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DEHYDRATION PROCESSES AND MORPHOLOGICAL CHARACTERISTICS OF NATURAL GYPSUM MINERALS FROM THE USTYURT PLATEAU, REPUBLIC OF KARAKALPAKSTAN

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Abstract

This paper investigates the thermal and morphological characteristics of natural gypsum minerals collected from the Ustyurt Plateau, Republic of Karakalpakstan. Three gypsum samples were selected as the objects of study. The dehydration processes of the gypsum minerals were examined using differential thermal analysis (DTA) and thermogravimetric analysis (TGA). The microstructural characteristics were analyzed by scanning electron microscopy (SEM).

The obtained data provide a basis for evaluating the thermal behavior and microstructural features of the gypsum minerals and can be used for further studies aimed at optimizing their processing conditions.

Keywords: Ustyurt Plateau, gypsum mineral, dehydration process, thermogravimetric analysis, differential thermal analysis, scanning electron microscopy, morphology, thermal stability.

Introduction

In the construction materials industry, gypsum mineral ($CaSO_4 \cdot 2H_2O$) is considered one of the important raw materials due to its economic efficiency, wide processing potential, and environmental safety. Gypsum-based binders are widely used in the production of various structural elements, interior finishing materials, and shaped products, thereby occupying a significant position in modern construction [1]. The performance properties of gypsum materials are largely determined by their response to thermal exposure and their structural state. Under thermal treatment, dehydration processes occurring in gypsum minerals lead to the formation of hemihydrate and anhydrite phases, and these phase transformations directly affect the subsequent technological properties of the material [2]. From this perspective, the analysis of the thermal behavior of gypsum is essential for scientifically substantiating its processing technologies.

In addition, the morphological characteristics of gypsum crystals-such as particle shape, structural density, and porosity-are among the key factors governing their mechanical and technological performance. The features of microstructure formation are closely related to phase transformations induced by thermal effects [3], and this relationship can be effectively investigated using scanning electron microscopy.

The present study is focused on a comprehensive analysis of the thermal characteristics and microstructural state of natural gypsum minerals from the Ustyurt Plateau, Republic of

Karakalpakstan. The obtained results provide a scientific basis for substantiating the processing routes of gypsum raw materials.

Materials and Methods

Three samples of natural gypsum stone (Samples No. 1, No. 2, and No. 3) collected from the Ustyurt Plateau, Republic of Karakalpakstan, were selected as the objects of investigation. The samples belong to the same mineralogical category but differ in their structural characteristics and degree of purity.

The thermal properties of the gypsum samples were evaluated using differential thermal analysis (DTA) and thermogravimetric analysis (TGA). The measurements were performed with a simultaneous thermal analyzer under an air atmosphere over a temperature range extending from ambient temperature to elevated temperatures. TGA was employed to determine mass loss behavior and dehydration stages under thermal exposure, while DTA enabled the identification of endothermic and exothermic effects associated with phase transformations. The thermal analysis results were evaluated for each sample in terms of dehydration temperature, mass loss fraction, and energy-related parameters.

The microstructural characteristics of the gypsum samples were investigated using scanning electron microscopy (SEM). The specimens were prepared under vacuum conditions and examined at magnifications ranging from 100× to 10,000×. SEM analysis was used to assess the morphology of gypsum crystals, including particle shape (needle-like and plate-like), mutual arrangement of crystals, porosity, and structural density. The obtained micrographs were compared with the thermal analysis results to identify correlations between morphological features and dehydration processes.

Results

The thermal properties of the gypsum minerals collected from the Ustyurt Plateau were determined using differential thermal analysis (DTA) and thermogravimetric analysis (TGA). The thermal analysis results indicated that the dehydration process in all investigated samples proceeds in two distinct stages [4].

For Sample No. 1, the TGA curve revealed a major mass loss within the temperature range of 20–180 °C. This stage is associated with the release of structurally bound water molecules from the gypsum crystal lattice and corresponds to the dehydration reaction $CaSO_4 \cdot 2H_2O \rightarrow CaSO_4 \cdot 0.5H_2O$. Within this temperature interval, a clearly defined endothermic peak was observed on the DTA curve, indicating the endothermic nature of the dehydration process.

At higher temperatures, a decrease in the rate of mass loss and a reduction in thermal effects were recorded, suggesting the transition of the structure toward a relatively stable phase state (Figure 1).

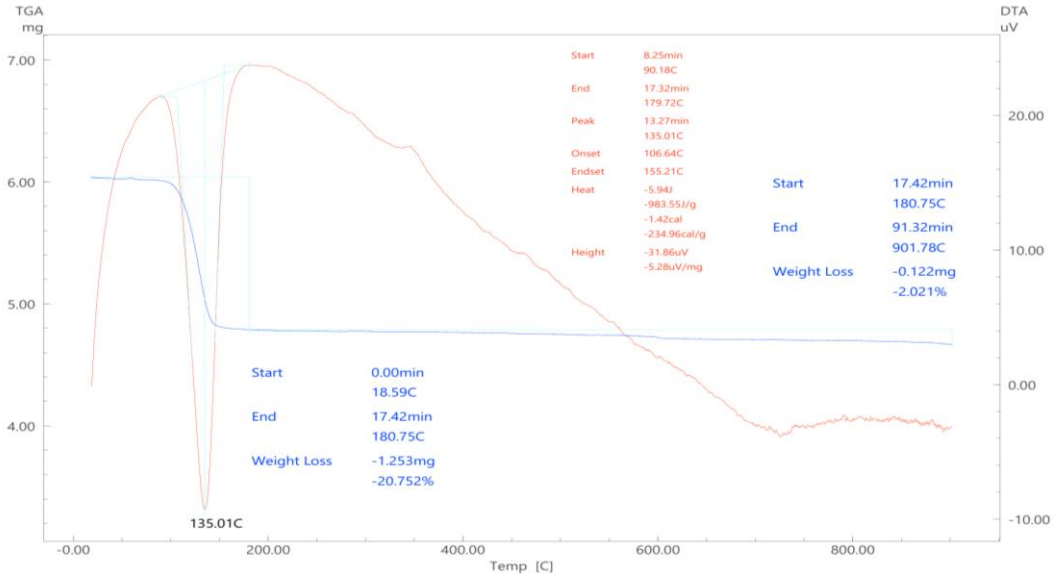

Figure 1. DTA and TGA analysis results of gypsum mineral Sample No. 1

For Sample No. 2, the thermal analysis results also demonstrated a two-stage dehydration behavior. Compared to Sample No. 1, the temperature interval corresponding to the first mass loss stage was shifted toward higher temperatures. This shift indicates a relatively stronger binding of water molecules within the gypsum crystal structure.

The endothermic peak recorded on the DTA curve exhibited high intensity, confirming the clearly pronounced endothermic nature of the dehydration process. The higher intensity of thermal effects and the occurrence of dehydration at elevated temperatures indicate that Sample No. 2 possesses greater thermal stability relative to the other investigated samples (Figure 2).

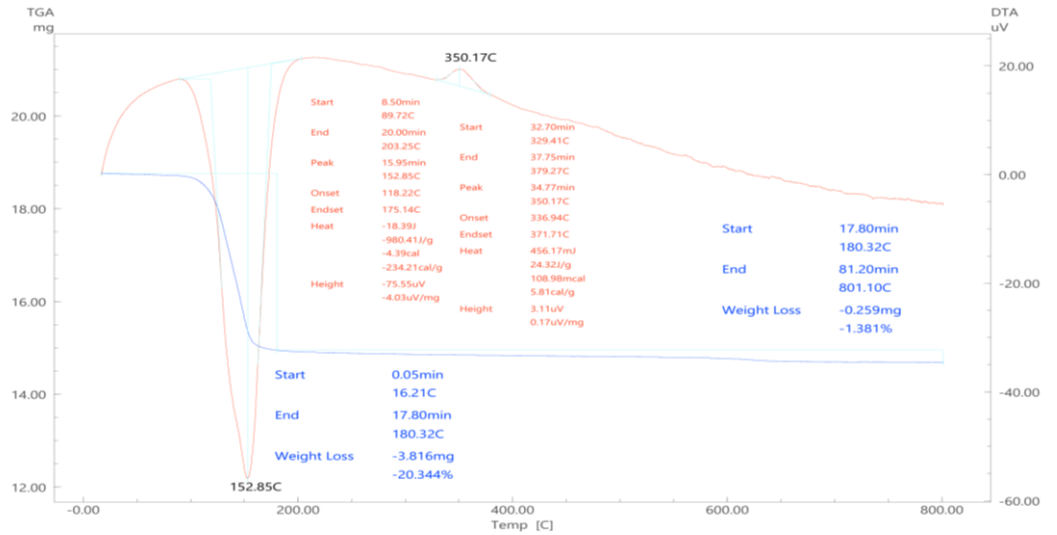

Figure 2. DTA and TGA analysis results of gypsum mineral Sample No. 2

For Sample No. 3, the thermogravimetric analysis results showed that the dehydration process begins in a lower temperature range. This behavior indicates that the release of bound water

molecules from the gypsum crystal lattice requires relatively lower energy, reflecting a weaker binding of water within the crystal structure.

The earlier appearance of the endothermic peak on the DTA curve and its lower intensity are associated with the presence of impurity components in the sample. At higher temperatures, the instability of thermal effects suggests that the structure does not reach a fully equilibrated phase state (Figure 3).

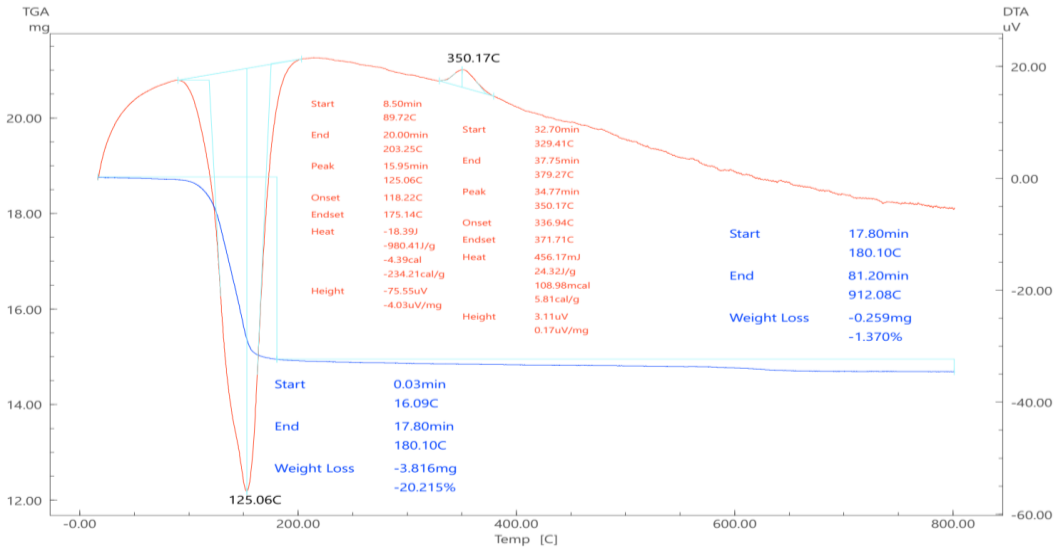

Figure 3. DTA and TGA analysis results of gypsum mineral Sample No. 3

According to the thermal analysis results, dehydration in Samples No. 1 and No. 2 proceeds within higher temperature intervals and is characterized by relatively greater energy consumption. In contrast, Sample No. 3 exhibits the onset of dehydration at lower temperatures, indicating a lower degree of thermal stability.

The microstructural characteristics of the gypsum samples were analyzed using scanning electron microscopy (SEM). SEM micrographs revealed distinct differences in crystal morphology and structural density among the samples. In Sample No. 1, gypsum crystals are predominantly needle-like, forming a dense and relatively well-ordered microstructure due to their compact mutual arrangement. In this structure, porosity is at a moderate level, and the crystalline phases are uniformly distributed. These morphological features are consistent with the relatively stable temperature interval observed for the dehydration process.

In Sample No. 2, the crystals are mainly plate-like, occasionally accompanied by needle-like elements. Owing to the multidirectional orientation of the crystals, the microstructure exhibits a relatively balanced and homogeneous distribution. The porosity ranges from low to moderate values, and this morphology is associated with stable behavior under thermal exposure.

In contrast, the microstructure of Sample No. 3 shows a lower degree of structural uniformity. The crystals are predominantly plate-like and are distributed in a dispersed and irregular manner. Increased porosity and reduced structural density correlate with the initiation of dehydration at lower temperatures. This morphological state indicates a lower thermal stability of Sample No. 3 (Figure 4).

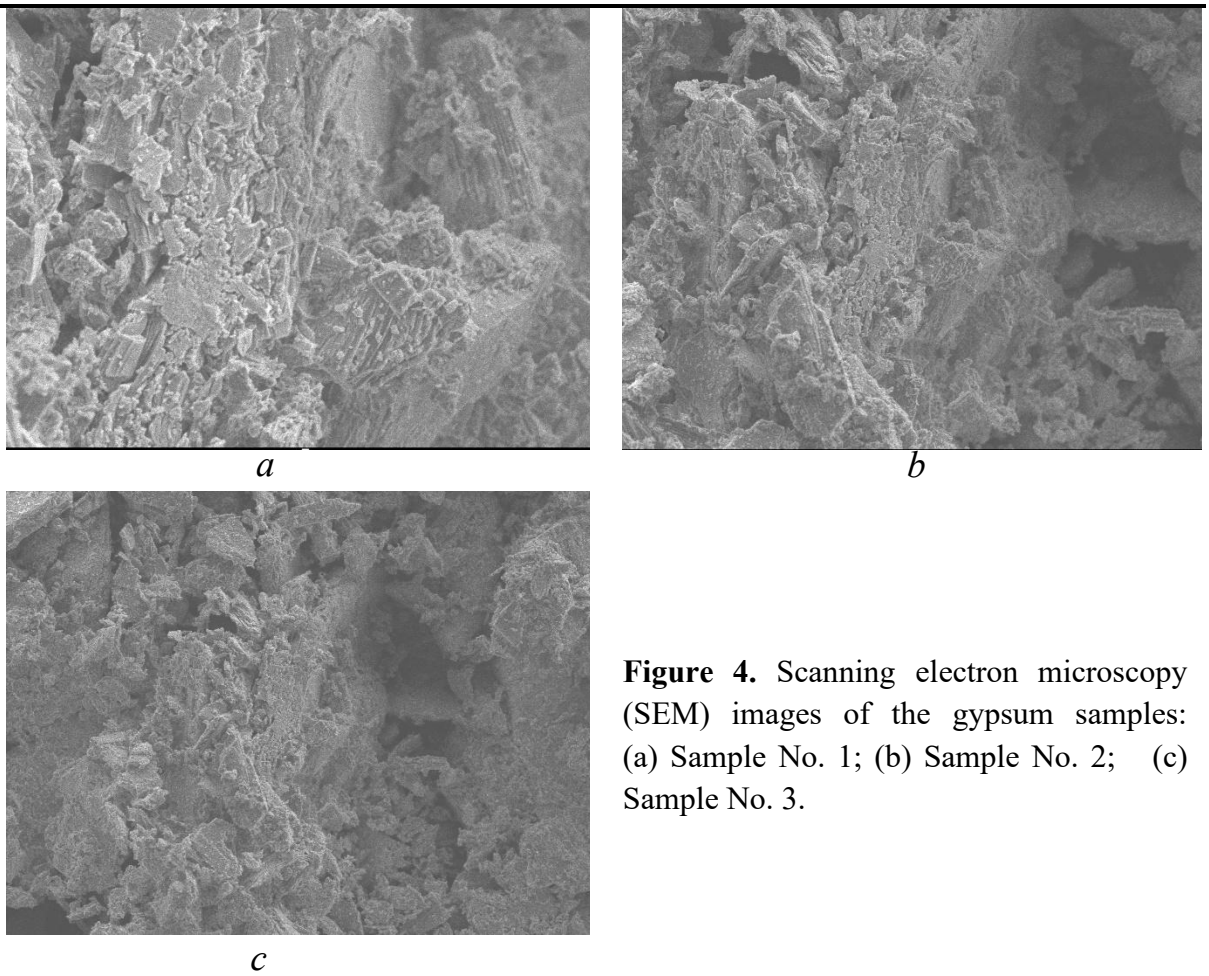

Figure 4. Scanning electron microscopy (SEM) images of the gypsum samples: (a) Sample No. 1; (b) Sample No. 2; (c) Sample No. 3.

Discussion

The obtained thermal and morphological analysis results demonstrate a close correlation between dehydration processes and microstructural characteristics of gypsum minerals from the Ustyurt Plateau. For all investigated samples, the dehydration process proceeds in two stages, corresponding to the phase transformations $CaSO_4 \cdot 2H_2O \rightarrow CaSO_4 \cdot 0.5H_2O \rightarrow CaSO_4$.

In Samples No. 1 and No. 2, the onset temperature of dehydration was observed within higher temperature intervals, and the endothermic effects were characterized by high intensity. This behavior can be attributed to the relatively strong binding of water molecules within the gypsum crystal structure and to the formation of a dense and well-ordered microstructure [5]. The scanning electron microscopy results support this interpretation, revealing a stable microstructure characterized by compactly arranged needle-like and plate-like crystals in these samples. In contrast, Sample No. 3 exhibited the initiation of dehydration at lower temperatures, accompanied by a lower intensity of the endothermic peak. This behavior is associated with the higher porosity and irregular crystal arrangement identified in the microstructural analysis. A porous and disordered structure reduces the energy required for the release of water molecules from the crystal lattice, resulting in decreased thermal stability.

Thus, the thermal analysis results are consistent with the morphological characteristics identified by SEM. When the crystal structure is dense and well ordered, the dehydration process occurs at higher temperatures and proceeds more stably. This relationship is of

particular importance for substantiating gypsum processing technologies, especially for selecting optimal temperature regimes for the production of hemihydrate and anhydrite phases.

Conclusion

The investigation of the thermal and morphological characteristics of natural gypsum minerals from the Ustyurt Plateau revealed that all analyzed samples exhibit a two-stage dehydration behavior. The first stage is characterized by the transformation of $CaSO_4 \cdot 2H_2O$ to $CaSO_4 \cdot 0.5H_2O$, while the second stage corresponds to the formation of the anhydrite phase. In Samples No. 1 and No. 2, dehydration occurs within higher temperature intervals and is accompanied by high-intensity endothermic peaks, indicating relatively greater thermal stability. In contrast, Sample No. 3 shows the onset of dehydration at lower temperatures, which is associated with higher porosity and structural irregularity in its microstructure.

Scanning electron microscopy confirmed that the shape, mutual arrangement of crystals, and structural density of gypsum have a direct influence on thermal stability. The obtained results provide a scientific basis for substantiating the processing routes of Ustyurt gypsum minerals and for selecting optimal temperature regimes in the production of high-quality gypsum-based binders.

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