the films, materials were processed at 200°C, for 5 minutes without applying pressure, followed by applying 2 tons for 0.5 min. The tensile test specimens were conditioned and tested according to ASTM D882 using an universal testing machine, EMIC model DL3000 with a load cell of 50kgf and 5mm/min of crosshead speed. Scanning Electron Microscopy - SEM (Philips SL-30 FEG microscope) was used to evaluate the interfacial adhesion in the composites.

Table 1 – '	The HAAK	E blending	compositions
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#	Samples	%wt
1	PPh	100
2	PPh/Cellulose	98/2
3	PPh/Cellulose/PP-MAH	96/2/2
4	PPh/PP-MAH	98/2
5	PPc	100
6	PPc/Cellulose	98/2
7	PPc/Cellulose/PP-MAH	96/2/2
8	PPc/PP-MAH	98/2
9	PP-MAH	100

### **Results and Discussion**

Torque rheometry tests were performed to evaluate the average melt viscosity of the materials used [6]. Results showed that torque values stabilized after 5 minutes. Although the differences observed among torque at 10 minutes for different compositions, they are significant, since they indicate that interaction processes seems to be occurring in the mixture.

Torque overshootings are observed during loading of solid components in the mix chamber, before softening and melt of the components occur. These values depend of addition rate, size and form of the particles during loading and may present different among experiments.

Torque rheometry results from homopolymer composites (Figure 1) showed that the incorporation of cellulose microfibers leads to a decrease in the torque measured at 10 min, as compared to the pure PPh. PP-MAH was used in this system as compatibilizing agent, since the presence of maleic anhydride could interact with cellulose hydroxyl groups. Addition of PP-MAH in PPh reduces the viscosity.

The torque was also reduced upon the addition of cellulose to the copolymer, as presented in Figure 2. It was observed in the conditions used that PP-MAH, alone, presents a viscosity lower than all the polymeric matrices and composites investigated, as expected. However, differently from the homopolymer, addition of PP-MAH to PPc does not reduce the viscosity of system.



Figure 1 – Torque curves as a function of time of the PP homopolymer composites obtained with the Haake at 200°C and 50rpm.



Figure 2 – Torque curves as a function of time of the PP copolymer composites obtained with the Haake at 200°C and 50rpm.

It can be seen in Table 2 that the addition of cellulose microfibers presented a slight increase in the tensile strength and modulus of the copolymer composites, whereas for the homopolymer a decrease was observed. In the latter one an improvement in this behaviour could be obtained only by using PP-MAH as compatibilizing agent. According to Qiu et al. [1] for these types of systems the interaction can be due to enhanced interfacial esterification between PP-MAH and active cellulose sites.

The elongation was decreased significantly in all composites, as expected, although it was much stronger for the copolymer. The elongation at rupture was reported at the maximum value obtained. This distinct mechanical behaviour should be due to differences on the macromolecular structure between polypropylene homopolymer and random polypropylene copolymer.

Samples	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Rupture (%)
PPh	$20.9 \pm 1.4$	$0.66 \pm 0.06$	568
PPh/Cellulose	$18.6 \pm 0.9$	$0.54 \pm 0.04$	380
PPh/Cellulose/PP-MAII	$20.6 \pm 1.1$	$0.68 \pm 0.06$	200
PPh/PP-MAII	$20.8 \pm 1.5$	$0.62 \pm 0.04$	474
ppe	$21.5 \pm 0.7$	$0.62 \pm 0.05$	985
PPc/Cellulose	$21.8 \pm 1.4$	$0.67 \pm 0.06$	30
PPc/Cellulose/PP-MAH	$19.5 \pm 0.9$	$0.62 \pm 0.04$	139
PPc/PP-MAII	$21.1 \pm 1.2$	$0.65 \pm 0.06$	530
PP-MAH	$16.3 \pm 1.0$	$0.70 \pm 0.03$	4

In Figure 3, one may notice a better adhesion between the components for the PPh/Cellulose/PP-MAH and PPc/Cellulose/PP-MAH systems, as compared to the PPh/Cellulose and PPc/Cellulose. The photomicrographs of composites with cellulose obtained by SEM indicate compatibilization effect of the interface when using PP-MAH in the composition of the composites.

XI International Macromolecular Colloquium



Figure 3 – SEM Photomicrographs of the composites: (A) and (B) PPh/Cellulose; (C) and (D) PPh/Cellulose/PP-MAH; (E) and (F) PPc/Cellulose; (G) and (H) PPc/Cellulose/PP-MAH.

# Conclusions

The compatibilizing agent PP-MAH showed different effects in the systems PPh/cellulose and PPc/cellulose, with also different tensile behaviour for PPc/Cellulose/PP-MAH composite. The SEM photomicrographs indicates improvement on the interfacial interaction. These results are corroborated by torque rheometry data.

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