



## THE EFFECT OF HYDROTHERMAL REGIME ON THE DEHYDRATION PROCESS OF AGJAKAND GYPSUM STONE

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### Abstract

The research paper utilizes Agjakend gypsum stone as the primary material. The effect of the hydrothermal regime on the dehydration process of the Agjakend gypsum stone was investigated. Following the drying stage, the amount of hydration water in the product and the construction-technical properties of the resulting binder were determined.

Analysis of the obtained results indicates that the pressure inside the autoclave significantly influences both the decomposition rate of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  and the amount of residual hydration water [1]. Subsequent experiments demonstrate that, in addition to the aforementioned advantages, the construction-technical properties of high-strength gypsum binders dried via the vacuum method are superior to those of gypsum binders obtained through alternative methods.

**Key words:** Agjakend gypsum stone, high-strength gypsum, hydrothermal treatment, drying proses, autoclave processing, compressive strength limit.

### INTRODUCTION

A hemihydrate gypsum obtained by treating Agjakend gypsum stone in an autoclave at a pressure of 0.5 MPa for 8 hours was utilized in the study. To examine the impact of the drying regime on the properties of the high-strength gypsum binder, the binder obtained under the specified conditions was dried using three different methods. Analysis of the presented data shows that the strength of the vacuum-dried gypsum was 5% and 12% higher compared to the oven-dried and autoclave-dried gypsum, respectively.

It is well known that in order to obtain  $\alpha$ -hemihydrate gypsum (high-strength gypsum), gypsum stone must be processed in an autoclave.

Given that the granulometric composition of the gypsum stone and the conditions of hydrothermal treatment have a significant impact on the quality and quantity of the obtained  $\alpha$ -hemihydrate, the influence of these indicators on the properties of high-strength gypsum was investigated under laboratory conditions [1]. Gypsum stone from the Agjakend deposit, which possesses the largest reserves in the territory of Azerbaijan, was utilized in the research work. The deposit is located in the Goranboy region, with confirmed reserves amounting to 318 million tons.

The gypsum stone of the Agjakend deposit is of high quality. According to literature data, the content of dihydrate gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) in the rocks belonging to this deposit reaches up to 98% [9].

The chemical composition of the gypsum stone is as follows (in %):  $\text{CaO}$  – 28.0–34.0;  $\text{SO}_3$ – 40.0–48.6;  $\text{SiO}_2$  – 0.38–0.40;  $\text{Al}_2\text{O}_3$  – 0.17–0.64;  $\text{Fe}_2\text{O}_3$  – 0.09–0.38;  $\text{MgO}$ – 0.07–0.3; [1, 3, 9,] Insoluble residue – 0.22–2.32. The average density of the rock varies within the range of 2.38–3.02  $\text{g/cm}^3$  [1, 9]

Considering that the granulometric composition of gypsum stone and hydrothermal processing conditions have a significant impact on the quality and quantity of the obtained  $\alpha$ -hemihydrate, the effect of the mentioned indicators on the properties of high-strength gypsum was studied in laboratory conditions [1].

### Main Part.

The gypsum stone was crushed to a size of 2.5–10 mm and treated in an autoclave. The steam pressure inside the autoclave was raised over a period of 1.5 hours. Isothermal treatment was carried out for 2, 4, 6, and 8 hours. During this process, the pressure in the autoclave was 0.2, 0.3, 0.5, and 0.7 MPa, with corresponding temperatures of 110, 130, 150 and 165 $^{\circ}\text{C}$ , respectively. Drying was performed at temperatures above 105–110 $^{\circ}\text{C}$ . Subsequently, the treated gypsum stone was ground in a laboratory mill to a specific surface area of 3000–4000  $\text{cm}^2/\text{g}$ .

Following the drying stage, the amount of hydration water in the product and the construction-technical properties of the resulting binder were determined [1]

Analysis of the obtained results indicates that the pressure inside the autoclave significantly influences both the decomposition rate  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  and the amount of residual hydration water.

**Table 1. The effect of autoclave regimes on the dehydration of Agjakend gypsum stone**

| Pressure, MPa                             | 0,2  |      |      |      | 0,3  |      |      |      | 0,5  |      |      |      | 0,7  |      |      |      |
|-------------------------------------------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|
| Treatment duration, hours                 | 2    | 4    | 6    | 8    | 2    | 4    | 6    | 8    | 2    | 4    | 6    | 8    | 2    | 4    | 6    | 8    |
| Loss in autoclave, %                      | 2,9  | 5,4  | 6,9  | 8,2  | 3,7  | 5,9  | 8,3  | 10,4 | 3,8  | 9,4  | 12,4 | 13,3 | 5,4  | 10,7 | 13,4 | 13,4 |
| Content of hydration water in the product | 16,8 | 14,4 | 12,8 | 10,5 | 16,0 | 13,8 | 11,4 | 9,3  | 15,0 | 10,3 | 7,3  | 6,4  | 14,3 | 9,0  | 6,3  | 6,3  |

At low pressure (0.2 MPa), the removal of crystalline water is insufficient [1, 9]. At a pressure of 0.2 MPa for a duration of 8 hours, the amount of hydrate water in the product was 10.5%. However, at a pressure of 0.3 MPa for 8 hours, this value constitutes 9.3%. As the pressure increases, the removal of crystalline water accelerates, and the dehydration time of the residual crystalline water corresponding to hemihydrate gypsum is completed in 8 hours at a pressure of 0.5 MPa, and in 6 hours at 0.7 MPa. At the specified pressure and duration, the gypsum stone completely loses its crystalline water and converts into an anhydrous form.

When the autoclave mode is carried out at a pressure of 0.5–0.7 MPa, the drying process proceeds intensively.

The regime with a steam pressure of 0.3 MPa and an autoclave treatment duration of 8 hours can be accepted as optimal. The compressive strength of the gypsum binder obtained under this regime was 32 MPa when testing half of the dry specimens with dimensions of 4x4x16 cm [7,8].

The removal of water separated as a result of calcium sulfate dehydration and steam condensation from the autoclave is one of the primary processes in the production of high-strength gypsum. Despite the relative simplicity of the process, different approaches exist for its implementation [7, 8].

During the drying process, the temperature should not be less than the transition temperature of dihydrate gypsum into hemihydrate gypsum (105°C) and should not exceed the transition point of the hemihydrate into  $\alpha$ -soluble anhydrite (220°C).

As a result of our investigations, the use of vacuum-drying is considered appropriate for drying  $\alpha$ -hemihydrate gypsum. Vacuum drying offers the following advantages:

- The drying duration does not exceed 2 hours;
- Fuel consumption is reduced.

Subsequent experiments demonstrate that, in addition to the aforementioned advantages, the construction-technical properties of high-strength gypsum binders dried via the vacuum method are superior to those of gypsum binders obtained through alternative methods.

In the next experiment, a hemihydrate gypsum obtained by treating Agjakend gypsum stone in an autoclave at a pressure of 0.5 MPa for 8 hours was utilized [4, 5]. To examine the impact of the drying regime on the properties of the high-strength gypsum binder, the binder obtained under the specified conditions was dried using three different methods.

One part of the  $\alpha$ -hemihydrate gypsum obtained via autoclave treatment was dried in a drying oven at 110-130°C for 12 hours [4]. Another part was dried in the same autoclave at a temperature not lower than 105-110°C [1]. The third part was dried in a vacuum dryer at a temperature of 120-140°C for 2 hours.

The testing results of the obtained gypsum binder are presented in Table 2.

**Table 2. The effect of drying methods on the construction-technical properties of gypsum binders**

| Drying methods   | Hydration water content in the product, % | Water/gypsum ratio | Compressive strength limit, MPa |
|------------------|-------------------------------------------|--------------------|---------------------------------|
| In a drying oven | 0,38                                      | 0,44               | 18,7                            |
| In the autoclave | 6,30                                      | 0,42               | 22,1                            |
| In vacuum        | 6,22                                      | 0,39               | 26,3                            |

An analysis of the results presented in Table 2 indicates that the strength of the vacuum-dried gypsum was 5% and 12% higher than that of the gypsum dried in the drying oven and the autoclave, respectively [5]. This can be attributed to the lower formation of calcium sulfate dihydrate during vacuum drying compared to the other drying methods [7, 8].

## Result

1. Analysis of the obtained results indicates that the pressure in the autoclave significantly increases the decomposition rate of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$
2. The findings demonstrate that the autoclave pressure has a significant impact on the residual hydration water content.
3. Based on our investigations, the use of vacuum drying is considered expedient for drying hemihydrate gypsum. This is due to the fact that the vacuum drying duration did not exceed 2 hours, which consequently led to a reduction in energy consumption.
4. Experimental results determined that the strength limit of the vacuum-dried gypsum reached 26.3 MPa. This strength is 5% and 12% higher compared to the gypsum dried in the drying oven and the autoclave, respectively.

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