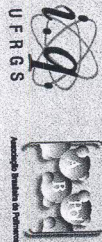


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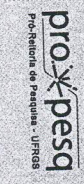
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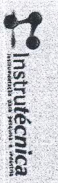
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XII INTERNATIONAL MACROMOLECULAR COLLOQUIUM

ISNaPol 2010

7th INTERNATIONAL SYMPOSIUM ON NATURAL POLYMERS AND COMPOSITES

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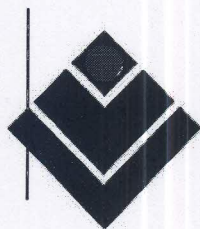
FOREWORD

It is a great pleasure for us to welcome all the participants of the **XII International Macromolecular Colloquium and the 7th International Symposium of Natural Polymers and Composites**. We are very pleased with your contributions which are very important to the success of the Meeting. During this time, researchers will have the opportunity to initiate and enhance fruitful interactions among different institutions around the world working in the field of Polymer Science and Technology. We hope this Meeting will also offer a good opportunity to improve the research on the field of natural polymer-based materials and composites developed in Brazil.

Without your participation and specially the contribution of those presenting the 30 lectures, 34 oral sessions and 411 posters it would not be possible to organize this Meeting. We would like to acknowledge also the support from BRASKEM, CAPES, CNPq, FAPERGS, FAPESP, Petrobras and PROPESQ-UFRGS and the participation of the exhibitors dpUnion, Instrutécnica, Polimate and Reoterm.

We wish all the participants lots of interesting discussions and important stimulus for their further work and a pleasant stay in Gramado.

Organizing Committee



THERMAL CHARACTERIZATION OF POLY(METHYL METHACRYLATE)/RICE HUSK SILICA COMPOSITES

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Talick C. Gozzoli¹, Scheila D. F. Alves¹, Vitor B. Carmona¹, Luiz H. C. Mattoso¹, José M. Marconcini^{1*}

^{1*}National Nanotechnology Laboratory for Agriculture (LNNA), Embrapa Agricultural Instrumentation, P. O Box 741, Zip Code: 13560-970, São Carlos - SP, Brazil, marconcini@cnpdia.embrapa.br

Composites of two grades of poly(methyl methacrylate) (PMMA) were formulated by casting method and studied in this work with rice husk silica. Infrared spectroscopy and thermogravimetric analysis were realized to characterize PMMAs. Thermal characterization by differential scanning calorimeter (DSC) was performed to raw materials and composites. It was observed changes of glass transition temperature (T_g) to studied composites, with increase of T_g to most composites studied.

Introduction

In recent years, there have been many attempts to develop polymeric composites and nanocomposites as advanced plastics materials. Poly(methyl methacrylate) (PMMA) is an important amorphous thermoplastic material with excellent transparency, chemical and UV resistance. However, one of its limitations is its thermal instability and dynamic mechanical properties at elevated temperatures which restrict its applications in high temperature environment.^{1,2}

Composites of polymer matrix and inorganic load have attracted considerable attention in the last two decades due to interest both academic and industrial fields.^{1,2}

In the case of poly(methyl methacrylate) (PMMA) and silica composites, improvements in the mechanics, thermo, optics, electric and barrier properties have been reported also for composites of bulk polymers and thin films³.

This paper intends to obtain the PMMA with silica composites and its thermal characterization. Injection molding PMMA grade and PMMA from industrial waste were polymer used, and as inorganic load, had been utilized commercial silica and rice husk silica.

Experimental

Materials

In this work it was used PMMA injection grade (synthesized from suspension polymerization), PMMA from industrial waste (synthesized from bulk polymerization) and chloroform in analytical grade. Silica from rice husks was obtained accordingly Souza et. al 2002⁴.

Methods

Spectroscopy on the infrared region

The identity of chemical substances, as changes in the structure can be obtained by infrared spectroscopy.

Films were made by solubilizing the polymer in chloroform and then casting on a glass surface and waited the evaporation of the solvent, during 48h. The films could be teared off and analyzed.

Thermogravimetric Analyses

It's important to understand how the heat affects the PMMA. One good way to measure it is the thermogravimetric analyses, which consists in heating the material and checking the loss e weight as the temperature rises.

Had been taken an amount about 7mg and put into the sample holder and set it on the TGA Q500, from TA Instruments. Heating rate had been adjusted to 10°C/min in nitrogen. With the results, the graphs temperature against loss weight were constructed so the characteristics of the films could be analyzed.

Differential Scanning Calorimeter (DSC)

It had been taken an amount about 7mg and put into the sample holder and set it on the DSC Q100, from TA Instruments. The heating rate had been adjusted to 20°C/min from room temperature to 180°C, in nitrogen atmosphere (50mL/min).

Composites preparation

Composites were prepared by solubilizing the polymers in chloroform, adding rice husk silica in the

following contents: 1, 2, 5 and 7.5% and stirring the solution during 1h.

Composite films were obtained by casting on a glass surface and waited the evaporation of the solvent, during 48h. The films could be teared off and analyzed

Results and Discussion

The infrared spectrum obtained to pure PMMA to injection and from industrial wastes is in Figure 1.

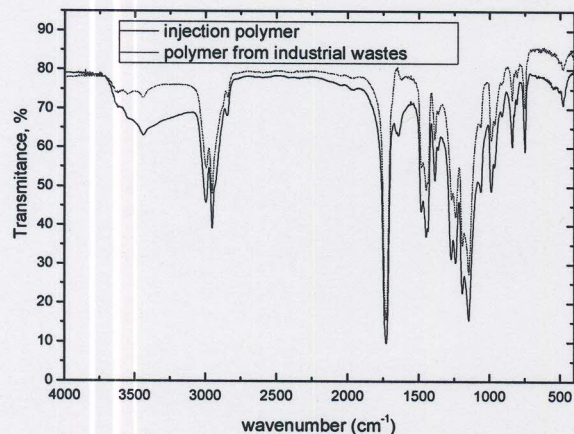


Figure 1. Infrared spectrum of PMMAs.

PMMA exhibit a strong vibration of the ester carbonyl stretch at 1736cm^{-1} , two CH stretching vibrations at 2975 and 3003, two strong bands at 1445 and 1494 from the vibration of angular deformation of the connection and an O-CH₃ very strong absorption at 1139cm^{-1} associated with the stretching of CO bond, and two bands at 1401 and 1234cm^{-1} angular deformation vibration of CH bond⁶.

Figures 2 and 3 show thermogravimetric analyses of the injection PMMA grade and from industrial wastes in nitrogen at heating rates of $10^\circ\text{C}/\text{min}$, respectively.

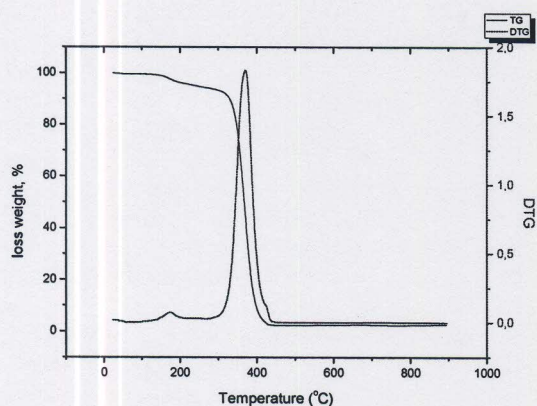


Figure 2: TG/DTG curve of the PMMA from industrial wastes at $10^\circ\text{C}/\text{min}$ in nitrogen.

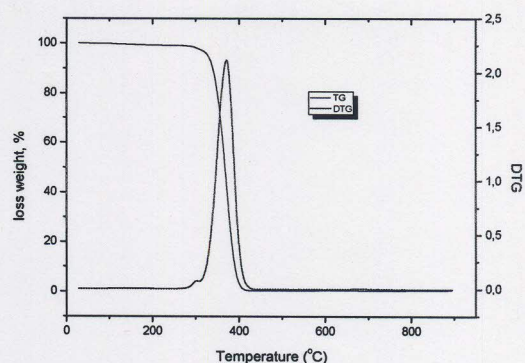


Figure 2: TG/DTG curve of the PMMA for injection at $10^\circ\text{C}/\text{min}$ in nitrogen

These curves permit identifying the initial temperatures of degradation ($^\circ\text{C}$), volatile content (%) at 267°C for the PMMA from industrial wastes and 300°C for the PMMA for injection, respectively. Volatile content observed for PMMA from industrial waste is 5.64% and for the PMMA injection grade is 1.74%. Possible volatiles observed to these materials are humidity and liquids, such as monomer and solvent residuals. Higher content volatile was observed to PMMA from industrial waste, and it was possible that this high value occurs due high content of residual monomer from bulk polymerization process.

Glass transition temperatures of the studied materials obtained by DSC are listed in the table 1.

Table 1: Glass transition temperature of pure PMMA and for PMMA and silica composites

Composition (PMMA / silica)	Glass Transition Temperature, T _g ($^\circ\text{C}$)
PMMA injection grade / 0% of silica	91
PMMA injection grade / 1,0% of silica	115
PMMA injection grade / 2,0% of silica	132
PMMA injection grade / 5,0% of silica	121
PMMA injection grade / 7,5% of silica	122
PMMA from industrial wastes / 0% of silica	121
PMMA from industrial wastes / 1,0% of silica	115
PMMA from industrial wastes / 2,0% of silica	130
PMMA from industrial wastes / 5,0% of silica	128
PMMA from industrial wastes / 7,5% of silica	110

Composites with PMMA injection grade showed an increase of T_g, comparing with raw material. This result should be explained by the interaction from silica and acrylic components, that reduces mobility of polymer chains. Composites with PMMA from industrial waste showed an increase of T_g to the compositions 2.0 and 5.0%, of rice husk silica, and a reduction to 1.0 and 7.5%. These results are under investigation, to understand this behaviour.

The increase of T_g to the composites is important, because it allows to use PMMA in a higher temperature.

Conclusions

It is possible to obtain composites of PMMA and silica from rice husks, with enhanced properties comparing with raw materials. Thermal properties were affected with inclusion of silica in the composites.

Besides infrared spectrum is similar for both PMMAs, the DSC results are different, it indicates that, even though there is no structural molecular change, there are some morphological alteration. It is imperative that more studies, like tensile and melt flow index tests, must be done in order to attest that the PMMA from industrial waste can be used instead of the PMMA for injection.

Acknowledgements

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References

1. M. Laachachi, E. Cochez, M. Leroy, J.M. Ferriol, Lopez-Cuesta *Polymer Degradation and Stability*, 2007, 92, 61.
2. M. Alexandre; P. Dubois *Materials Sci. & Eng.* 2000, 28, 1.
3. D. Stojanovic, A. Orlovic, S. Markovic, V. Radmilovic, P.S. Uskokovic, R. Aleksic, *Journal of Materials Science*, 2009, 44, 6223.
4. M.F. de Souza, W.L.E. Magalhães, M.C. Persegil *Materials Research*, 2002, 5, 467.
5. F.A. Zhang, D.K Lee., T.J.Pinnavaia, *Polymer*, 2009, 50, 4768.
6. J. A. Brydson, *Plastic Materials*, 7th edition, Butterworth and Heinemann ed., London, 1999, 398-424.