Dynamic rheological comparison of silicones for podiatry applications

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ABSTRACT

Purpose. This work shows an effective methodology to evaluate the dynamic viscoelastic behavior of silicones for application in podiatry. The aim is to characterize, compare their viscoelastic properties according to the dynamic stresses they can be presumably subjected when used in podiatry orthotic applications. These results provide a deeper insight which extends the previous creep-recovery results to the world of dynamic stresses developed in physical activity. In this context, it shoulod be taken into account that an orthoses can subjected to a set of static and dynamic shear and compressive forces.

Methods. Two different podiatric silicones, Blanda-blanda and Master, from Herbitas, are characterized by dynamic rheological methods. Three kinds of rheological tests are considered: shear stress sweep, compression frequency sweep and shear frequency sweep, all the three with simultaneous control of the static force at three different levels. The static force represents a static load like that produced by the weight of a human body on a shoe insole. In a practical sense, dynamic stresses are related to physical activity and are needed to evaluate the frequency effect on the viscoelastic behavior of the material. It is considered that the dynamic stresses can be applied in compression and shear since, in practice, the way the stresses are applied in real life depends on the orthoses geometry and its exact location with respect to the foot and shoe. The effects of static and dynamic loads are individualized and compared to each other through the relations between the elastic constants for isotropic materials.

Conclusions. The overall proposed experimental methodology can provide very insightful information for better selection of materials in podiatry applications. This study focuses on the rheological characterization to choose the right silicone for each podiatric application, taking into account the dynamic viscoelastic requirements associated to the physical activity of user. Accordingly, one soft and one hard silicones of common use in podiatry were tested. Each of the two silicones exhibit not only different moduli values, but also, a different kind of dependence of the dynamic moduli with respect to the static load. In the case of the soft sample a linear trend is observed but in the case of of the hard one the dependence is of the

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power law type. Moreover, these samples exhibit very different Poisson's coefficient values for compression stresses lower than 20 kPa, and almost the same values for stresses above 40 kPa. That different dependence of the Poisson's ratio on the static load should also be taken into account for material selection in customized podiatry applications, where static and dynamic loads are strongly dependent on the individual weight and activity.

Keywords: Silicones; Orthoses; Podiatry; Rheology; Dynamic; Load.

1. Introduction

An important part of the features of foot ortoses depends on the material of which they are made. Pathology, age and weight are important factors to choose the right type of orthoses and the material of which they are made (Nicolopoulos et al., 2000). Interactions between the foot and the insole/shoe have been studied but more studies are still needed to clarify the biomechanical effects of such devices (Chen et al., 2010). While loading rate and impact force were widely studied, the effect of foot orthoses on these variables remains unclear (McMillan and Payne, 2008). There is an important amount of studies where physiology or health are related to orthoses, the materials from which they are made and the physical activity. With respect to the stresses resulting from physical activity, axial compression and shear forces are developed in the foot during walking. Shear forces are on average 30% of the value of vertical forces (Laing et al., 1992). Both static and dynamic measurements of foot pressure seem important to devise suitable means of protecting the foot from ulceration (Boulton et al., 1983). Shear forces are also thought to be important in the pathogenesis of these ulcers (Pollard et al., 1983). For example, silicone insoles and heel cups are prescribed for plantar heel pain and other foot pathologies (Toomey, 2009). The use of gel materials proved to be better than foams for the reduction of plantar shear forces (Curryer and Lemaire, 2000). There are also reports where shoe inserts are recommended to correct mechanical imbalances of the foot (Caselli and George, 2003). An emphasis is made on the need that orthotics meet patient's individual needs (Whitney, 2003). In addition, the increasing use of silicones as a viscoelastic material for podiatry orthoses justifies the importance of performing viscoelastic studies reproducing the static and dynamic loads to which a material is subjected as a part of a orthoses during its normal use. Static and dynamic viscoelastic properties of materials such as silicones can be studied by dynamic mechanical analysis (Chartoff et al., 2009). There are also some relatively new instruments that allow to perform some tests that reproduce very realistically the stresses and deformations exerted on the material during the normal use of podiatric orthoses. That kind of situations may include simultaneous stresses, both compression and shear. Some of the newest rheometers allow to apply that simultaneous stresses: while a compression static load is applied in the axial direction of the rheometer shaft, the dynamic stresses can be applied in compression too or in shear. As a general rule in podiatry, hard silicone rubbers, which have relatively high elasticity modulus, are indicated for corrective orthoses, while soft silicones, with low elasticity modulus, are better for paliative treatments (Chadchavalpanichaya et al., 2018). Of course, that rule works in most cases where only static or very low frequency stresses are involved. A previous work of the authors was focused on the static viscoelastic properties of silicones for palliative and corrective podiatry orthoses (Janeiro-Arocas et al., 2016). That study was based on creep-recovery tests performed at room temperature with the aim to provide an insight for situations

where forces similar to those used in the work were statically applied on the orthoses. For example, while standing on the two feet the weight is distributed in both. However, orthotic materials will normally undergo dynamic loading. For instance, an insole is cyclically loaded as the individual walks or runs. In addition, the stiffness of the material changes as a function of the frequency when subjected to cyclic mechanical stresses. Also, the way the stiffness and, in general, the viscoelastic properties change with the frequency is specific for each material. The practical implications of that frequency dependence in the framework of podiatry applications was not completely evaluated yet and will be associated to human activities where mechanical stresses of relatively high frequency are involved. Sudden movements related to slip and fall, emergency brake pedal operation when driving, jumps in sports like basketball, where the height of the jump is affected by the leverage transmitted trough the shoes, running, since the leverage and the achieved speed are related to the stiffness of the soles or skiing, where vibrations of different frequencies are involved are just a few examples of activities where dynamic stresses are transmitted through the shoes (Gobbi et al., 2013; Jarboe and Quesada, 2003; Walsh and Tarlton, 2017; Zhang et al., 2014). Nevertheless, up to the moment, little work was dedicated to relate dynamic mechanical properties of materials to their performance as potential prosthetic elements. Maybe the main reason for that is that the frequency of the mechanical stresses involved in the human body movements fall in general into a very narrow range of frequencies.
The aim of this study is to demonstrate how dynamic viscoelastic characterization can be used to better suit materials to specific applications where dynamic stresses are involved. Being the viscoelastic response dependent on the frequency of the stimulus, this work will focus on the frequency range associated to human body activity. Another important aspect of this study is to consider that, normally, podiatry orthoses are simultaneously subjected to static loads and dynamic shear or compression stresses. Thus, three kinds of rheological tests are considered here: shear stress sweep, compression frequency sweep and shear frequency sweep, all the three with simultaneous control of static force. Three levels of static force were chosen in the range from zero to 90 kPa, which covers most of the mean peak plantar pressures developed during walking in the different regions of the foot under the conditions of barefoot and shod at different speeds (Burnfield et al., 2004).

2. Experimental

Silicone cylindrical samples of 17 mm diameter and about 4.5 mm thickness were prepared from commercial components. Two pre-polymer mixtures containing additives for easier handling and higher comfort were used: Blanda-blanda (TM) and Master (TM). These mixtures, respectively, correspond to the soft and hard types of podiatry silicones. According to the manufacturer, Herbitas-Spain, the Shore A hardness values that can be obtained with these products using the recommended procedure are: 4 for Blanda-blanda (TM) and 25 to 26 for Master (TM). A pre-polymer base and a curing agent, Reaktol, were manually mixed together at the recommended mass proportion of 1 drop of the curing agent per gram of pre-polymer. A metallic cylinder supported on a glass plate was used as a mold. The mold was completely filled with the mixture and let at room temperature overnight to ensure a full cure.

Rheological tests were performed in a commercial TA instruments Discovery Hybrid Rheometer DHR-2. This instrument is furnished with a magnetic bearing system which allows to perform oscillatory axial tests. All experiments were repeated to check for reproducibility. The experiments were carried out according to the following experimental setups:

1. Compression frequency sweep (CFS)

It consisted of applying a logarithmic frequency sweep from 0.1 to 10 Hz in the axial direction, using a strain amplitude of 0.085 and keeping a constant axial stress. This constant axial stress was controlled at 4.4, 8.8, 22.0 and 88.1 kPa.

2. Shear stress sweep (SSS)

It consisted of applying a logarithmic torque sweep in the 0.1 to 10000 μN·m range, which practically represents an oscillation stress in the 0.1 to about 8000 Pa range. The frequency was 1 Hz and a constant axial stress was applied. The experiments were performed with four constant axial stress values: 4.4, 8.8, 22.0 and 88.1 kPa.

3. Shear frequency sweep (SFS)

It consisted of applying a logarithmic frequency sweep in the 0.1 to 100 Hz range, a torque amplitude of 10 μN·m and a constant axial force. The experiments were performed with four constant axial stress values: 4.4, 8.8, 22.0 and 88.1 kPa.

3. Results and discussion

An important point in rheology is the reliability of the results because that depends on the instrument performance, the experimental setup, and on the properties of the sample. That becomes more important when too low or too high values of stress, displacement or frequency are involved since they may fall below the sensitivity limit or result in significant inertia noise. That is not the case of the compression frequency sweep since in this case, due to the instrument specification, the frequency is imited to the 0.1-100 Hz range. However, as it will be commented below, the situation is different for the shear tests.

3.1. Compression frequency sweep

Figure 1 shows a typical plot of the results obtained with experimental setup 1. The storage and loss modulus are respectively obtained as the in-phase and out-of-phase components of the complex modulus. As it will be discussed below, these results representthe ratio of axial stress to axial strain in a uniaxial strain state, which was referred to as bulk longitudinal modulus (Ferry, 1980), plate-wave modulus (J. Bobber, 1970), p-wave (Mavko et al., 2003) and constrained modulus (Lakes, 2009). Here we will use the term longitudinal modulus (M). In this case the frequency is limited to the 0.1 to 10 Hz range, which broadly covers the frequency values associated to the normal human physical activity. It can be observed that in that range of frequencies there is a slight increase of the storage modulus (M’), much higher than that of the loss modulus (M’’), considering the logarithmic scale. In fact, the storage modulus values are about 8 times those of the loss modulus. That trend of the modulus versus frequency was observed for both the Blanda-blanda and Master samples and is very normal as the elastic response results more dominant for shorter observation times.

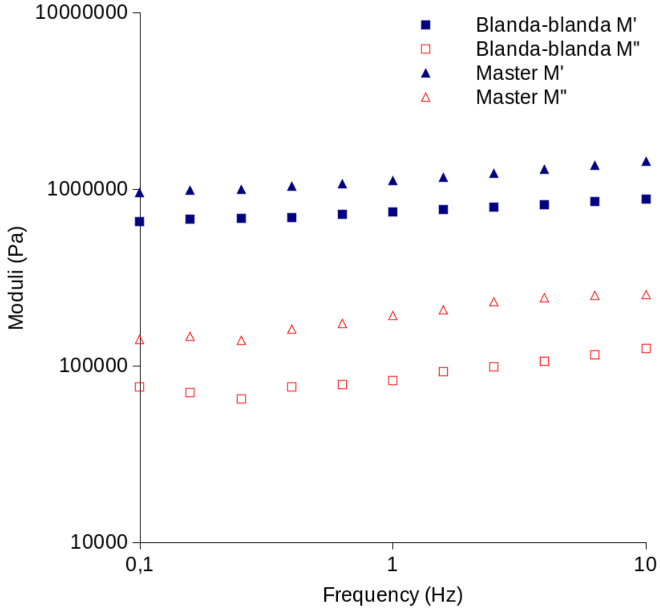

Figure 1. Plot of longitudinal storage and loss moduli obtained from the Blanda-blanda and Master samples in CFS mode using a static axial compression of 4.4 kPa

The results of the moduli versus the static axial compression stress in the linear viscoelastic region are displayed on Figure 2. Interestingly, while for the Blanda-blanda sample there is a linear dependence, a power law trend is observed for the Master sample. With this type of silicones a higher stiffness is normally achieved by increasing the crosslinking density. We may easily assume that the Master sample is more highly crosslinked than the Blanda-blanda one and assign that different behavior to their different structures. However we cannot discard that other structural factors such as the presence of aromatic rings, which typically imparts some stiffness to the polymeric chains, may also contribute to the difference of behavior between both samples. Anyway, it is clear that both silicones become very differently as the static stress increases. While for little static stresses both samples exhibit a similar response, for higher static stresses, as those involved in cases of overweight or when a human body is heavily loaded, the Master sample behaves much more rigidly.

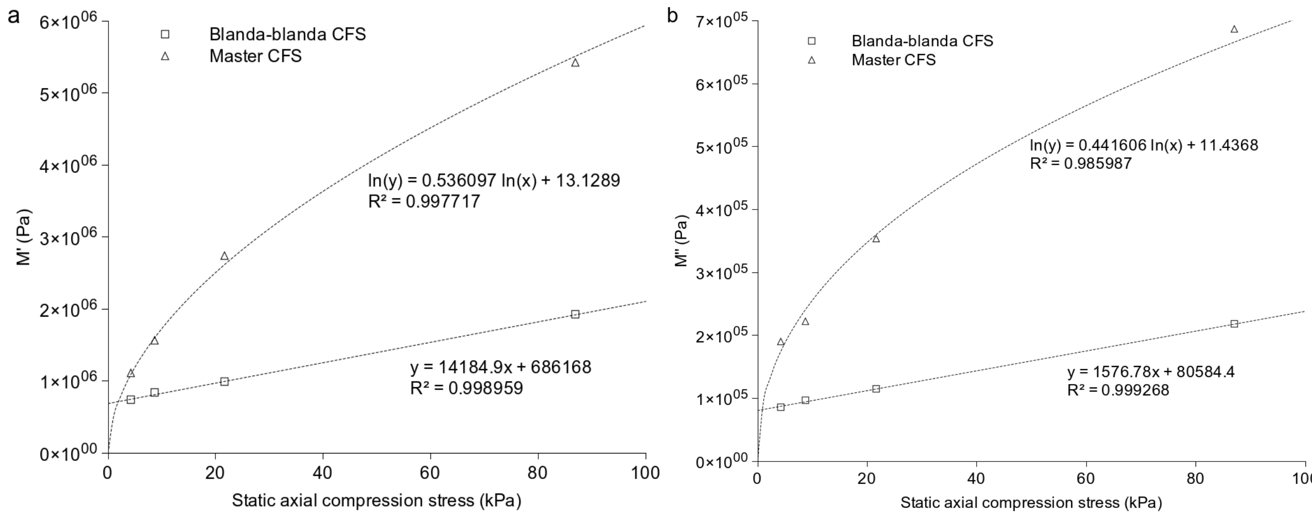

Figure 2. Longitudinal storage and loss moduli values obtained from the Blanda-blanda and Master samples in CFS tests at 1 Hz

3.2. Shear stress and shear frequency sweeps

Figure 3 shows the wave shape of the imposed torque and the resulting displacement obtained at three values of the oscillation stress in SSS. Both the torque and the displacement shapes should be sinusoidal to obtain the right storage and loss component values from the complex data. If the shape of the waves is out of control then the convoluted results are not reliable. For example, the results presented in Figure 4 are reliable in the 0.1 to 2600 Pa range. For higher oscillation stress values, the separation of the storage and loss components is not good. On the other hand, for oscillation stress values lower than 1 Pa, although the torque control is good, the displacement in that conditions is slightly noisy.

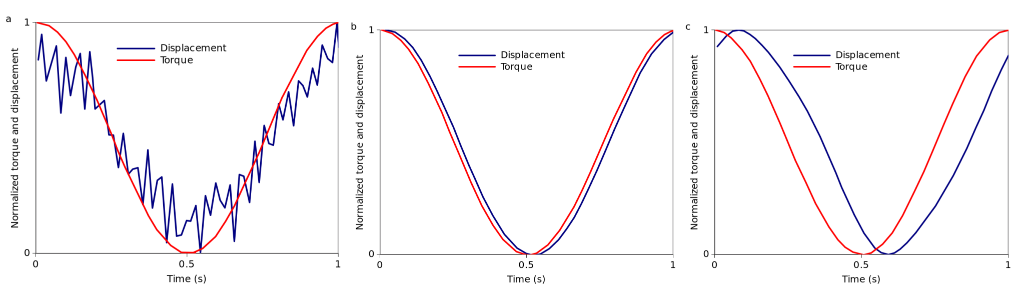

Figure 3. Wave shapes of torque and displacement corresponding to one cycle at three values of the oscillation stress: 0.5 Pa (a), 104 Pa (b), and 2621 Pa (c)

Figure 4 shows a typical plot of the results obtained with the SSS setup. The storage modulus represents the elastic or recoverable component of the modulus while the loss modulus represents the part of the modulus related to the plastic or viscous deformation. The complex modulus, not displayed, practically matches the storage modulus because G'' is one order of magnitude lower than G'. According to Figure 4, the storage and loss moduli are practically constant up to about 1000 Pa oscillation stress. In that range, the value of the storage modulus is about 10 times that of the loss modulus. For higher oscillation stress values the loss modulus increases and, simultaneously, the storage modulus decreases. It means that the material becomes softer at high dynamic deformations.

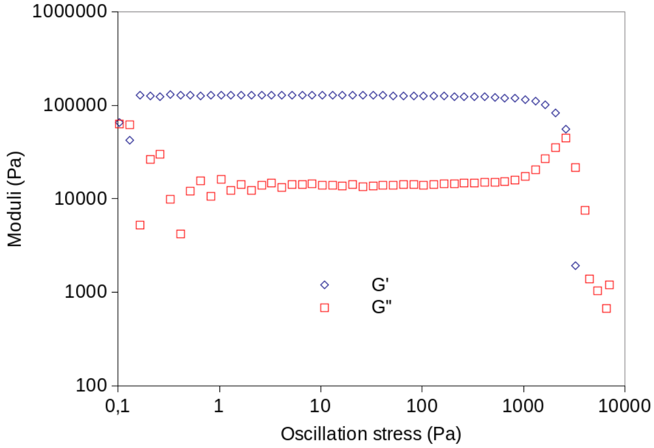

Figure 4. Plot of storage, and loss moduli obtained from the Blanda-blanda sample with SSS setup, 1 Hz and static axial compression of 4.4 kPa

Figure 5 shows a typical plot of the results obtained with the SFS setup. In this case, the frequency covers the 0.1 to 100 Hz range, although for frequency values higher than 30 Hz there is some noise. It can be observed that, as in the case of compression frequency sweep, there is a slight increase of the modulus with frequency. In this case the storage modulus values are about 7 times those of the loss modulus.

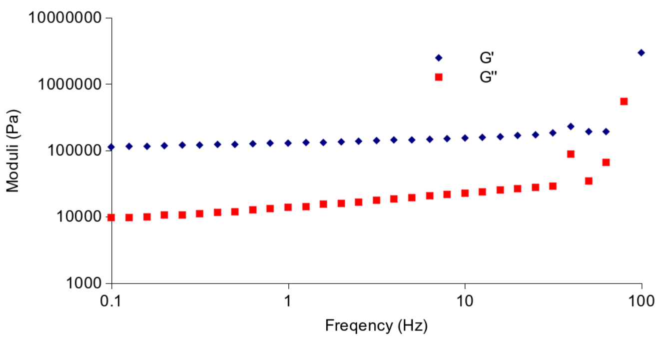

Figure 5. Plot of storage and loss moduli obtained with the Blanda-blanda sample, with SFS setup and static axial compression of 4.4 kPa

Figure 6 shows the resulting moduli values versus the applied static axial compression stress. It is important to note that data obtained with different experimental setups, SSS and SFS, match perfectly. In the case of SFS an oscillation stress of about 10.4 Pa was consistently observed along the experiment, except for the highest frequency values. In the case of SSS the moduli were practically constant in the 0.1-1000 Pa range, although some noise is observed in the loss modulus for oscillation stresses below 10 Pa. Similarly to what was observed in the compression tests, as presented in Figure 2, a power law trend is observed for Master with respect to the axial compression stress while for the Blanda-blanda sample there is a linear dependence. These results confirm that the dynamic shear response is affected by the static compressive stress in the same way than the dynamic compressive response. Thus, the implications for overweight situations are also similar and while for low pressure the dynamic response of both silicones is of the same order, when the pressure increases the increase of both the storage and loss moduli is more noticeable in the case of Master.

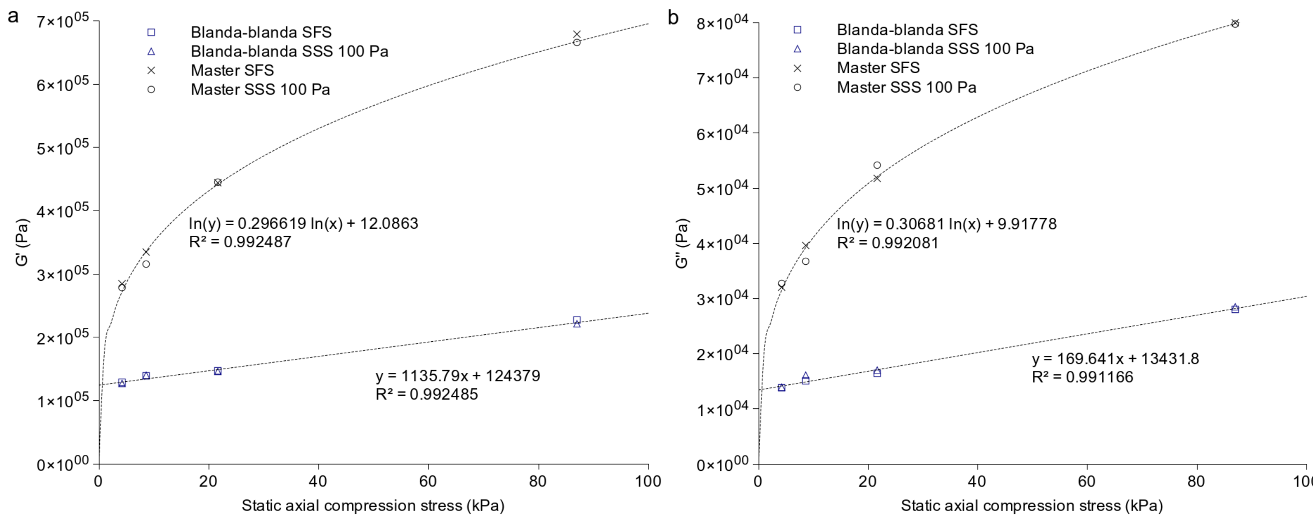

Figure 6. Moduli values obtained from the Blanda-blanda and Master samples in SSS and SFS tests at 1 Hz

3.3. Crosslinking density

The crosslinking density of both samples is estimated through the equation of rubber elasticity (Treloar, 2005):

$$M_c = \frac{\phi \cdot \rho \cdot R \cdot T}{G'}$$

where M_c is the molecular mass between crosslinking points, ϕ is the front factor, ρ the density of the sample, R the gas constant, and T temperature. For that, density of both samples was measured. It was verified through the Poisson's ratio and the gap measurements that density does not experience any significant change in the range of axial pressure considered. In addition a front factor value of 1 was adopted for the G' value obtained by extrapolation at the zero axial static load. That extrapolated value of G', obtained from the trend equations in Figure 6a, is 238690 kPa for Master and 124379 kPa for Blanda-blanda. The resulting values of M_c were 10.3 kg mol^{-1} for Master and 19.8 kg mol^{-1} for Blanda-blanda.

3.4. Compression modulus interpret

As mentioned before, the moduli values obtained from the dynamic analysis of the CFS tests fit better with the assumption that they actually represent the longitudinal modulus (M) instead of the Young's modulus (E).

As described in the Experimental section, this kind of tests basically consists of applying an static compressive force and, over it, a sinusoidal compressive force at given frequencies. That situation can be assimilated to a plane P-wave uniaxial strain (as it is formed from alternating compressions and rarefactions). Then, the modulus obtained from the dynamic signal should be considered as the longitudinal modulus (M), and the one obtained from the static load-strain relationship as the Young's modulus. The results obtained in the CFS experiments make sense in the context of this approach, considering the relations existing between the elastic constants for homogeneous isotropic materials (Birch, 1961; Mavko et al., 2009). It deserves to be mentioned that both E and M moduli could be simultaneously measured as the rheometer allows for separated analysis of the static and dynamic stress-strain data. However, a more precise measurement of E in compression can be obtained at the beginning of the experiment, just after applying the static compressive force but before applying the dynamic load. On the other hand, E can be calculated, by means of the relations existing between the elastic constants, from the G and M values obtained, respectively, in the SFS and CFS tests. As commented on Figures 1 and 4, the values of M' and G' are, respectively, about 8 and 10 times those of M'' and G''. Thus, the complex moduli, M, G and E, are practically represented by the storage moduli M', G' and E'. Figure 7 plots the E' modulus, calculated from G' and M' through the relations between elastic constants, versus the static compression load.

$$E=\frac{G\cdot(3\cdot M-4\cdot G)}{M-G}$$

These values of E' are consistent with those obtained by direct measurement of the initial gap change resulting from the static load.

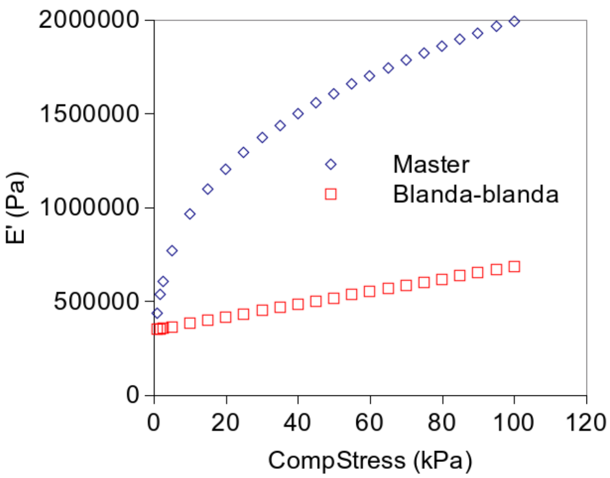

Figure 7. Plot of the E' modulus calculated from the G' and M' moduli through the relations between the elastic constants versus the static compression load

In order to better understand the different behavior of both samples, the Poissons coeeficient, ν, was also calculated from the same experimental data than the moduli. It is related with the other elastic constants through these expressions (Birch, 1961; Mavko et al., 2009):

$$\nu=\frac{E}{2\cdot G}-1=\frac{M-2\cdot G}{2\cdot M-2\cdot G}$$

Figure 8 shows how the Poisson's ratio changes much more with the static compression stress in the case of the Master sample than in the case of Blanda-blanda.

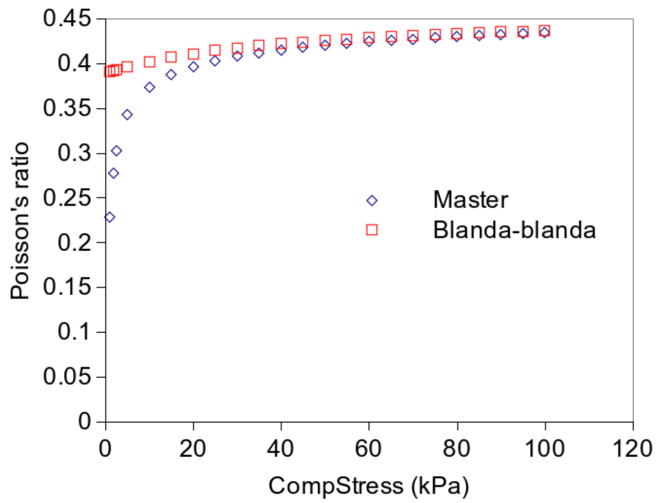

Figure 8. Plots of the Poisson's ratio versus the static compression stress

3.5. Important difference between both samples

Regardless the dynamic stresses are applied in compression or in shear, the same type of trends are observed for the loss and storage moduli with respect to the static compression stress for a given sample. But the trends are totally different from one to the other sample: In the case of Blanda-blanda all M', M'', G' and G'' moduli are linearly related to the applied static compression stress. However, in the Master case, where the density of crosslinking is much higher, a power trend is observed for the same moduli with respect to the axial compression stress. These different behaviors on compression are probably related to the higher tendency of the Blanda-blanda sample to easily spread in the lateral direction, which is known as barrelling. This feature makes the Blanda-blanda silicone more adequate for palliative application where reducing the pressure at each impact during gait is intended such as the special case of protection against ulcerations (Lavery et al., 1997, 2005).
E' was calculated from G' and M' through the relations between the elastic constans for isotropic materials. The trends obtained for both samples were of the same types than those obtained for G' and M' and match very well with the complex modulus, E, obtained before applying the dynamic stresses.
According to Figure 7, while for very little compression stresses the E modulus are of the same order for both materials, when increasing the compressive load, the Master sample becomes much more rigid. This behavior is clearly due to its much higher cross-linking density with respect to the Blanda-blanda sample. However, according to Figure 8, Blanda-blanda and Master samples exhibit a very different Poisson's coefficient for compression stresses lower than 20 kPa. In fact, while the Blanda-blanda values are in the range of the rubbers, the vary little values of Master are more typical of metals. Otherwise, for stresses above 40 kPa, their Poisson's coefficients become almost the same, about 0.43. These different features should be considered when designing an orthoses which will be confined between the foot and the shoe and subjected to a set of static and dynamic shear and compressive forces. In fact, these material features can be related to the final use of silicones for adaptive or corrective tasks.

4. Conclusions

A methodology is proposed to characterize and choose the best silicone for each podiatric application according to the dynamic viscoelastic requirements associated to the physical activity of the patient or user. Also, this methodology allows to evaluate how a given material will perform in different physical activities. The combined effect of the static load and the frequency on the loss and storage modulus is totally different for the two materials studied, which correspond to two of the most common silicone grades used in podiatry. The increase of the modulus with frequency was measured for both samples in compression and in shear using different levels of static compression in a range covering most of the plantar pressures developed during walking in the different conditions. That increase is higher for the storage than for the loss modulus. The effect of the static load in all dynamic modulus is very different in the Blanda-blanda and Master samples. In compression, for Blanda-blanda, the dependence of the uniaxial moduli M' and M'' with the static load is linear while for Master power law trends are observed. While for low compressive loads the moduli values are similar for both samples, for higher loads Master behaves much stiffer than Blanda-blanda. Similarly, in shear, G' and G'' vary linearly in the case of Blanda-blanda and with a power low trend in the case of Master. Also, while both samples present similar values of G' and of G'' for low static pressures, the increase of both moduli resulting from the increase of the static load is much more important for Master than for Blanda-blanda. However, these samples exhibit a very different Poisson's coefficient for compression stresses lower than 20 kPa, and almost the same for stresses above 40 kPa. These differences should be taken into account in order to select the right material for an specific orthoses. In addition, the overall proposed experimental methodology can provide very insightful information for better selection of materials in podiatry applications.

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