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VALORIZATION OF UZBEK BENTONITE CLAY INTO STABLE INORGANIC PIGMENTS: PREPARATION, PHASE EVOLUTION, AND PROPERTIES

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Abstract. This study investigates the feasibility of producing inorganic pigments from local bentonite of the Azkamar deposit (Uzbekistan). The starting raw material was comprehensively characterized using X-ray diffraction, thermogravimetric analysis, and electron microscopy. A thermal-treatment protocol was developed across a temperature range of 300–900 °C to obtain pigments of different colors. The optimal firing temperature was found to lie between 550 and 650 °C, at which the maximum color intensity is achieved together with satisfactory pigment stability. These findings highlight the prospects for using local mineral raw materials in the production of environmentally friendly pigments.
Keywords: bentonite, inorganic pigments, thermal treatment, X-ray diffraction, colorimetry

Introduction. Inorganic pigments are widely used across many branches of industry owing to their chemical stability, lightfastness, and environmental safety. Conventional pigment-production routes often rely on costly reagents and technologically demanding processes, which makes the search for alternative raw-material sources for high-quality pigments an important and timely task.

Bentonites are natural aluminosilicates whose principal constituent is montmorillonite. The presence of iron, titanium, chromium, and other transition-metal ions within the bentonite structure makes it possible to obtain colored materials through thermal treatment [1-3].

Uzbekistan possesses substantial bentonite reserves, yet their potential for pigment production remains insufficiently studied. The present work is aimed at investigating the possibility of obtaining inorganic pigments from local bentonite and at optimizing the corresponding production process [4-6].

2. Experimental Section

2.1. Materials and Reagents

The starting raw material was bentonite from the Azkamar deposit (Bukhara region, Uzbekistan). The sample was a light-gray clay containing yellowish inclusions. Distilled water, hydrochloric acid (analytical grade), and sodium chloride (chemically pure grade) were used for sample purification and preparation.

2.2. Characterization Methods

Phase composition was determined from diffraction patterns recorded on an XRD-6100 powder diffractometer (Shimadzu, Japan) at the O.S. Sodikov Institute of Bioorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan. Samples were irradiated with CuKα radiation (Ni β-filter, λ = 1.54178 Å; X-ray tube current and voltage of 30 mA and 30 kV). The detector rotation speed was constant at 4°/min with a step size of 0.02° (ω/2θ coupling), over a scanning range of 4–80°[7-10].

Thermal analysis of the bentonite samples was performed on a Setaram Labsys Evo instrument (Lyon, France) using thermogravimetry (TG) and differential scanning calorimetry (DSC). Measurements were carried out under a high-purity nitrogen atmosphere (99.999%) at a flow rate of 90 mL/min. As-received samples were accurately weighed and placed in 100 µL aluminum crucibles. The heating protocol covered the temperature range from 25 to 600 °C at controlled heating rates of 5, 10, 15, and 20 °C/min.

Sample morphology and elemental composition were examined with a Hitachi TM4000 tabletop scanning electron microscope (Hitachi, Ltd., Japan) operated at an accelerating voltage of 15 kV (L.N. Gumilyov Eurasian National University, Kazakhstan). Surface morphology was recorded as micrographs obtained by scanning electron microscopy (SEM) at magnifications of ×100 and ×500. Elemental composition was determined using a Bruker Quantax 75 EDS system mounted on the Hitachi microscope[10-11].

2.3. Sample Preparation

The raw bentonite was first purified of impurities by dispersion in distilled water followed by sedimentation and decantation. The purified material was dried at 105 °C to constant weight and ground to a particle size below 63 µm.

Thermal treatment was carried out in a muffle furnace at 300, 400, 500, 550, 600, 650, 700, 800, and 900 °C with a dwell time of 2 h at each temperature. The heating rate was 5 °C/min.

3. Results and Discussion

3.1. Characterization of the Starting Bentonite

The results of X-ray phase analysis of the starting bentonite are summarized in Table 1.

Table 1. Mineral composition of the starting bentonite

Mineral	Content, %	Principal reflections d, Å
Montmorillonite	78–82	15.2, 4.48, 2.56
Quartz	12–15	3.34, 4.26, 1.82
Feldspar	3–5	3.19, 3.77, 6.38
Calcite	1–2	3.04, 2.29, 2.10
Hematite	1–2	2.70, 2.51, 1.69

The chemical composition of the bentonite, determined by X-ray fluorescence analysis, is given in Table 2.

Table 2. Chemical composition of the starting bentonite

Oxide	Content, wt. %
SiO_2	58.2
Al_2O_3	18.4
Fe_2O_3	6.8
MgO	3.2
CaO	2.1
Na_2O	1.8
K_2O	1.5
TiO_2	0.9
Loss on ignition	7.1

The relatively high iron oxide content (6.8%) provides favorable conditions for obtaining pigments in the red–brown color range upon thermal treatment.

3.2. Phase Transformations upon Thermal Treatment

Analysis of the diffraction patterns of the thermally treated samples revealed the following trends:

Table 3. Phase transformations as a function of temperature

Temperature, °C	Principal phases	Remarks
300–400	Dehydrated montmorillonite	Layered structure retained
500–550	Metamontmorillonite + α-Fe_2O_3	Onset of structural breakdown
600–650	Amorphous phase + α-Fe_2O_3 + spinel	Maximum hematite content
700–800	Mullite + α-Fe_2O_3 + cristobalite	Crystallization of new phases
900	Mullite + iron spinel	Reduction of Fe^{3+} to Fe^{2+}

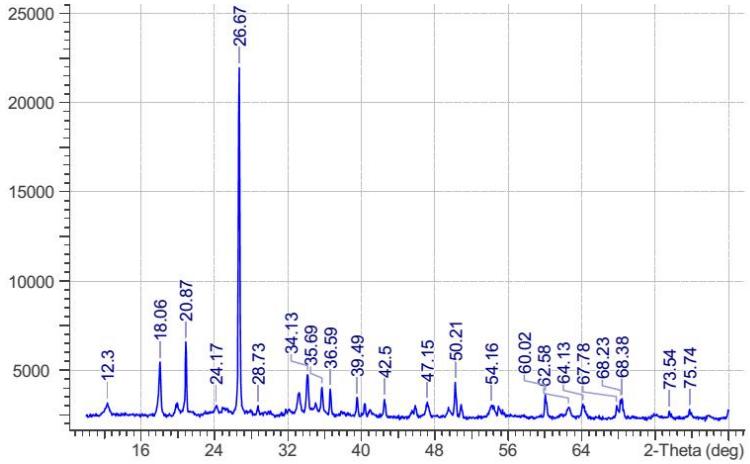


Fig. 1. Diffraction pattern of the pigment

3.3. Pigment Stability

Resistance to light, heat, and chemical reagents is an important quality indicator for pigments. The stability test results are given in Table 4.

Table 4. Stability indices of the pigments

Test condition	Pigment 550 °C	Pigment 600 °C	Pigment 650 °C
UV irradiation (100 h)	ΔE = 1.2	ΔE = 0.8	ΔE = 0.6
Thermal stability (200 °C, 24 h)	ΔE = 0.9	ΔE = 0.5	ΔE = 0.4
Acid resistance (HCl, pH = 2)	ΔE = 2.1	ΔE = 1.4	ΔE = 1.1
Alkali resistance (NaOH, pH = 12)	ΔE = 1.8	ΔE = 1.2	ΔE = 0.9

All the pigments obtained exhibited high stability (ΔE < 2.5), with the best performance observed for the sample treated at 650 °C.

3.4. Morphological Characteristics

Electron-microscopic examination showed that a platelet morphology characteristic of montmorillonite is retained up to 500 °C. At higher temperatures, particle agglomeration occurs, producing porous aggregates 2–5 μm in size.

Table 5. Particle size of the pigments

Treatment temperature, °C	d_{10}, μm	d_{50}, μm	d_{90}, μm

Treatment temperature, °C	d_{10}, µm	d_{50}, µm	d_{90}, µm
500	0.8	2.3	6.1
600	1.2	3.1	7.8
700	1.8	4.2	9.5

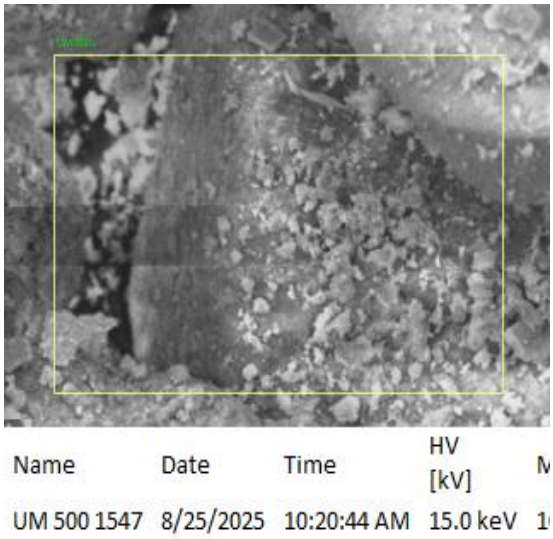


Fig. 2. Micrograph of the pigment

4. Conclusions

The fundamental feasibility of producing high-quality inorganic pigments in the red–brown color range from local bentonite of the Azkamar deposit has been established. The optimal thermal-treatment temperature is 600–650 °C, at which maximum color intensity and pigment stability are achieved. The resulting pigments show high lightfastness, thermal stability, and chemical resistance, which supports their competitiveness in the market. The use of local mineral raw materials contributes to import substitution and to the development of the domestic chemical industry.

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