Analytical methods for the determination of the input material quality for gypsum wallboard production

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Abstract. Determination of the input material quality is crucial for calcination and for gypsum wallboard production itself. The article focused on the practical use of analytical methods in building practice. It presents procedures on how to determine the content of calcium sulphate in the raw material as well as the results from real measurements of the raw material samples, further calculation and evaluation. Together four samples from three different sources located near the potential gypsum wallboard production line were analysed using X-ray Diffraction (XRD) and Thermogravimetry (TGA).

1. Introduction

Wide use of gypsum wallboard in the construction of buildings (residential and commercial) is due its relatively easy installation with minimal finishing, which saves time and financial resources.

Gypsum wallboard consists of a hardened gypsum – containing a core covered with paper. It creates a sandwich structure which is commonly manufactured by placing an aqueous slurry (comprised of calcined gypsum) between two sheets of paper. Hardening of the core slurry is caused by rehydration of the calcined gypsum (calcium sulphate dihydrate). Hardening of the final product is controlled by heat treatment in a dryer which removes redundant water from the wallboard.

The manufacturing process begins with the addition of pre-generated foam to the slurry mix in order to decrease the final weight of the board. Foam is generated in a foam generator where an appropriate foaming agent is mixed with air and water.

Paper located on a long moving belt creates a carrier for the foamed gypsum slurry flowing from the mixer. Slurry is placed on top of the first paper, covered by a second paper, thus the sandwich is complete and passes through a forming station. A forming station is setup to finalize the required thickness of the gypsum board which can be changed. The width of the board is also controlled by a forming station, but it is rarely changed because of the standard dimension of 1200 mm [1].

The setting of the whole mix is controlled by adding admixtures in order to facilitate the continuous operation of the plant. Setting of the slurry is fast and begins immediately after it passes through the forming station. Then, as the belt moves, the board passes on the belt several meters to provide sufficient time for hardening and subsequent cutting. As mentioned before, the final step in gypsum wallboard production is drying so the boards are moving continually into a board dryer where they acquire their final properties. For simplification, the whole process is depicted in the figure 1.
As mentioned previously the major ingredient consists of calcined gypsum, i.e. stucco, which is produced from natural rock. It is also possible to use a synthetic material (e.g. from desulphurisation of flue gases) with high content of gypsum.

The process of the stucco manufacture is in both cases similar. In the first stage the input material is put into a kiln to remove the moisture from the rock. As soon as the excess water is removed the material is ready for grinding to a required fraction. Next step consists from calcination, while the ground gypsum is put into a calcination unit (rotary kettle), where following chemical reaction takes place:

$$\text{CaSO}_4 \cdot 2H_2O + \text{heat} \rightarrow \text{CaSO}_4 \cdot 0.5H_2O + 1.5H_2O$$  \hspace{1cm} (1)

The hardening of stucco represents a reverse reaction after mixing of stucco with an appropriate amount of water connected with heat release. The quality of the input material is crucial for the calcination and also for the production of the gypsum wallboard itself [3].

According to the economics, in each case of gypsum wallboards plant design, the first and most important fact is proximity to the source of input rock or synthetic material with appropriate quality. This appropriate quality is defined by the content of gypsum (purity) and is, as mentioned above, key for the manufacturing of wallboard. High levels of purity are desired because lower masses of wallboard can be produced. That means the higher the purity the lower the mass of wallboard produced. From this fact it is needed to find an input material that contains at least 80% gypsum. Generally, the purity of natural rock varies usually from 80 – 96% according to local conditions. The purity of synthetic material is more than 95% and another benefit is its stable composition compared to natural sources.

The article is focused on the practical use of analytical methods for the determination of the input material quality for gypsum wallboard production. It presents a procedure on how to determine the content of calcium sulphate in the raw material as well as results from the real measurements of the raw material samples (suitable/unsuitable). Together four samples from three different sources were analysed using X-ray Diffraction (XRD) and Thermogravimetry (TGA).

2. Materials and methods

The content of the gypsum in the rock or synthetic material can be determined by several analytical methods including Differential Scanning Calorimetry/Thermal Gravimetry (DSC/TGA), X-Ray Fluorescence Spectroscopy, X-Ray Diffraction, SO$_3$ analysis and also their combination [4]. As mentioned before results from XRD and DSC/TG are presented. XRD was used to define the quality of the raw material.
of the samples and quantitative analyses were performed by TG. Four samples from different locations (near the location of potential gypsum wallboard plant) were analysed. Three of them were from the Slovak Republic and one was from Poland. Two samples were collected from the same location but from different layers in the mining locality.

Qualitative analyses of the samples were carried out with an X-Ray Diffraction (XRD) using diffractometer Bruker D2 Phaser (Bruker AXS, GmbH, Germany) in Bragg-Brentano geometry (configuration Theta-2Theta), using the 1.54060 Å CuKα radiation, Ni Kβ filters and scintillation detector at a voltage of 30 kV and 10 mA current. The XRD patterns were processed using the software Diffrac.EVA v. 2.1. The ICDD PDF database (ICDD PDF – 2 Release 2009) was utilized for the phase identification.

The thermogravimetric analysis experiments and differential scanning calorimetric measurements (DSC) were carried out by using an instrument (STA 449F3 Jupiter, Netzsch, Germany). The record of mass loss in response to the temperature was collected to determine both the TG and derivative thermogravimetric (DTG) curves. Thermal decomposition of the samples was monitored from an ambient temperature to 300°C at a constant heating rate of 10°C/min under a high-purity nitrogen atmosphere of 100 mL/min.

3. Results

Thermogravimetry can be a good analytical method for the determination of gypsum rock purity because of losing crystalline water bonded in calcium sulphate dihydrate during the heating. As the dihydrate is heated the elimination of the water takes place in two steps. In the first step dihydrate is transformed to a form of hemihydrate (see equation 1) and then to the anhydrous form. It is also possible to separate the determination of both forms, but it is slightly complicated because the temperature ranges of these reactions are close together. For testing of gypsum content in natural rock this approach is not necessary and it is only needed to know the total mass loss to anhydrous form. According to stoichiometry, complete dehydration of 100% pure material is equal to mass loss of 20.9% and it can be divided into two steps (as mentioned before): 15.7% mass loss from dihydrate to hemihydrate and subsequent 5.2% mass loss to anhydride [5]. The assumption is valid only in the case, that the natural rock does not contain any other compound in the hydrated form. That is the reason why it is needed to use another analytical method for quality determination (e.g. XRD).

The results of thermogravimetry and further calculation are summarized in table 1.

| Sample | Mass Loss [%] | Gypsum content [%] |
|--------|---------------|-------------------|
| DN     | 18.99         | 90.86             |
| GOS1   | 5.53          | 26.46             |
| GOS2   | 18.13         | 86.75             |
| SH     | 3.25          | 15.55             |

It is clearly visible that two of the samples with low content of gypsum are not applicable for potential gypsum wallboard production. On the other hand, two have a high potential and can be used as a source for stucco production.

For the comparison of different samples (applicable and not applicable) representative results from thermogravimetry measurement are depicted in the figure 2 and 3. According the DSC curve (figure 2) it is possible to observe two separate reactions in the dehydration of the sample. First is located in the temperature range approx. from 120°C to 150°C (transformation to hemihydrate) followed by another one to complete dehydration around 200°C.
The GOS samples were collected from the same locality with different results of gypsum content. Probably it is caused by heterogeneous structure of the deposit and the content of gypsum is varying.
through the layers. According the XRD, the GOS1 sample contains additional mineral phases e.g. calcite, dolomite or quartz (see figure 4).

Figure 4. XRD analysis of rock material (Sample GOS1 - low content of gypsum).

To confirm the thermogravimetric analysis of DN sample, XRD pattern is also presented (figure 5). From the pattern it is evident that the sample contains only one hydrated compound which is represented by the calcium sulphate dihydrate. No others phases were observed.

Figure 5. XRD analysis of rock material (Sample DN - high content of gypsum).
4. Conclusion
Based on the presented results, it can be stated, that the combination of XRD and TG can be useful for fast and precise determination of calcium sulphate dihydrate in natural rock material. It can also be used for the quality measurement of the material in the production itself. According to analyses, two of four analysed samples proved to be appropriate for use in gypsum wallboard production (content of CaSO$_4$.2H$_2$O more than 85%). Another two samples exhibit inadequate properties because of impurities content and must be excluded from the process of production planning.

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