Experimental Characterization of the Mechanical Properties of PETG and PA6 Thermoplastics

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Abstract

Simple thermoplastic materials and polymer matrix composites have been used in this work. Polyethylene terephthalate glycol (PETG) plates, a copolymer of CHDM- cyclohexane dimethanol modified polyethylene terephthalate [56], were used, which is an amorphous and therefore transparent material, which was available in plates or extruded sheets in a wide range of thicknesses. The PETG sheets are of commercial type and their production process is carried out by the company that supplied them. Also part of the study is polyamide 6.6 with 30% short glass fibre content obtained by injection moulding and compression moulding, which was supplied by the ESIS TC4, for the first Round Robin of short glass fibre composite materials.

Keywords: Mechanical properties, Thermoplastics, PETG, PA6, Fiber glass

1. Introduction

Glycol-modified PET copolyester is called PETG. PETG, introduced in 1972 by
Eastman, is a copolyester produced by the reaction of 1,4-cyclohexylene glycol with a mixture of isophthalic and terephthalic acids. When the three structural units react, the structural regularity is lost and a thermoplastic polymer is obtained (1). The structural units of PETG: 1,4-cyclohexandimethyloxide, isophthalate acid and isophthalic acid, are represented in Figure 1, (a), (b) and (c) respectively.

![Figure 1. Structure of PETG. (a) 1,4-cyclohexandimethyloxide, (b) Isothalate acid, (c) Isophthalic acid.](image)

PETG is a very transparent material, the surface of the plates tested is very shiny, with lower roughness than glass (2). It has been observed in tensile and bending tests that it is a very ductile material and has a high resistance to impact (3). These materials have several important characteristics to achieve the objectives of this research.

- The two simple materials (PETG and PA6) have different phases and their optical, mechanical and chemical properties are different.
- The procurement processes are different in each case. Since PETG plates are extruded, polyamide 6.6 is obtained by two different forms of molding, (injection molding and compression molding). Therefore, there is variety in the characteristics of simple and compound polymers and in the processes of obtaining each material (3).
- The materials have been the subject of previous studies and this work allows us to advance and deepen the relationships between process, structure and properties in each case.

Resistance to chemical attack is generally high, as they are resistant to oils, gasoline, glycerin and ethanol (4). On the other hand, its chemical resistance to ammonia, acetone and toluene is very poor. The material begins to degrade at around 290°C, which does not pose any potential danger of possible poisoning. The recommended working temperature should not exceed 70 degrees to avoid any kind of problem. Some of the applications of this polymer are: Orthopaedic parts and components, Bus stop shelters, Lighting covers for danger zones, Advertising panels, Protective screens for machines, Packaging for medical utensils, Displays and signage for external use, Equipment for refrigerators and chambers, Helmets for cyclists, Food packaging.

2. Methodology

The study of the properties of materials and the understanding of their structural response, require the prior characterization of their mechanical properties, so that an initial knowledge of them can be obtained and it can be a starting point to model
and predict their behavior, or simply to later compare the changes that have taken place in the material. In this sense, it is proposed to initiate, establishing the rigidity of the test tubes of this investigation (5). For this purpose, the response of the materials is studied using the test geometries, samples and production processes described below (6). As stated above, one of the materials under study is polyethylene terephthalate glycol PETG (7), which was supplied by NUDEC S.A. in the form of extruded rectangular plates (290 x 200 mm) and with thicknesses of 1, 2, 3, 4, 5, 8, 10. mm. (See figure 2).

Figure 2. PETG plate supplied by Nucdec S.A.

An extract of the properties that characterize Eastman's PETG and PA6 manufactured by Nudc is shown in (8, pages 66-72).

**Differential Scanning Calorimetry of PETG and PA6.**

In order to observe the thermal behaviour of PETG and to verify the characteristics given by the manufacturer. As well as erasing the thermal history of the extruded material with the first heating and then performing the second heating over a wide temperature range to observe whether its behaviour is completely amorphous or whether it initiates crystallization processes in the thermal evolution of the test, the differential scanning calorimetry test was performed. The results obtained from the test are shown in Figure 3. Differential Scanning Calorimetry (DSC) tests were performed on a Pyris 1 PE (Perkin Elmer) equipment, recording a complete heating cycle of the samples. In Figure 3, the glass transition temperature peak (Tg) of PETG can be seen. The PETG is a modified PET which presents changes of optical and mechanical type, and goes from a semicrystalline material to being an amorphous material, inducing an improvement in its appearance and obtaining a totally transparent material, with a Tg around 80ºC which makes this material work below the Tg, that is to say it behaves like a solid with characteristics similar to glass (hard and fragile) due to its amorphous condition.
A DSC thermal analysis is then performed on single and reinforced polyamide 6 in order to observe the behaviour of the polyamide in its simple state and the possible changes in its structure with the introduction of the glass fibre. And at the same time, assess the influence that the latter can exert on the crystallinity of the composite material, Figure 4.

**Obtaining the specimens**

The PETG, Polyamide 6 and Polyamide 6.6 specimens required for the study were obtained. The orientation of materials loaded in polymers is key to their final characteristics, because depending on where the injection point is located on the plate, the fibres will have a preferential orientation if the specimens are cut in one direction or another. See figure 5.

PETG and polyamide 6 specimens are shown in Figures 6 and 7.
3. Results and discussion

The DMTA technique (dynamic mechanical thermal analyzer) uses an alternating deformation (dynamic tests) to study the viscoelastic behavior. DMTA tests allow us to know the offset sinusoidal stress that causes an equally sinusoidal deformation to which the specimen is subjected as a function of time or temperature. The experimental unit compares the stress and deformation and finds the storage components ($E'$), losses ($E''$) and the tan δ. $E'$ represents the energy that the material stores as a consequence of the deformation, in a reversible and recoverable way. "$E''$" represents the energy that the material irreversibly dissipates and so δ (called loss factor) corresponds to the quotient between $E''$ and $E'$. In the texts, the complex dynamic modulus obtained in tensile or bending tests is usually referred to by $E^*$ and the complex dynamic modulus obtained in shear tests by $G^*$. To study the transitions associated with the different types of movements, sweeps can be made at different frequencies keeping the temperature constant and obtaining the so-called frequency spectrum (9). Usually you work between frequencies between 0.1 and 1 kHz. Sweeps can also be made by varying the temperature at a constant frequency and it is in this case that we can speak strictly of dynamic mechanical thermal analysis (DMTA) tests. In Figures 8 and 9, the results obtained by DMTA are shown for PETG and single and reinforced polyamide 6, the specimens were ordered in the single cantilever mode, at a frequency of 1 Hz and a temperature ramp of 2°C/min.

Figure 8 shows the evolution of the PETG storage module. It presents a stable behavior until reaching temperatures close to its glass transition temperature, around 80 degrees, temperature in which there is a change in the behavior of the storage module and a significant increase in the loss module which is associated with the viscous behavior of the material (10). In short, it is established that the PETG storage module, which is associated with the elastic behavior, behaves constantly up to temperatures close to Tg. This value, according to Figure 8, is approximately 1.9 MPa.

![Figure 8. Bending DMTA test on PETG specimens.](image)

![Figure 9. Flexure DMTA test on single PA6, PA6+22.5%FV and PA6+45%FV specimens.](image)
From Figure 9, some important considerations can be made regarding the behavior of simple and reinforced polyamide. The evolution of the storage module value as a function of the fibre content is directly proportional. Storage module increments based on fibre content clearly comply with the law of blends. The behavior of the composite material is governed by the polymer matrix (polyamide 6) as the temperature increases, since the storage module varies significantly for both single and reinforced polyamide 6. It could be expected that the higher the fiber content, the more stable the behavior of the composite material at the value of the modulus in the elastic zone, however, its evolution is determined by the polymer base. Finally, the mixture approaches degradation at around 220ºC when the test is stopped. Tensile tests were carried out on all materials at a separation rate of 5 mm/min and ambient temperature. From each test the stress-strain curves were obtained (σ-ε) and the elastic modulus (E) and yield stress (σy) were determined as characteristic parameters. These tests allow a preliminary mechanical characterization of the simple and composite materials, studied in the low speed range of solicitation. Figure 10 shows, as an example, representative curves of PETG specimen tests.

![Stress-strain curves of tests on PETG samples.](image1)

![Strain-strain curves of tensile tests on single and reinforced PA6.](image2)

To analyze the final properties of the material, simple PA6 and the composite materials formed by the PA6 matrix reinforced with fiberglass in percentages of 22.5% and 45% were subjected to traction. During the test, two types of load are distinguished, depending on whether the load is applied: a) longitudinal, on the axis where there is a greater degree of orientation of the fibre or b) transversal, perpendicular to the axis where there is a greater degree of orientation of the fibre. Figure 11 shows the change in the recorded stress-strain curves of the tests. Two factors influencing the material behaviour can be seen in these curves. As the fiber content increases, the behavior of the material changes. Moving from exhibiting the behavior of a ductile material to a rigid material, notably modifying the stress limit to creep and breakage. The stress on the longitudinal axis of the orientation of the fibres gives values of maximum stress higher than those obtained in the transverse orientation.
Fiberglass is characterized by its high tensile strength (3450 MPa) and high modulus of elasticity (70 GPa), and as shown in the above graph, it influences the final properties of the material, however, we must quantify the increase in the values of modulus of elasticity and stress limits as a function of the fiber content. (See figures 12 and 13). From figures 11, 12 and 13, some important considerations can be made. In the cases seen, with loads applied, either in the direction of the fibre orientation axis or perpendicular to it, it has been demonstrated that the capacity of the composite material to support loads is increased by the addition of reinforcement; so that the greater the volumetric fraction of the fibre, the greater the elastic modulus and the greater the yield stress. Furthermore, it is clear that the load-bearing capacity of a composite material depends first of all on the elastic modulus of the matrix and the volumetric proportion of the reinforcement. Figure 14 clearly shows the evolution of the elastic modulus as a function of the orientation of the fibre. In which it is established that the angle formed between the applied tension and the orientation of the fiber has a marked incidence in the mechanical response of the properties of the material, making it more resistant and tenacious in the direction of the orientation of the fiber. However, it is important to note that the greater orientation of the fiber unidirectionally makes the highly anisotropic composite material in this case. In the case of PA6 without fibre, a slight increase in the elastic modulus (E) is also observed due to the orientation induced by the injection flow which tends to orient the polymer chains.
4. Conclusions

The modulus of elasticity values obtained at low velocity of stress compared to those obtained at relatively high velocity of stress in simple materials are consistent with the theory of increasing strain rate in a polymer. However, in glass fibre reinforced materials the increase in the modulus of elasticity is less because the reinforcement restricts the movement of the matrix, thus reducing the effects of the viscous component of the material. The ratio of blends in all tests performed throughout the research is met for single PA6 and with short fiberglass content at 22.5% and 45%. The elastic modulus and tensile strength increase with the fiberglass content.

References


Experimental characterization of the mechanical properties


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