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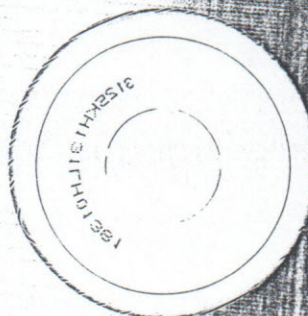
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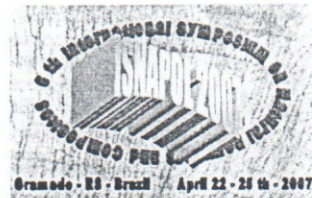


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## INTERFACIAL BEHAVIOUR OF COMPOSITES OF RECYCLED POLY(ETHYLENE TEREPHTHALATE) AND SUGAR CANE BAGASSE FIBERS



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In this work composites of recycled Poly(ethylene terephthalate) (PETr) and sugar cane bagasse fibers were produced with and without compatibilizing agents. The interfacial behaviour of these composites was studied using torque rheometry, tensile tests and scanning electron microscopy (SEM). Comparing the compatibilizing agents ethylene/n-butyl acrylate/glycidyl methacrylate copolymer (EBGMA) and a copolymer of ethylene and methyl acrylate (EMA), the torque values indicated that EBGMA increased the interaction between the constituents more effectively than a EMA. Addition of bagasse sugar cane fibers kept the tensile modulus and reduced the tensile strength and elongation of PETr, as usually observed for these types of composites. SEM showed that EBGMA improved adhesion between the constituents, consistent with the results of torque rheometry. All the composites showed a nice visual aspect and could be a good alternative to produce ecologically friendly products.

### Introduction

Recycling of Poly(ethylene terephthalate) represents one of the most successful and important example of polymer recycling [1]. The main driving forces responsible for the increased recycling of post-consumer PET are its widespread use, particularly in the beverage industry which has made PET the main target for plastics recycling [1].

The use of natural fibers as additives for composite materials presents a great potential for improving their performance and technological application, due to their low cost, abundance, biodegradability and high specific strength [2]. This is especially important if such fibers are residues of agro industrial process and if their raw-properties are acceptable for composites [3], since it also responds to ecological and societal appeals. This is the case of sugar cane bagasse fiber, which is largely produced in Brazil as a by-product from the sugar and bio-ethanol industry [4].

The aim of this work was to produce composites using recycled PET and sugar cane bagasse fiber and to study their interfacial behaviour with two different commercial interfacial compatibilizings agents.

### Experimental

#### Materials

In this work recycled Poly(ethylene terephthalate) (PETr) obtained from the beverage industry was used

in the form of pellets. The sugar cane bagasse fibers (FC) with length smaller than 3mm were kindly supplied by EDRA Ecosistema Ltda. A copolymer of ethylene and methyl acrylate (EMA) containing 24% methyl acrylate, commercially known as Elvaloy<sup>®</sup> 1224AC and an ethylene/n-butyl acrylate/glycidyl methacrylate copolymer (EBGMA), commercially known as Elvaloy<sup>®</sup> PTW from DuPont company were used as interfacial compatibilizing agents.

#### Processing and characterization

The compositions containing PETr, FC, EMA and EBGMA (Table 1) were blended in a Rheomix600 mixer connected to a HAAKE torque rheometer at 230°C and 50rpm for 10 minutes. All materials were previously dried at 80°C under vacuum before processing. The compositions PETr, PETr/EMA, PETr/EBGMA, PETr/FC, PETr/FC/EMA, PETr/FC/EBGMA were processed at 250°C for 3 minutes without pressure and after which a force at 4 tons for 0.5 min was used to produce molded sheets of (80x80)mm<sup>2</sup> and 0.3mm of thickness (Figure 1). 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 between PETr with the sugar cane bagasse fiber.

Table 1 – Compositions prepared at the HAAKE rheometer

#	Samples	%wt
1	PETr	100
2	EMA	100
3	EBGMA	100
4	PETr/EMA	95/5
5	PETr/EBGMA	95/5
6	PETr/FC	95/5
7	PETr/FC/EMA	90/5/5
8	PETr/FC/EBGMA	90/5/5
9	EMA/FC	95/5
10	EBGMA/FC	95/5

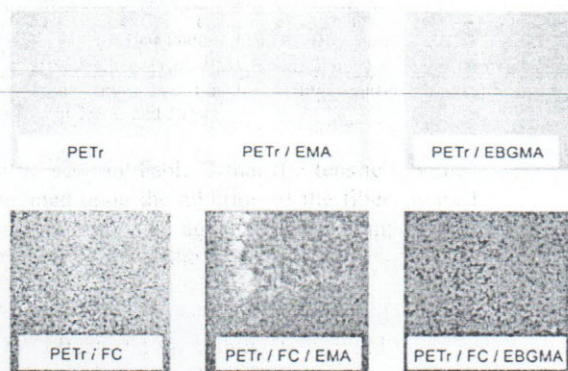


Figure 1 - Molded sheets used to produce the tensile tests samples.

## Results and Discussion

A very nice texture and visual aspect were obtained for all the composites investigated as shown in Figure 1. The torque rheometry results (Figure 2) show that the presence of 5%wt of the compatibilizer, EMA or EBGMA, increased the viscosity of the systems PETr/EMA or PETr/EBGMA. This seems to be an indication that some synergistic interaction is occurring in these compositions.

The synergistic effect was obtained from the experimental torque values by subtracting the calculated torque values using the additive rule. These values were 1.1N.m and 0.4N.m for PETr/EMA and PETr/EBGMA, respectively. These results also indicate that the interaction between PETr with EMA is higher than PETr with EBGMA.

Adding 5%wt of the FC to PETr matrix made the torque values to decrease, when it was compared to the pure PETr. On the other hand, addition of EMA to PETr/FC, leads to an increase in torques values to around 0.2N.m. An increase of 0.4N.m in torque was observed by adding EBGMA in the same system, indicating that PETr/FC/EBGMA system presents a higher interaction.

In Figure 3, one may notice a poor adhesion between the components for the EMA/FC system, whereas for EBGMA/FC a good adhesion seems to be obtained. These results indicate that different types of interaction are occurring. It is suggested that for EMA/FC the interaction is mainly formed by secondary bonds and for EBGMA/FC the interaction is predominantly formed by primary bonds.

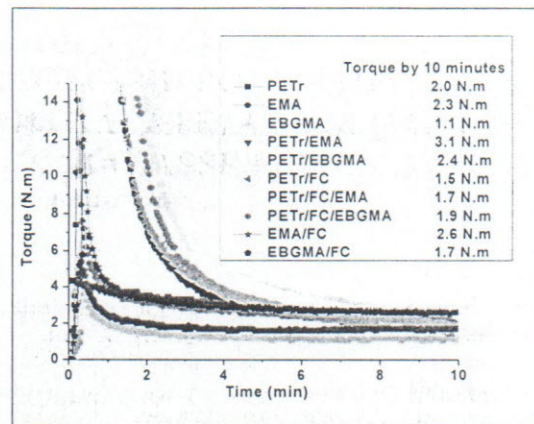


Figure 2 – Torque curves as a function of time, performed in a Haake rheometer at 230°C and 50rpm.

It can be seen in Table 2 that the tensile modulus was maintained upon the addition of the fiber, as well as the compatibilizing agent in all compositions investigated, with the exception of the PETr/FC/EBGMA composites, for which a slight decrease seems to be noticed. This might be due to the fact that the effect of compatibilization could be more pronounced for this compatibilizer.

On the other hand, the tensile strengths were decreased in all samples as compared with PETr. Even the two compatibilizing agents themselves also reduced the average tensile strengths when added to pure PETr. The reduction on tensile strengths upon FC incorporation could be explained by the poor mechanical properties of FC. Stael et al. [5] have observed similar effect in EVA polymer matrix, with reduction of deformation capacity and impact resistance of bagasse waste in EVA composites.

As it is usually expected the incorporation of fiber resulted in a reduction of the elongation at rupture, which it was also slightly decreased upon addition of the compatibilizing agents to the PETr.

Table 2 – The tensile tests results

Samples	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Rupture (%)
PETr	42.8 ± 1.5	1.3 ± 0.1	5.0 ± 0.4
PETr / EMA	38.6 ± 0.4	1.3 ± 0.1	4.7 ± 0.2
PETr / EBGMA	39.8 ± 0.9	1.3 ± 0.1	4.9 ± 0.1
PETr / FC	24.8 ± 1.7	1.3 ± 0.1	3.1 ± 0.2
PETr / FC / EMA	24.9 ± 1.7	1.3 ± 0.1	3.1 ± 0.1
PETr / FC / EBGMA	24.6 ± 1.1	1.1 ± 0.1	3.5 ± 0.2

Figure 3 shows the photomicrographs of composites with FC obtained by SEM. It is possible to observe the compatibilized interface when using EBGMA in the composition of the composites.

In the Figure 3 (G) and (H) the interaction between the sugars cane bagasse fiber and EMA and EBGMA can be observed. EMA does not show a good interaction but for EBGMA a good adhesion seems to be occurring as the matrix is covering the fiber completely.

Among the composites, the largest value of elongation at rupture was obtained for the system PETr/FC/EBGMA, because it showed the best

interfacial adhesion, consistent with the SEM micrographs.

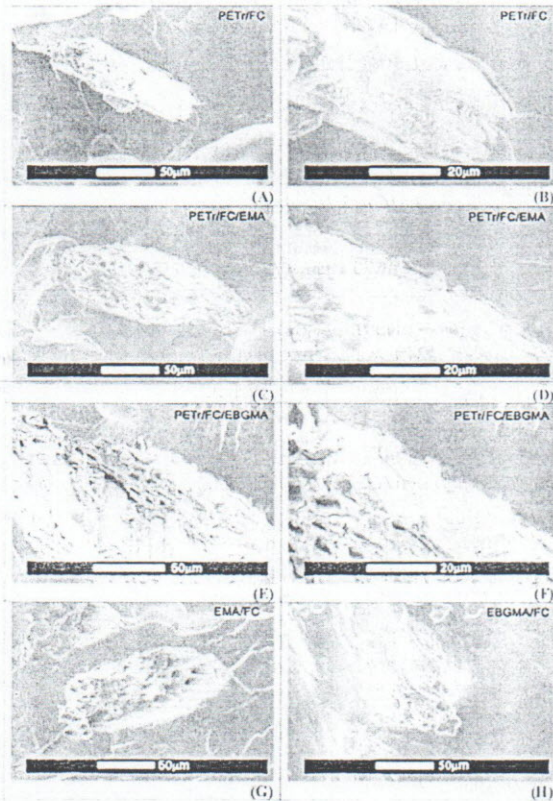


Figure 3 – SEM micrographs of composites: (A) and (B) PETr/FC; (C) and (D) PETr/FC/EMA; (E) and (F) PETr/FC/ EBGMA; (G) EMA; (H) EBGMA/FC.

## Conclusions

Torque rheometry and SEM have demonstrated that the EBGMA compatibilizer showed better adhesion effect than EMA for PETr/FC composites. Tensile modulus was maintained upon the addition of the fiber, as well as the compatibilizing agent in all compositions investigated, whereas the tensile strength was reduced. Nevertheless the level of the mechanical properties achieved for the composites is still adequate for their use in several types of products.

The texture and visual aspects of the composites in all the formulation were very satisfactory. Recycled PET filled with sugar cane bagasse fibers seems to be a good alternative for obtaining ecologically friendly products.

## Aknowledgements

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## References

1. J. Scheirs in *Polymer Recycling: Science, Technology and Applications*, John Wiley & Sons, New York, 1998.
2. V. Tserki, P. Matzinos, C. Panayiotou, *Composites A*, 2006, 37, 1231.
3. E. Garcia-Hernandes, A. Licea-Claverie, A. Zizumbo, A. Alvarez-Castillo, P. Herrera-Franco. *Polym. Compos.*, 2004, 25, 134.
4. T.C.C. Ripoli, W.F. Molina Jr., M.L.C. Ripoli, *Scientia Agricola*, 2000, 57, 677.
5. G.C. Stael, M.I.B.Tavares, J.R.M. d'Almeida, *Polym. Test.*, 2001, 20, 869.