Technical Report 3

Bulk compression testing of grape pomace vitreous biomaterial

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Abstract

The results of bulk compression tests of grape pomace and, a vitreous biomaterial obtained through Powder House's Vitreous Transformation Process of grape pomace, are reported, with the objective of comparing the grindability of the two materials.

I. INTRODUCTION

Previously, results of tests on diametral compression [1], uniaxial compression [2, 3] and uniaxial tension [4] of a grape pomace vitreous biomaterial have been reported. The information provided by these mechanical tests can be relevant to interpret the results of crushability or grindability of the vitreous biomaterial.

Grindability is a fundamental property in industrial processes where a fine powder is desired [5]. In physical terms, grindability is a composite property that encompasses other specific properties such as hardness, strength, toughness and fracture toughness [6]. Indeed, during the milling process, the vitreous biomaterial is fragmented by mechanical causes. The efficiency of the process is given by the amount of energy required to grind a certain amount of material [7]. This means that by reducing the energy required to disintegrate a vitreous biomaterial, less energy will be required to grind it in bulk. The property that determines the energy to break a material is *toughness*. It corresponds to the area under the stress-strain curve of a material before breakage. Stiff and brittle materials have, in general, a low toughness, since they do not undergo plasticity before breaking. Thus, a brittle behavior of a material contributes to a higher grindability.

The SCS, CU and TU tests reported above correspond to tests applied to individual vitreous biomaterial. This report presents the results of *bulk crushing strength* (BCS) tests on vitreous biomaterial and BCS tests on the raw pomace from which the biomaterial is made. The information provided by a BCS test should be more indicative of how the vitreous biomaterial fails under in-use conditions [8]. The objective is to compare the grindability of both materials, based on the strength response of each.

From the SCS, CU and TU tests, it is concluded that the vitreous biomaterial is quasi-fragile and that this property manifests itself to a progressively greater degree in the following order: 1) SCS, 2) CU, 3) TU. Of the three tests, SCS and CU are particularly relevant to the milling process. In the CU test, it is observed that once the compressive strength is reached, the strength, although it does not decrease abruptly as would be the case for a brittle material, decreases substantially, ruling out a ductile behavior. After this drop, a fluctuating force is observed, suggesting the existence of a cascade of failure events where the vitreous biomaterial disintegrates more and more (see second report).

II. METHODOLOGY

A. Sample holder

For the BCS test it was necessary to fabricate a device that would allow a compression to be applied to a bulk material. Based on ASTM D7084-18 (Standard Test Method for Determination of Bulk Crush Strength of Catalysts and Catalyst Carriers) a simplified device was manufactured consisting of a plastic sample holder and a metal piston, both of which are shown in Fig. 1. The sample holder has a cylindrical cavity with a diameter of 50 mm and a depth of 50 mm.

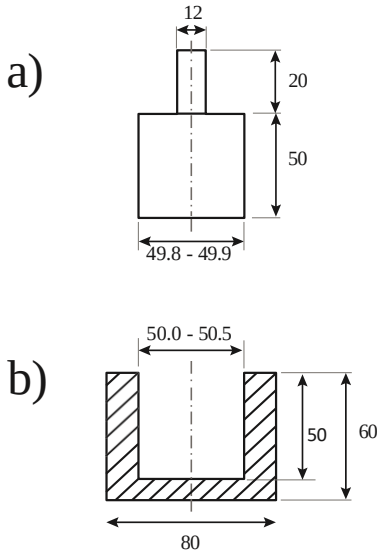

FIG. 1. BCS test apparatus: a) Piston; b) sample holder.

B. Tests

For the BCS tests, an Instron 3365 tensile/compression machine was used, equipped with a 2.5kN load cell. A 56 mm diameter compression stage is mounted in the fixed part and the piston (Fig. 1a) is attached to the load cell in the movable part (crosshead). The sample holder (Fig. 1b) with the bulk material inside is placed on the platen. Before placing the material in the sample holder, the inner walls of the sample holder are cleaned. Special care is taken to ensure that the material is flush. In the case of vitreous biomaterial, small grains that are likely to have been dislodged by transport and handling are left out. The piston is initially positioned 60 mm from the bottom of the sample holder. The piston is lowered in two steps: first a fast lowering (v = 120

mm/min) to manually fit the sample holder to the piston and to bring the piston within a few millimeters of the bulk sample; then a slow lowering (v = 1 mm/min, the same as in the SCS, CU and TU tests) to perform the BCS test itself. Software is used to record the crosshead position d and the compression force F, with a frequency of 100 measurements per second. Three (3) tests are performed with three vitreous biomaterial bulk samples and three tests with three pomace samples.

III. RESULTS

A. Compression at empty sample holder

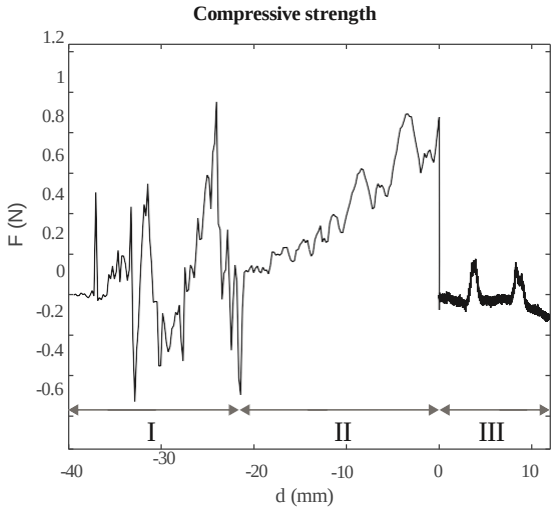

FIG. 2. Force measured in test with empty specimen holder. The crosshead is lowered in two stages with two different speeds: I and II) v = 120 mm/min; III) v = 1 mm/min.

First, a test was carried out with the sample holder empty, in order to evaluate the quality of the manufactured device and to quantify the effects of friction: being a closed sample holder, the only air evacuation path during compression is the gap between the piston and the sample holder. The force measured for this test is shown in Fig. 2 and can be divided into three stages (I-III). The force fluctuations in (I) are due to the piston being manually wedged in the specimen holder. In (II), the measured force suffers from the effects of the lubrication resistance between the piston and the inner wall of the sample holder. Finally, in (III), the force decreases considerably, remaining in the range of ±0.2 N. It is in this range of displacement that compression of the bulk material occurs. This preliminary test shows that the effects of friction during compression of the bulk material are of very small magnitude, at the chosen compression speed.

B. Compression tests

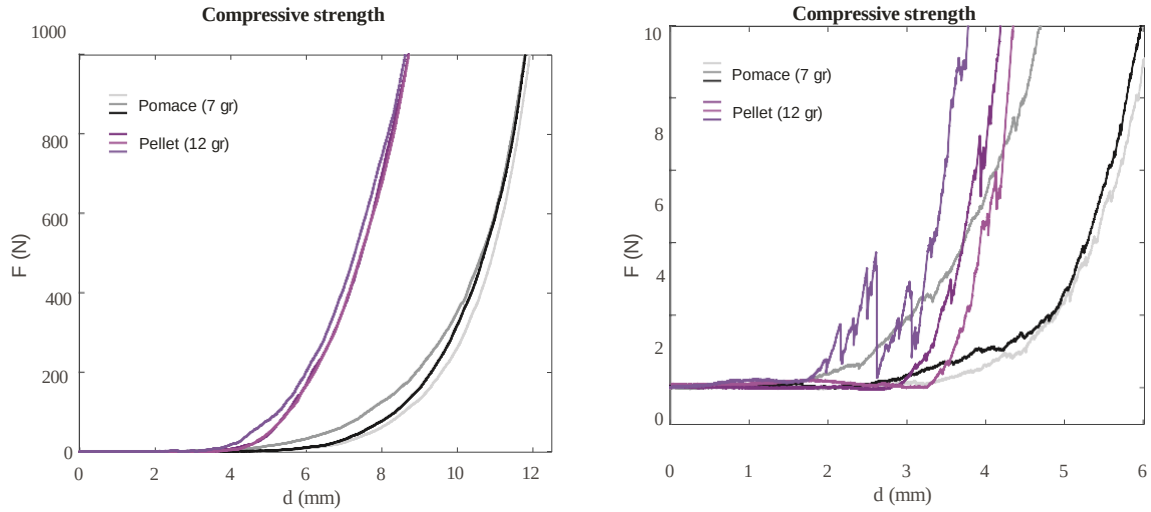

FIG. 3. Compression force at equal volume: 7 g of pomace (3 tests, gray scale) and 12 g of vitreous biomaterial (3 tests, lilac color scale). a) Force measured up to 1000 N; b) Detail of force measured up to 10 N.

A height $h \approx 16$ mm of the material layer, lower by a factor of 3 than the inner diameter of the sample holder, was chosen for the tests. This choice is due to the fact that the load on the material layer decreases with the distance from the surface where the load is applied, to the point that after a certain depth, the sample holder walls bear all the load [8] (Janssen effect). A simple estimate gives a ratio between the bulk densities of the vitreous biomaterial and the pomace of 1.7. This means that if 12 g of biomaterial are used, then about 7 g of pomace are required for the height of both bulk materials to be approximately the same inside the sample holder.

In Fig. 3, the *F vs. d* responses of the compression tests performed on equal volumes of vitreous biomaterial and pomace are plotted on the same graph. In all tests, a crosshead lowering speed of 1 mm/min is used. The test stops automatically when the force reaches 2000 N or the displacement, 52 mm. Fig. 3a shows that the compression force of the vitreous biomaterial is much higher than that of the pomace at the same distance of compression. The curves for each material are clearly distinguishable in spite of the scatter for each material. In Fig. 3b the force in the range of 20 N is plotted to appreciate what happens at the onset of compression. First of all, in this force range a greater relative dispersion is observed which tends to superimpose the vitreous biomaterial and pomace force curves. However, they are distinguished by a higher average rate of increase of the compressive strength of the vitreous biomaterial compared to the pomace. Finally, it can be seen that the force fluctuations for the vitreous biomaterial are of much greater amplitude than those for the pomace.

IV. ANALYSIS AND DISCUSSION

In the tests, a large variability of the measured strength was observed, despite the use of identical mass quantities with an error of ±0.1 g for each material. This is especially evident for the pomace. More tests would be needed to investigate this point further. For the pomace, a monotonic increase in compressive strength is observed, even at small forces (Fig. 3b), with small fluctuations in strength, hardly identifiable as fracture events; rather, they could be due to relative displacements and deformations of the particles (Fig. 3b).

relative displacements and deformations of the constituent particles of the pomace (skin, seeds, fiber, etc.) without fracture. In the vitreous biomaterial, the force fluctuations up to a deformation of about 12% resemble the same fluctuations observed in the individual vitreous biomaterial tests. In fact, their characteristic amplitude is of the same order of magnitude (1 N) as those undergone by the individual vitreous biomaterial in the uniaxial compression (UC) tests reported earlier. In contrast, the fluctuations experienced by the pomace in the same range of forces have a typical amplitude of an order of magnitude lower (0.1 N).

The higher rate of increase in vitreous biomaterial compressive strength at the beginning of the test is probably due to the fact that the bulk density of the vitreous biomaterial is higher than that of the pomace. Being more compact, the vitreous biomaterial is expected to exhibit a higher stiffness when compressed. On the other hand, at high compression a regime similar to that described in the two previous reports is reached, where the vitreous biomaterial, or the pomace in this case, is progressively compressed with a consequent substantial increase in the compression force (Fig. 3a). However, there is an important difference when removing the (crushed) material from the sample holder. In the case of the pomace, it can be removed very easily, with no evidence of fragmentation or agglomeration. Vitreous biomaterial, on the other hand, are visibly crushed and agglomerated. When trying to remove the material from the sample holder, the vitreous biomaterial disintegrates easily and small grains (fragments) that were not present before the test are observed.

V. CONCLUSIONS

BCS tests indicate brittle behavior of the vitreous biomaterial, totally absent in the raw pomace, at least at the onset of compression, within a range of deformation in compression of approx. 12 %. The higher amplitude of the force fluctuations in the vitreous biomaterial compression indicate fracture events characteristic of a material that breaks in a brittle manner. As several vitreous biomaterials are compressed at the same time, such events correspond to fractures of several individual vitreous biomaterials, resulting from mutual contacts. Above a certain level of compression, the vitreous biomaterial is left in conditions that do not correspond to those of a milling process. Indeed, in the BCS test, the available volume is reduced in the same proportion as the piston displacement. On the other hand, in a milling process, the available volume remains constant and the vitreous biomaterial is left in conditions that do not correspond to those of a milling process.

is subjected to mixed loading where compressive loading should be largely predominant. Therefore, the conclusions that can be drawn from the initial stage of the BCS tests are applicable to the milling process in the sense that the vitreous biomaterial can fragment due to its brittleness, unlike the raw pomace, which cannot, as observed under bulk compression conditions.

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