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end-of-life underground coal mines to  
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jobs

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### **Deliverable 3.1**

Dense fluids for pumped hydro



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The rheometric studies consisting on the determination of the stationary viscous and linear viscoelastic responses of the samples were carried out by Dr. Francisco José Rubio Hernández from the University of Málaga (Spain).

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## Executive Summary

This deliverable analysis the development of dense fluids required for unconventional pumped hydro by means of using fine coal waste from the three case studies of the project. The characteristic to be sought is a dense fluid with a density higher than three, in order to achieve a yield up to three times that of the conventional pumped hydro.

To achieve this goal, and after preparing the samples for the analysis, rheology studies consisting on determining the stationary viscous and linear viscoelastic responses of the samples were developed, using a MARS III controlled stress rheometer.

The rheometric test was specifically designed to obtain the stationary viscous response of the samples under study. The ability of the samples studied to dissipate and store the energy supplied to them, i.e. the determination of their viscous and elastic components, was obtained by performing an oscillating shear rheometric test (also known as mechanical-dynamic analysis).

The viscoelastic study was carried out to determine the characteristic time of each sample (relaxation time), which gives an idea of the dominant behaviour (viscous or elastic) depending on the duration of the mechanical action exerted on the material behaviour (viscous or elastic) as a function of the duration of the mechanical action exerted on the material.

Also, the pumping capacities of a high-density fluid material using the finite volume method (CFD) were analysed using ANSYS FLUENT software, version 2021 R1, focused on obtaining the mass flow curves as a function of pressure and pumping head and the maximum pumping head.

Finally, and using a sample of the test material, a pumping test was carried out in a lifting tower. The objective was to analyse the viability of this material for various applications in which, in all of them, it is necessary to pump it.

## 1 Introduction

Work Package No 3, “Deploying circular economy technologies”, mainly aims to analyse the deployment of circular economy technologies for the three case studies based on the valorisation of fine coal waste. Specific objectives are:

1. Assess the development of high-density fluids required for the unconventional pumped hydro.
2. Evaluate the development of soil substitutes to restore waste heaps using different combinations of fine coal waste with other industrial/organic waste.
3. Identify potential approaches for the concentration of mixed rare earth oxides.
4. Analyse the technical specifications, cost data and operational constraints of the selected alternatives for each circular economy technology.
5. Prepare a detailed assessment of the job creation potential of each alternative in terms of production capacity, both for commissioning and operation.

Task 3.1 Dense fluids for pumped hydro, led by M&B with the assistance of UNIOVI, is based on their previous cooperation within unconventional pumped hydro development and with the participation of the three coal mining companies: Węłokoks Kraj S.A. (WEGLO), Hulleras del Norte S.A. S.M.E. (HUNOSA) and Premogovnik Velenje d.o.o. (PV). Specific research was carried out using fine coal waste from the three case studies and suitable additives to develop the high-density fluids needed for the unconventional hydro energy storage system.

Low-density fraction separation by mineralurgical processes, as well as density, fluidity, water/slurry interface and remobilisation tailor-made tests, will be developed at TRL 5 (technology validated in a relevant environment, industrially relevant environment in the case of key enabling technologies) – TRL 6 (technology demonstrated in a relevant environment, industrially relevant environment in the case of key enabling technologies).

The characteristics to be sought for the fluid are a density  $>3$  kg/l, value retention after decommissioning, and 100% recoverable materials. At this density, the yield will be up to three times that of conventional pumped hydro, while the efficiency will be very similar. It will also be checked if the fluid is stable and can be remobilised.

Finally, technical specifications, cost data and operational constraints will be analysed, and a detailed assessment of the job creation potential of each case study in terms of dense fluid production capacity for both commissioning and operation will be prepared.

## 2 State of the Art

First, we compare the leading energy storage technologies. We intend to store clean energy, so the storage system must also be environmentally benign (Table 2-1).

**Table 2-1. Main energy storage technologies**

| TECHNOLOGY                                  | COST                               | EFFICIENCY | LIFETIME (CYCLES) | ENVIRONMENTAL IMPACT |
|---|------------------------------------|------------|-------------------|----------------------|
| H <sub>2</sub>                              | High                               | 25%        | ?                 | ?                    |
| Li-ion                                      | High                               | 70%        | 4.000             | High                 |
| Pumped Hydro (conventional)                 | Very Low                           | 80%        | 100K+             | High                 |
| <b>Unconventional Pumped Hydro, M&amp;B</b> | <b>Low or Very Low<sup>1</sup></b> | <b>80%</b> | <b>100K+</b>      | <b>Low</b>           |

<sup>1</sup> It is very competitive when dumpsite materials can be used in an existing cavern.

Because hydropower is a mature technology that has been providing dispatchable power since the very beginning of the electric era, it is the preferred solution for large-scale energy storage, with more than 90% of existing capacity, and it will most likely remain at the top, at least where topographic conditions are good. Conventional pumped hydro requires dams and reservoirs, which take away land for people and wildlife, so several initiatives can be considered unconventional pumped hydro besides Magellan & Barents:

### a) Hydrostor

This Canadian company uses water in a surface reservoir to compress air in an underground installation. It is already building commercial projects. Using a dense fluid would allow two or three times the power and energy storage capacity they achieve with water. <https://www.hydrostor.ca/>. The opportunities for synergies are considerable, particularly in their more advanced projects (Figure 2-1).

### b) Pyhäsalmi

The Pyhäsalmi energy storage project is a 75 MW/530 MWh project in a mine about 1450 meters deep. It got EUR 26.3 million from the Government of Finland in 2022 (Figure 2-2). The Pyhäsalmi project is exciting because it is developing high-head hydropower equipment very similar to our needs, with an excellent opportunity to share development and production costs. <https://www.epv.fi/en/project/a-pump-storage-station-for-pyhasalmi-mine/>. There are exciting overlaps; we are very interested in their ultra-high-head Pelton turbines, and can probably contribute to our water-tight solutions.



Figure 2-1. Scheme of Hydrostor, HYDROSTOR

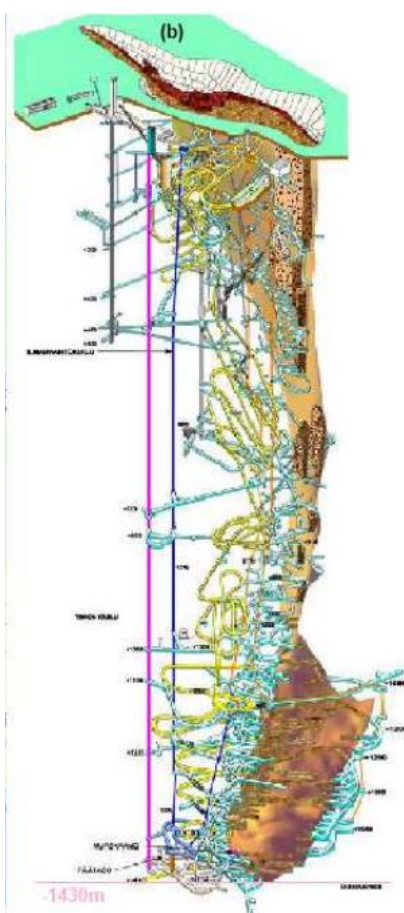
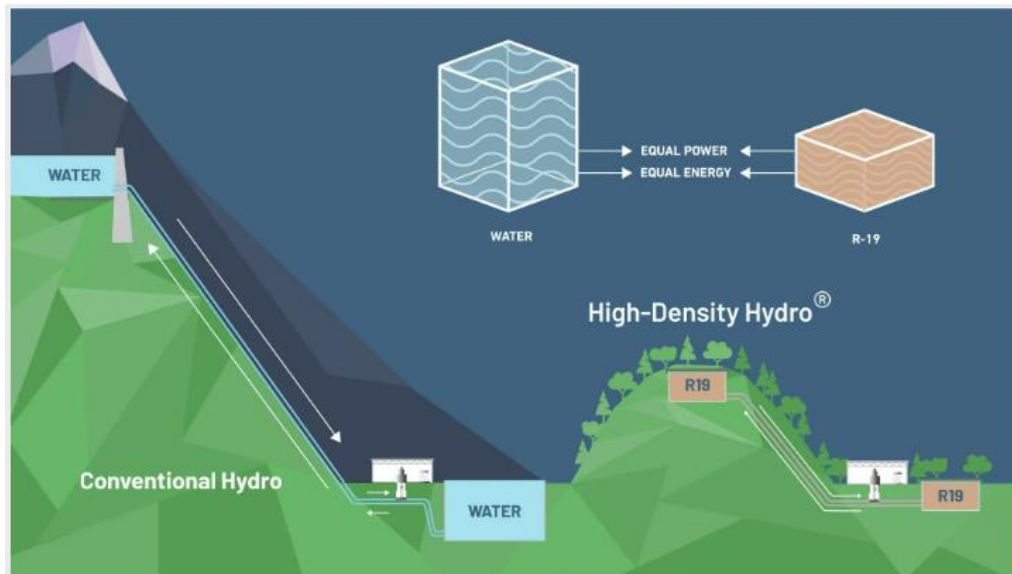


Figure 2-2. Scheme of Pyhäsalmi, EPV

**c) Rheenergise**

RheEnergy uses a dense fluid likely made from magnetite fines and water. The density is 2.5, which can be easily achieved (Figure 2-3). The company claims the reduction in tank or reservoir volume and associated civil works as their main advantage. <https://www.rheenergise.com/>



**Figure 2-3. Scheme of Rheenergise, RHEENERGISE**

**d) Shell International**

Shell prepared and patented a project in The Netherlands, EP0191516A1, but the project was cancelled after the 1986 Chernobyl NPP accident. It could not use any escarpments, so it needed two underground reservoirs for dense fluid (Figure 2-4). <https://patents.google.com/patent/US4691524>

These projects point to a diverse, disruptive, relatively mature technological field, attracting multimillion Euro investments in several countries. Typically for any R+D ecosystem, M&B’s technology acts as a force multiplier for some of those projects and benefits from specific developments by others.

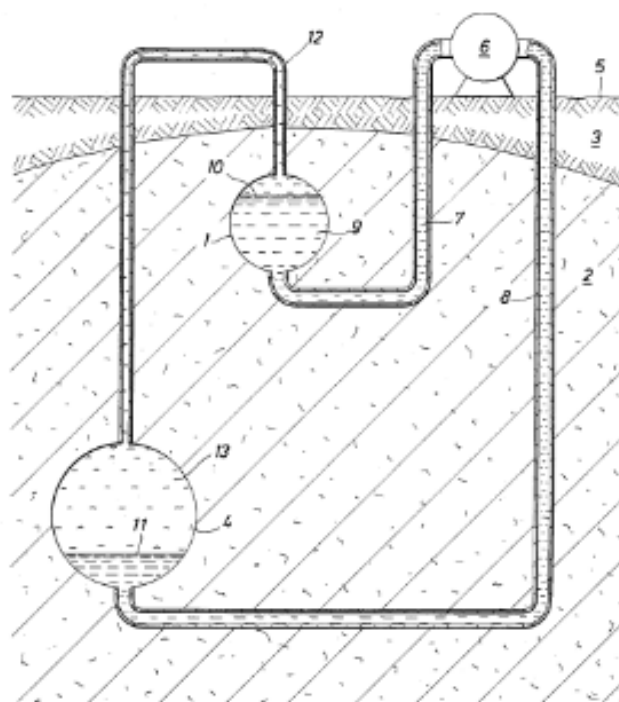


Figure 2-4. Scheme of Shell International, SHELL INTERNATIONAL

### 3 Unconventional pumped hydro storage characteristics

Renewable energies, such as those harnessed from the sun, wind and water, are popular forms of energy to generate electricity since they have minimal impact on our environment. For example, renewable energy does not pollute the environment such as CO<sub>2</sub> emissions.

Although renewable energy has advantages, there are also disadvantages. For example, renewable energy is highly dependent on nature, which is undependable or unreliable. Solar power requires sunlight, which can be affected by clouds; wind power relies on the wind, which can come and go; water power relies on water, which relies on a limited number of waterways and has numerous challenges. This unreliability or inconsistencies of renewable energy contribute to imbalances in supply and demand. Such imbalances cause huge swings in energy pricing.

Conventional pumped hydro energy relies on water flowing from an upper reservoir to a lower reservoir through a penstock. The water then turns into a turbine to generate electricity sent to the grid. Water is pumped up the penstock to recharge the upper reservoir. Pumped hydro energy storage, since it has, besides a turbine, a pump to recharge the system, provides controllability and reliability. This stabilises the imbalances of supply and demand, which are inherent in traditional renewable energy sources.

Furthermore, an essential consideration for conventional hydropower energy systems and pumped hydro storage is the footprint required by the reservoirs.

Unconventional pumped hydro storage is directed to a small footprint pumped hydro energy storage system and method with high power output.

Embodiments generally relate to an unconventional pumped hydro storage system and application of the pumped hydro storage system. The system has a smaller footprint and higher energy density than conventional pumped hydropower energy systems.

The system uses a high-density fluid and allows for different configurations where upper and lower reservoirs may be at the same elevation. Hydraulic pumps and turbines may be placed higher than the lower reservoir, for example, on the surface above an underground mine.

In particular, an embodiment relates to a pumped hydro storage system, which includes a first and second reservoir, disposed below the first reservoir. The system also includes a turbine unit. The turbine unit includes a first turbine unit flow port and a second turbine unit flow port.

A penstock is provided in fluid communication with the first and the second reservoirs. The penstock includes a first portion coupled to the first reservoir, the first turbine unit



flow port, and a second portion coupled to the second reservoir and the second turbine unit flow port.

The turbine unit is disposed of proximate to the second reservoir. A slurry circulates through the system. The slurry is a high-density fluid which has a density greater than water. The slurry flows through the turbine in a first or forward direction from the first reservoir to the second reservoir to cause the turbine unit to generate energy.

In the recharge mode, the slurry flows through the turbine unit in the second or reverse direction from the second reservoir to the first reservoir to recharge the system. The high-density slurry increases the system's power output compared to water systems.

The lower-density fluid flowing into the lower reservoir causes the turbine to turn in the first direction, generating power. To recharge the system, the pump pumps the lower-density fluid down to the cavity tank in the second direction, causing the high-density fluid to flow back into the upper reservoir.

Providing the high-pressure cavity tank below ground is advantageous as it can utilise the lithostatic pressure, thereby countering the pressure caused by the fluid. This reduces the construction costs of the lower reservoir. In addition, the mountain terrain provides a natural elevation for the upper reservoir. The height at which the upper reservoir is elevated can be configured based on output requirements. For example, lower elevations may be helpful to reduce costs associated with building the upper reservoir and penstock if output requirements are met.

The fluid of the pumped hydro storage system is a high-density fluid. The high-density fluid has a density greater than water. For example, the high-density fluid may have a density of  $> 3x$ , where  $x$  is the density of water. In one embodiment, the high-density fluid is a slurry mixture.

Various types of slurry mixtures may be employed. The slurry mixture may include, for example, metal oxide particles mixed with a lower-density fluid, such as water.

Other types of particles and lower-density fluids may also be helpful. The volume of particles in the slurry may equal or exceed 50%.

For example, the percentage of particles may be about 50 -85%. In other embodiments, the percentage of particles may be 50 - 75%. The higher the volume of particles, the higher the density of the slurry. All percentages are volume percentages. Other percentages may also be helpful.

A small surfactant may be added to prevent the slurry from coalescing and improve flow. For example, less than 1% of surfactant can be added. In some cases, antifreeze may be added to prevent freezing of the slurry. The concentration of antifreeze should be sufficient to prevent the slurry from freezing.

An example of a high-density fluid is a magnetite slurry mixture. The magnetite slurry



mixture may achieve a density of 3 to 4 tons/m, more than 3 times the density of water. Other types of slurry mixtures, as discussed, can also be employed as high-density fluid. The density may depend on the mineral content and composition.

A more compact pumped hydro energy storage system can be achieved by employing high-density fluid. For a given reservoir or tank volume, the energy storage capacity is proportional to the density of the fluid. For example, in the case where the high-density fluid has a density of 3x, the energy storage capacity of the system is 3 times that when water is used. This is because the mass flow rate is about 3 times more than water's.

Alternatively, the system can produce the same energy output using less fluid volume and a lower height differential between the upper and lower reservoirs. This results in lower costs and more flexibility in designing a system to satisfy output requirements.

An advantage, as discussed with using a high-density fluid, is higher power output. A high-density fluid can be easily retrofitted into existing pumped hydro storage systems by modifying the penstock and pump to handle the high-density fluid, thereby increasing the power output. Furthermore, existing designs of hydro storage systems can be modified to serve as models for highly efficient hydro storage systems which handle a high-density fluid. The cost to build, for a given power output requirement, would be reduced due to less volume needed, smaller penstocks and reduced elevation or height between the reservoirs.

## 4 Treatment of samples for initial analysis

This section will describe the procedure for preparing samples for initial analysis in an accredited laboratory. Two samples have been taken from company Hulleras del Norte S.A. S.M.E. (HUNOSA - Spain), one sample from company Węglokoks Kraj S.A. (WEGLO - Poland) and one sample from company Premogovnik Velenje d.o.o. (PV - Slovenia).

It is necessary to point out that in the case of HUNOSA, some samples have been taken since the El Batán coal preparation plant only carries out a pre-treatment by screening the material from the San Nicolás well, the company's only active mine.

To compare, two samples have been taken, one from the El Batán coal preparation plant and another from the old coal preparation plant tailings accumulated in the La Matona dump. Five bags of between 20 and 25 kg of material were taken from the El Batán coal preparation plant and as many from the La Matona dump.

### 4.1 Sample drying procedure and humidity calculation

The procedure, using the UNE 32-001-81 Standard, for drying the sample at laboratory temperature and calculating the **imbibition humidity (X)** was as follows:

- Place the material from a sample bag in an appropriate number of large trays, with a volume between 0.015m<sup>3</sup> (0.50m x 0.30m x 0.10m) and 0.013m<sup>3</sup> (0.51m x 0.34m x 0.075m), so that it dries in a time of approximately one or two weeks. Between 5 and 10 kg of the sample will be placed per tray.
- The quantity of 1 and 3 kg of sample is taken in a smaller tray, with a volume of 0.004m<sup>3</sup> (0.30m x 0.24m x 0.055m), to determine imbibition humidity.
  - The empty tray is weighed (P<sub>1</sub>), and its value is recorded.
  - The tray with the sample is weighed again (P<sub>2</sub>).
  - The weight of the empty tray is subtracted, and the weight of the sample (P<sub>3</sub>) is obtained.

$$P_3 = P_2 - P_1$$

- It is left to dry at laboratory temperature until the weight stabilises, checking every 3 days the variation in weight in grams and calculating the percentage of humidity lost. This process can take approximately 1-2 weeks.
- Once the weight stabilises, the imbibition humidity is obtained.

The procedure, using the UNE 32-001-81 Standard, for drying the sample in a laboratory drying oven and calculating the **hygroscopic humidity (M)** was as follows:

- An empty tray is weighed, and its value is noted (m<sub>2</sub>).
- The empty tray is placed in the oven at a temperature between 378 and 383 K (105 and 110°C), its air atmosphere is renewed 3 to 5 times per hour, and it is weighed again when hot, noting its new value (m<sub>4</sub>).

- The sample from the imbibition humidity is placed in the tray and distributed evenly over the entire surface (maximum 1 g/cm<sup>2</sup>). The tray plus the sample (m<sub>1</sub>) is weighed and placed in the oven to dry.
- It is kept at that temperature until constant mass. The drying time ranges between 3 and 6 hours.
- Once the weight of the tray plus the hot sample has stabilised, it is taken to the balance and its value (m<sub>3</sub>) is recorded.
- The hygroscopic humidity of the analysed sample is obtained employing the following expression:

$$M = \frac{(m_1 - m_4) - (m_3 - m_2)}{(m_1 - m_4)}$$

m<sub>1</sub> = mass, in grams, of tray and sample as received

m<sub>2</sub> = mass, in grams, of the empty tray

m<sub>3</sub> = mass, in grams, of tray and sample after heating

m<sub>4</sub> = mass, in grams, of the empty, dry tray

When the sample, as in this case, has been air-dried, the **total humidity (H<sub>T</sub>)**, in percentage, is calculated using the following formula:

$$H_T = X + M (1 - (X / 100))$$

X = Imbibition humidity in per cent

M = Hygroscopic humidity in per cent

## 4.2 Sample grain reduction procedure

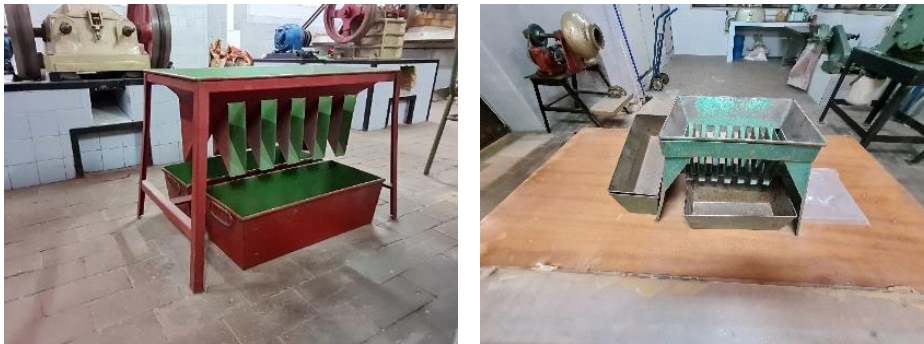
Next, once dry, the procedure used to reduce the grain of the samples to obtain an optimal size will be described.

The machines used are the following:

- Single-acting jaw crusher (Figure 4-1, left).
- Disc mill (Figure 4-1, right)
- Jones or channel sampler (Figure 4-2).
- Scales (Figure 4-2).



**Figure 4-1. Single-acting jaw crusher and Disc mill**



**Figure 4-2. Jones or channel sampler for coarse and fine**



**Figure 4-3. Scales**

Initially, the machines must be cleaned to eliminate any impurities from previous procedures that could contaminate the samples. Initially, the sample is passed through the jaw crusher, which crushes it, giving it a maximum size of 2 cm, optimal for introducing it into the disc mill, which reduces it to a maximum size of 5 mm. Once this size has been achieved, the desamplers will be used to obtain the appropriate sample size for analysis.

#### 4.2.1 Sample from the La Matona dump (HUNOSA)

Next, the drying procedure of the sample extracted from the La Matona of the HUNOSA dump will be described. Figure 4-4 shows the state of the La Matona sample.



**Figure 4-4. Initial state of the La Matona sample**

Initially, one of the bags, of approximately 20 kg of the sample, was placed in two large trays with a volume of 0.015m<sup>3</sup> each and the amount of 2526.60 g in another small tray, 0.004m<sup>3</sup>, which weighs 551.50 g when empty, as seen in Figure 4-4. After 4 days, the small tray was weighed again, after eight days it was weighed again, verifying that the weight had not yet stabilised. After 12 days, the same operation was carried out, observing the final stabilised weight and being able to obtain the imbibition humidity of the 12.6%. The results obtained are those presented in the following table (Table 4-1):

**Table 4-1. Variation in the weight of the La Matona sample and calculation of imbibition humidity**

| DATE                    | WEIGHT (g)  |         | VARIATION      |               |
|-------------------------|-------------|---------|----------------|---------------|
|                         | Sample+tray | Sample  | g              | %             |
| 28/02/2022              | 3075.10     | 2523.60 | 0.00           | 0.00%         |
| 04/03/2022              | 2830.40     | 2278.90 | -244.70        | -10.70%       |
| 07/03/2022              | 2792.10     | 2240.60 | -38.30         | -1.70%        |
| 09/03/2022              | 2787.70     | 2236.20 | -4.40          | -0.20%        |
| 11/03/2022              | 2787.70     | 2236.20 | 0.00           | 0.00%         |
| <b>TOTAL VARIATION:</b> |             |         | <b>-287.40</b> | <b>-12.6%</b> |



Before placing the sample in the tray for humidity calculation, it was placed in the oven at a temperature between 378 and 383 K (105 and 110°C) and said tray was weighed once dry, obtaining a value of 550.7g. This value is obtained to calculate the hygroscopic humidity, which turned out to be 0.9% once the formula was applied. The values used for the calculation are detailed in Table 4-2.

**Table 4-2. Calculation values of hygroscopic humidity in the sample of La Matona**

| CONCEPT                                      | SYMBOL | MASS (g) |
|--|--------|----------|
| Initial mass in grams of tray + sample       | $m_1$  | 2787.7   |
| Mass in grams of the initial empty tray      | $m_2$  | 551.5    |
| Mass in grams of tray + sample after heating | $m_3$  | 2768.3   |
| Mass in grams of an empty and dry tray       | $m_4$  | 550.7    |

Once the imbibition and hygroscopic humidity are obtained, the **total humidity** is obtained, which turns out to be 13.4%.

Next, once the sample was dry, it was passed through the jaw crusher, obtaining the optimum size for the disc mill. Figure 4-5 shows the different machines through which the La Matona sample was passed and the grain size obtained in the jaw crusher.



**Figure 4-5. Grinding treatment of the La Matona sample**

After grinding the material, it is passed through the large desampler and the finer one to obtain a sample bag for analysis (Figure 4-6).



**Table 4-3. Weight variation of the El Batán sample and imbibition humidity**

| DATE                    | WEIGHT (g)  |         | VARIATION     |               |
|-------------------------|-------------|---------|---------------|---------------|
|                         | Sample+Tray | Sample  | g             | %             |
| 28/02/2022              | 1923.30     | 1357.80 | 0.00          | 0.00%         |
| 04/03/2022              | 1845.20     | 1279.70 | -78.10        | -6.10%        |
| 07/03/2022              | 1844.50     | 1279.00 | -0.70         | -0.05%        |
| 09/03/2022              | 1844.70     | 1279.20 | 0.20          | 0.02%         |
| 11/03/2022              | 1844.50     | 1279.00 | -0.20         | -0.02%        |
| <b>TOTAL VARIATION:</b> |             |         | <b>-78.80</b> | <b>-6.16%</b> |

Before placing the sample in the tray for humidity calculation, it was placed in the oven at a temperature between 378 and 383 K (105 and 110°C) and said tray was weighed once dry, obtaining a value of 564.7g. Once the formula was applied, this value was obtained to calculate the hygroscopic humidity of 0.6%. The values used for the calculation are in Table 4-4.

**Table 4-4. Calculation values of hygroscopic humidity in a sample from El Batán**

| CONCEPT                                      | SYMBOL         | MASS (g) |
|--|----------------|----------|
| Initial mass in grams of tray + sample       | m <sub>1</sub> | 1844.5   |
| Mass in grams of the initial empty tray      | m <sub>2</sub> | 565.5    |
| Mass in grams of tray + sample after heating | m <sub>3</sub> | 1840.7   |
| Mass in grams of an empty and dry tray       | m <sub>4</sub> | 564.2    |

Once the imbibition and hygroscopic humidity have been obtained, the **total humidity** is obtained, which turns out to be 6.6%.

Next, once the sample was dry, it was passed through the jaw crusher, obtaining the optimum size for the disc mill. In Figure 4-8, you can see the difference in the grain size of the sample passed through the jaw crusher and after passing it through the disc mill.



**Figure 4-8. Grinding treatment of the El Batán sample**

After grinding the material, it is passed through the large sampler and the finer one to obtain a sample bag for analysis (Figure 4-9).





**Figure 4-9. Final sample of La Matona**

#### **4.2.3 Sample from the WEGLO dump**

Next, the drying procedure of the sample extracted from WEGLO will be described. Figure 4-10 shows the state of the WEGLO sample.



**Figure 4-10. The initial state of the WEGLO sample**

In this sample, it was not necessary to calculate the moisture content because it was scorched, and there was no variation in weight.

Next, the sample was passed through the disc mill, obtaining the optimum size for the analysis.

After grinding the material, it is passed through the large sampler and the finer one to obtain a sample bag for analysis.

#### **4.2.4 Sample from the PV dump**

Next, the drying procedure of the sample extracted from PV will be described. Figure 4-11 shows the state of the VP sample from Slovenia.



**Figure 4-11. The initial state of the VP sample**

Initially, one of the bags, of approximately 25 kg of the sample, was placed in 3 large trays with a volume of 0.013m<sup>3</sup> each and 518,3g in another small tray, 0.004m<sup>3</sup>, which weighed 567.10g. After three days, the small tray was weighed again. After seven days, it was weighed again, and the same after 14 days verifying that the weight had already stabilised and obtained imbibition humidity of 6.16%. The results obtained are those presented in the following Table 4-5.

**Table 4-5. Weight variation of the El Batán sample and imbibition humidity**

| DATE                    | WEIGHT (g)  |        | VARIATION     |                |
|-------------------------|-------------|--------|---------------|----------------|
|                         | Sample+Tray | Sample | g             | %              |
| 21/02/2023              | 1085.40     | 518.30 | 0.00          | 0.00%          |
| 24/02/2023              | 1031.80     | 464.7  | -53.60        | -10.34%        |
| 28/02/2023              | 1031.00     | 463.9  | -0.80         | -0.17%         |
| 14/03/2023              | 1031.00     | 463.9  | 0.00          | 0.00%          |
| <b>TOTAL VARIATION:</b> |             |        | <b>-78.80</b> | <b>-10.51%</b> |

Before placing the sample in the tray for humidity calculation, it was placed in the oven at a temperature between 378 and 383 K (105 and 110°C) and said tray was weighed once dry, obtaining a value of 565.7g. This value is obtained to calculate the hygroscopic humidity, which turned out to be 3.7% once the formula was applied. The values used for the calculation are in Table 4-6.

**Table 4-6. Calculation values of hygroscopic humidity in a sample from El Batán**

| CONCEPT                                      | SYMBOL         | MASS (g) |
|--|----------------|----------|
| Initial mass in grams of tray + sample       | m <sub>1</sub> | 1028.6   |
| Mass in grams of the initial empty tray      | m <sub>2</sub> | 567.1    |
| Mass in grams of tray + sample after heating | m <sub>3</sub> | 1012.7   |
| Mass in grams of an empty and dry tray       | m <sub>4</sub> | 565.7    |

Once the imbibition and hygroscopic humidity have been obtained, the **total humidity** is obtained, which turns out to be 13.1%.

Next, once the sample was dry, it was passed through the disc mill, obtaining the optimum size.

#### 4.2.5 Samples for Dense Fluids

Four 150 g samples with a grain size <0.1 mm were taken for the dense fluid and viscosity tests. The samples were labelled as shown in Figure 4-12:

- FD-ESL for the PV sample (Slovenia)
- FD-PO for the WEGLO sample (Poland)
- FD-MA for the La Matona sample (HUNOSA-Spain)
- FD-BA for the El Batán sample (HUNOSA-Spain)
- FD-PT-1 for one of the control standards
- FD-PT-2 for one of the other control standards



**Figure 4-12. Samples for Dense Fluid and Viscosity Tests**

## 5 Results of the rheology test

The object of the Rheometric studies consisted of determining the stationary viscous and linear viscoelastic responses of six samples. Dr Francisco José Rubio Hernández carried it out from the University of Málaga (Spain).

### 5.1 Rheometer

A MARS III controlled stress rheometer (Thermo-Haake, Germany) was used. (Figure 5-1). The span geometry is the one best suited to the characteristics of the samples supplied (Barnes & Carnalli, 1990). Figure 5-2 shows a picture of the span geometry and its dimensions. The spacing between the rotor and stator complied with the single shear condition ( $R_1=1,2R_2$ ). The spacing between the rotor and the bottom of the outer cylinder was four *mm*, complying with the condition that this distance is equal to or greater than ten times the particle size (0.1 *mm*) present in the problem fluid.

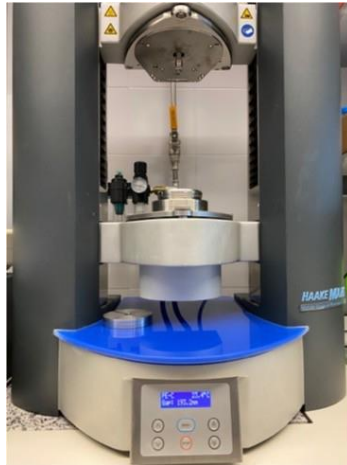


Figure 5-1. Controlled stress rheometer Haake MARSIII. Dr Francisco José Rubio Hernández. University of Málaga (Spain).

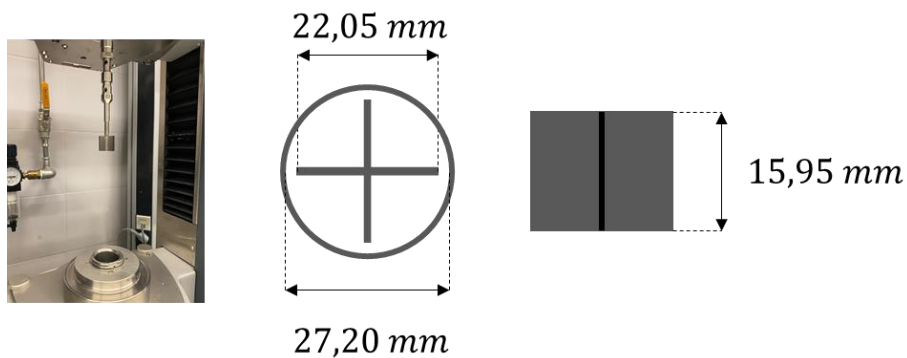
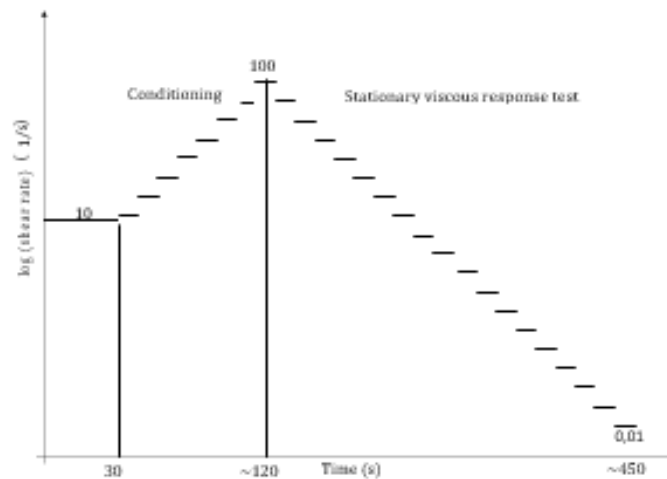


Figure 5-2. The geometry of spans. Dr Francisco José Rubio Hernández. University of Málaga (Spain).

## 5.2 Rheometric test

Figure 5-3 describes the rheometric test specifically designed to obtain the stationary viscous response of the samples under study. After a conditioning phase of the sample after being placed in the span geometry, a decreasing sequence of logarithmically distributed shear velocities (velocity gradient) is applied. The measured magnitude is the stress the specimen opposes to the rotation in the same shear direction. The accepted value for each shear rate ( $\dot{\gamma}$ ) is the stationary stress ( $\tau$ ), accepting as such the value that shows a variation of less than 1% for ten  $s$ .



**Figure 5-3. The test was designed to study the stationary viscous response. Dr Francisco José Rubio Hernández. University of Málaga (Spain).**

The apparent viscosity ( $\eta$ ) of the sample, corresponding to each shear rate, is calculated from its definition,

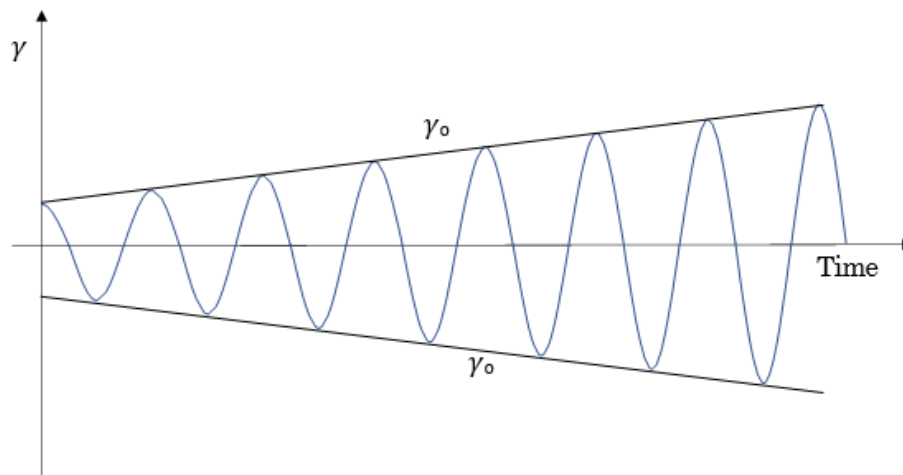
$$\eta = \frac{\tau}{\dot{\gamma}} \quad (1)$$

In the case of a Newtonian fluid, this viscosity, for a given temperature and pressure, will be constant. However, when the viscosity depends on the shear rate, i.e.  $\eta = \eta(\dot{\gamma})$ , the fluid will be non-Newtonian. It should be noted that there are other reasons why a fluid may be non-Newtonian (viscosity dependence on time or the fluid's ability to store energy). The test described in Figure 3 was designed to determine the dependence  $\eta = \eta(\dot{\gamma})$ .

The ability of the samples studied to dissipate and store the energy supplied to them, i.e. the determination of their viscous and elastic components, was obtained by performing an oscillating shear rheometric test (also known as mechanical-dynamic analysis),

$$\gamma = \gamma_0 \text{sen } \omega t \quad (2)$$

where  $\gamma$  is the shear strain applied to the sample,  $\gamma_0$  is the maximum value of this strain, and  $\omega$  is the angular frequency of the oscillation ([F.J. Rubio Hernández, *Flujos no-Newtonianos y Reología*, UMA Editorial, Universidad de Málaga, Málaga 2022]. The test consists of two parts. First, an amplitude sweep is performed on the deformation of the specimens, starting from a minimum value and increasing logarithmically in value while keeping the oscillation frequency constant (Figure 5-4).



**Figure 5-4. Amplitude sweep. The first part of the test was designed for the viscoelastic study of the samples. Dr Francisco José Rubio Hernández. University of Málaga (Spain).**

In this way, we can determine the value of  $\gamma_0$  that, at most, we can apply to the sample so that the microstructure it develops in the resting state responds linearly. In other words, whenever we study the response of the fluid in this region, it will be of the type,

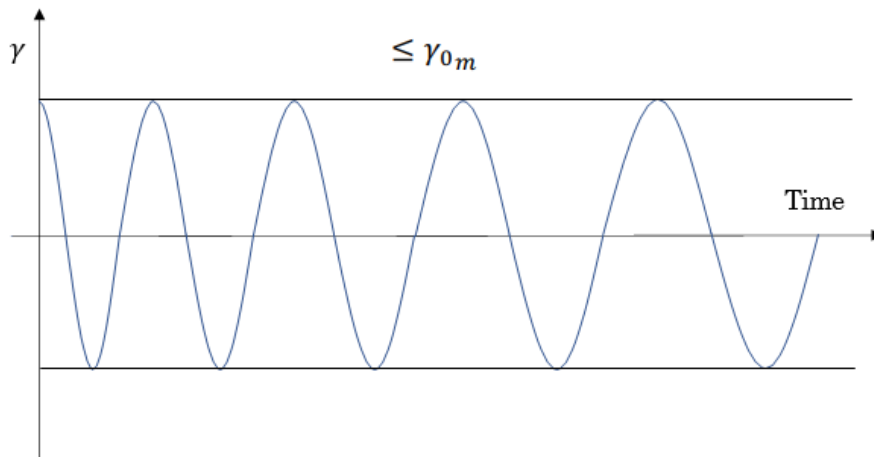
$$\tau = \tau_0 \text{sen} (\omega t + \varphi) \quad (3)$$

where  $\tau_0$  is the maximum stress supported by the fluid when a maximum deformation  $\gamma_0$  is applied, and  $\varphi$  is the phase difference between the harmonic signals given by (2) and (3).

Once the maximum value of the amplitude  $\gamma_{om}$  has been obtained, we proceed to the second part of the mechanical-dynamic analysis. In this case, keeping the oscillation amplitude below the value  $\gamma_{om}$ , a frequency sweep is performed (Figure 5-5). In this way, we will be able to know which component (viscous or elastic) of the viscoelastic response of the fluid dominates and in what proportion when carrying out short (high frequencies) or long-lasting (low frequencies) tests. The frequency value corresponds to a 50% split of both components of the viscoelastic response, and, more appropriately, the resulting time value is a characteristic parameter of the material (relaxation time). The viscous and elastic components are obtained from the development of the trigonometric function (3),

$$\tau = \tau_0 \cos \varphi \text{sen} \omega t + \tau_0 \text{sen} \varphi \cos \omega t \quad (4)$$





**Figure 5-5. Frequency sweep. The second part of the test was designed for the viscoelastic study of the samples. Dr Francisco José Rubio Hernández. University of Málaga (Spain).**

The first summand corresponds to the part of the stress in phase with the strain, i.e. it results from the elastic component of the fluid, while the second summand is in phase with the strain rate ( $\dot{\gamma} = \gamma_0 \omega \cos \omega t$ ), i.e. it results from the viscous component of the fluid. Consequently, two new rheological parameters are defined to quantify both components,

$$\begin{aligned} G' &= \frac{\tau_0}{\gamma_0} \cos \varphi \\ G'' &= \frac{\tau_0}{\gamma_0} \sin \varphi \end{aligned} \quad (5)$$

While  $G'$  gives an idea of the elasticity of the fluid (elastic modulus), the value of  $G''$  gives the importance of the viscous response of the fluid (viscous modulus).

### 5.3 Results

The experimental results show an average of at least three measurements in each case. A precondition for fluid under rheometric study is that it must be homogeneous. Since the samples presented a specific coagulation state upon receipt (Figure 5-6), they were manually shaken for at least 5 minutes before starting the rheometric studies. The reproducibility of the obtained rheometric results confirmed that the samples' homogeneity was achieved this way.





**Figure 5-6. The initial physical state of samples in the AMU rheology laboratory. Dr Francisco José Rubio Hernández. University of Málaga (Spain).**

## 5.4 Viscous response

The apparent viscosity curves are shown in Figure 5-7. In all cases and in the range of shear rates that were accessible (reproducible results), the viscosity decreases with increasing shear rate. This is the non-Newtonian behaviour known as rheo-fluidising (shear-thinning).

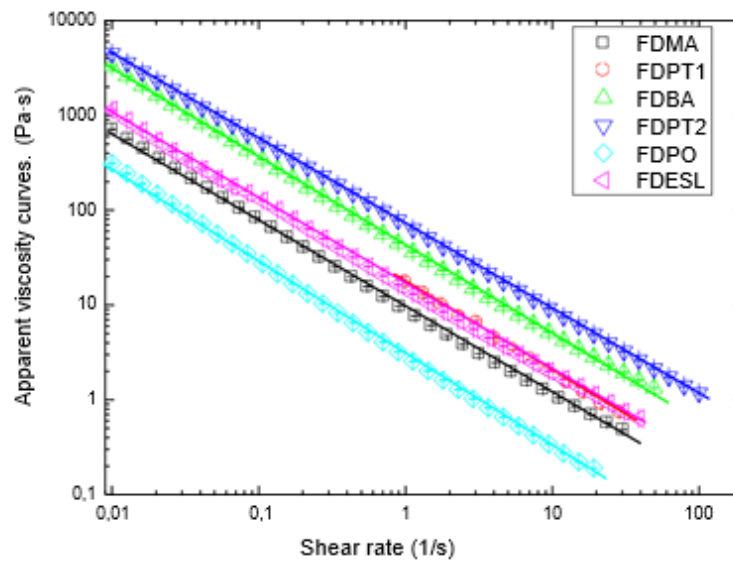


Figure 5-7. Apparent viscosity curves

The experimental results are presented in Appendix 1 and conform, in all cases, to a power law (Ostwald-de Waele),

$$\eta = K\dot{\gamma}^{n-1} \quad (6)$$

Where  $K$  is the consistency index ( $Pa \cdot sn$ ), and  $n$  is the flow index (dimensionless).

Table 5-1 shows the values of these parameters, as well as the correlation coefficient ( $r^2$ ) corresponding to each fit of the experimental points with equation (6).

Table 5-1. Parameters resulting from the power-law fit

| SAMPLE | $K (Pa \cdot s)$ | $n (-)$           | $r^+$  | $\eta . (Pa \cdot s)$ |
|--------|------------------|-------------------|--------|-----------------------|
| FDPO   | $3.0 \pm 1.0$    | $0.011 \pm 0.004$ | 0.9995 | $3.2 \pm 0.1$         |
| FDMA   | $9.5 \pm 1.0$    | $0.076 \pm 0.004$ | 0.9993 | $9.6 \pm 0.1$         |
| FDESL  | $15.5 \pm 1.0$   | $0.093 \pm 0.004$ | 0.9994 | $16.7 \pm 0.1$        |
| FDPT1  | $17.3 \pm 1.0$   | $0.049 \pm 0.009$ | 0.9989 | $19.0 \pm 0.1$        |
| FDDBA  | $42.5 \pm 1.0$   | $0.073 \pm 0.003$ | 0.9995 | $43.5 \pm 0.1$        |
| FDPT2  | $70.5 \pm 1.0$   | $0.102 \pm 0.001$ | 0.9999 | $73.8 \pm 0.1$        |

Since the flow index values for each sample are different, varying even by order of magnitude, a comparison based on the consistency index is impossible since the units are different in each case ( $Pa \cdot sn$ ). However, taking into account that when the shear rate is  $1 \text{ s}^{-1}$   $\log \dot{\gamma}=0$ , we can use the viscosity value corresponding to this shear rate value as a valuable parameter to compare the viscous responses of the six samples supplied (Naranjo-Herrera, 2022).

Thus, we will conclude that the viscous response of the samples follows the increasing order,

$$\text{FDPO} < \text{FDMA} < \text{FDESL} < \text{FDPT1} < \text{FDMA} < \text{FDPT2} \quad (7)$$

### 5.5 Viscoelastic response

The viscoelastic study was carried out to determine the characteristic time of each sample (relaxation time), which gives an idea of the dominant behaviour (viscous or elastic) depending on the duration of the mechanical action exerted on the material behaviour (viscous or elastic) as a function of the duration of the mechanical action exerted on the material. This study is defined in the linear viscoelastic response region. This is why an amplitude sweep was applied (Figure 5-4), obtaining the results shown in Figure 5-8. These experimental results are presented in Appendix 2,

$$\text{FDPO} < \text{FDMA} < \text{FDESL} < \text{FDPT1} < \text{FDMA} < \text{FDPT2} \quad (8)$$

Which, moreover, confirms the goodness of sequence obtained from the purely viscous response (7).

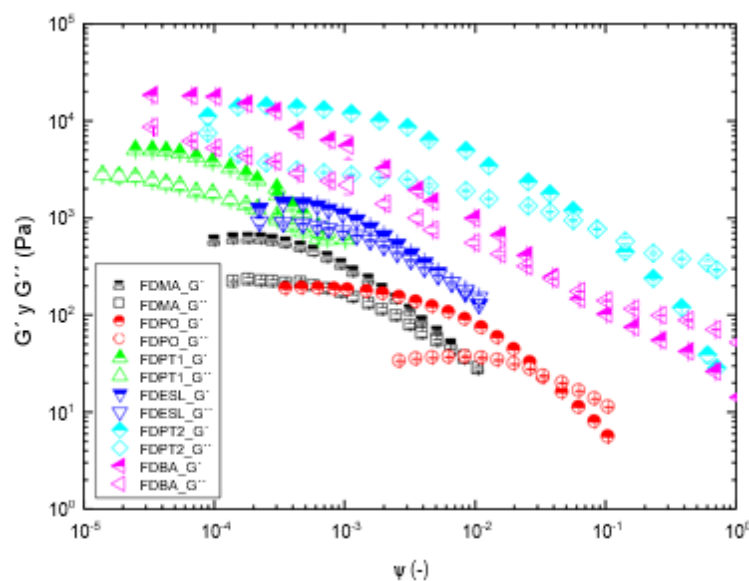


Figure 5-8. Amplitude sweeps corresponding to the six samples studied. Dr Francisco José Rubio Hernández. University of Málaga (Spain).

The dominant response of the substances when subjected to tests of different durations was determined after obtaining the corresponding maximum amplitude values delimiting the respective regions of linear viscoelastic and non-linear viscoelastic behaviour. This information is obtained from the results obtained with the so-called frequency sweep test (Figure 5-5). Figure 5-9 shows the results of the frequency sweeps in the region of linear viscoelastic behaviour. These experimental

results are presented in Appendix 3. The inverse of the frequency value where both viscoelastic moduli coincide is used as the characteristic time of the material. When the duration of the mechanical action exerted on a sample is less than this characteristic time, its response will be closer to that of a solid.

In contrast, the response will be reminiscent of that of a liquid when the duration of the mechanical action is greater than the characteristic time of the material. In either case, the responses are, strictly speaking, viscoelastic. This characteristic time indicates the influence of the duration time of the mechanical action on the dominance of one or the other viscoelastic component. According to the results in Figure 5-9, the characteristic times (indicated in the exact figure) follow the increasing order,

$$FDPT1 < FDESL < FDMA < FDDBA < FDPO < FDPT2 \quad (9)$$

For the FDPT2 sample, due to the experimental limitation, we could only conclude that its relaxation time will be higher than 1000 s

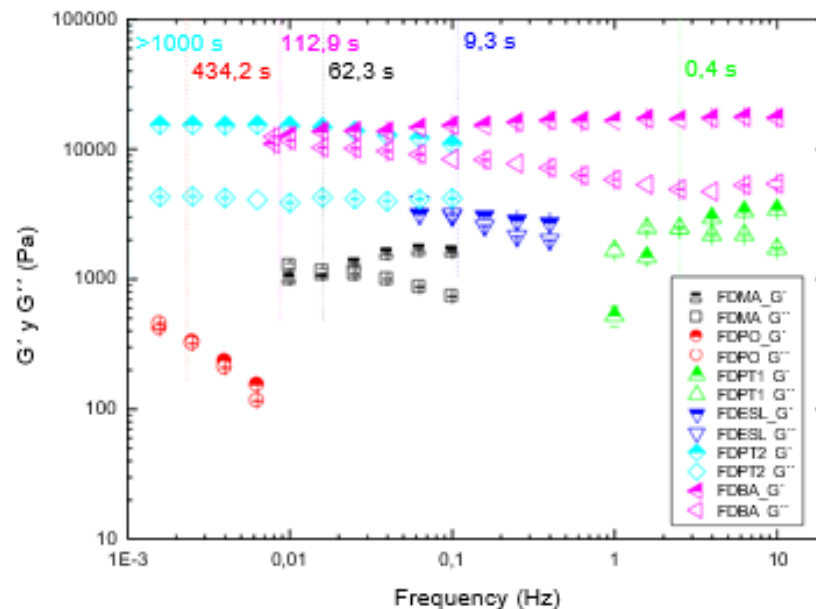


Figure 5-9. Amplitude sweeps corresponding to the six samples studied. Dr Francisco José Rubio Hernández. University of Málaga (Spain).

## 6 Report of fluidity in test installation with h=40 m

Magellan & Barents has asked the IDONIAL Foundation to analyse the pumping capacities of a high-density fluid material using the finite volume method (CFD). A sample of the test material was used, and a pumping test was carried out in the lifting tower owned by IDONIAL, located in GIJON.

The objective is to analyse the viability of this material for various applications in which, in all of them, it is necessary to pump it.

Initially, laboratory tests were carried out to obtain the plastic properties of the fluid, which were subsequently used in the simulation.

The analyses, by the finite volume method using ANSYS FLUENT software, version 2021 R1, focused on obtaining the mass flow curves as a function of pressure and pumping head and the maximum pumping head. For this purpose, the following studies and the subsequent post-processing of results were carried out:

- **Analysis 1 - Study of the maximum pumping head.** This study was carried out by applying an inlet pressure on the fluid inside a pipe of virtually infinite length. The equilibrium height of the fluid was established for different pressures.
- **Analysis 2 – Mass flow study.** This study was carried out by applying an inlet pressure to the fluid inside a pipe of a given length. The outlet flow rates were measured for the different combinations of pressure and length.

The rheological characterisation of the high-density fluid material under study is necessary for the abovementioned analyses. This characterisation was carried out employing laboratory tests.

### 6.1 Material properties

Laboratory rheological tests were carried out on the sample to characterise the high-density fluid under study. The density and plastic properties of the material were determined, and flow curves were obtained to determine the variation of the viscosity of the material as a function of shear rate.

A 10 ml test tube was used to measure the density, flush with the material under study, obtaining a value of 2.180 mg/ml.

On the other hand, the graph in Figure 6-1 shows the values obtained from the rheological test. The material has a pseudo-plastic behaviour with thixotropic properties, i.e. the viscosity is time-dependent, especially at low speeds.

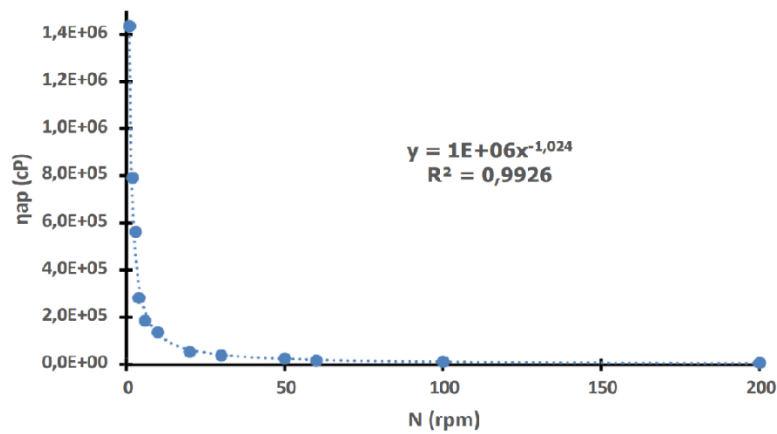


Figure 6-1. Viscosity of the material. Isaac Fernández, IDONIAL C.T.

The resulting curve is fitted with an exponential model of the equation:  $\eta = k \cdot \dot{\gamma}^{(n-1)}$

Where k is the consistency index [N·s/m<sup>2</sup>], and n is the flow behaviour index. The values of n determine the type of fluid, where:

- n > 1 -> dilatant fluid
- n = 1 -> Newtonian fluid
- n < 1 -> pseudoplastic

In this case, n takes a value of -0.024, indicating a pseudo-plastic behaviour as indicated above.

Figure 6-2, Figure 6-3 and Figure 6-4 below show the behaviour versus time plots corresponding to the thixotropic model:

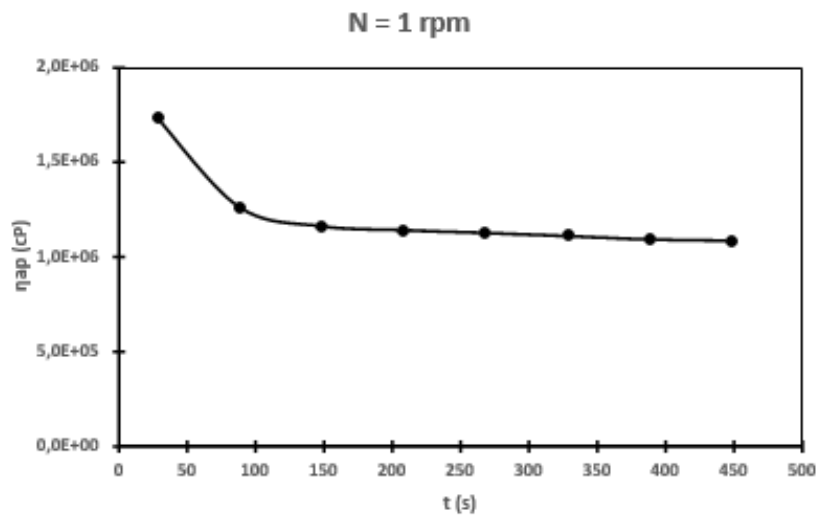


Figure 6-2. Behaviour thixotropic behaviour (N = 1 rpm). Isaac Fernández, IDONIAL C.T.

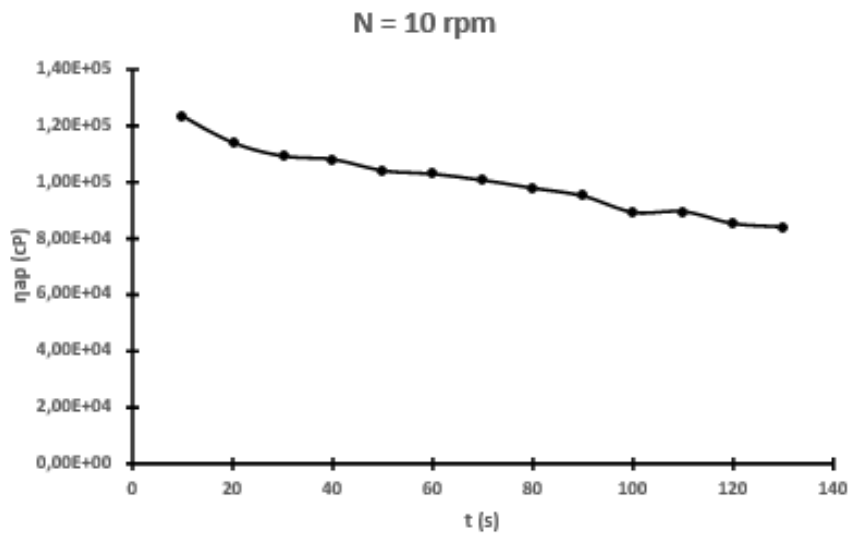


Figure 6-3. Thixotropic material behaviour (N = 10 rpm). Isaac Fernández, IDONIAL C.T.

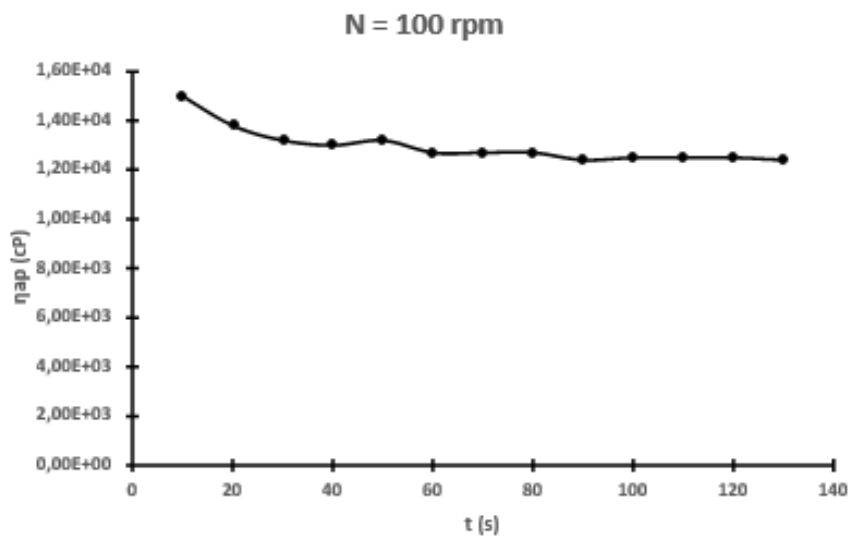


Figure 6-4. Thixotropic material behaviour (N = 100 rpm). Isaac Fernández, IDONIAL C.T.

## 6.2 Description of the model

### 6.2.1 Description of the geometric model



The 3D geometric model is based on the test at the IDONIAL company facilities in the Roces Industrial Estate, Gijón lift tower (Asturias, Spain).

The test consisted of pumping the material through a pipe from ground level to a tank located on the roof of the building using a mobile pumping station with pressure regulation.

The pumping station is replaced in the simulation model by a cylinder of 144 mm diameter and length 400 mm where the pressure is applied as a boundary condition, see Figure 6-5. The pipe has a nominal diameter of 50 mm, corresponding to an NPS of 2 inches.

Different slope heights have been tested so that the length of the pipe varies between the different models, respecting the rest of the dimensions.

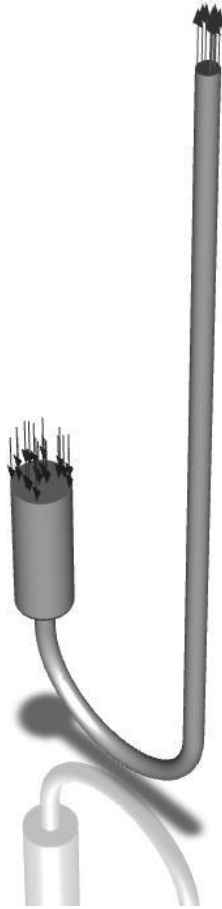


Figure 6-5. A geometric model for analysis. Isaac Fernández, IDONIAL C.T.

### 6.2.2 Description of the finite volume model

The finite volume model was developed based on the geometric model described in the previous subsection. This was generated using a structured mesh with hexahedral elements of 15 mm in length. Figure 6-6 presents a detail of the generated model. The number of elements varies depending on the case study, directly associated with the length of the pipe.

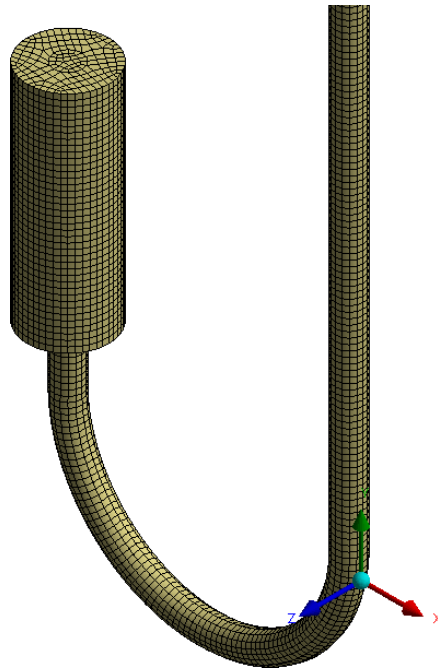


Figure 6-6. Detail of the mesh of the finite volume model. Isaac Fernández, IDONIAL C.T.

## 6.3 Boundary conditions

For the analysis of the model, the boundary conditions that allow the proposed installation to be simulated have been considered. At the inlet of the pipe, a pressure is introduced that simulates the pressure exerted by the pumping station on the fluid. A zero-gauge pressure is set at the outlet of the pipe, which simulates the outlet to 'free air' (atmospheric pressure). In both analyses, gravity considers the self-weight of the fluid under study.

### 6.3.1 Boundary conditions

The different boundary conditions used in the analyses are presented below:

- **Analysis 1. Pumping head.** A pressure sweep was carried out to obtain the fluid's different equilibrium heights. The pressure is introduced through a pressure inlet in the cylinder applied to the fluid. The outlet condition is set with a pressure outlet of zero value.
- **Analysis 2. Mass flow rate.** Different pipe lengths were used, varying the gradient from the ground-level pumping station to the maximum head. A sweep was carried out at different pressures for each length. Again, the pressure-inlet condition was used for fluid injection and zero pressure outlet for the outlet.

### 6.3.2 Time step

The simulations utilise transient analysis with a variable time step, keeping the Courant number constant and of value 0.2. The time intervals ranged from 0.001 s for the initial instants of the simulation to 1 s when reaching the equilibrium points.

## 6.4 Results of the fluid-dynamic analysis

The results obtained from the fluid-dynamic analyses for the described geometry and boundary conditions are presented below.

### 6.4.1 Pumping head

As mentioned in the previous section, a pressure sweep was applied to determine the equilibrium head in the pipe. These pressures start at  $1 \times 10^5$  Pa and reach a maximum of  $8 \times 10^5$  Pa, referred to as manometric values. The graph in Figure 6-7 represents the results obtained.

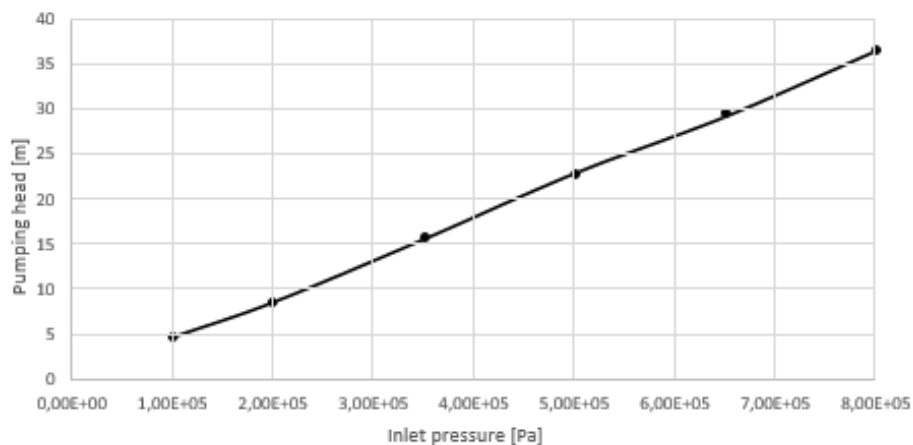
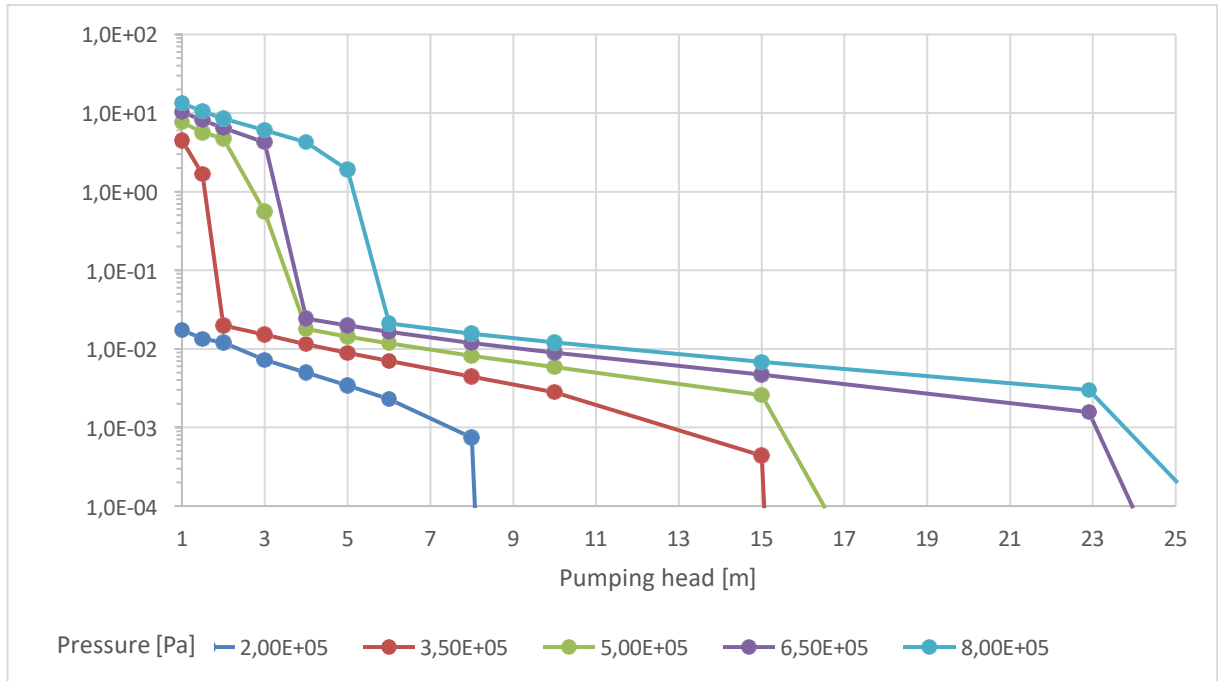


Figure 6-7. Maximum pumping head for different pressures. Isaac Fernández, IDONIAL C.T.

The results show a linear relationship between inlet pressure and maximum pumping head.

### 6.4.2 Mass flow rate

Utilising a pressure sweep, varying the length of the pipe and thus the height, the mass flow rate is obtained for the different combinations. The results are represented in the following graph in logarithmic scale, Figure 6-8.



**Figure 6-8. Mass flow rate for different pressures and heads. Isaac Fernández, IDONIAL C.T.**

A logarithmic behaviour is observed for high flow rates. As the pumping head increases, there is a sharp decrease in flow rate up to about 0.02 kg/s in all cases. From that point on, it continues again with a logarithmic adjustment that continues up to a limit at zero, where the equilibrium pressure is reached. This behaviour could be related to the pseudo-plastic properties of the fluid.

## 7 Estimate of cost for the dense fluid

The cost analysis is based on the 80 MW / 320 MWh project in San Nicolás pit. This includes 80,000 cubic meters of dense fluid with density 2.5, or 200,000 tons.

Cost breakdown of the dense fluid is presented in Table 7-1.

**Table 7-1. Cost breakdown of the dense fluid.**

| Item   |  | EUR/ton   | % by weight |
|--|--|-----------|-------------|
| Production of components:  | Cuttings from cavern enlargement <sup>1)</sup> . | 10        | 40          |
|  | Base dense fluid                                 | 75        | 60          |
| Production cost per ton of dense fluid:                            |  | <b>50</b> |             |
| Cost per ton of transport and blending of components <sup>2)</sup> |  | 2         |             |
| <b>Total</b>   |  | <b>52</b> |             |

<sup>1)</sup>The material from cuttings is free, but it has to be crushed and sieved. We have found that up to 40% of cuttings can be included in the blend with acceptable fluidity. The remaining 60% of cuttings from cavern enlargement can be sold as aggregates (good quality limestone puddingstones) in the local market in central Asturias.

<sup>2)</sup>Transport distance to the top reservoir in Pico Llosorio is 10 km. Blending costs are well known from concrete plants. Base amount is 200,000 ton.

## 8 Assessment of the job creation potential

We keep our focus on the jobs created during the formulation and production of the dense fluid, the remaining job potential of an energy storage project with dense fluids is discussed in our previous work, D2.4 Deploying unconventional pumped hydro. It must be noted that there would also be an opportunity to do land reclamation work, which has been studied by our colleagues from Slovenia in their work, D3.2 Soil substitutes for restoration (Table 8-1).

**Table 8-1. Examples of job positions for formulation and production of dense fluid**

| Job position              | Roles   | Skills  |
|---------------------------|---|---|
| Project Managers          | Coordinating production projects, managing budgets, timelines, and stakeholder communication.       | Strong organizational, leadership, and communication skills             |
| Heavy Equipment Operators | Operating machinery used in reclamation such as bulldozers, excavators, crushers, conveyor belts... | Proficiency in heavy machinery operation and maintenance.               |
| Truck Drivers             | Distribution of materials to the design locations.  | Precision in driving and operating dump trucks.                         |
| Ecologists                | Restoring and managing ecosystems, designing strategies to reintroduce flora and fauna              | Ecological assessment, habitat restoration, and biodiversity management |

In-situ production of a dense fluid is similar to operating a fresh concrete plant plus a quarry for aggregates, but the aggregates would be recovered from waste heaps instead of carrying out drill and blast operations. Rock crushing is typically necessary.

The estimated job positions are shown in table 7.2, for 200,000 ton of dense fluid, produced in one year and transported 10 km to the upper reservoir in Pico Llosorio are presented in Table 8-2.

**Table 8-2. Assessment of job creation potential, production of dense fluid**

| Job position                  | Number of workplaces |
|-------------------------------|----------------------|
| Truck Driver                  | 6                    |
| Heavy equipment operator      | 6                    |
| Blender operator              | 3                    |
| Worker for road maintenance   | 4                    |
| Engineer or higher technician | 2                    |
| Manager                       | 1                    |

|                    |           |
|--------------------|-----------|
| Production Manager | 1         |
| <b>Total</b>       | <b>26</b> |



## 9 Conclusions & lessons learnt

Hydropower is the preferred solution for large-scale energy storage as it is a mature technology that has been providing dispatchable power since the beginning of the electric era. With over 90% of the existing capacity, it will most likely remain at the top, at least where topographic conditions are good.

An essential consideration for conventional hydropower energy systems and pumped hydro storage is the footprint required by the reservoirs. Unconventional pumped hydro storage is directed to a small footprint pumped hydro energy storage system and method with high power output. The system uses a high-density fluid and allows for different configurations where upper and lower reservoirs may be at the same elevation. Hydraulic pumps and turbines may be placed higher than the lower reservoir, for example, on the surface above an underground mine.

A more compact pumped hydro energy storage system can be achieved by employing high-density fluid. For a given reservoir or tank volume, the energy storage capacity is proportional to the density of the fluid. For example, in the case where the high-density fluid has a density of 3x, the energy storage capacity of the system is 3 times that when water is used. This is because the mass flow rate is about 3 times more than water's.

Various types of slurry mixtures may be employed. The slurry mixture may include, for example, metal oxide particles mixed with a lower-density fluid, such as water. Other types of particles and lower-density fluids may also be helpful. The volume of particles in the slurry may equal or exceed 50%.

Two samples have been taken from company Hulleras del Norte S.A. S.M.E. (HUNOSA - Spain), one sample from company Węglokoks Kraj S.A. (WEGLO - Poland) and one sample from company Premogovnik Velenje d.o.o. (PV - Slovenia) to analyse the viability of developing slurry mixtures with material from waste heaps via rheometric tests specifically designed to obtain the stationary viscous response and linear viscoelastic response of the samples under study.

The lessons relevant to the Project from this deliverable can be summarised as follows:

1. A small surfactant (less than 1%) should be added to prevent the slurry from coalescing and improve flow. In some cases, antifreeze may be added to prevent freezing of the slurry. The concentration of antifreeze should be sufficient to prevent the slurry from freezing.
2. The reproducibility of the obtained rheometric results confirmed that the samples' homogeneity was achieved by shaking them manually for at least 5 minutes before starting the studies.

3. In all cases, in the viscous response and in the range of shear rates that were accessible (reproducible results), it is observed that viscosity decreases with increasing shear rate.
4. The viscoelastic study was carried out to determine the characteristic time of each sample (relaxation time), which gives an idea of the dominant behaviour (viscous or elastic) depending on the duration of the mechanical action exerted on the material behaviour (viscous or elastic) as a function of the duration of the mechanical action exerted on the material. This study is defined in the linear viscoelastic response region.
5. The results of the fluid-dynamic analysis, in conjunction with the rheology test data, show the pumping capabilities of the high-density fluid, particularly in the upward direction.
6. The dumpsite minerals from the mines are initially attractive as base materials for dense fluids. The samples from Slovenia and Spain show values in the interval bounded by our contrast formulations, which have already been used to prepare dense fluids. The sample for Poland is out of bounds but might also be helpful.
7. These results may serve as a reference in subsequent phases of the project, allowing the dimensioning of installations.

## 10 Glossary

AM – Ante Meridiem

E&M – Electrical and Mechanical

FAEN – Fundación Asturiana de la Energía

GW – Gigawatt

GWh – Gigawatt hour

HUNOSA – Hulleras del Norte, S.A.

m.a.s.l. – meters above sea level

M&B – Magellan & Barents

MW – Megawatt

MWh – Megawatt hour

OMIE – Operador del Mercado Ibérico de Electricidad

Solar PV – Solar Photovoltaics

UNIOVI – Universidad de Oviedo

USA – United States of America

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## Appendix

### Appendix 1. Experimental measurements of stationary viscosity.

| FDPT1                |                   |                   |                   |
|----------------------|-------------------|-------------------|-------------------|
| $\dot{\gamma}$ (1/s) | $\eta^*$ (Pa · s) | $\eta_+$ (Pa · s) | $\eta_-$ (Pa · s) |
| 50                   | 0.718             | 0.6027            | 0.718             |
| 37.81                | 0.6563            | 0.6562            | 0.6563            |
| 28.59                | 0.7444            | 0.7347            | 0.7444            |
| 21.62                | 0.9106            | 0.9179            | 0.9106            |
| 16.35                | 1.204             | 1.198             | 1.204             |
| 12.37                | 1.576             | 1.558             | 1.576             |
| 9.35                 | 2.057             | 1.991             | 2.057             |
| 7.071                | 2.671             | 2.705             | 2.671             |
| 5.348                | 3.379             | 3.404             | 3.379             |
| 4.044                | 4.468             | 4.544             | 4.468             |
| 3.058                | 6.811             | 5.925             | 6.811             |
| 2.313                | 7.395             | 7.718             | 7.395             |
| 1.748                | 10.15             | 9.972             | 10.15             |
| 1.322                | 12.77             | 14.27             | 12.77             |
| 1                    | 17.31             | 17.6              | 17.31             |

| FDPT1                |                   |                   |                   |
|----------------------|-------------------|-------------------|-------------------|
| $\dot{\gamma}$ (1/s) | $\eta^*$ (Pa · s) | $\eta_+$ (Pa · s) | $\eta_-$ (Pa · s) |
| 50                   | 0.4225            | 0.4266            | 0.4225            |
| 38.92                | 0.4353            | 0.4399            | 0.4353            |
| 30.3                 | 0.4839            | 0.4907            | 0.4839            |
| 23.58                | 0.5696            | 0.5784            | 0.5696            |
| 18.36                | 0.6922            | 0.7028            | 0.6922            |
| 14.29                | 0.8477            | 0.8618            | 0.8477            |
| 11.12                | 1.042             | 1.059             | 1.042             |
| 8.658                | 1.283             | 1.304             | 1.283             |
| 6.739                | 1.584             | 1.611             | 1.584             |
| 5.246                | 1.964             | 1.997             | 1.964             |
| 4.084                | 2.444             | 2.484             | 2.444             |
| 3.179                | 3.052             | 3.103             | 3.052             |
| 2.474                | 3.824             | 3.888             | 3.824             |
| 1.926                | 4.805             | 4.886             | 4.805             |
| 1.499                | 6.051             | 6.15              | 6.051             |
| 1.167                | 7.639             | 7.768             | 7.639             |
| 0.9084               | 9.660             | 9.819             | 9.66              |
| 0.7071               | 12.23             | 12.45             | 12.23             |
| 0.5504               | 15.51             | 15.82             | 15.51             |
| 0.4285               | 19.7              | 20.02             | 19.7              |
| 0.3335               | 25.05             | 25.51             | 25.05             |
| 0.2596               | 31.88             | 32.41             | 31.88             |
| 0.2021               | 40.51             | 41.15             | 40.51             |
| 0.1573               | 51.45             | 52.34             | 51.45             |
| 0.1224               | 65.28             | 66.28             | 65.28             |
| 0,09531              | 82.8              | 84.36             | 82.8              |
| 0,07419              | 104.9             | 107.1             | 104.9             |
| 0.05775              | 133.1             | 135.5             | 133.1             |
| 0.04496              | 168.8             | 171.3             | 168.8             |
| 0.03499              | 213.3             | 217.4             | 213.3             |
| 0.02724              | 271.7             | 276.2             | 271.7             |
| 0.0212               | 346.1             | 353.9             | 346.1             |
| 0.01651              | 438.4             | 449.2             | 438.4             |
| 0.01285              | 556.8             | 569.3             | 556.8             |
| 0.01                 | 704.7             | 724.1             | 704.7             |



| FDBA                 |                   |                   |                   |
|----------------------|-------------------|-------------------|-------------------|
| $\dot{\gamma}$ (1/s) | $\eta_*$ (Pa · s) | $\eta_+$ (Pa · s) | $\eta_-$ (Pa · s) |
| 100                  | 0,8772            | 0,8499            | 0,8352            |
| 78,97                | 0,9692            | 0,9329            | 0,9106            |
| 62,35                | 1,143             | 1,096             | 1,066             |
| 49,24                | 1,377             | 1,32              | 1,283             |
| 38,88                | 1,659             | 1,593             | 1,55              |
| 30,7                 | 1,999             | 1,923             | 1,873             |
| 24,24                | 2,413             | 2,326             | 2,269             |
| 19,14                | 2,921             | 2,821             | 2,755             |
| 15,12                | 3,551             | 3,437             | 3,358             |
| 11,94                | 4,338             | 4,203             | 4,111             |
| 9,427                | 5,319             | 5,155             | 5,049             |
| 7,444                | 6,543             | 6,343             | 6,22              |
| 5,878                | 8,076             | 7,833             | 7,682             |
| 4,642                | 9,99              | 9,705             | 9,514             |
| 3,665                | 12,38             | 12,04             | 11,8              |
| 2,894                | 15,38             | 14,96             | 14,67             |
| 2,285                | 19,13             | 18,63             | 18,27             |
| 1,805                | 23,82             | 23,22             | 22,77             |
| 1,425                | 29,67             | 28,97             | 28,39             |
| 1,125                | 36,95             | 36,21             | 35,44             |
| 0,8886               | 46,14             | 45,14             | 44,35             |
| 0,7017               | 57,63             | 56,37             | 55,41             |
| 0,5541               | 72,01             | 70,68             | 69,06             |
| 0,4375               | 89,98             | 89,12             | 86,58             |
| 0,3455               | 112,1             | 110,9             | 109,7             |
| 0,2729               | 141,9             | 138,8             | 137,3             |
| 0,2154               | 177               | 171,8             | 171,3             |
| 0,1702               | 223,2             | 219               | 216,6             |
| 0,1344               | 276,5             | 269,4             | 266,7             |
| 0,1061               | 347,2             | 337,9             | 336,9             |
| 0,08374              | 435,5             | 436,6             | 423,2             |
| 0,06622              | 541,8             | 528,1             | 527,9             |
| 0,05225              | 675,9             | 668,6             | 659               |
| 0,04127              | 838,9             | 808,6             | 803,4             |
| 0,03263              | 1047              | 1017              | 1002              |
| 0,02569              | 1342              | 1230              | 1277              |
| 0,02033              | 1692              | 1579              | 1590              |
| 0,01608              | 2130              | 2007              | 2012              |
| 0,01269              | 2692              | 2561              | 2536              |
| 0,009993             | 3452              | 3221              | 3246              |

| FDPT2                |                   |                   |                   |
|----------------------|-------------------|-------------------|-------------------|
| $\dot{\gamma}$ (1/s) | $\eta^*$ (Pa · s) | $\eta_+$ (Pa · s) | $\eta_-$ (Pa · s) |
| 100                  | 1,261             | 1,144             | 1,134             |
| 78,97                | 1,491             | 1,375             | 1,37              |
| 62,35                | 1,822             | 1,7               | 1,71              |
| 49,24                | 2,239             | 2,09              | 2,079             |
| 38,88                | 2,756             | 2,57              | 2,581             |
| 30,7                 | 3,374             | 3,169             | 3,196             |
| 24,24                | 4,153             | 3,899             | 3,948             |
| 19,14                | 5,122             | 4,786             | 4,874             |
| 15,12                | 6,315             | 5,892             | 6,033             |
| 11,94                | 7,788             | 7,28              | 7,434             |
| 9,427                | 9,609             | 9,013             | 9,18              |
| 7,444                | 11,87             | 11,14             | 11,32             |
| 5,878                | 14,64             | 13,79             | 14,03             |
| 4,642                | 18,12             | 17,06             | 17,35             |
| 3,665                | 22,45             | 21,11             | 21,44             |
| 2,894                | 27,84             | 26                | 26,61             |
| 2,285                | 34,56             | 32,07             | 33,05             |
| 1,805                | 42,92             | 39,61             | 41,07             |
| 1,425                | 53,31             | 49,1              | 51,07             |
| 1,125                | 66,31             | 60,94             | 63,46             |
| 0,8887               | 82,31             | 75,56             | 78,88             |
| 0,7018               | 102,1             | 93,61             | 98,18             |
| 0,5542               | 126,9             | 116               | 121,8             |
| 0,4376               | 157,5             | 143,5             | 150,6             |
| 0,3456               | 194,4             | 177,5             | 186,8             |
| 0,273                | 240,7             | 219               | 229,9             |
| 0,2155               | 297,4             | 271,1             | 283,4             |
| 0,1702               | 367,9             | 334               | 346,7             |
| 0,1344               | 455,1             | 414,5             | 423,8             |
| 0,1061               | 558,4             | 504,9             | 523,9             |
| 0,08389              | 684,5             | 620,6             | 633,6             |
| 0,06619              | 851,3             | 764,7             | 790,2             |
| 0,05231              | 1047              | 941,1             | 963,4             |
| 0,04129              | 1288              | 1162              | 1189              |
| 0,03258              | 1596              | 1421              | 1492              |
| 0,02568              | 2016              | 1750              | 1844              |
| 0,02034              | 2526              | 2189              | 2274              |
| 0,01608              | 3099              | 2725              | 2847              |
| 0,01271              | 3807              | 3421              | 3588              |
| 0,01001              | 4758              | 4305              | 4514              |

| FDPO                 |                   |                   |                   |
|----------------------|-------------------|-------------------|-------------------|
| $\dot{\gamma}$ (1/s) | $\eta^*$ (Pa · s) | $\eta_+$ (Pa · s) | $\eta_-$ (Pa · s) |
| 100                  | 0,264             | 0,2642            | 0,2621            |
| 78,97                | 0,2076            | 0,2045            | 0,2042            |
| 62,35                | 0,19              | 0,1868            | 0,1847            |
| 49,24                | 0,189             | 0,1856            | 0,1833            |
| 38,88                | 0,1928            | 0,189             | 0,1863            |
| 30,7                 | 0,1973            | 0,1949            | 0,1933            |
| 24,24                | 0,1888            | 0,1871            | 0,1854            |
| 19,14                | 0,1929            | 0,1915            | 0,1867            |
| 15,12                | 0,227             | 0,2209            | 0,2168            |
| 11,94                | 0,2794            | 0,2721            | 0,2671            |
| 9,427                | 0,3491            | 0,3396            | 0,334             |
| 7,444                | 0,4356            | 0,4241            | 0,4172            |
| 5,878                | 0,5432            | 0,5291            | 0,5206            |
| 4,642                | 0,6742            | 0,6573            | 0,6472            |
| 3,665                | 0,8404            | 0,8185            | 0,807             |
| 2,894                | 1,047             | 1,022             | 1,009             |
| 2,285                | 1,311             | 1,278             | 1,264             |
| 1,805                | 1,644             | 1,603             | 1,584             |
| 1,425                | 2,065             | 2,013             | 1,992             |
| 1,125                | 2,59              | 2,531             | 2,52              |
| 0,8886               | 3,257             | 3,185             | 3,181             |
| 0,7017               | 4,124             | 4,04              | 4,019             |
| 0,5541               | 5,218             | 5,107             | 5,093             |
| 0,4375               | 6,592             | 6,437             | 6,461             |
| 0,3455               | 8,308             | 8,13              | 8,193             |
| 0,2728               | 10,48             | 10,25             | 10,39             |
| 0,2154               | 13,3              | 13,04             | 13,24             |
| 0,1701               | 16,91             | 16,61             | 16,87             |
| 0,1343               | 21,47             | 21,11             | 21,6              |
| 0,1061               | 27,33             | 26,87             | 27,48             |
| 0,08377              | 34,91             | 34,15             | 35,2              |
| 0,06615              | 44,35             | 43,4              | 44,84             |
| 0,05223              | 56,37             | 55,38             | 56,9              |
| 0,04125              | 71,91             | 70,46             | 72,76             |
| 0,03257              | 90,86             | 89,81             | 92,82             |
| 0,02572              | 116,1             | 114,1             | 118,9             |
| 0,02031              | 147,2             | 146,2             | 150,7             |
| 0,01604              | 187,3             | 186,7             | 194,6             |
| 0,01266              | 240,4             | 238,7             | 250,1             |
| 0,01                 | 309,3             | 306               | 318,9             |

| FDESL                |                   |                   |                   |
|----------------------|-------------------|-------------------|-------------------|
| $\dot{\gamma}$ (1/s) | $\eta^*$ (Pa · s) | $\eta_+$ (Pa · s) | $\eta_-$ (Pa · s) |
| 100                  | 0,5844            | 0,5938            | 0,6002            |
| 78,97                | 0,5791            | 0,5897            | 0,5962            |
| 62,35                | 0,5683            | 0,5774            | 0,5836            |
| 49,24                | 0,5657            | 0,5777            | 0,5868            |
| 38,88                | 0,6242            | 0,6331            | 0,6459            |
| 30,7                 | 0,7256            | 0,7505            | 0,7689            |
| 24,24                | 0,8999            | 0,9289            | 0,9513            |
| 19,14                | 1,104             | 1,137             | 1,161             |
| 15,12                | 1,339             | 1,373             | 1,4               |
| 11,94                | 1,621             | 1,66              | 1,694             |
| 9,427                | 1,968             | 2,013             | 2,054             |
| 7,444                | 2,394             | 2,451             | 2,501             |
| 5,878                | 2,93              | 2,995             | 3,057             |
| 4,642                | 3,595             | 3,673             | 3,75              |
| 3,665                | 4,43              | 4,52              | 4,611             |
| 2,894                | 5,463             | 5,578             | 5,688             |
| 2,285                | 6,752             | 6,896             | 7,024             |
| 1,805                | 8,366             | 8,516             | 8,683             |
| 1,425                | 10,36             | 10,56             | 10,75             |
| 1,125                | 12,87             | 13,13             | 13,34             |
| 0,8886               | 16,03             | 16,35             | 16,61             |
| 0,7017               | 19,93             | 20,4              | 20,72             |
| 0,5541               | 24,9              | 25,49             | 25,91             |
| 0,4376               | 31,24             | 31,83             | 32,49             |
| 0,3455               | 39,19             | 39,84             | 40,65             |
| 0,2729               | 49,03             | 50,04             | 51,01             |
| 0,2154               | 61,87             | 62,75             | 64,08             |
| 0,1701               | 78,48             | 79,47             | 80,98             |
| 0,1343               | 98,63             | 100,2             | 102,5             |
| 0,1061               | 121,5             | 126,2             | 129,9             |
| 0,0838               | 143,9             | 144,7             | 163,8             |
| 0,06615              | 172,1             | 170,6             | 187,8             |
| 0,05226              | 208,8             | 209,5             | 225               |
| 0,04124              | 259,8             | 265               | 281,4             |
| 0,03259              | 319,7             | 324,8             | 348,1             |
| 0,02573              | 406,8             | 415               | 429,8             |
| 0,0203               | 527,8             | 534,4             | 550,4             |
| 0,01603              | 663               | 682,7             | 715,5             |
| 0,01267              | 851,2             | 860,3             | 928,3             |
| 0,009993             | 1094              | 1125              | 1194              |

## Appendix 2. Experimental measurements corresponding to the amplitude sweeps

| FDMA         |            |              |            |              |            |              |
|--------------|------------|--------------|------------|--------------|------------|--------------|
| $\gamma$ (-) | $G^* (Pa)$ | $G''^* (Pa)$ | $G^+ (Pa)$ | $G''^+ (Pa)$ | $G^- (Pa)$ | $G''^- (Pa)$ |
| 0.00009608   | 1947       | 490.3        | 1977       | 491.2        | 1971       | 479.1        |
| 0.0001311    | 1915       | 499.6        | 1903       | 511.5        | 1962       | 522.7        |
| 0.0001704    | 1852       | 493.7        | 1854       | 504.1        | 1862       | 507.1        |
| 0.0002215    | 1787       | 490.7        | 1799       | 498          | 1860       | 505.8        |
| 0.000282     | 1727       | 495.7        | 1728       | 499.2        | 1770       | 514.8        |
| 0.0003603    | 1635       | 494          | 1631       | 496.6        | 1540       | 465.4        |
| 0.0004599    | 1504       | 479.5        | 1495       | 478.1        | 1456       | 452.8        |
| 0.0005869    | 1365       | 457.7        | 1355       | 454.3        | 1359       | 454.6        |
| 0.0007489    | 1221       | 436          | 1226       | 436.4        | 1229       | 438.1        |
| 0.0009557    | 1079       | 409.5        | 1084       | 412.7        | 1095       | 417.6        |
| 0.001218     | 939.2      | 382          | 942.4      | 384.9        | 946.8      | 392.4        |
| 0.001553     | 806.3      | 350.6        | 811.4      | 355.1        | 817.2      | 352.4        |
| 0.00198      | 682.4      | 319.3        | 686.8      | 322.1        | 686.6      | 318          |
| 0.002521     | 570.6      | 285.7        | 571.2      | 289.3        | 576.7      | 289.9        |
| 0.003211     | 468.2      | 252.8        | 468        | 255.2        | 474.2      | 256.4        |
| 0.003948     | 367,1      | 217.2        | 373.2      | 222          | 377.8      | 223          |
| 0.005206     | 283.5      | 189.7        | 285.1      | 191.3        | 288.6      | 192.9        |
| 0.006626     | 207.9      | 162.4        | 203.3      | 161.9        | 210        | 164.4        |
| 0.008422     | 154.3      | 139.4        | 152.1      | 139          | 154.9      | 140.1        |
| 0.01072      | 120.2      | 121          | 120.2      | 121.5        | 119.6      | 121.1        |

| FDPO             |              |               |              |               |              |               |
|------------------|--------------|---------------|--------------|---------------|--------------|---------------|
| $\gamma_{\&(-)}$ | $G'_{*}(Pa)$ | $G''_{*}(Pa)$ | $G'_{+}(Pa)$ | $G''_{+}(Pa)$ | $G'_{-}(Pa)$ | $G''_{-}(Pa)$ |
| 0,0001054        | 147,1        | 20,09         | 154,7        | 21,49         | 154,2        | 21,66         |
| 0,000148         | 165,8        | 19,27         | 168,8        | 20,52         | 172,1        | 20,85         |
| 0,000199         | 174,9        | 19,9          | 177,7        | 20,35         | 181,7        | 21,4          |
| 0,0002667        | 178,7        | 20,62         | 183,1        | 21,12         | 187,3        | 22,3          |
| 0,0003573        | 181,6        | 21,3          | 186,6        | 21,91         | 191,1        | 22,73         |
| 0,000474         | 184,3        | 22,16         | 187,4        | 22,3          | 193          | 23,37         |
| 0,0006315        | 184          | 22,41         | 186,9        | 23,58         | 192,4        | 24,44         |
| 0,0008432        | 181,3        | 24,34         | 185,1        | 25            | 189,8        | 26,22         |
| 0,001062         | 178,5        | 25,57         | 180,5        | 26,62         | 185,9        | 27,85         |
| 0,001493         | 171,6        | 27,83         | 173,8        | 28,53         | 178,2        | 30,09         |
| 0,00198          | 162,8        | 29,87         | 164,2        | 30,94         | 168,1        | 32,28         |
| 0,002654         | 150,9        | 32,12         | 151,4        | 33,14         | 154,6        | 34,53         |
| 0,003555         | 134,7        | 33,95         | 135,4        | 34,81         | 138,2        | 36,29         |
| 0,004745         | 120          | 35,08         | 120,6        | 35,98         | 122,9        | 37,32         |
| 0,006329         | 104,7        | 35,87         | 105,2        | 36,71         | 108,8        | 37,62         |
| 0,008442         | 88,99        | 35,99         | 89,28        | 36,79         | 91,38        | 38,09         |
| 0,01127          | 73,32        | 35,2          | 73,43        | 35,95         | 74,38        | 37,04         |
| 0,01502          | 58,3         | 33,41         | 58,09        | 34,06         | 58,44        | 34,91         |
| 0,01999          | 44,63        | 30,63         | 44,12        | 31,04         | 43,6         | 31,82         |
| 0,0266           | 33,35        | 27,02         | 31,83        | 27,6          | 31,33        | 28,16         |
| 0,03343          | 23,51        | 22,78         | 22,24        | 23,67         | 22,02        | 24,04         |
| 0,04699          | 16,11        | 19,09         | 15,72        | 20,03         | 15,66        | 20,27         |
| 0,0628           | 11,23        | 15,96         | 11           | 16,49         | 11,22        | 16,85         |
| 0,08308          | 7,91         | 13,26         | 7,81         | 13,58         | 8,049        | 13,95         |
| 0,1052           | 5,513        | 11,01         | 5,509        | 11,27         | 5,58         | 11,44         |

| FDPT1            |              |               |              |               |              |               |
|------------------|--------------|---------------|--------------|---------------|--------------|---------------|
| $\gamma_{\&(-)}$ | $G'_{*}(Pa)$ | $G''_{*}(Pa)$ | $G'_{+}(Pa)$ | $G''_{+}(Pa)$ | $G'_{-}(Pa)$ | $G''_{-}(Pa)$ |
| 8,827E-06        | 2333         | 181,1         | 2316         | 343,3         | 2283         | 482,6         |
| 7,472E-06        | 2474         | 1179          | 3116         | 1998          | 3168         | 1859          |
| 0,00001393       | 4473         | 3334          | 4319         | 2595          | 4233         | 2408          |
| 0,00001873       | 5402         | 3462          | 4537         | 2385          | 4385         | 2200          |
| 0,00002492       | 5571         | 3154          | 4864         | 2454          | 4814         | 2305          |
| 0,00003356       | 5497         | 2820          | 4930         | 2326          | 4649         | 2126          |
| 0,00004291       | 5569         | 2762          | 4659         | 2105          | 4546         | 1930          |
| 0,00005468       | 5273         | 2516          | 4475         | 2050          | 4358         | 1876          |
| 0,00007282       | 4709         | 2091          | 4078         | 1761          | 3963         | 1721          |
| 0,00009712       | 4332         | 2106          | 3599         | 1657          | 3550         | 1599          |
| 0,0001334        | 3672         | 1809          | 3194         | 1481          | 2971         | 1325          |
| 0,0001756        | 3108         | 1500          | 2749         | 1340          | 2656         | 1246          |
| 0,0002144        | 2717         | 1415          | 2352         | 1174          | 2283         | 1136          |
| 0,0003037        | 2171         | 1133          | 1973         | 1012          | 1923         | 1004          |
| 0,0003064        | 1795         | 1037          | 1745         | 944,8         | 1609         | 904,9         |
| 0,0003961        | 1452         | 903,7         | 1196         | 774,1         | 1327         | 777,5         |
| 0,00048          | 1212         | 788,2         | 1107         | 700,3         | 1109         | 689,1         |
| 0,0006252        | 799,3        | 639,4         | 795,8        | 616,3         | 826,2        | 604,8         |
| 0,0007383        | 761          | 585,8         | 703,7        | 586,7         | 679,3        | 631,7         |
| 0,001016         | 640,7        | 629,1         | 594,5        | 591,2         | 576,1        | 585,6         |

| FDES L           |              |               |              |               |              |               |
|------------------|--------------|---------------|--------------|---------------|--------------|---------------|
| $\gamma_{\&(-)}$ | $G'_{*}(Pa)$ | $G''_{*}(Pa)$ | $G'_{+}(Pa)$ | $G''_{+}(Pa)$ | $G'_{-}(Pa)$ | $G''_{-}(Pa)$ |
| 0,0002227        | 1149         | 807           | 1380         | 873,1         | 1265         | 1063          |
| 0,0003377        | 1380         | 847,3         | 1555         | 877,5         | 1463         | 955,5         |
| 0,0004785        | 1351         | 849,7         | 1462         | 863,3         | 1504         | 907           |
| 0,0006175        | 1282         | 823,8         | 1385         | 837,1         | 1381         | 824,7         |
| 0,0007516        | 1237         | 794,1         | 1239         | 787,6         | 1224         | 766,3         |
| 0,0009666        | 1095         | 737,9         | 1149         | 742,1         | 1115         | 714,9         |
| 0,001233         | 944,2        | 671,4         | 987,5        | 674,1         | 937,8        | 641,4         |
| 0,001569         | 792,6        | 597,7         | 822,1        | 595,8         | 789,4        | 571,4         |
| 0,001996         | 652,8        | 519,2         | 693,4        | 524           | 640,2        | 493,5         |
| 0,002534         | 530          | 443,4         | 568,4        | 450,5         | 512,2        | 418,1         |
| 0,003221         | 424,4        | 372,3         | 455,4        | 379,8         | 414          | 352,8         |
| 0,004094         | 337,1        | 310,7         | 372,4        | 319,9         | 329,5        | 295,7         |
| 0,005205         | 264,1        | 257,3         | 301,6        | 269           | 260,6        | 247           |
| 0,006621         | 204,7        | 213,1         | 241,8        | 227,4         | 205,3        | 206,6         |
| 0,008422         | 158          | 177,4         | 192,2        | 192,9         | 161,3        | 174,2         |
| 0,01072          | 122,2        | 149,7         | 152,2        | 165,3         | 115,1        | 142,2         |

| FDPT2            |              |               |              |               |              |               |
|------------------|--------------|---------------|--------------|---------------|--------------|---------------|
| $\gamma_{\&(-)}$ | $G'_{*}(Pa)$ | $G''_{*}(Pa)$ | $G'_{+}(Pa)$ | $G''_{+}(Pa)$ | $G'_{-}(Pa)$ | $G''_{-}(Pa)$ |
| 0,00008917       | 13110        | 7277          | 7505         | 8253          | 12890        | 7061          |
| 0,0001521        | 14270        | 4638          | 13920        | 4583          | 13760        | 4345          |
| 0,0002514        | 14620        | 3754          | 14250        | 3691          | 14170        | 3617          |
| 0,0004301        | 14090        | 3174          | 13890        | 3182          | 13510        | 3027          |
| 0,0006938        | 13280        | 2965          | 13020        | 2902          | 12970        | 2926          |
| 0,001128         | 12160        | 2835          | 11940        | 2765          | 11750        | 2735          |
| 0,00185          | 10300        | 2653          | 10230        | 2612          | 9707         | 2507          |
| 0,003038         | 8836         | 2571          | 8407         | 2467          | 8662         | 2454          |
| 0,004435         | 5692         | 2012          | 6834         | 2286          | 6496         | 2170          |
| 0,00849          | 4908         | 1870          | 4966         | 1913          | 5076         | 1953          |
| 0,01274          | 3516         | 1590          | 3429         | 1597          | 3388         | 1551          |
| 0,02545          | 2451         | 1367          | 2236         | 1273          | 2413         | 1352          |
| 0,0374           | 1810         | 1176          | 1747         | 1132          | 1784         | 1160          |
| 0,05632          | 1116         | 916,4         | 1259         | 967,2         | 1232         | 955,8         |
| 0,08596          | 792,5        | 781,1         | 784,1        | 773,9         | 739,7        | 747           |
| 0,1412           | 485,8        | 622,3         | 480,6        | 613,9         | 378,5        | 495,1         |
| 0,2321           | 251,8        | 453,7         | 240,9        | 434,4         | 223,9        | 410,9         |
| 0,3819           | 121,2        | 383,3         | 117,9        | 376,4         | 120,8        | 376,6         |
| 0,6027           | 47,67        | 370,4         | 42,95        | 360,7         | 26,41        | 300,9         |
| 0,7118           | 31,07        | 298,5         | 26,82        | 290,7         | 27,58        | 286,4         |



| FDBA             |              |               |              |               |              |               |
|------------------|--------------|---------------|--------------|---------------|--------------|---------------|
| $\gamma_{\&(-)}$ | $G'_{*}(Pa)$ | $G''_{*}(Pa)$ | $G'_{+}(Pa)$ | $G''_{+}(Pa)$ | $G'_{-}(Pa)$ | $G''_{-}(Pa)$ |
| 0,00001528       | 785,8        | 1313          | 706          | 1157          | 746,7        | 909,8         |
| 0,00001037       | 106,7        | 2230          | 44,63        | 1321          | 76,32        | 1871          |
| 0,00001362       | 9075         | 12250         | 4651         | 10690         | 9019         | 12190         |
| 0,00003354       | 18560        | 8861          | 18450        | 8556          | 18320        | 8636          |
| 0,00006761       | 18150        | 6035          | 17570        | 6234          | 18650        | 6373          |
| 0,0001028        | 17860        | 5335          | 17470        | 5424          | 17850        | 5057          |
| 0,0001788        | 15010        | 4282          | 14530        | 4340          | 15620        | 4381          |
| 0,0002952        | 12650        | 3813          | 12440        | 3714          | 13100        | 3884          |
| 0,0004419        | 7212         | 2634          | 9183         | 3220          | 7916         | 2760          |
| 0,0007701        | 6558         | 2324          | 6185         | 2538          | 6340         | 2380          |
| 0,001062         | 3223         | 1422          | 4951         | 2166          | 8488         | 2991          |
| 0,00204          | 2845         | 1255          | 2487         | 1310          | 4411         | 1625          |
| 0,003624         | 1809         | 1014          | 2305         | 1153          | 1724         | 826           |
| 0,004754         | 1498         | 797,2         | 1574         | 831,4         | 1389         | 632,3         |
| 0,009877         | 905,1        | 534           | 1085         | 623,2         | 981,7        | 512,2         |
| 0,01559          | 627,2        | 409,8         | 707,3        | 464,1         | 661,6        | 403,4         |
| 0,0248           | 403,3        | 312,5         | 411,7        | 325,2         | 420,7        | 315,3         |
| 0,03953          | 244,3        | 234           | 244,9        | 236,7         | 252,9        | 239,9         |
| 0,06323          | 149,8        | 176,5         | 149,8        | 177,8         | 142,7        | 172,3         |
| 0,1017           | 102,3        | 139,6         | 102,8        | 140,6         | 103,2        | 142,6         |
| 0,1642           | 74,19        | 114,6         | 75,17        | 116,2         | 75,84        | 118,4         |
| 0,2654           | 54,6         | 98,2          | 55,92        | 100,1         | 55,06        | 99,63         |
| 0,4291           | 40,77        | 86,66         | 45,3         | 92,1          | 40,46        | 86,91         |
| 0,6928           | 25,55        | 69,79         | 27,44        | 73,15         | 25,4         | 70            |
| 1,058            | 14,69        | 53,16         | 14,43        | 53,27         | 13,46        | 50,9          |

### Appendix 3. Experimental measurements corresponding to the frequency sweeps

| FDMA     |             |              |            |             |            |             |
|----------|-------------|--------------|------------|-------------|------------|-------------|
| $f$ (Hz) | $G'^*$ (Pa) | $G''^*$ (Pa) | $G'+$ (Pa) | $G''+$ (Pa) | $G',$ (Pa) | $G'',$ (Pa) |
| 0,1      | 1666        | 733,2        | 1526       | 744,4       | 1666       | 733,2       |
| 0,0631   | 1672        | 859,4        | 1635       | 896,2       | 1672       | 859,4       |
| 0,03981  | 1554        | 980,5        | 1573       | 1041        | 1554       | 980,5       |
| 0,02512  | 1289        | 1066         | 1323       | 1143        | 1289       | 1066        |
| 0,01585  | 1049        | 1111         | 1167       | 1249        | 1049       | 1111        |
| 0,01     | 959         | 1209         | 1080       | 1387        | 959        | 1209        |

| FDPT1    |             |              |            |             |            |             |
|----------|-------------|--------------|------------|-------------|------------|-------------|
| $f$ (Hz) | $G'^*$ (Pa) | $G''^*$ (Pa) | $G'+$ (Pa) | $G''+$ (Pa) | $G',$ (Pa) | $G'',$ (Pa) |
| 10       | 3449        | 1741         | 3507       | 1735        | 3394       | 1691        |
| 6,31     | 3360        | 2220         | 3409       | 2116        | 3224       | 2262        |
| 3,981    | 3001        | 2296         | 3061       | 2166        | 2875       | 2138        |
| 2,512    | 2392        | 2541         | 2533       | 2464        | 2450       | 2459        |
| 1,585    | 1276        | 2321         | 1735       | 2677        | 1427       | 2425        |
| 1        | 367,7       | 1420         | 682,6      | 1925        | 518,6      | 1626        |

| FDPO     |             |              |            |             |            |             |
|----------|-------------|--------------|------------|-------------|------------|-------------|
| $f$ (Hz) | $G'^*$ (Pa) | $G''^*$ (Pa) | $G'+$ (Pa) | $G''+$ (Pa) | $G',$ (Pa) | $G'',$ (Pa) |
| 0,00631  | 153,9       | 115,7        | 154,9      | 114,7       | 152,9      | 116,7       |
| 0,003981 | 235,8       | 210,2        | 232,8      | 208,2       | 231,8      | 207,2       |
| 0,002512 | 325,2       | 319,6        | 330,2      | 319,6       | 332,2      | 317,6       |
| 0,001585 | 434         | 450          | 404        | 450         | 414        | 460         |

| FDES�    |             |              |            |             |            |             |
|----------|-------------|--------------|------------|-------------|------------|-------------|
| $f$ (Hz) | $G'^*$ (Pa) | $G''^*$ (Pa) | $G'+$ (Pa) | $G''+$ (Pa) | $G',$ (Pa) | $G'',$ (Pa) |
| 0,3981   | 2710        | 2005         | 2720       | 2010        | 2725       | 2005        |
| 0,2512   | 2845        | 2156         | 2835       | 2146        | 2825       | 2136        |
| 0,1585   | 3050        | 2582         | 3050       | 2592        | 3055       | 2582        |
| 0,1      | 3093        | 3168         | 3083       | 3178        | 3063       | 3188        |
| 0,0631   | 3173        | 3971         | 3183       | 3991        | 3163       | 3961        |

| FDPT2    |            |              |            |              |             |              |
|----------|------------|--------------|------------|--------------|-------------|--------------|
| $f$ (Hz) | $G^*$ (Pa) | $G''^*$ (Pa) | $G^+$ (Pa) | $G''^+$ (Pa) | $G^-,$ (Pa) | $G''-,$ (Pa) |
| 0,1      | 10730      | 4149         | 10720      | 4249         | 11720       | 4149         |
| 0,0631   | 12190      | 4059         | 12290      | 4159         | 12290       | 4179         |
| 0,03981  | 13200      | 3979         | 13100      | 3979         | 12100       | 3999         |
| 0,02512  | 14170      | 4126         | 14190      | 4126         | 13190       | 4226         |
| 0,01585  | 14920      | 4318         | 14720      | 4218         | 14720       | 4218         |
| 0,01     | 15230      | 3784         | 15250      | 3884         | 15250       | 3984         |
| 0,00631  | 15280      | 4081         | 15380      | 4081         | 15580       | 4081         |
| 0,003981 | 15330      | 4358         | 15230      | 4158         | 15230       | 4188         |
| 0,002512 | 15310      | 4473         | 15380      | 4273         | 15580       | 4263         |
| 0,001585 | 15360      | 4376         | 15460      | 4376         | 15460       | 4176         |

| FDDBA    |            |              |            |              |             |              |
|----------|------------|--------------|------------|--------------|-------------|--------------|
| $f$ (Hz) | $G^*$ (Pa) | $G''^*$ (Pa) | $G^+$ (Pa) | $G''^+$ (Pa) | $G^-,$ (Pa) | $G''-,$ (Pa) |
| 10       | 17390      | 5752         | 17490      | 5752         | 17590       | 4752         |
| 6,31     | 17760      | 5148         | 17760      | 5348         | 17860       | 5348         |
| 3,981    | 17430      | 4721         | 17450      | 4721         | 17430       | 4721         |
| 2,512    | 16940      | 4853         | 16940      | 4953         | 16940       | 4953         |
| 1,585    | 17140      | 5340         | 17240      | 5340         | 17340       | 5340         |
| 1        | 16640      | 5837         | 16640      | 5837         | 16640       | 5867         |
| 0,631    | 16570      | 6226         | 16670      | 6426         | 16570       | 6226         |
| 0,3981   | 16790      | 7100         | 16790      | 7100         | 16490       | 7500         |
| 0,2512   | 16140      | 7757         | 16180      | 7757         | 16140       | 7757         |
| 0,1585   | 15230      | 8288         | 15230      | 8388         | 15230       | 8188         |
| 0,1      | 15180      | 8396         | 15380      | 8396         | 15280       | 8396         |
| 0,0631   | 14720      | 9018         | 14720      | 9018         | 14720       | 9418         |
| 0,03981  | 13430      | 9683         | 13530      | 9883         | 14430       | 9583         |
| 0,02512  | 13880      | 10140        | 13780      | 10040        | 13880       | 10340        |
| 0,01585  | 13520      | 10440        | 13920      | 10340        | 13620       | 10240        |

