Alternative dynamic torsion test to evaluate the elastic modulus of polymers

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Abstract

This work presents an alternative for the determination of the torsion modulus, G, of polymers. These materials may be subjected to shear stresses in some structural applications; thereby, the knowledge of G is of great interest. For this purpose, a mechanical system featuring a simplified torsion pendulum version and a rotational motion sensor (RMS) coupled to it was used to establish an angular position as a function of time. The applied technique is considered non-destructive and makes it possible to obtain G without the Poisson’s ratio through an equation derived from mechanical spectroscopy and material strength. The main goal is to present and validate the employment of this method for polymers. Therefore, circular cross-sectional samples of extruded polytetrafluoroethylene (PTFE) were subjected to torsional stresses, in which a physical and quantitative explanation is given for the frequency and G curves as a function of the prefixed rotational inertia (I), length (L), and diameter (d). Differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) were also made to ensure the reliability of data. It is possible to establish an L/d ratio, which explains why G converges to a single value when the sample dimensions are different from each other. It was found that G is approximately 350 MPa for an L/d ratio equivalent to 10.64. Such a value is within limits found in the literature, opening the possibility of assessing other polymers.

1. Introduction

Polymeric materials are commonly used in engineering applications loaded in shear [1]. The data about the torsion or shear modulus (G) is of great importance to design polymer products and whether they have the strength required to avoid fatigue or mechanical failure [2]. Dynamic torsional tests are generally performed to characterize the shear behavior and the viscoelastic properties of polymers [3, 4]. The viscoelastic behavior of such materials can be measured by the forced vibrations method in the dynamic mechanical analysis (DMA) or systems of free oscillation with a torsion pendulum. Free oscillation pendulum methods have the advantage of simplicity and secure handling when compared to other dynamic measures [5]. In this work, we make use of a torsion pendulum designed and built into our laboratory for evaluation of the torsion modulus, G, of circular cross-sectional samples. This mechanical system has been utilized for assessing biomaterials (titanium alloys) [6] and was now applied to polymeric materials. In particular, polytetrafluoroethylene (PTFE), a well-known fluoropolymer with outstanding physical and chemical properties, has been chosen to validate the use of this technique. Owing to its chemical inertness, thermal stability, non-stick property, and robust mechanical strength [7, 8], this polymer has been employed in different fields, from more straightforward applications, such as seals [9], to more complex structural applications in the aerospace [10], nuclear [11–13] and biomedical industries [14–16]. In such situations, the material may be subject to shear stresses, thereby it being necessary to know G to predict its mechanical behavior. The measure consists of subjected samples with different diameters of PTFE to torsional stresses varying their effective length. The main goal is to validate the equation for the
calculation of G, establishing an L/d ratio that provides the same G values for different sample diameters. This equation is derived from mechanical spectroscopy studies [17] and the theory of material strength [18]. When compared to other dynamic methods, the advantage of this technique is that it is not necessary to know the Poisson’s ratio to obtain G. The system is composed of a torsion pendulum and a rotational motion sensor (RMS) coupled to it and is capable of recording the angular position as a function of time. The measure is considered dynamic and non-destructive, maintaining both physical and chemical properties of the sample after the test, therefore of great practical interest.

There are several torsion pendulum designs found in the literature [19–22] since the first reported by Kuhn and Künzle [23]. Each has its advantages and disadvantages while being able to measure different types of samples. For example, Li et al [24] used a torsion pendulum with a vacuum chamber and temperature control to measure G in a cylindrical sample of Molybdenum (Mo) coated with TiN / Ti(C, N) multilayers. In another work, Liu et al [25] developed a modified torsion pendulum apparatus to measure single filaments with a micro-sized diameter. At the same time, Yu et al [26] measured G of adhesives also in a modified torsion pendulum. Although these works have made a significant contribution, they present complex systems and measures for a specific group of samples. Alternatively, the torsion pendulum shown in this work is intended to be a low-cost system that is easy to use and has the potential to measure different classes and sample sizes. This system has a simplified setup without a vacuum chamber or temperature control. The configuration used herein is different from the Kê-type inverted torsion pendulum [27] used in other studies [28, 29], making the system mechanically more stable without tensile stress acting on the specimen. The sample relaxation curve is measured automatically by the RMS, eliminating the laser beam and its detection by photocells, as in the case of traditional systems [30, 31]. The results of validating the G equation for PTFE not only provides a way to investigate its shear behavior but also opens up the possibility of assessing other polymer samples using this technique.

2. Materials and methods

2.1. Experimental apparatus

The torsion pendulum used in the measurements is shown in figure 1. The pendulum is fixed to a rotational platform in a rigid structure and oscillated concentric to its axis passing through a bearing (figure 1(a)). The base moves by four screws on the structure (figures 1(a), (b)), permitting modification of the effective length. Figure 1(b) shows a schematic drawing of the elements composing the measurement system. Electromagnets are attached to the upper structure and placed in a proper position to torsion the pendulum (figures 1(b), (c)). A rotational motion sensor (RMS-PASCO CI6538, 1°, and 0.25° of resolution, ±0.09 degrees accuracy) is used to record the relaxation curve utilizing an inextensible wire attached to a pendulum shaft rod (figure 1(d)). While one end of the sample is attached to a static three jaw-chuck, the other is fixed to the torsion pendulum by screws and is free to oscillate (figure 1(e)).

2.2. Materials

Cylindrical bars extruded of pure, commercial PTFE supplied by DuPont with dimensions of 8.00 × 500 mm, 6.50 × 500 mm, and 5.00 × 500 mm were subjected to torsion as received. The samples were fixed to the pendulum with effective lengths of L₁ = 120 mm, L₂ = 104 mm, L₃ = 85.15 mm, L₄ = 69.20 mm and L₅ = 53.20 mm. For each effective length, we used four rotational inertias (I) for torsion and free-sample oscillation. Three measurements were carried out for each set of L, R, and I fixed values. We established a torsion angle of 8 degrees for all measurements, enough to generate the relaxation curve and extract the data from it. For differential scanning calorimetry (DSC) analysis, samples were cut from the extruded bars with a scalpel and cleaned in an ultrasonic bath for 600 s at a power of 50 W, first in isopropyl alcohol and then in deionized water. This measure is carried out to estimate de crystalline percentage as well as investigate the presence of impurities from the extrusion process. Density measures are conducted to assess the integrity of the PTFE sample, ensuring reliability for the mechanical measurement of G.

PTFE is a thermoplastic polymer considered as an engineering plastic owing to its versatility and high performance [32]. Its polymer chain is composed of multiple carbon-fluorine bonds, wherein the protection of the fluorine atoms surrounding the carbon chain is responsible for the properties of a low coefficient of friction, thermal stability, and chemical inertness [7]. PTFE is a semi-crystalline material that exhibits four pressure- and temperature-generated phase transitions [33, 34]. When undergoes to sinusoidal stress at different temperatures, PTFE presents three relaxation peaks: β, as the first-order peak containing phase transitions II (triclinic), IV (hexagonal), and I (pseudo-hexagonal), and the second-order peaks, α, and γ [35]. Most fluoropolymers, except for PTFE, are processed by traditional melt-processing routes, such as injection molding, blow and screw extrusion [36, 37]. However, PTFE parts are not manufactured by these processing routes due to its high melt viscosity (10⁶ to 10¹⁵ Pa s⁻¹ at 360 °C) [38], with the ram extrusion process being one
of the required processing technique [39–42]. Mechanical properties of PTFE parts are much dependent on the degree of crystallinity [33, 43, 44], which may be altered by changing the processing route (thermal history) during its production [45, 46]. Thus, there must be an accurate estimation of the crystalline proportion of the material subject to mechanical tests.

2.3. Methods

2.3.1. Determining $G$

The equation for the calculation of $G$ was developed by Pintão et al [6] and first validated with commercially pure titanium CP-Ti. Mechanical spectroscopy [17], i.e., the torsion motion of the pendulum, is defined by the next equation:

$$\ddot{\theta} + G_0 (1 + i \omega \theta) \theta = 0$$

(1)

Where $I$, $G_0$ and $i$ are the rotational inertia, the torsion constant of the sample attached to the pendulum, and the imaginary number, respectively. On these terms, the air resistance and the friction of the bearings from the RMS and rotational system are negligible as they don’t significantly impact the movement [6, 47]. The solution from equation (1) has the appearance of $\theta = \theta_0 e^{-i \omega \theta}$, where $\omega^* = \omega_0 \left(1 + i \frac{\delta}{2\pi}\right)$ and $\omega \theta = \frac{\delta}{\pi}$. Then, $G_0$ can be expressed as

$$G_0 = I \omega^2 \left(1 - \frac{\delta^2}{4\pi^2}\right)$$

(2)

From equation (2) one can see that the rotational inertia of the pendulum, $I$, the angular velocity, $\omega_0$, and the expression $\delta/2\pi$ can be used to compute $G_0$. Shifting the masses position relative to the torsion pendulum’s rod causes $I$ to change, which consequently changes $\omega_0$. Therefore, obtaining $G_0$ with respect to the oscillation system frequency makes the measurement be considered as a dynamic measurement. Using small oscillation angles, in which the measuring system has a linear behaviour, $G_0$ can be determined:

$$G_0 = \frac{\text{applied torque}}{\theta}$$

(3)
The aim of the study is to determine the torsion modulus, $G$, of a solid uniform circular cross-sectional sample with length, $L$. Therefore, considering the mechanics of solids and the strength of the material [18, 48, 49], one can calculate the sample resistive torque when it undergoes torsion through an applied torque via pendulum (see figure 1). The following equation calculates this torque:

$$ \text{resistive torque} = T_R = \frac{GL}{L} \theta $$

(4)

Where $J_c$ is the polar moment of inertia for a circular cross-section given by:

$$ J_c = \frac{\pi d^4}{32} $$

(5)

When the applied torque is equal to the resistive torque, the system is said to be in static equilibrium, and thus equations (3) and (4) can be written as:

$$ G_0 = \frac{GL}{L} \theta $$

(6)

The magnitude of $G$ is, therefore, obtained by substituting equation (2) into equation (6) and, as a result:

$$ G = \frac{L}{J_c} \frac{G_0}{J_c} = \frac{L}{J_c} \frac{G_0}{J_c} \left( 1 - \frac{\delta^2}{4\pi^2} \right) $$

$$ = \frac{32}{\pi d^3} \frac{G_0}{J_c} \left( 1 - \frac{\delta^2}{4\pi^2} \right) $$

(7)

Taking $\omega^2 = \omega_0 \left( 1 + \frac{i \delta}{2\pi} \right)$ in the solution, $\theta = \theta_0 e^{-i\omega_0 t}$, relative to the differential equation that describes the motion of the free pendulum [17]:

$$ \theta = \theta_0 e^{-i\omega_0 t} \left( 1 + \frac{i \delta}{2\pi} \right) $$

$$ = \theta_0 e^{-i\omega_0 t} \left( \cos \omega_0 t + i \sin \omega_0 t \right) $$

Considering the real part from equation (8), the following equation is given:

$$ \theta = \theta_0 e^{-\frac{\delta}{2\pi} t} \cos \omega_0 t $$

(9)

For a given $L$, $d$, and $I$, the physical parameters $\delta$ and $\omega_0$ are taken from the experimental curve $\theta$ versus $t$ using the RMS software and the Pasco interface. By means of a fit function similar to equation (9) from Origin 7.0 software environment, selecting a 'waveform' function such as the following:

$$ \varphi = A \exp \left( -t/t_0 \right) \sin \left( \frac{\pi W t}{W} - \frac{\pi t_0}{W} \right) $$

(10)

Comparing the later equation with equation (9) makes possible to compute the parameters $t_0$ and $W$, by fitting equation (10) to the experimental values, and then obtain the parameters $\delta$ and $\omega_0$:

$$ T = \frac{2\pi}{W} = 2W \quad \text{and} \quad \delta = \frac{2W}{t_0} $$

(11)

Finally, knowing $W$ and $t_0$, and using equation (11), the parameters $\delta$ and $\omega_0$ can be calculated, and the equation (7) use to determine $G$.

The method for measuring $G$ consists of applying a torsion of 8 degrees at the end of the sample attached to the pendulum. In contrast, the other is attached to a static base, and for each fixed value, the process is repeated. Employing an RMS attached to a pendulum shaft rod (figure 1(d)), the angular position of the pendulum ($\theta$) is recorded as a function of time ($t$). For a solid circular cross-sectional sample with diameter $d$ and effective length $L$, the equation for $G$ is described for equation (7).
\[
\tau_{\text{MAX}} = \frac{T_R d}{2L} \quad (12)
\]
\[
\gamma_{\text{MAX}} = \frac{d\theta}{2L} \quad (13)
\]

Therefore, the value of \( G \) can also be expressed as:

\[
G = \frac{\tau_{\text{MAX}}}{\gamma_{\text{MAX}}} = \frac{T_R L}{I_0 \theta} \quad (14)
\]

2.3.2. Determining rotational inertia

Masses are attached to the upper torsion pendulum rod, the same configuration relative to the rotational axis (figure 1). By changing the relative position, the rotational inertia is also altered. Four configurations of rotational inertia were defined and used to calculate \( G \) as well as verify the validity of equation (7). A few different techniques to determine the rotational inertia of a body can be found in the literature [47, 50, 51]. However, our measurements were based on independent methods developed in the laboratory [52]. Thus, the resulting torque (\( \tau \)) responsible for the movement of the pendulum is given by:

\[
\tau = \tau_T - \tau_{\text{ATR}} = I \alpha_0 \quad (15)
\]

In equation (15), \( \tau_{\text{ATR}} \), \( I \) and \( \alpha_0 \) are the rotating shaft torque friction force, the rotational inertia and the pendulum’s angular acceleration, respectively. A pulley with a radius \( r = (18.75 \pm 0.03) \times 10^{-3} \text{ m} \) is concentrically fixed to the pendulum shaft with a wire wrapped around it. Suspending a mass, \( m \), connected to the other end of the wire, the torque of the traction force is \( \tau_T = m(g - \alpha_0 r) r \) with \( g = (9.79 \pm 0.01) \text{ m s}^{-2} \). Equation (15) indicates that the graph, \( \tau_T \) versus \( \alpha_0 \), is linear. Therefore, the slope, \( \Delta \tau_T / \Delta \alpha_0 \), is equal to \( I \). We used 13 values of \( m \), starting with 25 g and increasing 20 g until reaching 265 g, repeating the process for each configuration.

2.3.3. Dynamic mechanical analysis (DMA)

The viscoelastic behavior of PTFE can also be assessed by forced vibration methods, such as the dynamic mechanical analysis (DMA). The technique consists of applying oscillating stress (sinusoidal) to the sample at a given frequency and creating a stress-strain curve with an angle defining the lag between them, known as a phased angle [5]. The elastic behavior is related to the storage modulus (\( E' \)), which corresponds to the mechanical energy stored by the material in a load application cycle, whereas the viscous part is given by the loss modulus (\( E'' \)), representing the damping. The ratio between \( E' \) and \( E'' \) corresponds to the property known as internal friction (\( \tan \delta \)), mainly related to intermolecular friction [5, 47]. The elastic modulus from the torsion pendulum is computed over the total relaxation curve, while \( E' \) originates from almost one point [53]. Furthermore, the elastic modulus from the pendulum presented herein differs from the DMA, as it considers not only the internal friction but also the friction from the ball bearing and the air. It is worth mentioning that in order to converge the elastic modulus that comes from the DMA into \( G \) would require the Poisson’s ratio, which is not necessary when using the torsion pendulum technique presented.

The measure was performed on the Metravib DMA 25 equipment in tension-compression mode. Extruded circular samples of PTFE with 8 × 38 mm were used in the tests. Internal friction (\( \tan \delta \)) data were collected from −115 °C to 300 °C using liquid nitrogen (LN2) with a heating rate of 2 °C min \(^{-1} \) and frequencies of 1, 10, and 40 Hz. These internal friction data were compared to the total friction results from the pendulum in order to verify if they are within the same magnitude, ensuring that the parameters used in equation (7) are reliable.

2.3.4. Density

PTFE density was evaluated with the Archimedes principle [54] according to the following equation:

\[
\rho = \frac{m_s}{(v_f - v_0)} \quad (16)
\]

The method consists of inserting a sample mass \( (m_s) \) into an initial volume of liquid \( (v_0) \) and displacing it to a final volume \( (v_f) \). The difference between them corresponds to the sample volume. Calculating the ratio between sample mass \( (m_s) \) and sample volume, the PTFE density is obtained. Deionized water was utilized as the displaced liquid inside a 10.0 ml graduated glass cylinder. With an analytical balance, the mass of the polymer was measured in an air-conditioned environment at 25 °C. We measured the density of three samples of each extruded bar as described herein and then computed their mean density values. In the literature [7, 8, 55], PTFE density is between 2140.0 and 2200.0 kg m \(^{-3} \), and the calculated values for the extruded PTFE samples should be within this range.
2.3.5. Differential scanning calorimetry (DSC)

Differential scanning calorimetry (DSC) was performed with the Mettler-Toledo model DSC 1 Stare® system. The sample mass was 7.15 mg placed in closed 40 μl crucibles with a hole in the cover, heating from 25 °C to 350 °C at a ratio of 10 °C min⁻¹ with a dry air atmosphere flow at 50 ml min⁻¹. A thermal cycle was conducted, cooling to 30 °C and heating again until 350 °C under the same ratio of 10 °C min⁻¹. The crystalline percentage ($X_c$) of PTFE could be estimated using the heat of fusion ($\Delta H_f$) from DSC measurements, calculating the ratio with a theoretical 100% crystalline sample ($\Delta H_f^{0}$) of 80 J g⁻¹, according to equation (17):

$$X_c = \frac{\Delta H_f^{(sample)}}{\Delta H_f^{0}}$$

The value, $\Delta H_f^{0}$, is taken directly from the works of Rae and Dattelbaum [44] along with Jordan et al [10], who considered the mean values of PTFE $\Delta H_f$, ranging from 57 to 104 J g⁻¹ according to Lehnert [56].

3. Results and discussion

3.1. Density and differential scanning calorimetry (DSC)

The DSC curve as a function of temperature can be seen in figure 2.

At the beginning of the heating of the DSC curve, an endothermic peak is observed at 31.6 °C, corresponding to a hexagonal crystal phase transition to pseudo-hexagonal (phase $\beta$). The melting temperature ($T_m$) is 330.7 °C, specifically very close to literature values [8, 9, 55]. Impurities and metastable phases that could produce additional pre-melt peaks were not seen, ensuring that the sample is free of impurities that could influence the mechanical results. The heat of fusion of the melting event is $\Delta H_f^m = 35.5$ J g⁻¹, provided from the crystal formation during the cooling of the extrusion process with a calculated crystalline percentage ($X_c$) of 44%, based on equation (17). During cooling, the recrystallization event takes place at $T_c = 310.0$ °C. This crystalline percentage influences mechanical properties since the crystalline phase can be considered more rigid than the amorphous phase [10]. Regions of higher crystallinity have more significant interaction between the intermolecular chains. The proximity of the chains affects the Van der Waals bond strength and, consequently, the elastic modulus [55]. The computed values are found in table 1, along with the density calculated as 2156.3 kg m⁻³, which is within the limits of the literature [7, 8, 55].

![Figure 2. Curve experimental (DSC) obtained of extruded PTFE.](image)

| Material (extruded) | Density (kg m⁻³) | $\Delta H_f^m$ (J g⁻¹) | $X_c$ (%) | $T_m$ (°C) | $T_c$ (°C) |
|--------------------|------------------|------------------------|-----------|------------|------------|
| Pure PTFE          | 2156.3 ± 0.1     | 35.5                   | 44 ± 1    | 330.7      | 310.0      |

Table 1. Physical characteristics of pure extruded PTFE obtained from DSC and density measurements.
3.2. The measure of torsion pendulum’s rotational inertia

The results from the torque applied to the torsion pendulum (τ_T) and angular acceleration (α_0) were used to plot figure 3. It is observed that straight lines do not commence from the origin, intercepting the Y-axis at the same point. This point is the linear coefficient, corresponding to the torque of the friction force acting on the rotating shaft, τ_ATR = (11.3 ± 3) 10^−4 kgm^2. As expected, the inertia values are dependent on the distribution of the masses relative to the rotational axis. Thus, higher inertia values were measured for Configurations 1 and 2 and smaller values for Configurations 3 and 4. These values were used to calculate G and assess equation (7).

3.3. Validating the equation for G

3.3.1. Varying effective length with a fixed diameter

The validation of the proposed equation (7) is done using a measure of I (section 2.3.2), independent of that used in the determination of G. This measure is compared to the measure of G. For this, it is verified if the ratio between the values of G or I, measured independently of each other, present the same value. For the sake of brevity, only the results with the 8 mm diameter sample will be displayed (see [57] for all diameters). The results for varying the L of the PTFE sample with a diameter of 8 mm are presented in tables 2–6, which will be discussed in this section as well as sections 3.3.2 and 3.3.3, and were used to build the graphs within these sections. The TR value was calculated using equation (4) with an SMR torsion angle of θ = 8 degrees (0.14 rad), taking into account that the angle on the sample axis corresponds to 0.222 of θ (0.031 rad) [57] and, thus assuming the hypothesis that there is only elastic deformation.

As expected and based on equation (7) the value of G depends on the L of the sample. Besides, a dependence of the frequency values concerning the inertia configuration and L is observed. The results from determining G as a function of L^2 with d = 8 mm are presented in figure 4.

By calculating the ratio between the rotational inertias used and the slope of the straight lines (B) obtained from the linear adjustment of the experimental points, it can be shown that equation (7) describes G for the considered interval of L. If we verify that the values are the same, the experimental curves of figure 4 provide a means to verify the validity of equation (7). Table 7 lists the calculated proportions and percentage deviations.

Figure 3. Four values of I corresponding to the four configurations used. The values were determined through a linear fit with the experimental results.

Table 2. Results - PTFE: d = 8.00 ± 0.05 mm, L = 123.80 ± 0.05 mm, \( I_L = 4.02 \times 10^{-10} \text{m}^2 \).

| Physical quantity | Config. 1       | Config. 2       | Config. 3       | Config. 4       |
|-------------------|-----------------|-----------------|-----------------|-----------------|
| I (kgm^2 × 10^−4) | 526 ± 4         | 398 ± 1         | 207 ± 1         | 98.1 ± 0.5      |
| G (MPa)           | 427 ± 11        | 424 ± 11        | 425 ± 11        | 426 ± 11        |
| TR (Nm×10^−4)     | 4.26            | 4.23            | 4.24            | 4.25            |
| f_0 (Hz)          | 0.818           | 0.937           | 1.30            | 1.89            |
| TR (kgm^2 Hz^2)   | 0.0352          | 0.0349          | 0.0350          | 0.0350          |
| δ                 | 0.274           | 0.281           | 0.281           | 0.269           |
| QT^{-1}           | 0.044           | 0.045           | 0.045           | 0.043           |
between them in absolute values. As can be seen in table 7, the values for the percentage deviations are very small, correctly less than 2%. This shows that the parameter, δ, does not significantly interfere, and the ratio between the values of G reflects the ratio between the values of I. In this case, equation (7) can be employed to determine G. For diameters of 6.5 mm and 5 mm, the percentage deviations were even lower, being less than 1% [57].

3.3.2. Frequency varying effective lengths and fixed diameters
Figure 5 depicts oscillation frequency (f0) as a function of the rotational inertia for the L values used with a fixed diameter, d. For smaller rotational inerties measured, the difficulty in changing the state of motion from the torsion pendulum was low and, consequently, f0 values were higher. This fact can be verified in the results of Table 3.

Table 3. Results - PTFE: d = 8.00 ± 0.05 mm, L2 = 104.00 ± 0.05 mm, Jc = 4.02 × 10^{-10} m^2.

| Physical quantity | Config. 1 | Config. 2 | Config. 3 | Config. 4 |
|-------------------|-----------|-----------|-----------|-----------|
| I (kg m^2 s^{-2}) | 526 ± 4   | 398 ± 1   | 207 ± 1   | 98.1 ± 0.5|
| G (MPa)           | 364 ± 10  | 363 ± 9   | 365 ± 9   | 366 ± 9   |
| Tg (Nm x 10^{-6}) | 4.32      | 4.31      | 4.34      | 4.35      |
| f0 (Hz)           | 0.822     | 0.946     | 1.31      | 1.91      |
| Jf (kg m^2 Hz^2)  | 0.0355    | 0.0356    | 0.0355    | 0.0358    |
| δ                 | 0.271     | 0.263     | 0.262     | 0.261     |
| Qr^{-1}           | 0.043     | 0.042     | 0.042     | 0.042     |

Table 4. Results - PTFE: d = 8.00 ± 0.05 mm, L3 = 85.15 ± 0.05 mm, Jc = 4.02 × 10^{-10} m^2.

| Physical quantity | Config. 1 | Config. 2 | Config. 3 | Config. 4 |
|-------------------|-----------|-----------|-----------|-----------|
| I (kg m^2 s^{-2}) | 526 ± 4   | 398 ± 1   | 207 ± 1   | 98.1 ± 0.5|
| G (MPa)           | 350 ± 9   | 350 ± 9   | 351 ± 9   | 353 ± 9   |
| Tg (Nm x 10^{-6}) | 5.08      | 5.08      | 5.09      | 5.12      |
| f0 (Hz)           | 0.892     | 1.03      | 1.42      | 2.07      |
| Jf (kg m^2 Hz^2)  | 0.0419    | 0.0422    | 0.0417    | 0.0420    |
| δ                 | 0.324     | 0.377     | 0.323     | 0.339     |
| Qr^{-1}           | 0.052     | 0.060     | 0.051     | 0.054     |

Table 5. Results - PTFE: d = 8.00 ± 0.05 mm, L4 = 69.20 ± 0.05 mm, Jc = 4.02 × 10^{-10} m^2.

| Physical quantity | Config. 1 | Config. 2 | Config. 3 | Config. 4 |
|-------------------|-----------|-----------|-----------|-----------|
| I (kg m^2 s^{-2}) | 526 ± 4   | 398 ± 1   | 207 ± 1   | 98.1 ± 0.5|
| G (MPa)           | 342 ± 9   | 341 ± 9   | 341 ± 9   | 340 ± 10  |
| Tg (Nm x 10^{-6}) | 6.11      | 6.09      | 6.09      | 6.07      |
| f0 (Hz)           | 0.979     | 1.12      | 1.56      | 2.26      |
| Jf (kg m^2 Hz^2)  | 0.0504    | 0.0499    | 0.0504    | 0.0501    |
| δ                 | 0.324     | 0.331     | 0.327     | 0.324     |
| Qr^{-1}           | 0.052     | 0.053     | 0.052     | 0.052     |

Table 6. Results - PTFE: d = 8.00 ± 0.05 mm, L5 = 53.20 ± 0.05 mm, Jc = 4.02 × 10^{-10} m^2.

| Physical quantity | Config. 1 | Config. 2 | Config. 3 | Config. 4 |
|-------------------|-----------|-----------|-----------|-----------|
| I (kg m^2 s^{-2}) | 526 ± 4   | 398 ± 1   | 207 ± 1   | 98.1 ± 0.5|
| G (MPa)           | 284 ± 8   | 283 ± 7   | 281 ± 7   | 282 ± 7   |
| Tg (Nm x 10^{-6}) | 6.59      | 6.57      | 6.52      | 6.55      |
| f0 (Hz)           | 1.02      | 1.17      | 1.61      | 2.35      |
| Jf (kg m^2 Hz^2)  | 0.0547    | 0.0545    | 0.0537    | 0.0542    |
| δ                 | 0.376     | 0.36      | 0.396     | 0.391     |
| Qr^{-1}           | 0.060     | 0.057     | 0.063     | 0.062     |
Tables 2–6, as $I_0^2$ decreases when $L$ increases. Then, for a fixed $L$ where $I_0^2$ is practically constant, decreasing $I$ leads to a higher frequency $f_0$.

By varying the value $L$ and maintaining the inertia fixed, $f_0$ increases as $L$ decreases because the torque required to torsion the sample at 8 degrees also raises. It was observed that the $f_0$ values for the effective length $L_1$ and $L_2$ are practically the same. The sample diameters of 6.5 mm and 5 mm exhibited the same tendency [57].
The $T_R$ value also presents the same value for $L_1 = 123.80$ mm and $L_2 = 104.00$ mm (see tables 2 and 3). Using equation (12), with $d = 8.00$ mm and $I_C = 4.02 \times 10^{-10}$ m$^4$ constant and fixed, it can be observed that $\tau_{\text{MAX}}$ decreases when $L$ increases, because $\tau_{\text{MAX}}$ depends on the value of $T_R$. Therefore, by increasing $L$, the value of $\tau_{\text{MAX}}$ is reduced, and this causes $f_0$ to decrease. In the case where $L_1 = 123.80$ mm and $L_2 = 104.00$ mm, $\tau_{\text{MAX}}$ is practically the same, and the frequencies must have equal values, as $T_R$ values are very close to each other and do not present significant differences. On the other hand, if $I$ is kept fixed and with the fact that $I_C$ is decreasing when $L$ increases, the frequency value of $f_0$ is expected to decrease. As the effective length values, $L_1$ and $L_2$, were the highest measured, the torque required to torsion the sample at 8 degrees was very small. Hence, it was practically not detected significant changes by the mechanical system. The $f_0$ is also related to the torsional stiffness of the sample [5]. For CP-Ti [6], the values of $f_0$ reached approximately 6 Hz for a $G = 30 \pm 4$ GPa, while the PTFE values did not exceed 2.5 Hz for a much lower $G$ measured in MPa. Free-oscillation method frequencies must be between $10^{-1}$ and 10 Hz [5] as can be observed by the values founded by this technique.

### 3.3.3. Values of $G$ for different effective lengths and fixed diameters

Figure 6 portrays the results of $G$ concerning rotational inertia for the effective lengths studied. The value of $G$ depends on $L$. It is also possible to observe that $G$ is independent of the values of $I$ considering the associated error. Looking at equation (7), $G$ is expected to increase when $L$ increases, keeping $d$ and $I$ previously fixed. The value of $Q^{-1}$ is small and does not significantly influence $G$. Also, under this same condition ($d = 8$ mm and pre-fixed $L$), the $I_C$ product is constant for four values of $I$ (see tables 2–6). So, it is expected that $G$, according to equation (7) does not change. According to equation (4) the value $T_R$ is expected to have the same value for different rotational inertias as we fixed $\theta$, $L$, and $I_C$. Thus, using equation (14), $G$ is predicted to have the same value, which reinforces the values obtained by equation (7).

The validation of the equation was also intended to standardize this technique for measurements with polymers. By definition, we know that the elastic modulus is a constant, and the findings presented here may confuse. However, it is possible to find an $L/d$ ratio with a value that is aligned with the elastic modulus of the studied material. For example, ASTM A 938–18, the standard test method for torsion testing of wire, provides a measurement procedure along with a table of the ratio versus diameter of the sample [38]. Additionally, the impulse excitation technique, a non-destructive dynamic method of elastic modulus characterization, has norms [59–61] that establish an $L/d$ ratio for cylindrical samples as $L/d \gtrsim 2$. Therefore, we can find the relation that provides the constant of $G$ by subjecting PTFE samples to successive torsion tests while varying their diameter and effective length. When observing figure 6, it is apparent that the values of $G$ tend to converge to effective lengths $L_5$, $L_3$, and $L_4$. The convergence of the $G$ values indicates which values for $L/d$ provide a single value for the torsion modulus, $G$, for PTFE samples. The value with the smaller length, $L_5$, is subject to stresses within the specimen fixation region (screws and three jaw-chuck) because it is below the recommended ratio of $L/d = 10$ [5]. Furthermore, the largest effective length, $L_1$, was subject to a significant influence of factor $L$ in equation (7). Therefore, both must be disregarded. A convergence trend with $G$ values was also observed by Pintão et al [6]. The values for the samples with the smallest effective lengths (17.50 mm) and those with the largest effective lengths (72.40 mm) were those that showed the most significant differences with the literature.
values. In our results, the same convergence trend was observed for all diameters analyzed. Comparing the values of $G$ obtained for different sample diameters, we observed that the $L/d = 10.64$ ratio elicited the same value of $G$ considering the associated error \[57\]. For samples with diameters of 5.00, 6.50, and 8.00 mm used in this work, the values were $G = (352 \pm 14) \text{MPa}$, $G = (354 \pm 11) \text{MPa}$ and $G = (351 \pm 9) \text{MPa}$, respectively. These values reflect how the $L/d = 10.64$ ratio provides a $G$ value of approximately 350 MPa for PTFE, being close to the expected value \[43, 44\]. Figure 7(a) depicts the curve of $G$ as a function of $L/d$, where it is shown that $G$ is the same for different sample diameters when the $L/d$ ratio reaches the value of 10.64.

Another analysis can be done based on the strength of the materials \[18\] using equation (14), changing $L$, $d$, and keeping $\theta$ fixed. In this case, it can be seen that what ensures that $G$ is the same for all three samples is when the $(T_{RL})/(I_{Cl})$ ratio has a single value. Thus, choosing an $L/d = 10.64$ ratio is based on checking the previous ratio, which can be expressed by $T_{RL}/d^4$. By doing so, it is found that $T_{RL}/d^4$ has a value of approximately 1.06 for all sample diameters, as shown in figure 7(b). On the other hand, figure 7(b) shows that $T_{RL}/d^4$ is 1.15, which is slightly higher than the previous values when the $L/d$ ratio = 13.84. In this situation, it is verified that $G = (384 \pm 16) \text{MPa}$ for the sample diameter of 5 mm, and that $G$ is different for the other diameters of 8 and 6.5 mm.

3.3.4. Varying the diameter and maintaining the fixed effective length for each rotational inertia

The results surrounding the PTFE sample with a fixed effective length of $L_4 = 69.20 \text{ mm}$ with diameters of 5.00 mm, 6.50 mm, and 8.00 mm are featured in tables 5, 8, and 9. Figure 8 was crafted from these tables, portraying the behavior of the values by varying the sample diameter with a fixed effective length for the rotational inertias used. A linear fit was applied for all experimental data for each inertia, $I$. To not overextend the paper, we merely present the results $L_4 = 69.20 \text{ mm}$ for diameters 5.00 mm, 6.50 mm, and 8.00 mm for $I = 207 \times 10^{-4} \text{ kgm}^2$. 

![Figure 7](image-url)
The other results for different rotational inertias and effective lengths are contained in [57]. The objective was the same as in section 3.3.1—verifying the validity of equation (7) by calculating the ratio between the rotational inertia used and slope, B.

As expected and based on equation (7), the value of G is reliant on the diameter, d, of the sample. The error associated with the linear coefficient is always higher than the set value (see figure 8), which means that it can be disregarded. At the same time, the adjusted line passes through the origin. Table 10 lists the calculated ratios and percentage deviations in absolute value for all rotational inertias used in tables 5, 8, and 9 and slopes accessed in [57]. It can be seen that the slope (B) and the relevant percentage deviations are higher than the deviations determined in section 3.3.1 (see table 7). According to equation (7) G depends on the value of d⁻⁴, and the errors associated with the values of G are higher than those in section 3.3.1. These values justify these higher percentage deviations. Nevertheless, the deviations were not higher than 5% for any of the effective lengths, confirming that equation (7) can be utilized to determine G.

Table 8. Results - PTFE: d = 5.00 ± 0.05 mm, L₄ = 69.20 ± 0.05 mm, \( I_c = 6.136 \times 10^{-11} \text{m}² \).

| Physical quantity | Config. 1         | Config. 2         | Config. 3         | Config. 4         |
|-------------------|-------------------|-------------------|-------------------|-------------------|
| I \( (\text{kgm}² \times 10^{-4}) \) | 526 ± 4           | 398 ± 1           | 207 ± 1           | 98.1 ± 0.5        |
| G (Mpa)           | 382 ± 16          | 383 ± 16          | 384 ± 16          | 385 ± 16          |
| \( T_0 \) (Nm×10⁻⁴) | 1.04              | 1.04              | 1.03              | 1.05              |
| \( \epsilon \) (Hz) | 0.405             | 0.467             | 0.647             | 0.942             |
| \( I \epsilon (\text{kgm}²\text{Hz}²) \) | 0.00863           | 0.00868           | 0.00867           | 0.00871           |
| \( \delta \)      | 0.488             | 0.431             | 0.403             | 0.458             |
| Q₁¹                | 0.078             | 0.069             | 0.064             | 0.073             |

Table 9. Results - PTFE: d = 6.50 ± 0.05 mm, L₄ = 69.20 ± 0.05 mm, \( I_c = 1.75 \times 10^{-10} \text{m}² \).

| Physical quantity | Config. 1         | Config. 2         | Config. 3         | Config. 4         |
|-------------------|-------------------|-------------------|-------------------|-------------------|
| I \( (\text{kgm}² \times 10^{-4}) \) | 526 ± 4           | 398 ± 1           | 207 ± 1           | 98.1 ± 0.5        |
| G (Mpa)           | 351 ± 12          | 354 ± 11          | 355 ± 12          | 356 ± 14          |
| \( T_0 \) (Nm×10⁻⁴) | 2.731             | 2.754             | 2.761             | 2.769             |
| \( I \epsilon (\text{kgm}²\text{Hz}²) \) | 0.0225            | 0.0227            | 0.0228            | 0.0229            |
| \( \epsilon \) (Hz) | 0.654             | 0.756             | 1.05              | 1.53              |
| \( \delta \)      | 0.488             | 0.431             | 0.403             | 0.458             |
| Q₁¹                | 0.078             | 0.069             | 0.064             | 0.073             |

Figure 8. Values of G as a function of the relation, \( (d^4)/d^4 \), with \( L_4 = 69.20 \text{ mm} \) and diameters of 5.00 mm, 6.50 mm, and 8.00 mm. Rotational inertia of \( I = (207 \pm 1) \times 10^{-4} \text{kgm}² \) was used.
3.3.5. Total friction $Q_{T}^{-1}$ and dynamic mechanical analysis (DMA)

As discussed in section 3.3.1, the parameter, $\delta$, should not significantly interfere with the values of $G$. This can be seen by using the expression, $\delta = \frac{\beta}{2\pi}$, to calculate the total friction of the pendulum. Accounting for the highest value of $\delta = 0.078$ (table 8) and calculating the factor $(1 - \frac{\delta^2}{4\pi^2})$, we yielded 0.999. In terms of percentage, this means a modification of 0.01\% into the value of $G$, demonstrating that the influence of $\delta$ is not significant.

The three PTFE relaxations first reported by McCrum [35] are plotted in the DMA measure (figure 9) alongside the first-order central peak, $\beta$, and the second-order side peaks, $\alpha$ and $\gamma$. It can be seen in figure 9 that the phase transitions are dependent on the rates employed for measurement as well as Young’s modulus ($E$), which is also dependent on temperature. Different from the free-oscillation pendulum test, the elastic modulus varies with frequency in the DMA, especially at cryogenic temperatures. Table 11 lists some parameters at room temperature ($25\,^\circ\text{C}$).

Comparing the total friction values, $Q_{T}^{-1}$, measured by the pendulum with the DMA internal friction values (tan $\delta$) within the nearest frequency range of the tests (1 Hz), they are found to be of the same magnitude (table 11). Being that $Q_{T}^{-1}$ is the sum of the internal friction of the sample, the friction of the ball bearing, and air friction, it is expected that this value would be higher than 0.0410, as the DMA measurements only consider the sample’s internal friction. Thus, the DMA measurements confirmed that the parameter $Q_{T}^{-1}$ used in the validation of the equation to determine $G$ is reliable. The central peak $\beta$ is the first-order peak, as it contains the phase transitions of the PTFE, which is also presented in the DSC curve (see figure 2). The peak from the phase transition from II to IV (triclinic to hexagonal crystal) at $19\,^\circ\text{C}$ is not seen in the DSC curve, as the measurement

Table 10. Proportions of values for $I$ and $B$ and percentage deviations with $L_{4} = 69.20 \pm 0.05\,\text{mm}$, using all rotational inertias from tables 5, 8, and 9 and slopes accessed in [57] (reproduced with permission).

| Proportion between $I$ | Proportion between $B$ | Percentage deviation (%) |
|------------------------|------------------------|--------------------------|
| 207/98.1 = 2.11        | 569507/260203 = 2.18870 | 3.73                     |
| 398/98.1 = 4.05        | 1047900/260203 = 4.02720 | 0.740                    |
| 398/207 = 1.92         | 1047900/569507 = 1.84000 | 4.30                     |
| 526/98.1 = 5.36        | 1403460/260203 = 5.39370 | 0.590                    |
| 526/207 = 2.54         | 1403460/569507 = 2.46430 | 3.02                     |
| 526/398 = 1.32         | 1403460/1047900 = 1.33930 | 1.34                     |

Table 11. Anelastic relaxation parameters for extruded PTFE at $25\,^\circ\text{C}$.

| Frequency (Hz) | $\tan \delta$ | $E$ (GPa) |
|---------------|---------------|-----------|
| 1 Hz          | 0.0410        | 1.07      |
| 10 Hz         | 0.0340        | 1.12      |
| 40 Hz         | 0.0320        | 1.14      |
starts at room temperature. Compared with the DSC curve, the acute peak \( \beta \) in figure 9 is usually closer to the endothermic transition at 31.6 °C, as shown in figure 2. In this case (figure 9), the peak \( \beta \) is around 45 °C, which happens to be a higher value when compared to other works from the literature [10, 35]. This is an effect related to the frequency used in the measurements, which causes the difference between the peaks found in the DMA and DSC [62]. It is possible to see in figure 9 that the three relaxation peaks shift to the right as the frequency increases from 1 Hz to 40 Hz. For example, Calleja et al [63] performed a DMA measurement with PTFE samples using lower frequency values (from 0.16 to 16 Hz) than the values used in this work (from 1 to 40 Hz), and the \( \beta \) peak in [63] was found to be at around 24 °C, which in this case shifts the curve to the left.

It is also possible to calculate \( G \) with Young’s modulus obtained from the DMA. If we use the relation, 
\[
G = \frac{E}{2(1 + \mu)},
\]
valid only for isotropic solids, as an approximation to evaluate \( G \) and the Poisson’s ratio (\( \mu \)) as 0.46 [64], the value for \( G \) is 366 MPa, which happens to be very close to that obtained via torsion pendulum. However, different values of Poisson’s ratio were reported in tensile tests (~0.36) in Rae and Brown’s work [43] and compression (~0.46) in Rae and Dattelbaum’s paper [44]. The authors argue that this is based on the difference in strain mechanism between tension and compression, making it questionable to use the value of 0.46 to calculate \( G \). Other authors have also reported that depending on the stress applied to the extruded PTFE, the Poisson’s coefficient can range from −14 to 0 [65–67]. As the technique presented in this study is independent of the Poisson’s ratio, the measurements will not be influenced by this factor for any applied stress, thereby serving as an advantage of the present technique. Dessi et al [68] also reported an elastic modulus dependency on the aspect ratio (\( L/d \)) of cylindrical polymeric rubber specimens undergoing torsion. However, their analysis was restricted to just one sample diameter, which limited their applicability range, especially when compared to the measure via torsion pendulum presented in this work. Furthermore, the correction coefficient proposed by the authors relies on the sample Poisson’s ratio, which does not happen with equation (7). Kumar et al [69] used a freely oscillating torsion pendulum configuration with a vertical and horizontal disk to stretch the sample and measure its torsion modulus. The deviations of around 10% in their results might be related to the tensile stress induced by the inverted configuration adopted by the authors. In contrast, the \( G \) deviations found herein are less than 5%, showing that the pendulum’s configuration is mechanically more stable and does not induce the sample to any considerable tensile stress. Besides, in a recent article [70], the authors have shown that it is possible to determine \( G \) using a method that takes into account the polymer deformation energy and the force work by a quasi-static torsion, and the values of \( G \) are in perfect agreement with the results reported in this work.

4. Conclusion

This work presented an alternative method in the determination of \( G \) for PTFE samples. The torsion pendulum given herein has a unique configuration that enabled the measurement of different sizes of PTFE samples, expanding its use beyond the metallic biomaterials previously evaluated in another study. All the results obtained from this measurement technique showed that the equation to determine \( G \) is valid. This was verified by the determination of rotational inertia using a method independent of that used to establish \( G \). The values of \( I \) were compared with those of \( G \), proving that the ratio between the values obtained by equation (7) reflects the ratio between the values of \( I \). The deviations, in this case, was not higher than 5%. On the other hand, it was observed that the \( G \) values converge to a typical value close to 350 MPa for an \( L/d \) ratio of 10.64, with smaller deviations for higher diameters. The rationale of choosing \( L/d = 10.64 \) is explained quantitatively based on the strength of materials. With the use of this technique, the values of \( G \) were not influenced by the Poisson’s ratio, as equation (7) does not rely on this factor. In this way, the main purpose of the study is accomplished, presenting a non-destructive technique capable of measuring samples of different sizes that differs from others reported to determine the \( G \) modulus. This argument reinforces the method as an alternative to study the elastic modulus of PTFE and, for future investigations, might be applied to other polymer samples or even tested on composite materials. Furthermore, it opens the possibility to assess new polymeric materials in which the Poisson’s ratio is unknown.

Acknowledgments

The authors thank FAPESP, proc. 2007/04094-9, proc. 2017/08820-8 and proc. 2018/12463-9, CAPES proc. 024/2012 and 011/2009 Pro-equipment. Also, we would like to express our gratitude to the ‘Laboratório de Análise Térmica e Polímeros, UNESP-Bauru’ group.
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