Technical Report 2

Uniaxial compression and tensile tests of vitreous biomaterial obtained through grape pomace treatment.

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Abstract

The results of compression and uniaxial tensile tests of vitreous biomaterial obtained through Powder House's Vitreous Transformation Process of grape pomace, are reported. The objective is to determine some mechanical properties of this vitreous biomaterial, such as their tensile strength (quantitative) and brittleness (qualitative). These properties, together with others of physical and chemical character may be important to determine their grindability or triturability.

i. INTRODUCTION

Previously, diametral compression tests (SCS [1]) were reported as a first approach towards the mechanical characterization of a vitreous biomaterial obtained through Powder House's Vitreous Transformation Process of grape pomace, specifically in terms of its hardness and brittleness. The SCS test takes advantage of the fact that the diameter of the vitreous biomaterial is constant, a product of its manufacturing process, which facilitates its conditioning for the test. However, the tests do not show brittle behavior but rather semi-fragile (Smith et al. [2]) or plastic behavior with gradual fracture (Procopio et al. [3]), characterized by fluctuations in compressive strength, attributable to multiple failure events of small˜ magnitude, without a clearly identifiable critical strength limit.

The uniaxial compression (UC) test is used to determine the compressive strength of a material. Brittle materials such as concrete often fail by axial rupture, shear or buckling (in the case of long specimens). Therefore, this test, applied to the vitreous biomaterial, can give information about its brittleness. The CU test, unlike the SCS test, does not allow to infer the tensile strength. However, it allows comparisons to be made with the same test applied to brittle and ductile materials, where the failure mode in each case is known. Unlike the SCS test, uniaxial compression requires flat faces of the cylindrical specimen. Compression is performed at constant speed. The ASTM D4179 - 22 standard for vitreous biomaterial compression indicates a specimen height h less than or similar to its diameter D ($h \leq D$). It is customary to report the strength of the specimen (maximum force at breakage). In the case of pomace vitreous biomaterial, most specimens have a height approximately equal to twice the diameter ($h \approx 2D$).

The tensile test (TU), due to its simplicity, is the most widely used test in the mechanical characterization of materials. It is also the one that provides the most direct information on mechanical properties such as stiffness (Young's modulus), yield strength, tensile strength, fracture toughness, among others. In particular, a sudden drop in tensile strength is a manifestation of the brittle character of a material. It occurs by the sudden propagation of a fracture initiated in some superficial defect of the specimen. A tensile test applied to the pomace vitreous biomaterial presents the difficulty that it is not possible to take it by the jaws of the testing machine without it being crushed when squeezed. In Alam et al. [5] a

methodology is described for subjecting roots to tension without breaking them at their ends.

We propose a similar technique to subject the pomace vitreous biomaterial to a TU test.

ii. UNIAXUAL COMPRESSION (UC)

A. Methodology

1. Test tubes

In the literature we find several authors describing the specimen preparation for a UC test. For example, Gilvari et al [6] and Kazimirova et al [7] present the methodology for conditioning biomass. In both works, the aim is to leave the biomaterial with flat ends. In our case, the most cylindrical biomaterial with a length greater than 12 mm were chosen. Specifically, units of biomaterial that did not have a straight axis of symmetry or those that were visibly inhomogeneous were discarded. The ends of the vitreous biomaterials were cut with a multipurpose tool (Dremel) and then sanded until they were flat and perpendicular to the axis of symmetry of each unit. An auxiliary piece consisting of a 10 mm high plastic block with a 5 mm diameter perforation in which the vitreous biomaterial was inserted to immobilize it was used for this purpose. The biomaterial was sanded by moving the block horizontally and exerting light pressure on the sandpaper. The vitreous biomaterial was sanded until both ends of each unit were flush with the block. If the unit of biomaterial did not withstand this process it was discarded. Approximately 1 of 3 units was left with an acceptable shape for testing. The dimensions of the units used in the test specimens were D = 4.8mm and h = (10.0 ± 0.1) mm. The vitreous biomaterial diameter is a quantity that, being determined by the elaboration process, remains relatively constant and, therefore, was measured only once.

2. Testing

For the CU tests, an Instron 3365 tensile/compression machine was used, equipped with a 2.5kN load cell. Two compression platens, each 56 mm in diameter, were mounted. Initially,

the upper platen was positioned about 11 mm from the lower platen. The specimen was placed in the center of the lower platen. The lowering speed of the crosshead was 1 mm/min, the same as in the SCS test (previous report).

The position *d* of the crosshead and the compression force *F* were recorded by software, with a frequency of 100 measurements per second. Five (5) identical tests were performed with five specimens.

five specimens.

B. Results

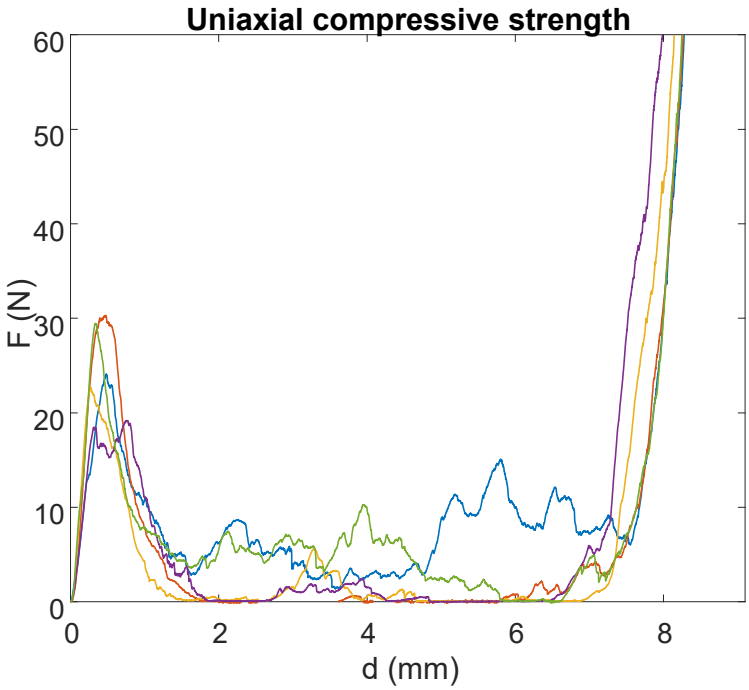

Fig. 1 Uniaxial compressive strength *vs.* displacement for the five vitreous biomaterial specimens.

The *F vs. d* plots of the five uniaxial compression tests are shown in Fig. 1. In all of them, the compression was performed at a constant speed of 1 mm/min and the test was automatically stopped when the force reached 2000 N. Note that the force is represented in a range lower than the maximum reached during the test to appreciate what happens in the zone of interest.

C. Analysis and discussion

A large variability of the force is observed despite the fact that the height *h* of the vitreous biomaterial units is the same. However, it is possible to identify three successive regimes in the response in compression.

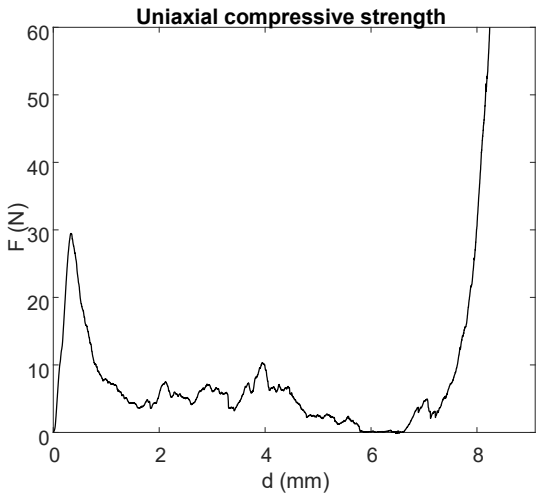

Fig. 2 Axial compressive strength *vs.* displacement for one of the specimens.

In Fig. 2 we have plotted the force measured for one of the five vitreous biomaterial units, which is representative of the response of these units, which is representative of the response of the other units. First we observe a continuous increase of the force with distance up to $d \approx 0.34$ mm, which corresponds to a percentage deformation of 3.4%. From this point the force drops, revealing a failure process. The drop, although not abrupt, is much steeper than the similar drop observed in some of the diametral compression tests (see previous report). After the drop, the force undergoes a series of dips and rises, indicative of successive failure events. However, the force remains constant on average, even reaching almost zero at around 6 mm displacement. Of the five specimens, the same phenomenon is observed in four of them. Only for one of the specimens the force never reaches zero (see Fig. 1). This can be interpreted as a total failure of the vitreous biomaterial, where over a distance interval it does not offer any compressive strength. Finally, from a distance of about 7 mm, the force again increases steadily. We can interpret the sequence of three behaviors as i) a response resulting from the resistance of the vitreous biomaterial to elastic, plastic or viscous deformation (due to the presence of fluid); ii) the formation of fractures or other localized

defects that reduce the compressive strength of the vitreous biomaterial; there may also be buckling and fracture effects as reported by Salas-Bringas et al. [4]; iii) a "granular" regime, identical to that observed in the SCS tests (see previous report), where the vitreous biomaterial, significantly fragmented, behaves like a "pancake" that flattens and widens more and more, which explains the substantial increase in strength for distances greater than 8 mm. In regime (i) additional tests would be needed to determine the degree of elasticity (reversible, independent of the compression rate), plasticity (irreversible, independent of the compression rate) or viscosity (irreversible, dependent on the compression rate). One of the vitreous biomaterial units in regime (ii) is shown in Fig. 3. The vitreous biomaterial has a splintered appearance, evidence of successive failure events and possibly buckling followed by fracture. Because of the latter, it is possible that the strength response would be qualitatively different if a test were performed with $h < D$ [4].

In comparison to the SCS tests reported in the first report, the CU tests, although performed with a lower number of repetitions, indicate a relatively more brittle behavior, due to the existence of a first failure event characterized by a much steeper drop than in some of the SCS tests (see previous report).

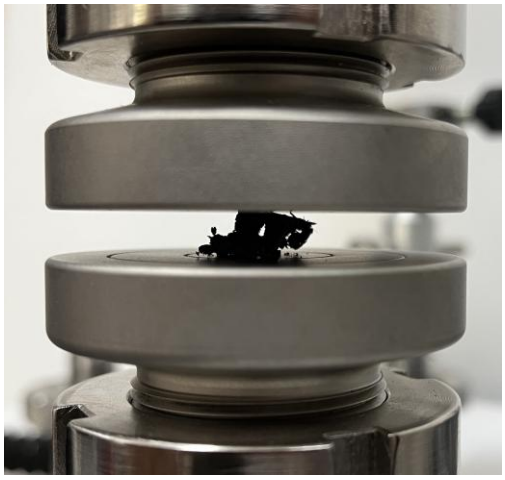

Fig. 3 Test specimen during uniaxial compression (UC) test with visible signs of splinter formation.

Compressive strength

From the results of the five tests it is possible to obtain a measure of compressive strength or *hardness*, determining for each test the maximum force reached between regimes (i) and (ii). The resistance obtained, with its standard deviation is $< F_{max} >= (25.0 \pm 4.9)$ N. From this we determine the compressive strength as

$$< \sigma_c >= \frac{4 < F_{max} >}{\pi D^2},$$

which gives $< \sigma_c > = (1.40 \pm 0.27)$ MPa [D = (4.8± 0.1) mm]. This strength value is below the strength of weaker masonry materials such as brick ($\sigma_c \approx 7$ MPa) and is slightly higher than that of Styrofoam or plumavit ($\sigma_c \approx 0.5$ MPa).

III. UNIAXIAL TRACTION (TU)

A. Methodology

1. *Test tubes*

The longest vitreous biomaterial unit with the straightest possible axis were chosen. It was not necessary to condition them. The ends of each unit were inserted into a piece of polyethylene tubing of 5 mm inner diameter, previously filled with epoxy resin. The resin was left to dry with the heads (the pieces of hose) of the specimen resting between two parallel rigid planes. The purpose of this procedure was to leave the heads aligned, at least in a common plane. This allowed each specimen head to be clamped simultaneously in each clamp, without the vitreous biomaterial running the risk of breaking by bending. In this way, 7 specimens were obtained. Six of them are shown in Fig. 4. No care was taken to leave the exposed part of the vitreous biomaterial of the same length; instead, its integrity was privileged during the fabrication of each specimen.

2. Testing

Standard grips of or reduced size were used. The specimen was fixed by tightening each jaw, taking care that the heads were not crushed to avoid giving to the vitreous biomaterial. Special care was taken to ensure that the specimen was not subjected to traction. Two of the specimens were left in traction and were discarded. Of the remaining five specimens, two were left in compression, with initial compression forces of– *1.*7 N and– *6.*7 N (yellow and light blue curves, respectively, in Fig. 5). Both forces are lower than the compression force that the vitreous biomaterial withstands in diametral compression, before it starts to break (see report above).

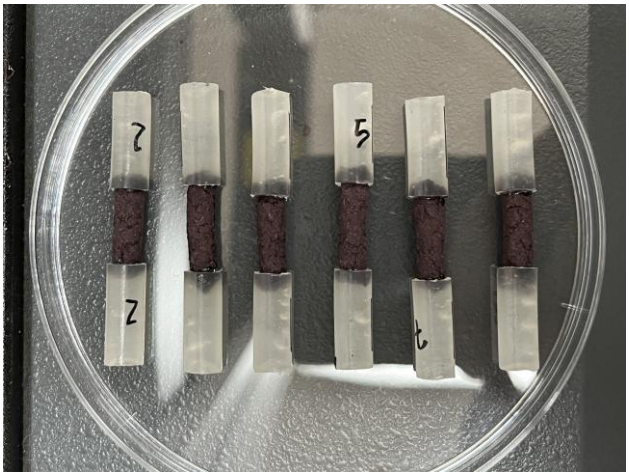

Fig. 4 Specimens for uniaxial tensile tests. The vitreous biomaterial is fixed by epoxy resin to two heads consisting of pieces of polyethylene hose.

Although it is not the same test, the comparison gives some support to affirm that the vitreous biomaterial did not suffer a damage or by compression, prior to applying the traction. In the remaining three specimens, the initial force remained in the range [– *0.*07, 0.13] N.

B. Results

The *F vs. d* plots of the five TU tests are shown in Fig. 5. In all of them, compression was performed at a constant speed of 1 mm/min and stopped manually once the specimen was visibly split in two. Photos of the five specimens after breaking are included (Fig. 6a-e). The remaining two specimens are shown in Fig. 6f. It can be seen that the fracture plane occurs at random heights, which rules out end effects. The inclined plane failure with respect to the tensile axis of the specimen in the upper subfigure in Fig. 6f is striking. In an isotropic material the fracture plane would be perpendicular to the tensile direction. This indicates a clear anisotropy of the material, specifically a strength that depends on the orientation of the fracture plane.

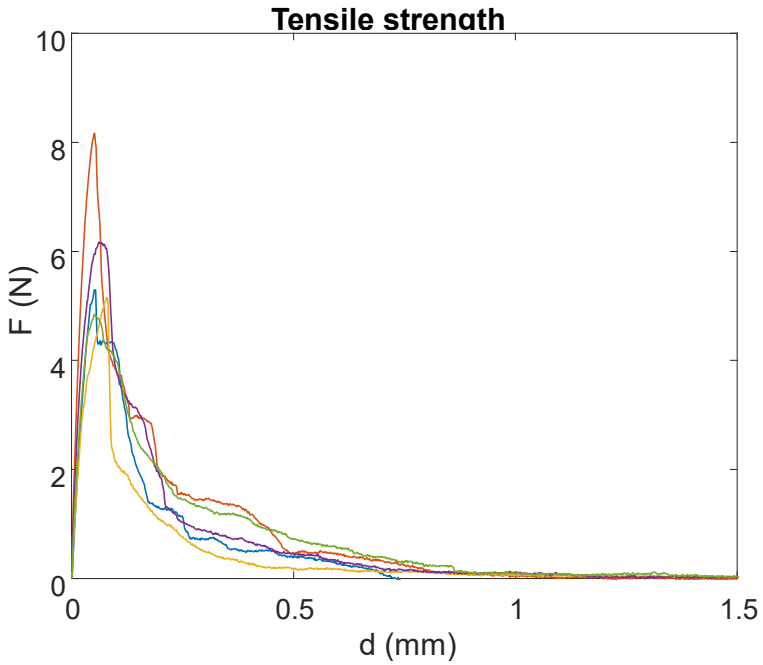

Fig. 5 Uniaxial tensile strength *vs.* displacement for five vitreous biomaterial specimens.

C. Analysis and discussion

From Fig. 5, it can be seen that the tensile force has a higher reproducibility than the compressive force in the CU tests. First, we observe a continuous increase of the force with distance up to $d \approx (0.05–0.08)$ mm, corresponds to a percentage deformation of (0.4-0.6) %.

From this point the force drops monotonically to an extension of about 1 mm. The corresponding deformation is ~6 %, although this does not correspond to an effective deformation of the vitreous biomaterial. The drop, while not as steep, is more pronounced than the similar drop observed in the CU tests. In addition, it occurs earlier. Finally, the failure is more singular than in the CU test. Indeed, in the CU test, the initial rise and fall reach an extension in compression of about 1 mm, while in the TU test they are located between 0 and 0.2 mm. The subsequent monotonic drop can be interpreted as the result of a series of minor rupture events, in which, unlike the compression tests where the vitreous biomaterial is increasingly confined between the compression platens, now, as the extension progresses, the biomaterial can only break more and more. We can interpret the sequence of two behaviors as i) a response resulting from the strength that allows the vitreous biomaterial to deform in an elastic, plastic or viscous manner.

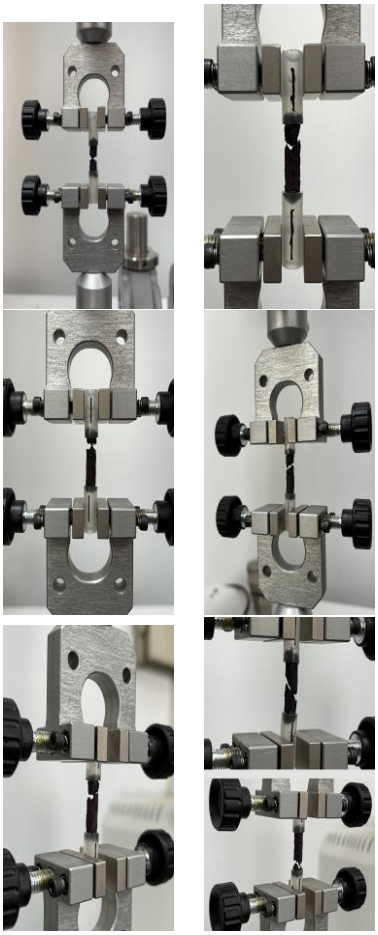

Fig. 6 Post-mortem vitreous biomaterial specimens corresponding to the five TU tests (a-e) and two specimens not considered in the analysis because they are in the initial tensile state (f).

(ii) the formation of a major failure from which the tensile response only decreases monotonically until the vitreous biomaterial splits in two. As for the CU test, in regime (i) additional tests are needed to determine the degree of elasticity, plasticity or viscosity.

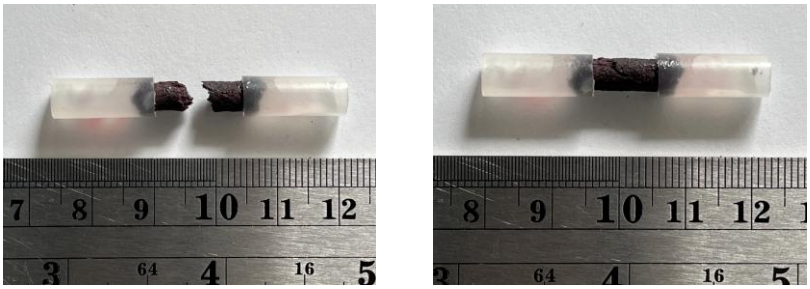

Fig. 7 Post-mortem vitreous biomaterial test tube (left) and reattachment (right) to demonstrate the quasi-fragile nature of the tensile rupture.

The drop in strength cannot be identified as being characteristic of a brittle material. However, when the two parts of the vitreous biomaterial unit are joined together, the unit looks, in appearance, the same as it did before the test (Fig. 7). This means that there was no permanent deformation, at least at the visible scale. This behavior is characteristic of brittle materials. However, since no abrupt drop in strength was observed, one could speak of quasi-fragile behavior in traction.

Tensile strength

From the results of the five tests it is possible to obtain a measure of tensile strength, determining for each test the maximum force reached between the regimes (i) and (ii). The resistance obtained, with its standard deviation, is $< F_{max} >$ = (5.9 ±1.3) N. From this we determine the tensile strength as follows

$$< \sigma_t >= \frac{4 < F_{max} >}{\pi D^2},$$

which gives $< \sigma_t >$ = (7.5 ± 2.0) MPa. This strength value is similar to that of the brick ($\sigma_t \approx$ 5 MPa).

IV. DISCUSSION (COMPARATIVE ANALYSIS)

Having subjected the vitreous biomaterial to SCS, CU and TU tests, a comparative analysis between the three is worthwhile. The CU test shows that the vitreous biomaterial is more brittle in uniaxial compression than in diametral compression. Indeed, in a SCS test, it is not possible to identify a clear fracture event as would occur in a brittle material (see first report).

On the contrary, in the five CU tests performed, a significant drop in strength is observed, followed by a regime where the strength is on average constant, which could be described as a pseudo-plastic behavior, in which the strength of the material does not increase on average with deformation, which is indicative of irreversible deformation and failure, although to verify this it would be necessary to perform additional tests to rule out deformation rate-dependent effects such as viscosity. This behavior differs from the behavior of the vitreous biomaterial in diametral compression, where, in global terms and in spite of slight dips and rises, product of successive random failures, the force increases monotonically. The difference between the two tests could be explained by the greater disintegration experienced by the vitreous biomaterial in the CU test. To better understand the processes responsible for the observed differences, it would be necessary to perform CU tests with different h, particularly with $h \sim D$ and $h < D$, for which it is more difficult to induce buckling events, which apparently tend to splinter the vitreous biomaterial (Fig. 3). In the SCS test, it appears that compression breaks and rejoins the vitreous biomaterial, which could explain the force fluctuations superimposed on a monotonic increase.

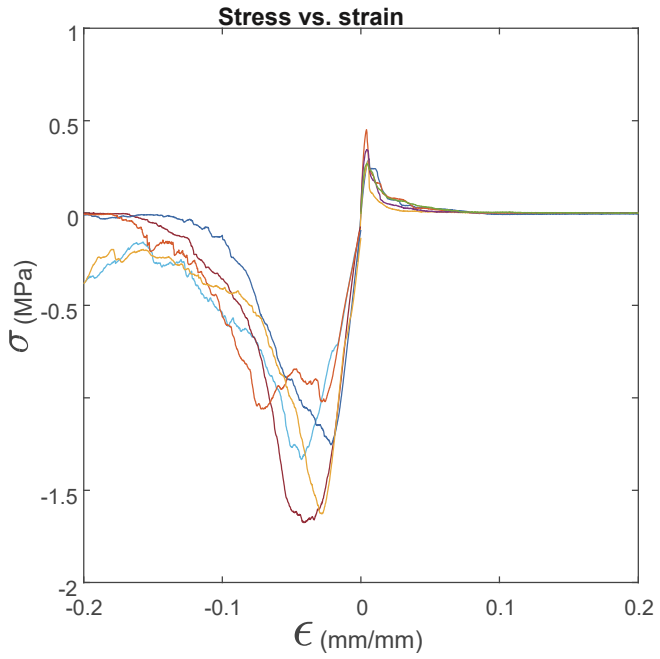

Fig. 8. CU and TU tests in a single stress-strain graph.

In the TU test, the behavior of the vitreous biomaterial is even more brittle than in the CU test, which indicates an asymmetry between compression and tension. To show this more clearly, in Fig. 8 the compressive (CU tests, Fig. 1) and tensile (TU tests, Fig. 5) force responses have been combined in a single stress-strain graph. For this it was necessary to adjust the extension offset of the compression tests because at the beginning of the compression there is an error associated with the fact that the vitreous biomaterial faces are not perfectly flat and require a small˜ or adjustment time. The stress is defined as σ= F/A, where A= $\pi D^2/4$ is the cross-sectional area of the vitreous biomaterial specimen; the strain is given by $\epsilon = d/h$. The higher compressive strength and the higher tensile brittleness are clearly seen.

It is interesting to compare the elastic stiffness in the two tests. It can be seen that the tensile stiffness is higher than the compressive stiffness. An estimate gives E_- ~ 57 MPa for compression while E_+ ~ 110 MPa for tension. In Williams et al [8] the Young's modulus obtained in an SCS test is compared with that of a CU test for vitreous biomaterial. The comparison is obvious that it is difficult to extract Young's modulus from an SCS test because

the contact area during compression increases in a non-linear way due to the cylindrical shape of the vitreous biomaterial. On the other hand, it is much more reasonable to compare Young's modulus between a CU test and a TU test, since in both tests the nominal cross section of the specimen does not change during the test.

V. CONCLUSIONS

The diametral compression, uniaxial compression and uniaxial tension tests allow us to affirm that the mechanical behavior of the vitreous biomaterial presents different degrees of brittleness, depending on the type of load. The vitreous biomaterial is more brittle in traction than in uniaxial compression, while it is less brittle in diametral compression than in uniaxial compression. This is indicative of a material with complex mechanical characteristics. Along the lines of Smith et al [2] and Procopio et al [3], it can be said, in the light of the CU and TU tests, that the vitreous biomaterial has quasi-fragile behavior. Once the compressive or tensile strength is reached, the strength decreases progressively and not abruptly as in brittle materials. This behavior is indicative of a progressive weakening of the material. However, it is much faster in tension than in diametrical compression, where, as mentioned above, failure and vitreous biomaterial reconstruction events are likely to occur simultaneously.

The initiation and propagation of a fracture in the vitreous biomaterial depends on the presence of defects: the larger the defect, the lower the strength (and vice versa) and therefore the load required to initiate a fracture [9]. When the number of defects is important, as seems to be the case of the vitreous biomaterial, many fracture events occur at higher and higher stresses. While the measured compressive or tensile force decreases, the effective cross-section that holds the vitreous biomaterial together can generate increasing stresses as the area of the cross-section decreases with the progressive breakage of the vitreous biomaterial. This provides the conditions for new fractures to initiate in smaller and smaller defects.

The tests suggest that the defects and inhomogeneities in the failure and vitreous biomaterial reconstruction events are likely to occur simultaneously are of size˜ or much larger than its elementary constituents (grains and cellular structures). If this were not the

case, a higher reproducibility of the assays would be observed. In order to physically interpret the results, it would be necessary to do more tests by i) increasing the number of repetitions, ii) varying the length of the specimens and iii) using different deformation rates. Under milling conditions, the vitreous biomaterial is subjected to mixed loads where compressive loads should be largely predominant and of sufficient magnitude to be considered non-linear (beyond the elastic regime). Moreover, due to the inhomogeneities of the vitreous biomaterial, these stresses are inhomogeneous and anisotropic [9]. In conclusion, the failure of the vitreous biomaterial is complex, gradual, with multiple fractures and internal friction. Mechanical properties such as Young's modulus, strength and brittleness are test dependent. However, each type of test contributes information to the mechanical characterization of the vitreous biomaterial.

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