

Doped Bio-Based Polyurethane Foam Tensile Characterization

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Abstract: This paper aims the tensile characterization of a bio-based polyurethane foam (PUF) when doped with alumina trihydrate (ATH) as flame retardant. The tensile test methods used in this work were adapted from an ASTM standard and previous works in order to obtain the strain field showed with the aid of digital image correlation (DIC). The results were similar for the pure PUF and PUF with a small amount of ATH. However, the addition of over 20% of ATH presented specimens with the highest stiffnesses and smallest strains.

Keywords: Polyurethane foam, tensile test, digital image correlation, flame retardancy.

INTRODUCTION

The first polyurethane (PU) was synthesized by Professor Otto Bayer in 1937 when he was working at the I. G. Farbenindustrie company. It was one of the major advances in polymer history at that time. The polyurethane foams (PUF) were firstly produced mixing water in the polyol structure, then, PUFs were soon introduced into the aeronautical industry (SZYCHER, 2013).

Polyurethanes are generally products of the chemical reaction between isocyanate and polyol which ends up in repeated groups of urethane chains. Generally, ester, ether and aromatic rings are also present along the backbone of the urethane bonds (SHARMIN; ZAFAR, 2012). Polyols are reactive substances, generally liquid, with a plurality of hydroxyl groups. They are the main responsible for most of the different bonds, changing significantly the PU properties. Polyurethanes generally have polyester or polyether based polyols (ASHIDA, 2007).

PUFs are produced by the reactions between isocyanate, polyol and water. The reaction between isocyanate and polyol produces urethane bonds while the reaction between isocyanate and water produces carbon dioxide. During the expansion, the carbonic gas ends up trapped inside the polymer, thus, at the end of the polymerization, the material presents gas filled cells, which characterize it as cellular polymer (WANG, 2006).

Bio-based raw materials such as castor, palm, canola and soybean oils have been used as source for polyols. The number of researches surrounding green raw materials for polyurethane have grown in the last years (HADJADJ et al., 2016; YU et al., 2014).

Since polyurethanes are highly flammable and are in touch with people all the time, the concern about its combustion is spreading throughout people (COSTES et al., 2017). Nevertheless, one way to minimize this issue involves the addition of flame retardants inside the polymer.

The tensile properties of foams are rather hard to precisely be obtained, although, some methods and standards have already been used to help the acquisition of these data, such as ASTM D1623 (ASTM, 2017) and the methods proposed by Viana and Carlsson (2002) and Tita et al. (2012). Therefore, this paper seeks the investigation around the tensile properties of the PUF when doped with different mass fractions of ATH as flame retardant. The application for these materials is the manufacture of ceiling plates for general usage in buildings. The PUF used in this work is obtained from a blend of vegetable oils. Mechanical properties were obtained by the tensile test which were done according to previous researches and standards.

MATERIALS AND METHODS

Kehl Company kindly offered the isocyanate (KT1106) and polyol (KT1106-R) used as the PUF raw materials. The ATH, which was used as flame retardant, was generously offered by Alcoa Corporation.

PUF specimens were made by mixing isocyanate and polyol (Fig. 1a) at 3000 RPM, and the proportion of each reagent was 1.2:1, respectively. When ATH was added, it was firstly placed within the polyol and homogenized, then, isocyanate was poured into the mixture and homogenized again. The density value chosen was 180 ± 5 kg/m³, which was the one that presented an easily seen pellicle of dense PU above the entire plate. The mass fraction of ATH in each

sample was proportional to the mass of PUF. The total sample mass and density was increased as more ATH was added into the mixture. However, the polymer mass amount in the material remained the same.

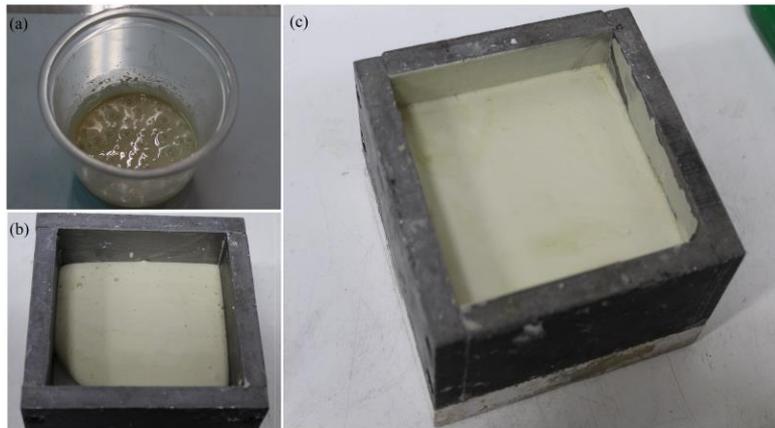


Figure 1 – (a) Reagents mixture; (b) mixture inside the mold; (c) PUF plate after expansion

The mass fractions of ATH added into the mixture were 0% (pure PUF); 10% (PUF+10%ATH); 20% (PUF+20%ATH); and 30% (PUF+30%ATH). Therefore, since the mass of each reagents was 24 g of isocyanate and 20 g of polyol, the ATH masses were 4,4 g (10%); 8,8 g (20%); and 13,2 g (30%).

A mold with specific dimensions was manufactured to produce plates of PUF with the desired volume. After the insertion of PUF (Fig. 1b), the molds were closed for two hours at room temperature until the PUF full expansion, then, the mold was opened (Fig. 1c) and the plate extracted. Finally, the plates were cut and milled in order to obtain specimens with dimensions (in mm) according to Fig. 2a with its thickness being 20 mm.

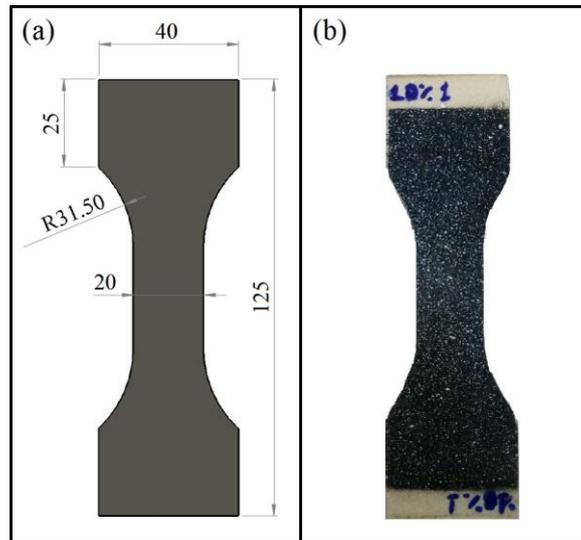


Figure 2 – (a) Tensile specimen dimensions; (b) Specimens after machining

Three specimens of each material were tested. The testing speed was set to 0.5 mm/min because the material fracture was expected to be fragile, once ATH and pores act as stress concentrator inside the polymer. Digital image correlation (DIC), aided by the software GOM Correlate, was used in order to obtain the strain properties of the foams. Therefore, the specimens were painted (Fig 2b), as required by the software, in order to achieve the best response as possible.

RESULTS AND DISCUSSIONS

The tensile data are given as mean stress-strain curves for Pure PUF, PUF+10%ATH, PUF+20%ATH, and PUF+30%ATH. Fig. 3 exhibits the mean stress-strain curve obtained for the pure PUF.

Fig 3 shows easy seen stress concentrators in the whole central specimen area, which implies in a more distributed elongation, moreover, there are explicit elastic and plastic regions, furthermore, the standard deviation shown by the three specimens are pretty small in the elastic region, implying in a well distributed stiffness along the PUF plate.

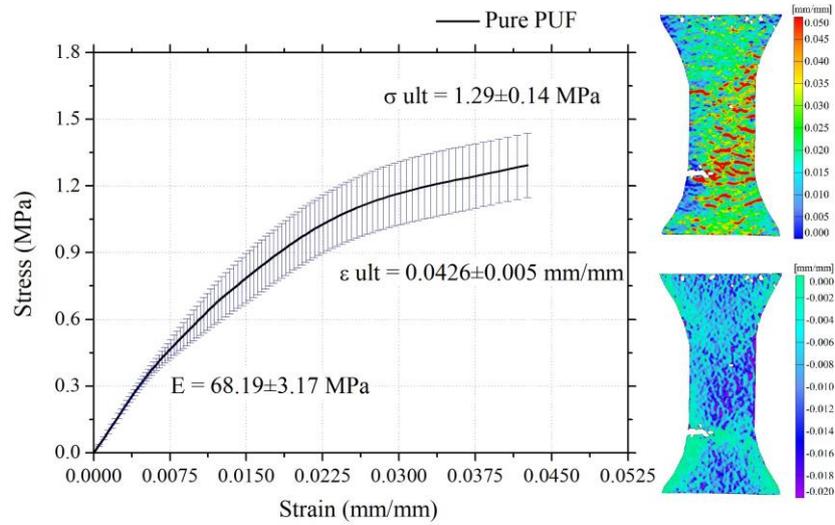


Figure 3 – Pure PUF mean stress-strain curve with DIC at the fracture time

The doped specimen shown in Fig. 4 presents local and longer stress concentrator areas and its rupture was given exactly in the center. The Young modulus (E) is a little higher than the one for the pure PUF, furthermore, this plate showed higher standard deviation values than the pure PUF plate in the elastic zone. It may be explained by the ATH non-linear distribution over the plate, which made some specimens tougher than others. The ultimate strength (σ_u) and strain (ϵ_u) were smaller than the pure porous polymer, although, the σ_u values may be considered equivalent if the deviation is observed, since σ_u is 1.29 ± 0.14 MPa and 1.06 ± 0.11 MPa for the pure PUF and PUF+10%ATH respectively. The smaller ϵ_u data are easily explained by the ATH presence harming the specimen elongation.

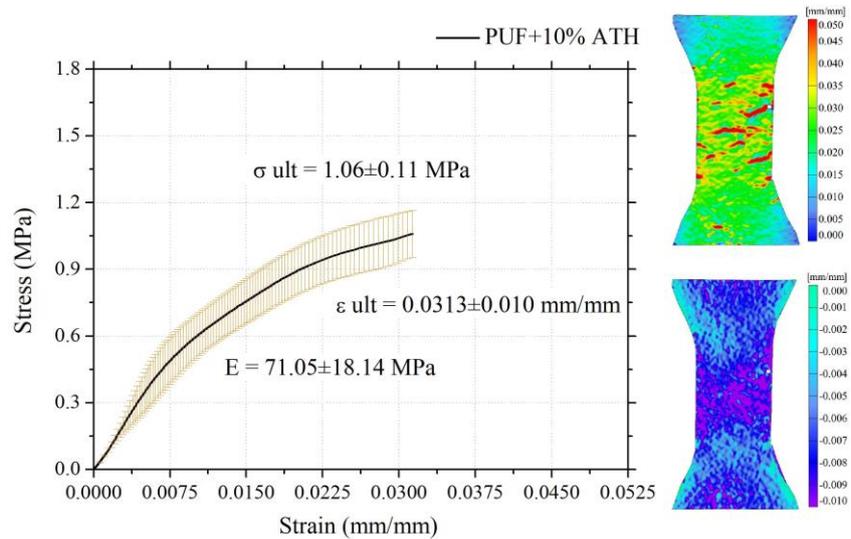


Figure 4 – PUF+10%ATH mean stress-strain curve with DIC at the fracture time

Fig. 5 and Fig. 6 show the stress-strain curve for PUF+20%ATH and PUF+30%ATH respectively. The PUF+20%ATH showed an enhancement in stiffness with a little loss in σ_u compared to the pure PUF, actually, its ultimate strength (1.12 ± 0.06 MPa) can be said to be the same as the pure PUF's (1.29 ± 0.14 MPa) if the standard deviation is considered. The ϵ_u suffered a decrement if compared to both materials previously discussed. It is also explained by the higher presence of ATH inside its foam's structure. The appearance of non-centralized stress concentrators, shown by the strain field, indicate a possible higher concentration of ATH in specific regions of the specimen, which may harm the maximum strain that this material could achieve.

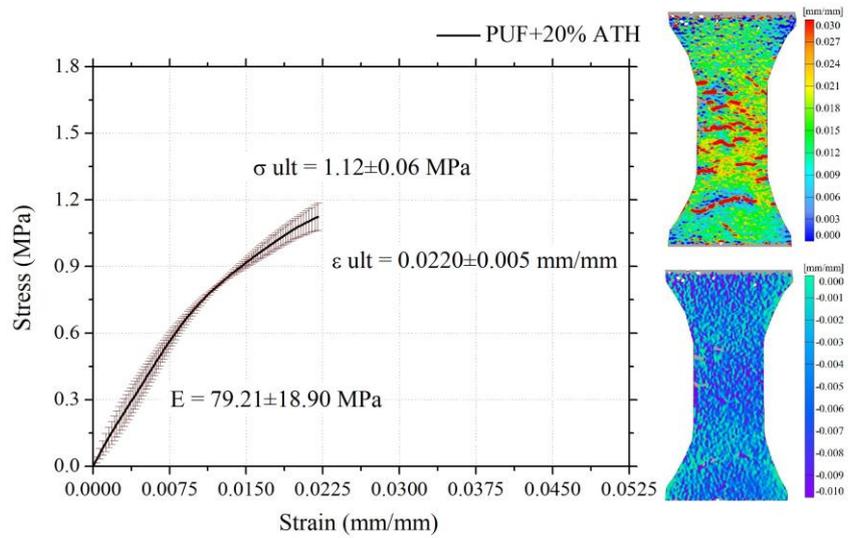


Figure 5 – PUF+20%ATH mean stress-strain curve with DIC at the fracture time

The most doped tested material presented mechanical properties similar to the 20% doped one, with rather similar E , σ_{ult} and ϵ_{ult} values. Moreover, the stress concentrators shown by PUF+30%ATH specimens had a better distribution along the sample bodies, as shown by the DIC in Fig. 6, than the presented by the PUF+20%ATH ones. It is a possible explanation of the reason why the ϵ_{ult} shown by the PUF+30%ATH (0.0237 mm/mm) is slightly higher than the one shown by the PUF+20%ATH (0.0220 mm/mm) because the first, containing higher amount of ATH, has a more distributed strain field than the second.

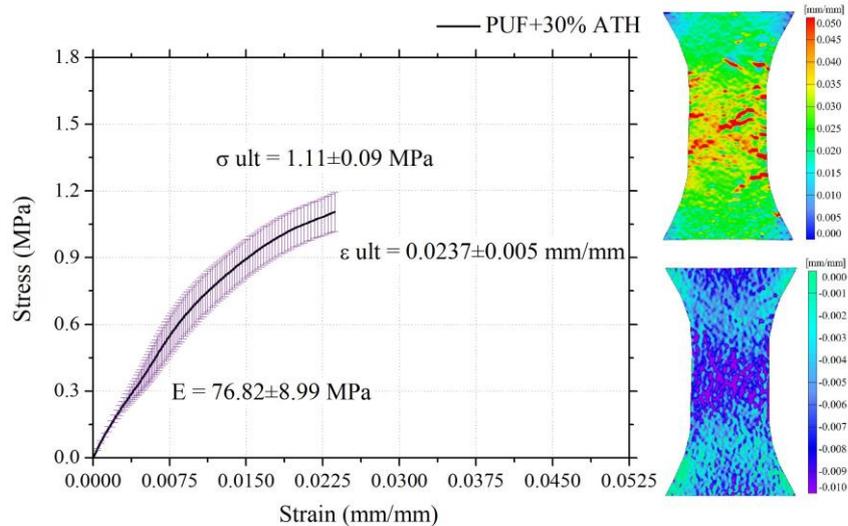


Figure 6 – PUF+30%ATH mean stress-strain curve with DIC at the fracture time

The mean values obtained for each material are shown in Tab. 1 in order to facilitate the comparison and show the behavior of the material as more ATH was added into its structure.

Table 1 – Summarized mean PUF tensile results

Material	E (MPa)	σ_{ult} (MPa)	ϵ_{ult} (mm/mm)
Pure PUF	68.19 ± 3.17	1.29 ± 0.14	0.0426 ± 0.005
PUF+10%ATH	71.05 ± 18.14	1.06 ± 0.11	0.0313 ± 0.0010
PUF+20%ATH	79.21 ± 18.90	1.12 ± 0.06	0.0220 ± 0.005
PUF+30%ATH	76.82 ± 8.99	1.11 ± 0.09	0.0237 ± 0.005

As shown in Tab. 1, the σu values had less considerable changes with the ATH addition. The stiffness and strain of the PUF+20%ATH and PUF+30%ATH are basically the same and the PUF+10%ATH showed intermediate values for both properties, while the pure PUF showed the smallest elastic modulus and the highest ultimate strain.

CONCLUSIONS

The addition of ATH inside the PUF structure should enhance its flammability, although, since polymeric foams mechanical properties are quite hard to predict, this paper investigated the changes in tensile properties that ATH would cause in a polyurethane foam. The results showed an enhancing elastic modulus and a decreasing strain at ATH doping percentages over 20%. Furthermore, no stiffness variations were observed regarding the less doped samples. The only value that showed considerable decrements, with the ATH addition, was the ultimate strain. Therefore, the 20% over doped PUF showed out as more rigid materials with small differences in σu compared to the pure PUF. Consequently, ATH doped vegetable-based PUFs have potential to be inserted in applications where materials with better flammability responses are required with no concerning regarding their tensile mechanical structure.

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