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| **ZEROBRINE\_D3.2\_DATA\_CRIEM\_v0.1** | | **Laboratory test of Mg(OH)2 precipitation using the UNIPA’s crystallizer** | | | | | | | | |
| Researchers: | | Daniele La Corte (CrIEM tests), Paweł Skóra (TGA analysis) | | | | | Date: | | October 2019 | |
| Equipment: | | Bench-scale CrIEM crystallizer made by UNIPA (BCr-3 equipment obtained from Italian BEC) – see Fig. 1.  Fig. 1: a) feed&bleed system; b) CrIEM zoom; c) buffer tank zoom | | | | | | | | |
| Protocol: | | Samples of 1st pass nanofiltration retentate working at the pilot plant in “Bolesław Śmiały” coal mine were collected at 70% and 80% of permeate recovery and used for the test. The composition of feeds was as follows:   |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | | Feed | Permeate recovery | Ionic composition | | | | | | Na+ | Mg2+ | Ca2+ | Cl- | SO42- | | NF1-70% | 70% | 14.62 | 0.74 | 0.70 | 19.13 | 0.36 | | NF1-80% | 80% | 16.31 | 1.13 | 1.12 | 19.85 | no data |   Each test consists of two steps: start-up and feed&bleed (see Fig. 2) experiment. First, 1L of NF retentate was placed in the buffer tank and 2 L of Ca(OH)2 suspension was placed in another vessel. Both solutions were recirculated inside the crystallizer until all magnesium reacted. A pH-meter was used to check the course of the reaction.  Next, when the reaction ended and Mg(OH)2 precipitated in the bottom part of conical buffer tank, the fresh NF retentate was pumped to the top of of buffer tanks, while the magnesium hydroxide suspension was being removed from the bottom. Sampling was carried out at regular intervals.    Fig. 2. The general scheme of feed & bleed process.  Every sample was filtered. The filtrates and the solid were analyzed with EDTA titration. For solids analysis, 40 mg of it was dissolved in 100 ml of HCl 1M. The composition of obtained solid Mg(OH)2 was analysed using thermogravimetric method (TGA), which showed that the sample contains 3.38% H2O and pure Mg(OH)2 (which decomposes) – see Fig. 3. | | | | | | | | |
| **Experiments with NF retentate collected at 70% permeate recovery** | | | | | | | | | | |
| Sample | Filtrate concentration [g/L] | | | Conversion [%] | Solid analysis | | | Sample volume [mL] | | Magma density [g/L] |
| Ca2+ | | Mg2+ | Ca [%] | Mg [%] | |
| S1 | 0.5 | | 0.05 | 93.1 | 7.6 | 92.4 | | 300 | | 2.01 |
| S2 | 0.56 | | 0.08 | 88.9 | 5.3 | 94.7 | | 300 | | 0.68 |
| S2 | 0.55 | | 0.12 | 83.4 | 3.8 | 96.2 | | 300 | | 0.79 |
| S4 | 0.52 | | 0.14 | 80.1 | 11.5 | 88.5 | | 240 | | 0.4 |
| S5 | 0.57 | | 0.06 | 91.3 |  |  | | 240 | | 0.04 |
| S6 | 0.59 | | 0.07 | 90.6 |  |  | | 240 | | 0.37 |
| S7 | 0.61 | | 0.01 | 99 | 1.2 | 98.8 | | 2000 | | 1.42 |
| **Experiments with NF retentate collected at 70% permeate recovery** | | | | | | | | | | |
| Sample | Filtrate concentration [g/L] | | | Conversion [%] | Solid analysis | | | Sample volume [mL] | | Magma density [g/L] |
| Ca2+ | | Mg2+ | Ca [%] | Mg [%] | |
| S1 | 1.00 | | 0.07 | 94.1 | 2.4 | 97.6 | | 1000 | | 0.66 |
| S2\* | 0.34 | | 0.09 | 92.5 | 15.6 | 79.6 | | 1000 | | 0.98 |
| S2 | 1.00 | | 0.00 | 100.0 | 0.0 | 100.0 | | 800 | | 1.94 |
| S4 | 0.96 | | 0.07 | 94.1 | 3.8 | 96.2 | | 850 | | 2.27 |

\* sample contaminated with tap water

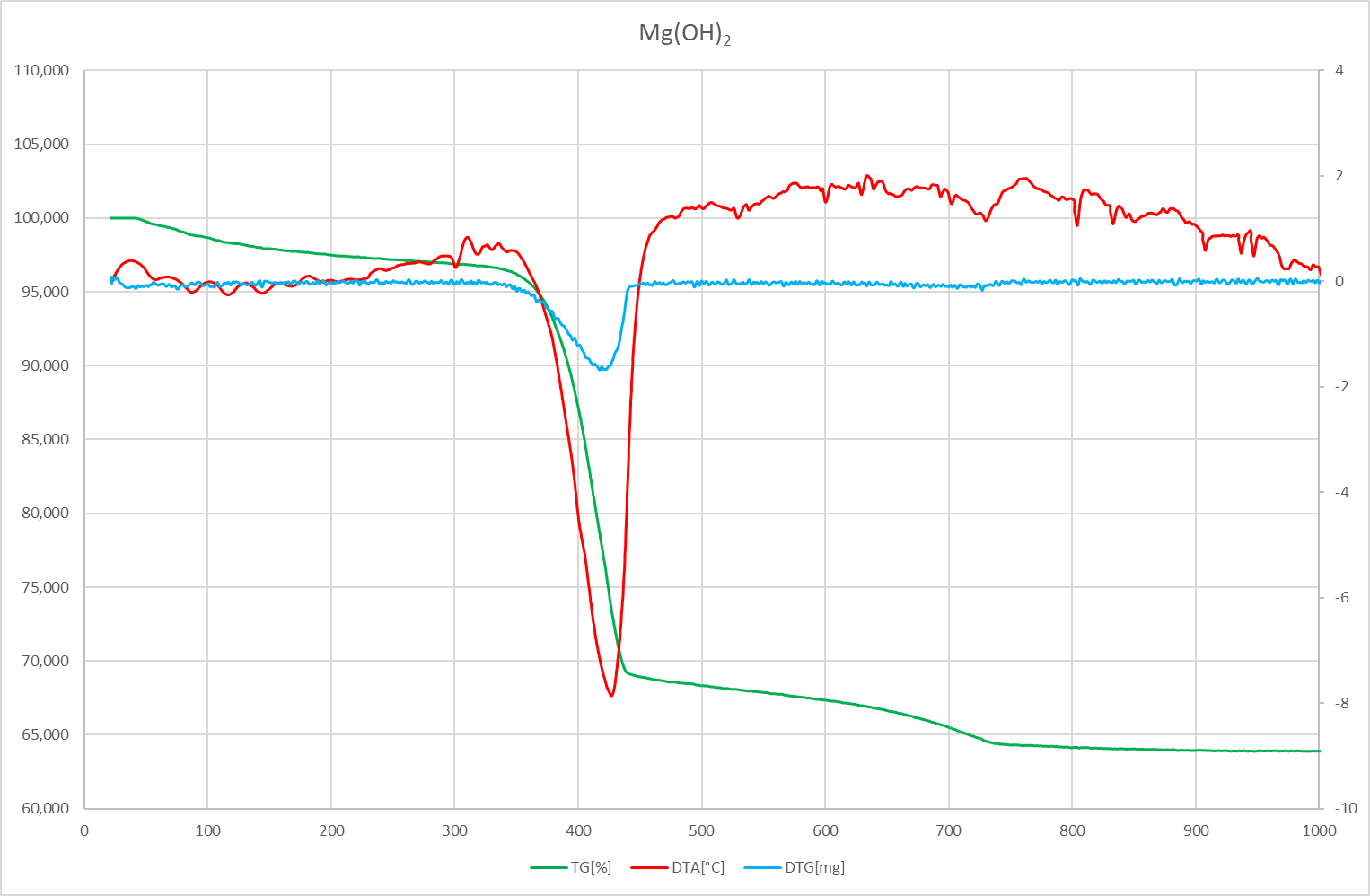


Fig. 3. Results of the TGA test of the solid sample