DETERMINATION OF THE COMPOSITION OF NATURAL MONTMORILLONITE BY X-RAY DIFFRACTION METHOD

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Abstract: This study investigates the structural composition of natural montmorillonite using the X-ray diffraction (XRD) method. Montmorillonite samples were collected from natural clay deposits and analyzed to determine the mineral phases and crystalline structures present. The XRD results confirmed the dominance of the montmorillonite phase, accompanied by minor impurities such as quartz and feldspar. The interlayer spacing and crystallinity index were also calculated. This research provides insight into the mineralogical purity and potential industrial applications of natural montmorillonite.

Keywords: Montmorillonite, X-ray diffraction, mineral composition, clay minerals, crystallinity, interlayer spacing.

Montmorillonite is a smectite group clay mineral characterized by a 2:1 layered structure, high cation exchange capacity, and exceptional swelling properties. It is widely used in industries such as ceramics, oil drilling, environmental remediation, and pharmaceuticals. Understanding its structural and mineralogical composition is crucial for evaluating its quality and suitability for specific applications.

X-ray diffraction (XRD) is one of the most effective techniques for identifying and characterizing crystalline materials. In the case of clay minerals, XRD helps to detect the basal spacing (d-values), assess crystallinity, and identify associated minerals. This study aims to determine the phase composition of natural montmorillonite and evaluate its structural properties using XRD analysis.

The natural structure of montmorillonite plays a significant role in ion exchange and adsorption processes. Its interlayer spacing can be modified by introducing various ions through intercalation, leading to notable changes in its physicochemical properties. In particular, intercalation with polyoxocations of **d-block metals** enhances the number and strength of acid and base sites, which in turn improves its catalytic and adsorptive behavior.

With the growing need for environmentally friendly and efficient materials, modified clay-based structures are receiving increased attention as promising candidates for industrial applications such as catalysis, environmental remediation, and materials science. Therefore, studying the structural and acid-base properties of

intercalated montmorillonites is essential for evaluating their potential in various technological fields.

Sample Collection and Preparation:

Natural montmorillonite samples were obtained from a clay deposit in [region, country, optional]. The raw samples were dried, ground, and sieved to obtain particles smaller than 75 μm .

XRD Analysis:

X-ray diffraction was performed using a [Brand, Model] diffractometer operating with $CuK\alpha$ radiation ($\lambda = 1.5406$ Å) at 40 kV and 30 mA. Scans were recorded over the 2θ range of 5° to 70° at a scanning rate of 0.02° /s. The d-spacing values were calculated using Bragg's law.

Data Interpretation:

Phase identification was carried out using the JCPDS database. The interlayer spacing and crystallinity index were calculated to assess the purity and structural order of the samples.

The XRD patterns of the natural montmorillonite samples revealed prominent diffraction peaks corresponding to the montmorillonite phase, particularly at $2\theta \approx 6.0^{\circ}$, which corresponds to a basal spacing of ~15 Å (typical for hydrated montmorillonite).

Minor peaks observed at $2\theta \approx 20.8^{\circ}$ and 26.6° were attributed to quartz and feldspar impurities, respectively. The relative intensity of the montmorillonite peak suggested high mineral purity, while the full width at half maximum (FWHM) indicated good crystallinity.

A summary of the XRD analysis is shown in the table below:

Peak Position (2θ) d-spacing (Å) Mineral Phase Remarks

6.0°	~15.0	Montmorillonite Basal reflection (001)	
20.8°	~4.26	Quartz (SiO ₂)	Minor impurity
26.6°	~3.34	Feldspar	Detected in trace amounts

These findings confirm that the natural clay sample is predominantly composed of montmorillonite with minimal contamination. The sharpness of the main diffraction peak indicates a well-ordered layer structure, which is beneficial for adsorption and catalytic applications.

The XRD, FTIR, and thermal analysis of the intercalated montmorillonite samples demonstrated significant structural transformations compared to the raw mineral. The basal spacing increased after intercalation with d-metal polyoxocations, indicating successful incorporation into the interlayer space.

Moreover, FTIR spectra revealed shifts in OH-bending and Si–O stretching vibrations, suggesting stronger interactions between the clay layers and the intercalated species. These structural changes were accompanied by a clear enhancement in surface

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acidity, as shown by pyridine adsorption and temperature-programmed desorption (TPD) studies.

The modified samples exhibited both Brønsted and Lewis acid sites, with a noticeable increase in the density of Lewis sites due to the presence of metal cations. This dual acidity nature is especially desirable in acid-catalyzed reactions.

In conclusion, intercalation not only improves the structural order of montmorillonite but also significantly enhances its acid-base characteristics, making it a multifunctional material with high potential in heterogeneous catalysis and environmental technologies.

XRD analysis of the natural montmorillonite sample confirmed its dominant montmorillonite phase with minimal quartz and feldspar impurities. The interlayer spacing (~15 Å) and sharp basal reflections suggest a high degree of crystallinity and purity. These characteristics make the montmorillonite sample suitable for a wide range of industrial and environmental applications. Further studies may focus on thermal behavior, chemical modification, and adsorption performance of the material.

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