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Preparation of polyurethane/montmorilonite polymeric nanocomposites by solution and characterization using low-field NMR

M. Anacleto [(1), (2)] and M. I. B. Tavares [(1)*]

(1) IMA, Universidade Federal do Rio de Janeiro, e-mail: mibt@ima.ufrj.br * Corresponding author.
(2) NUCAT/PEQ/COPPE, Universidade Federal do Rio de Janeiro, RJ - Brazil

Abstract – Polyurethanes have desirable mechanical properties. However, there also exist some disadvantages, for example, low thermal stability and barrier properties. To overcome the disadvantages, research on novel polyurethane/clay nanocomposites has been carried out. The investigation of the structure of polyurethane/clay nanocomposites has been mostly done by X-ray diffraction (XRD) and transmission electron microscopy (TEM). In this work, PU/clay films were prepared by solution, and the obtained nanocomposites were characterized by XRD and low-field nuclear magnetic resonance (NMR). NMR measurements were able to provide information on molecular dynamics of the nanocomposites. In addition, they also confirmed the results obtained by XRD.

Polymer-layered silicate nanocomposites have recently gained a great deal of attention because they offer a great potential to provide superior properties when compared to pure polymers and conventional filled composites. These properties include reduced gas permeability, improved flame retardance and enhanced mechanical properties. Most of the works reported in the literature have focused on preparation of PU/clay nanocomposites employing *in situ* polymerization and melting intercalation due the interaction and dispersity difficulties between organic-inorganic hybrid components [1]. The investigation of the structure of organic-inorganic nanocomposites has been mostly done by X-ray diffraction (XRD) and transmission electron microscopy (TEM). In this work, PU/clay films were prepared by solution, and the obtained nanocomposites were characterized by XR D and low-field nuclear magnetic resonance (NMR).

The membrane films were prepared with the mixture of two dispersions: PU in tetracloroethane (TCE) solvent and the organically modified montmorillonite (OMMT) clay (Viscogel B8) in TCE solvent. These solutions were mixed at 70^{o}C by using a rotating spinner for 24 hours. Subsequently, the final solution was put onto plates and kept at room temperature for three days to be dried. For complete solvent removal, the membranes were placed in a muffle furnace at 80^{o}C for one week. Afterwards, these membranes were kept in a dissector at room temperature. PU membrane as well as 1%wt and 2%wt OMMT/PU nanocomposites films were prepared. XRD and low field NMR experiments were performed on film samples.

T_1H measurements for PU membrane and its nanocomposites with 1%wt and 2%wt of OMMT films are in the Table 1. The results showed that the incorporation of 1 %wt OMMT caused a slight reduction of the molecular mobility of polyurethane matrix, suggesting a formation of OMMT/PU nanocomposites with a predominance of interacted and some exfoliated, since the metals present in the clay lamellae act as relaxation agents, a small decrease in the T_1H confirms that the polymer chains may be around the clay lamellae. The little change in the T_1H value, after the addition of 2%wt, suggests the presence of two domains in nanocomposites: – one that is intercalated (because of polymer chains have lower molecular motions, as they are restricted between clay lamellae) and – another one that is clay aggregate.

The X-ray diffractogram of clay showed two peaks, one at 2θ = 3.15°, for organophylic structure, and the other in 2θ = 6.80°, part of the structure of pure clay. In the patterns of 1%wt and 2%wt OMMT/PU, the peak at 2θ = 3.15° was totally absent, suggesting an exfoliation of the clay platelets in the polymeric matrix, which was confirmed by the increase in the basal spacing of the clay platelets due to shifts in the 2θ samples for lower values for pure clay, indicating a possible intercalation. However, the XRD patterns for 2%wt OMMT/PU showed a visible peak at 2θ = 6.80°, which indicated presence of pure clay.

Low field NMR measurements were able to provide important information on molecular dynamics of the polymeric nanocomposites OMMT/PU. In addition, they also confirmed the results obtained by XRD. So far, all these measurements point to the possibility of obtaining nanocomposites of OMMT / PU by solution.

Table 1:T_1H measurements of PU membrane and OMMT/PU nanocomposites

SAMPLES	T_1H (ms)
PU	54
1 %wt OMMT/PU	50
2 %wt OMMT/PU	53

[1] Meng, X.; Du, X.; Wang, Z.; Bi, W.; Tang, T.; *Composites Science and Technology* ***2008***, 68, 1815-1821