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METHODS OF THERMAL AND MICROWAVE EXPANSION OF EXPANDED VERMICULITE

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Key words: vermiculite, sorbent, microwave, expanded, thermal, absorption capacity, modification.

Annotation: vermiculite from a local mine was processed for expansion. Thermal and microwave expansion processes were carried out. The advantages and disadvantages of thermal and microwave processing were considered. Complete dehydration was observed during thermal processing. The removal of molecular water from the mineral composition using the microwave method is preferable due to its low energy and time consumption.

Introduction. In the contemporary era of rapid development of the chemical industry, the practical implementation of scientific research has become more relevant than ever. Among the key chemical products widely utilized in various sectors of chemical production are sorbents. Sorbents play a crucial role in the purification of metals, the treatment of wastewater containing heavy metals, bactericides, and organic contaminants, the removal of CO_2 at elevated temperatures, the production of low-temperature resistant composite materials, sealants, and numerous other industrial applications [1, pp. 95–171].

Vermiculite was first identified in 1824 by the American scientist H. Webb. This mineral occurs naturally and is primarily formed through hydrothermal processes, groundwater percolation, or a combination of both, which leads to the transformation of various minerals such as vermiculite, hydrobiotite, and phlogopite. Vermiculite is characterized by the presence of exchangeable cations within octahedral and tetrahedral sites in the interlayer space [2, pp. 161–170].

Structurally, vermiculite is a 2:1 type phyllosilicate with a higher layer charge density (0.6–0.9 eq./formula unit) than montmorillonite. Each layer consists of a single octahedral $Mg\text{-}O_4(OH)_2$ sheet positioned between two opposing Si-O tetrahedral sheets. The layer charge primarily arises from the substitution of Al^{3+} for Si^{4+} within the tetrahedral sheet and is balanced by exchangeable interlayer cations such as Mg^{2+}, Na^+, Ca^{2+}, and K^+. Due to its elevated charge density, vermiculite exhibits a greater capacity to accommodate

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cationic surfactants within its interlayer space compared to montmorillonite. Consequently, organo-vermiculite with high carbon content and large basal spacing can be synthesized through the exchange of interlayer cations with cationic surfactants (e.g., quaternary ammonium compounds), enabling its application as an effective adsorbent for organic pollutants [3, pp. 132–133].

Furthermore, water molecules present within the interlayer spaces of vermiculite can be removed through thermal or microwave treatments. The removal of structurally bound water converts the mineral into a low-density, highly porous material, enhancing its suitability for various industrial and environmental applications.

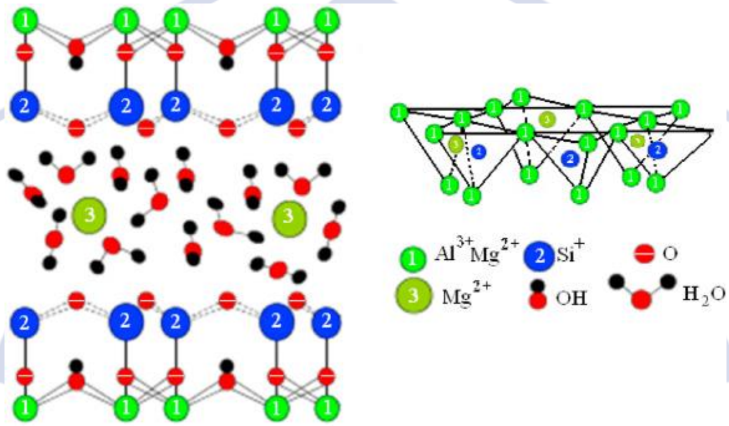

Figure 1. Molecular structure of vermiculite: silicate layers and interlayer water phases [4, pp. 122–795].

Experimental Section. Thermal expansion of vermiculite is based on rapid heating, which causes the physically bound and interlayer water within the vermiculite to convert into vapor and escape. The process of thermal expansion begins at temperatures exceeding 200°C. The vapor pressure generated by high heat enlarges the interlayer spaces, resulting in exfoliation of particles into an accordion-like morphology. The rapid release of water from the structure creates fissures within the vermiculite layers, leading to a 10–20-fold increase in volume.

Exposure to elevated temperatures induces structural changes in vermiculite, including the loss of physically bound and interlayer water [5, p. 15258]. Thermally treated expanded vermiculite can be approximately represented by the following structural formula:

$$(K_{0.5}Mg_{0.3})_{0.6}(Mg_{0.76},Fe_{0.2},Al_{0.15},Ti_{0.05})_6(Si_{0.68},Al_{0.32})_8O_{20}(OH)_{0.46}(H_2O)_{0.93}$$

Depending on bulk density and particle size, which range from 64 to 160 kg/m³, expanded vermiculite exhibits a significantly enhanced water absorption capacity. Additionally, it

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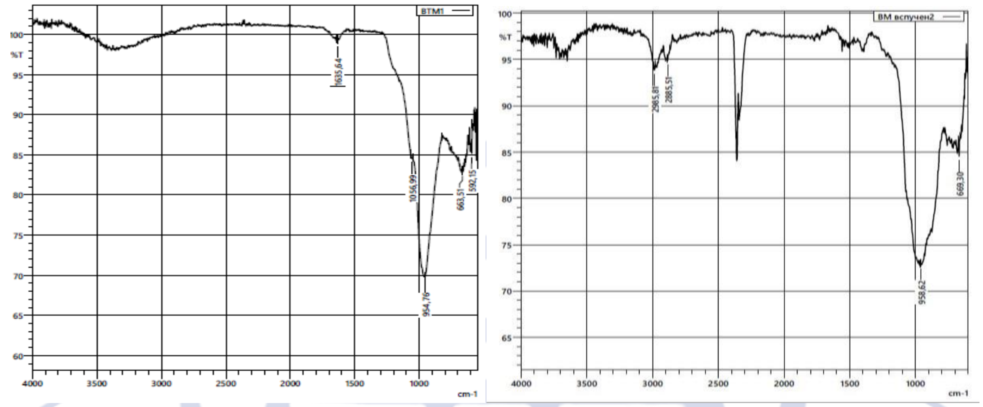
demonstrates low thermal conductivity, high temperature resistance, and excellent sound absorption properties.

The physical and chemical changes induced by thermal expansion are clearly observed in the infrared (IR) spectra of raw and expanded vermiculite, as shown in Figure 2.



(a) (b)

Figure 2. IR spectra of Tebinbuloq cone vermiculite in natural (a) and expanded (b) states.

In the analysis of natural vermiculite (a), vibration peaks at 1056.99 cm^{-1} correspond to the stretching vibrations of Si–O bonds and hydroxyl groups. In the thermally expanded vermiculite sample, slightly shifted, broad, and intense stretching peaks were observed at 2985.81 and 2885.51 cm^{-1}, which are attributed to O–H groups involved in hydrogen bonding.

For exfoliation of vermiculite via microwave heating, a laboratory-scale microwave oven (OLT-CR-5) was employed. To enhance expansion efficiency, the samples were pretreated for 20 minutes with 10% and 20% hydrogen peroxide solutions and water. The wet vermiculite was then spread on special numbered paper with high water absorption capacity, and surface moisture was removed. From each sample, 1.5 g was taken and placed into specially prepared polyethylene containers.

The microwave oven was set to operate at 650, 950, and 1000 W for durations of 10, 20, 30, 60, 90, and 120 seconds, at a frequency of 2400 MHz. During microwave heating, the decomposition of hydrogen peroxide produced oxygen, which induced fissures in the silicate layers. The escaping water vapor caused exfoliation of the vermiculite particles. After microwave treatment, the expanded vermiculite samples were placed in a desiccator and allowed to cool to room temperature. The samples were then dried in an oven at 110°C, and their mass and density were determined.

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The experiments demonstrated that, under the applied conditions, natural vermiculite achieved the highest expansion efficiency when processed in the microwave oven at 1000 W. Table 1 summarizes the amount of water lost during expansion of natural vermiculite pretreated with various aqueous solutions.

During microwave-assisted modification of natural vermiculite, pretreatment with water and hydrogen peroxide solutions facilitated the removal of water in different binding states within the mineral. The effectiveness of water removal increased in the follo wing order: natural state < water < 10% H_2O_2 < 20% H_2O_2 ≤ 25% H_2O_2.

Table 1. Water loss of natural raw vermiculite samples treated with water, 10%, 20%, and 25% H_2O_2 at 1000 W microwave power.

Water loss (%)					
Sample	**Microwave exposure time (s)**				
	10	**20**	**30**	**90**	**120**
Natural state	2	2,5	3	5	10
Treated with water	4	5	7	7,1	7,5
Treated with 10% H_2O_2	8	8,5	9	9,3	12
Treated with 20% H_2O_2	10	12	14	15	17
Treated with 25% H_2O_2	10,5	12	14,3	15	17,1

The process of expanding or activating vermiculite by removing water from its structure requires a significant amount of energy. **Figure 3** compares the energy consumption of microwave heating of vermiculite at the laboratory scale, industrial scale, and in packed-bed reactors [136, pp. 601–611]

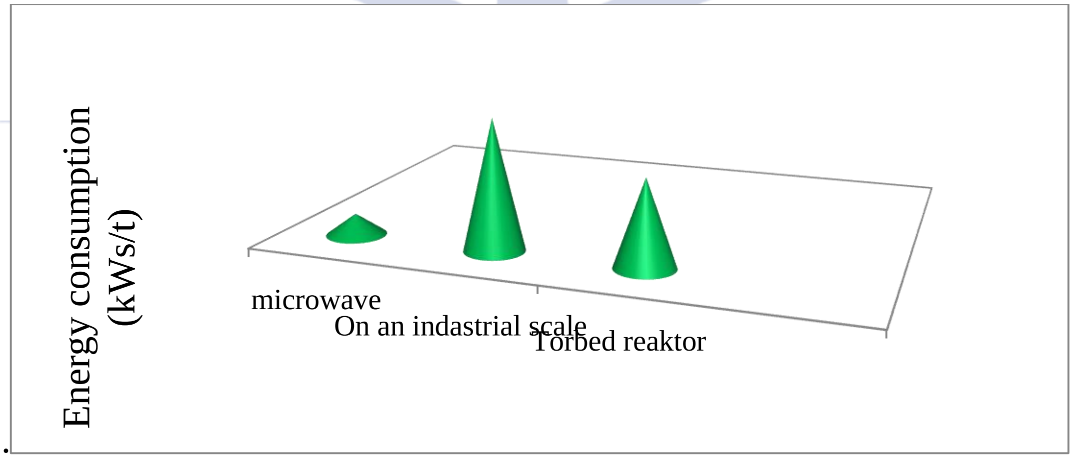

Figure 3. Energy consumption during the vermiculite expansion process.

Conclusion. Local vermiculite from the Tebinbuloq deposit was thermally treated at 850–900°C to achieve expansion. As a result, metallic-colored, glossy samples with a density of 150 kg/m³ were obtained, and their properties were investigated. Microwave-assisted expansion of vermiculite yielded samples with a lower density of approximately 100 kg/m³. The main drawback of thermal expansion is the high energy consumption required for the process. In contrast, microwave treatment offers advantages in terms of time and energy efficiency. However, the retention of moisture, i.e., the release of molecular water to the external layers, complicates the production process and necessitates additional drying.

Currently, vermiculite expansion is predominantly performed using conventional thermal treatment. Nevertheless, applying microwave-assisted expansion on a large scale could significantly reduce energy consumption and processing time, offering a more efficient alternative for industrial production**.**

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