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THERMOMECHANICAL TESTS APPLIED TO THE CHARACTERIZATION OF GREEN POLYURETHANE FOAMS

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ABSTRACT

In recent years, there has been an increasing demand for products of renewable origin with the aim of providing solutions to environmental problems, eliminate or minimize wastes and provide alternatives for the synthesis of new materials, due to the exhaustion of fossil sources. Renewable resources can provide a sustainable alternative to totally or partially replace petroleum-based polymers. Through the design of biobased polymers, which are an innovative technology, it is possible to replace traditional materials with ecofriendly substances. The new polymers are expected to exhibit similar performance to those of petrochemical origin. The most used renewable resources are polysaccharides (mainly cellulose and starch), proteins and vegetable oils, the latest being one of the most important raw materials for the production of polymeric materials.

Keywords: Fossil sources; Environmental problems

INTRODUCTION

The objective of this work is to complement previous studies [1] of polyurethanes characterization obtained from oily fractions of olive oil not suitable for human consumption without previous refinement

MATERIALS & METHODS

Materials Biopolymers were synthesized by reacting 4,4'-diphenylmethyldiisocyanate with polyols derived from three low-quality oily fractions: olive-pomace oil (PU OOHT), lamp oil (PU ALHT), and clear oil lees (PU CBHT). Polyols were obtained from these oily fractions through a modification process that involves steps of epoxidation, hydrolysis and transesterification, consecutively. In addition, samples of polyurethane foams using a commercial polyol of non-renewable origin, Jeffol G30- 650, were made for comparative purposes (PU Jeffol G30).

Methods: Dynamic mechanical analysis (DMA) and differential scanning calorimetry (DSC) tests were performed. DMA tests were carried out in torsion at a frequency of 1 Hz and an amplitude of 0.1% in a rheometer Anton Paar model MCR 301. The samples were prepared according to the rectangular geometry of 13.72 mm long, 7.1 mm wide and 3.36 mm thick and were conditioned by convection in a CTD600 chamber with compressed air. The thermal transitions of the polyurethanes were studied by DSC using a Shimadzu DSC-50 calorimeter. The samples (5-7 mg) were placed in aluminum capsules. The heating rate was 10 °C/min and the studied temperature range was -63 to 190 °C.

RESULTS

Differential Scanning Calorimetry

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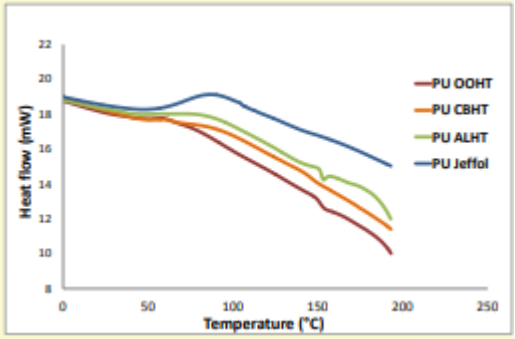

Figure 1: Comparison of DSC curves for tested samples.

As a consequence of the high crosslinking density of the samples, they adopt an amorphous morphology that can be corroborated by the absence of melting peaks in the DSC profiles, thus indicating the absence of crystallinity in the samples [3]. As the degree of crosslinking increases, the number of tie points between the chains increases and the segments that can move decrease. By decreasing the mobility of the chains, the glass transition temperature Tg increases [4]. The small endothermic peaks present in the PU OOHT and PU ALHT samples around 150°C may be due to certain densifications of the material (this usually appears when the material is thermoset) [5]. Table 2 (third column) summarizes the Tg values of the samples, determined at the beginning of the transition.

Dynamic Mechanical Analysis

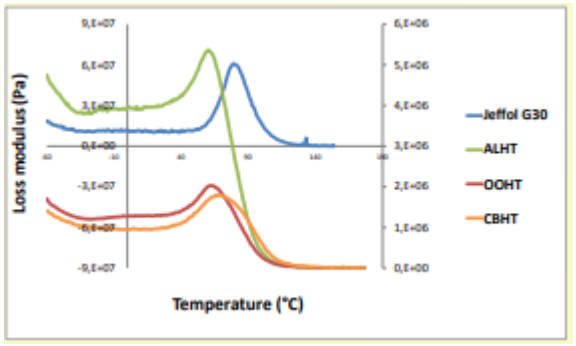

Figure 2: Loss modulus curves as a function of temperature.

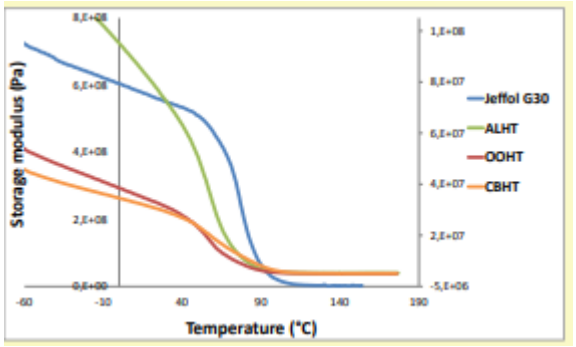

Figure 3: Storage modulus curves as a function of temperature.

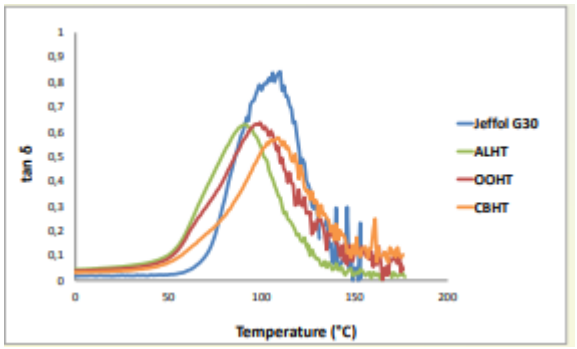

Figure 4: Tan δ curves as a function of temperature.

Table 1: DMA data for foams obtained from green polyols and from commercial polyol.

Samples	Tg (°C)	Max tan δ
PU ALHT	92	0.63
PU OOHT	99	0.64
PU CBHT	109	0.58
PU Jeffol G30	110	0.84

The characteristic changes of the modules and tan δ caused by thermally activated molecular movements begin at defined temperatures. The glass transition temperature (Tg) of the polymer was calculated from tan δ versus temperature curve (Figure 4). Table 1 summarizes the viscoelastic properties of PU foams measured by DMA. First column of the table lists glass transition temperatures, which are associated with the α relaxation of the soft domains in the foams. Both Tg and the maximum of tan δ represent the mobility of polymer chain. These values can be associated with diisocyanate conversion in foams, a greater conversion of diisocyanate results in higher Tg values and, therefore, restricts the mobility of the chain. Obviously, a lower conversion of diisocyanate leads to a lower Tg, and a greater chain mobility. Therefore, these results show that reaction conversion is directly connected to chain mobility and to the viscoelastic behavior of the foams [6]. The loss factor value is a measure of the material's ability to dampen mechanical vibrations, which propagate as waves in it. Although DSC testing is the traditional technique used to measure polymer Tg, DMA has always been used as another alternative method. Because this measurement has a strong mechanical dependence, the Tg value obtained is always higher than that found by DSC. Values obtained by these methods can differ by up to 25 °C from each other (Table 2). That is why Tg values derived from DSC and DMA differ and are generally only complementary, but not identical, since the results come from a calorimetric method on the one hand, and a dynamic mechanical method on the other.

Table 2: Comparison of Tg determined by DSC and DMA.

Samples	Tg DMA (°C)	Tg DSC (°C)

PU ALHT	92	85
PU OOHT	99	63
PU CBHT	109	92
PU Jeffol G30	110	87

CONCLUSIONS

Considering that one of the main applications of rigid polyurethane foams is as thermal insulation, Tg values found in the samples are promising. In addittion, the polyurethane Tg obtained from the oils do not differ significantly of that obtained from non-renewable polyol. The Tg values found by both characterization techniques are corroborated. It can be concluded that the samples have amorphous morphology since the glass transition occurs only in this type of material. In thermoset materials nothing happens after the Tg.

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