

# Report

# "Millability analysis: Dried grape pomace versus grape pomace vitreous biomaterial"

Prepared by:
Pia Lois-Morales
Assistant Prof.

Client: Biograpes SpA Santiago Elgueta

Date: 09/01/2023



### Department of Mining Engineering (DIMin), Universidad de Chile

Universidad de Chile has a long history in forming Mining Engineers, a tradition that started in 1853. The Department of Mining Engineering is responsible for the Mining Engineering Degree Program, Master programs in Mining and the Doctorate Degree Program in Mining Engineering. The Department has a prestigious academic staff, conducting research in areas such as mineral resource evaluation, geostatistics, mine planning, mining technology, rock mechanics, mineral processing, hydro-electrometallurgy, pyrometallurgy, sustainability and mineral economics, and collaborates with the Advanced Mining Technology Centre in R&D and in the generation of technologies for the mining industry. The Department of Mining Engineering has an active Continuing Education program, with Diplomas in the different areas of the mining, metallurgical process, and minerals economics. DIMin is ranked 1st in Chile and in Latin America in mining and minerals engineering, 8th in QS Ranking, and ranked 43rd in Shanghai Ranking of 2022.

Learn more by visiting minas.uchile.cl



#### **EXECUTIVE SUMMARY**

It was requested by the client to design and apply a milling protocol to two biomaterials coming from grape pomace. Sample 1, pomace obtained after a drying process and Sample 2, vitreous biomaterial obtained through Powder House's Vitreous Transformation Process of grape pomace.

A grinding protocol was designed in an ESSA pulverizer with a 3.7 kg steel ball charge, which consisted of a sequence of grinding at different times (0.5 to 24 min) for previously homogenized and quartered grape pomace of both samples.

Results indicate that sample 2, vitreous biomaterial, has a considerably different behavior than sample 1, dry pomace. A twenty times greater size reduction is observed in the case of sample 2, when using the same energy and ball load with both samples. This result implies considerable energy savings in the dry pomace milling process when in vitreous biomaterial form.

The results presented in this report indicate that there is a considerable change in milling behavior associated with the Vitreous Transformation Process of grape pomace that results in a twenty times faster size reduction in the case of the vitreous biomaterial.



#### 1. BACKGROUND

During the visit made by the client Santiago Elgueta of Biograpes & Fruits SpA to the Mining Engineering Department of the University of Chile, the company's need to carry out a comparative millability test of two biomaterials made from grape pomace was discussed. The first one (Sample 1) corresponds to dry pomace, while the second one (Sample 2) corresponds to dry pomace that has been processed through a Vitreous Transformation Process.

The mineral processing laboratory of the University of Chile, directed by Professor Dr. Pia Lois, is requested to carry out a test methodology to evaluate the effect of vitreous transformation on the grindability of grape pomace.

#### 2. OBJECTIVES

- To design an experimental methodology to determine the degree of millability that can be applied to both samples.
- To compare the results obtained in both samples, through the application of the experimental methodology developed.

#### 3. SAMPLES RECEIVED AND EXPLORATORY TESTS

To carry out the experimental plan, 1675 g of dry pomace sample (Sample 1) and 639 g of dry vitreous biomaterial sample (Sample 2) were received. Both samples were homogenized by quartering and rolling and cut into 100 g bags. Since there was a larger quantity of sample 1, the experimental plan was designed using this sample. The initial granulometry of the samples is as follows (Figure 1)

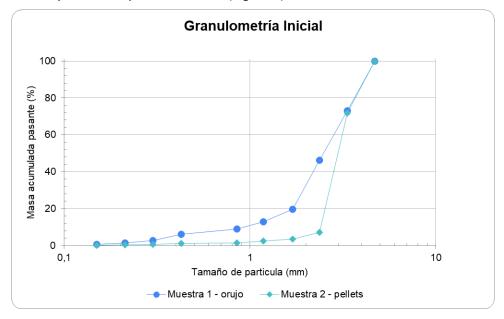


Figure 1: Initial granulometry of samples 1 and 2.



It is obserbed that, for both samples, 80% of the particles are under 3.7 mm. However, sample 2 is slightly coarser and equigranular than sample 1. For the sample grindability study, an ESSA LM2 vibratory pulverizer was used (see Figure 2). This mill has a power of 2.2 kW and is equipped with a pot that allows processing up to 1.8 kg of biomaterial. This pot can also incorporate a variable grinding media load (e.g. balls, steel disc), which is adjusted according to the experiment.



Figure 2: ESSA pulverizing equipment and holding vessel

A capacho of Sample 1 was placed in the pot and several grindability tests were carried out. First, a 3 min grinding was performed with a set of ceramic balls of 1.6 cm in diameter with a total weight of 250 g, where the material was obtained with a low grindability. Subsequently, another sample 1 was tested by grinding it with a 5 kg metal disc for 1 min, obtaining a result close to 300  $\mu$ m at one minute of grinding as shown in Figure 2.

As the objective was to observe how the granulometry of the samples varied and it was also visually observed that sample 2 was more fragile, it was decided to look for an intermediate weight between the ceramic balls and the disc to carry out the experiment. The selected weight was 3.8 kg made up of a load of steel balls distributed in: 54 balls of 15 mm, 31 balls of 16 mm and 22 balls of 28 mm. A 1 min grinding test was carried out with the defined load of balls.

The results of the initial tests carried out with the dry pomace sample (Sample 1) plus its initial particle size are presented below in Figure 3 and Table 1.



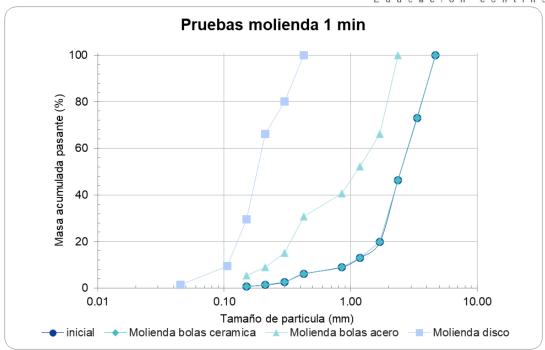


Figure 3: Initial milling test results

Table 1: Grinding results obtained with dry pomace (Sample 1) in the initial tests.

Method	Size 80% through (mm)
Initial	3.69
Disc (1 min)	0.30
Steel balls (1 min)	1.97
Ceramic balls (1	
min)	3.65

The above results show that by using the steel ball charge, an intermediate particle size is obtained for the pomace sample, which will allow observing the evolution of the particle size curve in both the pomace sample (Sample 1) and vitreous biomaterial sample (Sample 2).

#### 4. EXPERIMENTAL PLAN DEVELOPED

According to the results obtained, the degree of grinding to be achieved is defined at a particle size close to 200  $\mu$ m, which is estimated to be achievable with 24 minutes of grinding in both samples.

The grinding times with the load chosen for sample 1 are 1, 3, 6, 12, 18 and 24 minutes. The grinding times with the charge chosen for sample 2 are 30 seconds, 1 minute, 6 minutes and 24 minutes. For sample 2, a shorter milling time was used since it is visually observed that the vitreous biomaterial sample was more fragile than the dry grape pomace.



In addition, after each of the milling times, a granulometric analysis of the samples was carried out by ro-tap sieving, for which meshes from 3.36 mm (6 mesh) to 0.075 mm (200 mesh) were used.

For the material accumulated below 0.15 mm (100 mesh), wet sieving was performed. That is, the sieve was washed with water to favor the passage of the fine material. In this process, differences in the behavior of the materials were also observed. Sample 1 (pomace) generated an oil that prevented the particles from passing through the fine sieves, since it formed lumps in it, preventing their passage through the sieve. This phenomenon was not observed in sample 2 (vitreous biomaterial); however, in sample 2, slight static was observed at the time of sieving. This phenomenon is probably associated with the grinding method used in this study from the friction produced between the steel balls and the ore particles. However, this does not generate a problem neither in the grinding method nor in the sieving of this sample (See Figure 4).





Figure 4: Milling result before screening. Left. Sample of pomace in lumps. Right: Sample of vitreous biomaterial result with static.

#### 5. RESULTS AND ANALYSIS

The results of the milling and sieving process are shown below:

# 5.1 Granulometry Sample 1 Pomace

Figure 5 shows how the particle size of sample 1 changes as the pulverizing time increases between 1 min and 24 min. The P80 and P50 indicators are used, which correspond to the particle size at which 80 and 50% of the mass is below this size (see Table 2). It is observed that the particle size decreases from a P80 of 3.69 mm to 290  $\mu$ m in 24 min of milling. This grinding time corresponds to 0.88 kWh.



It is also observed in the results that the main size reduction is generated in the first 6 minutes of milling, where the sample is reduced to 440  $\mu$ m, and then in the remaining 18 minutes the size only goes down to 290  $\mu$ m. This result is observed in Figure 6, where the slope becomes more horizontal after 6 minutes.

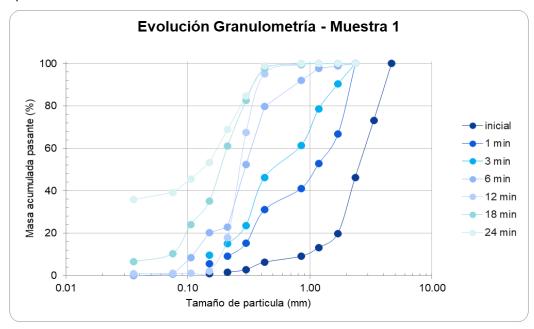
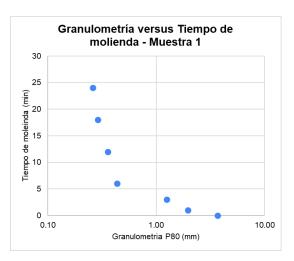


Figure 5: Evolution of the particle size of the pomace sample as the milling time increases.



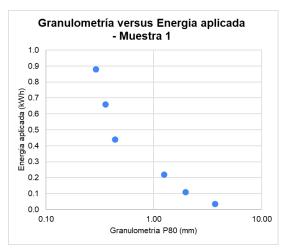


Figure 6: Granulometry of sample 1 versus different grinding times and applied energy.



Table 2: Results of particle size, sample 1

Grinding time (min)	80% through size (mm)	Size 50% through (mm)	Energy applied (kWh)
0	3.69	2.50	
1	1.96	1.10	0.04
3	1.25	0.53	0.11
6	0.44	0.29	0.22
12	0.36	0.27	0.44
18	0.29	0.19	0.66
24	0.26	0.12	0.88

#### 5.2 Particle size Sample 2 vitreous biomaterial

Figure 7 shows how the particle size of sample 1 changes as the grinding time increases between 0.5 min and 24 min. As in the previous sample, the P80 and P50 indicators are used, which correspond to the particle size at which 80 and 50% of the mass is below this size (see Table 3). It is observed that the particle size decreases from a P80 of 3.76 mm to  $110 \, \mu \text{m}$  in 24 minutes of milling. This grinding time corresponds to  $0.88 \, \text{kWh}$ .

It is also observed in the results that the main size reduction is generated in the first minute of milling, where the sample is reduced to 360  $\mu$ m, and then in the remaining 18 minutes the size only decreases to 110  $\mu$ m. This result is observed in Figure 8, where the slope becomes more horizontal after 6 minutes.

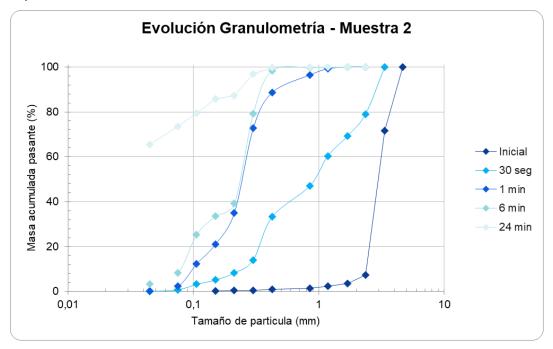
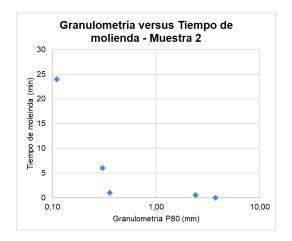


Figure 7: Evolution of the vitreous biomaterial sample particle size as the milling time increases.





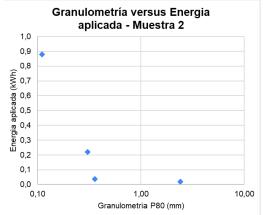


Figure 8: Granulometry of sample 1 versus different grinding times and applied energy.

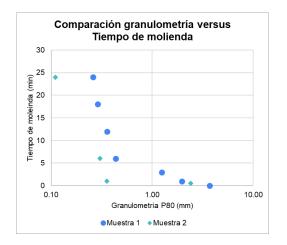
Table 3: Results of particle size, sample 2

Grinding time (min)	Size 80% through (mm)	Size 50% through (mm)	Energy applied (kWh)
0	3,74	3,02	
0,5	2,41	0,92	0,018
1	0,36	0,25	0,037
6	0,31	0,24	0,220
24	0,11	0,10	0,880

# 5.3 Comparison of Grindability of Samples

Figure 9 shows the progression of the particle size of the samples in time and according to the energy applied. It is observed that sample 2 (vitreous biomaterial) reaches smaller particle sizes in the same milling times and energy compared to sample 1 (pomace). In both samples a break in the size reduction slope is observed, which is at 6 minutes in the case of sample 1 and at one minute in the case of sample 2. This result implies that the grinding of sample 2, using the same ball charge and applied energy, is more efficient since a finer product is obtained with less grinding time than sample 1. In addition, it is observed that the reduction ratio of sample 1 is 14.17 and the reduction ratio of sample 2 is 34 times in the same unit of time with the same ball charge and energy applied in grinding.





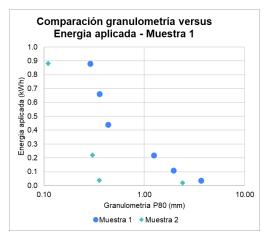


Figure 9: Particle size comparison of Sample 1 versus Sample 2 at different grinding times and applied energy.

# 6. CONCLUSIONS

The results of this study conclude that the vitreous biomaterial sample is considerably more brittle than the pomace sample when subjected to a grinding process. The vitreous biomaterial sample reduces twenty times more its size than the pomace sample (considering the reduction ratio as the quotient between the initial P80 and the P80 after the milling process established in this report).

Since the same milling times were used for both samples, which means the same energy input, the vitreous biomaterial sample is also more efficient in the energy consumption associated with the size reduction of this biomaterial in the milling equipment used.



# LOCATION AND CONTACT

University of Chile Department of
Mining Engineering
School of Engineering - Faculty of Physical and Mathematical Sciences
Beauchef 850 - Santiago Center

Anisse Pizarro

Head of Mineral Processing Laboratory e-mail:
apizarro@uchile.cl

Dr Pia Lois-Morales Assistant Professor.

e-mail: plois@uchile.cl