

## RESEARCH OF KARAÇEVA BENTONITE IN NATURAL STATE AND AFTER TREATMENT

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### ABSTRACT

The aim of this paper is to research the mineralogical and structural properties of Karaçeva bentonite, as well as to determine that how acid activation changes the structural and mineralogical properties of bentonite. From the obtained results was found that Karaçevabentonite contains montmorillonite as the main mineral, accompanied by other minerals, such as illite, quartz, feldspar, dolomite and calcite. The natural bentonite fractions with a particle size smaller than 2 µm are characterized by increased montmorillonite content and reduction of quartz content. The results of diffractometric and thermogravimetric measurements shows that the Karaçevabentonite contains very little calcite and dolomite. The diffractometric studies has shown that the basic activation of these bentonites has not resulted in any change in their mineral composition. Acid activation, has changed the structural and mineralogical properties of Karaçevabentonite.

**Keywords:** bentonite, acid activation, diffractometric analysis, DTA and TGA analysis

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### INTRODUCTION

Clays whose basic clay mineral is montmorillonite, or smectite, are generally called bentonites<sup>1</sup>. The physico-chemical properties of bentonites plays a major role in all these applications<sup>2</sup>.

Bentonites are seldom found as monomineralic clays and may contain other clay and nonclay minerals and also some organic impurities<sup>3</sup>. Owing to their structural properties, clays are used in many ways including construction, foundry, insulation, adsorption, filter aids, catalysis<sup>4</sup>.

The properties of bentonite clays depends on the structure and chemical composition, exchangeable ion type and small particle size of smectites.

These properties include large specific surface area, high cation exchange capacity, and others physical-chemical properties, such as: swelling, plasticity, cohesion, compressibility, strength, particle size, adsorptive properties, pore structure, surface acidity, and catalytic activity<sup>5</sup>.

As it is well known, the clay minerals are hydrous aluminum silicate and are classified as phyllosilicates. They have a layered structure which can be described as constructed from two modular units: a sheet of corner-linked tetrahedra and a sheet of edge-linked octahedra<sup>6</sup>.

Bentonite clays have the best adsorption properties, into which composition enters montmorillonite. Montmorillonite is capable of essentially increasing initial volume on account of water adsorption in the interlayer sites<sup>7</sup>.

### EXPERIMENTAL

#### Materials and Methods

In this paper, the research object was bentonite of Karaçeva source, which is in the eastern part of Kosovo.

Experimental research of bentonite samples is carried out using these methods: acidic and basic treatment, diffractometric analysis, DTA and TGA analysis.

#### Acidic and Basic Treatment

The acid treatment, besides leaching cations from octahedral and tetrahedral sheets, dissolves impurities such as calcite and replaces the exchangeable cations with hydrogen ions<sup>8</sup>.

Acid activation is carried out with the boiling of bentonite in the sulfuric acid digestion, with concentrations of 10% and 20%, for 3 hours. Samples are basically activated with a solution of 3% Na<sub>2</sub>CO<sub>3</sub>.

### Diffractometric Analysis

The diffractometric analysis of the powders with X-ray is the basic technique for clay mineral analysis. This method is used to identify the mineral components of bentonite, such as montmorillonite, feldspar, quartz, calcite, gypsum and zeolites. By means of the diffractometric analysis, is determined the mineralogical composition of the natural sample, the sample with fraction smaller than 2 μm, the sample activated with 10% H<sub>2</sub>SO<sub>4</sub> and with 3% Na<sub>2</sub>CO<sub>3</sub>.

### Differential thermal analysis and Thermogravimetric Analysis

Differential thermal and thermogravimetric analyzes have been used as thermal methods for characterizing the mineralogical composition of the analyzed samples, for quantitative determination of constitutional water, and water contained in the pore and between the layers<sup>9</sup>. With differential thermal and thermogravimetric analysis were investigated the natural sample and activated sample with 3% Na<sub>2</sub>CO<sub>3</sub>.

Differential thermal analysis curves of smectite are usually divided into three areas:

1. Low-temperature areas (<300 °C),
2. Dehydroxylation zone (400-750) °C and
3. Areas with high temperature (> 800 °C)<sup>10</sup>.

## RESULTS AND DISCUSSION

The summarized semi-quantitative representation of the mineralogical analysis for various Karaçevabentonite samples is given in Table-1.

In figures, 1-4 have presented the diffractograms of Karaçevabentonite in a natural state, fractionated and activated in the acidic and basic way.

Table-1: Results of the diffractometric analysis.

Sample	M	I	C	Q	D	F
Natural sample	++++	++	+	+	+	+
Fraction ≤2μm	++++	++	+	-	-	+
Sample activated with 10% H <sub>2</sub> SO <sub>4</sub>	++++	++	-	++	+	+
Sample activated with 3% Na <sub>2</sub> CO <sub>3</sub>	+++	++	-	++	+	+

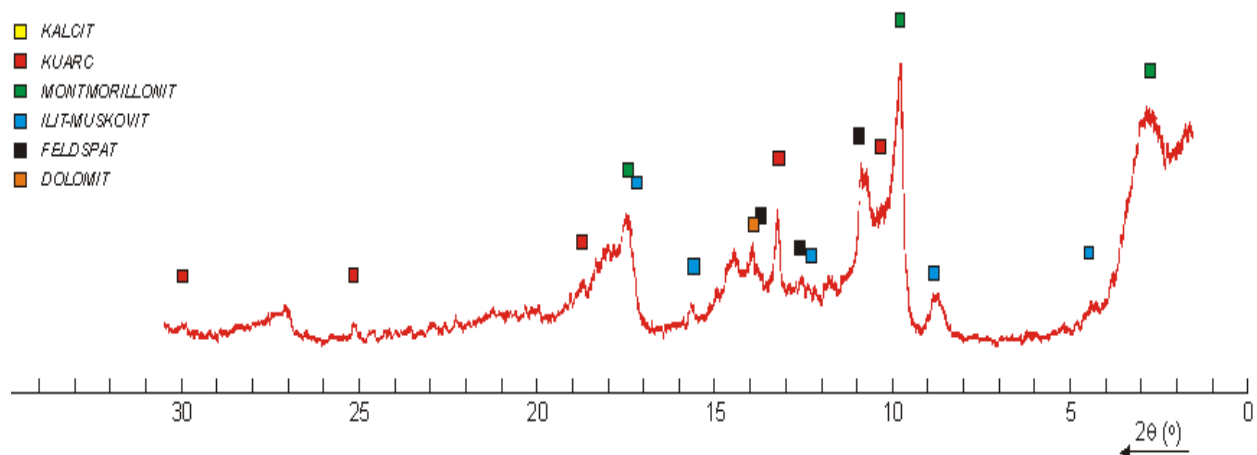


Fig.-1: The diffractogram of a natural sample of Karaçeva bentonite.

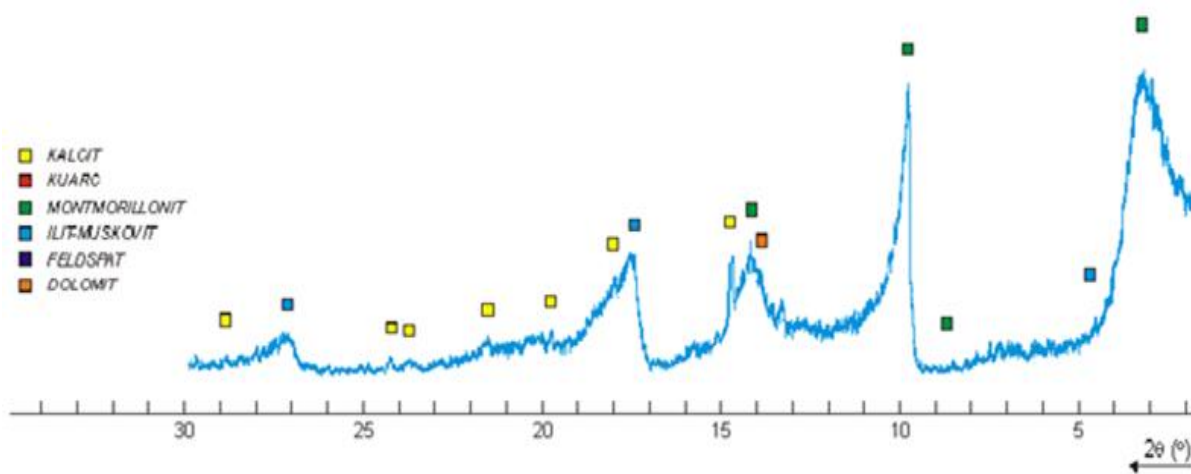


Fig.-2: The diffractogram of Karaçeva bentonite–fraction less 2  $\mu\text{m}$ .

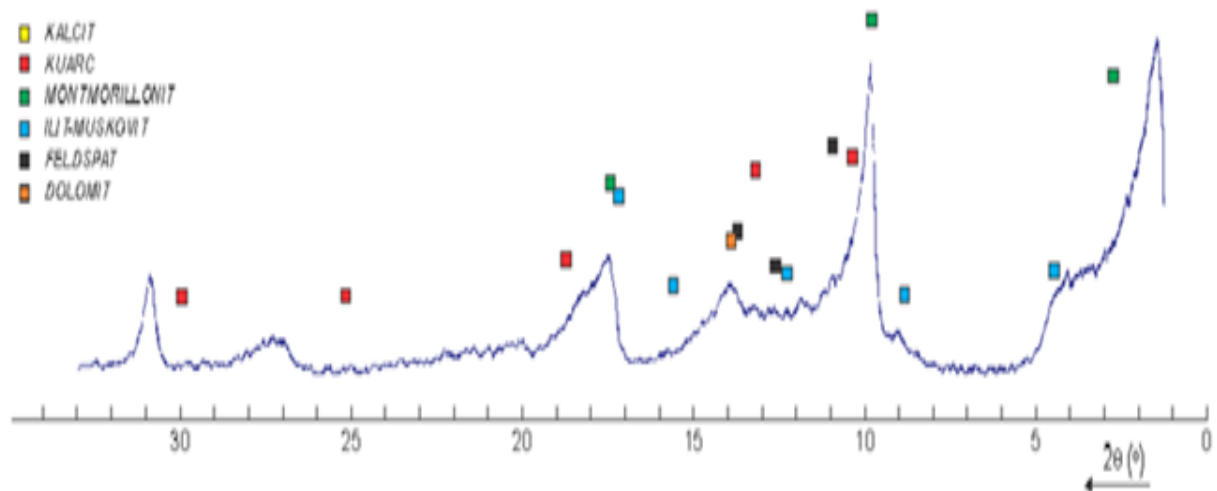


Fig.-3: The diffractogram of Karaçeva bentonite activated with 10%  $\text{H}_2\text{SO}_4$ .

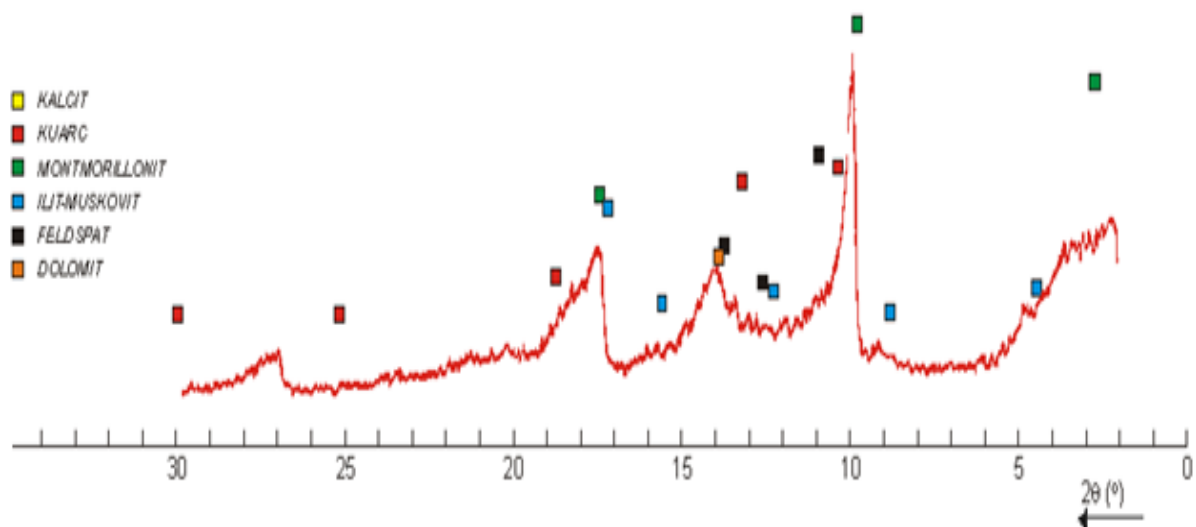


Figure 4. The diffractogram of Karaçeva bentonite activated with 3%  $\text{Na}_2\text{CO}_3$ .

#### Results of DTA and TGA analysis

Figure 5 shows the differential thermal and thermogravimetric analysis of Karaçeva natural bentonite, while in figure 6 are presented the differential thermal and thermogravimetric analysis of bentonite activated with 3%  $\text{Na}_2\text{CO}_3$ . In the Table-2 are presented the results of different parameters of DTA and TGA analysis.

Table-2: Results of DTA and TGA analysis

Parameters	Natural sample	Activated sample with 3% $\text{Na}_2\text{CO}_3$
$t_v$ ( $^{\circ}\text{C}$ )	160	140
$t_q$ ( $^{\circ}\text{C}$ )	580	570
$t_h$ ( $^{\circ}\text{C}$ )	700	700
$t_c$ ( $^{\circ}\text{C}$ )	780	780
$t_r$ ( $^{\circ}\text{C}$ )	870	850
$W_v$ (%)	8.14	7.06
$E_h$ (%)	3.14	4.25

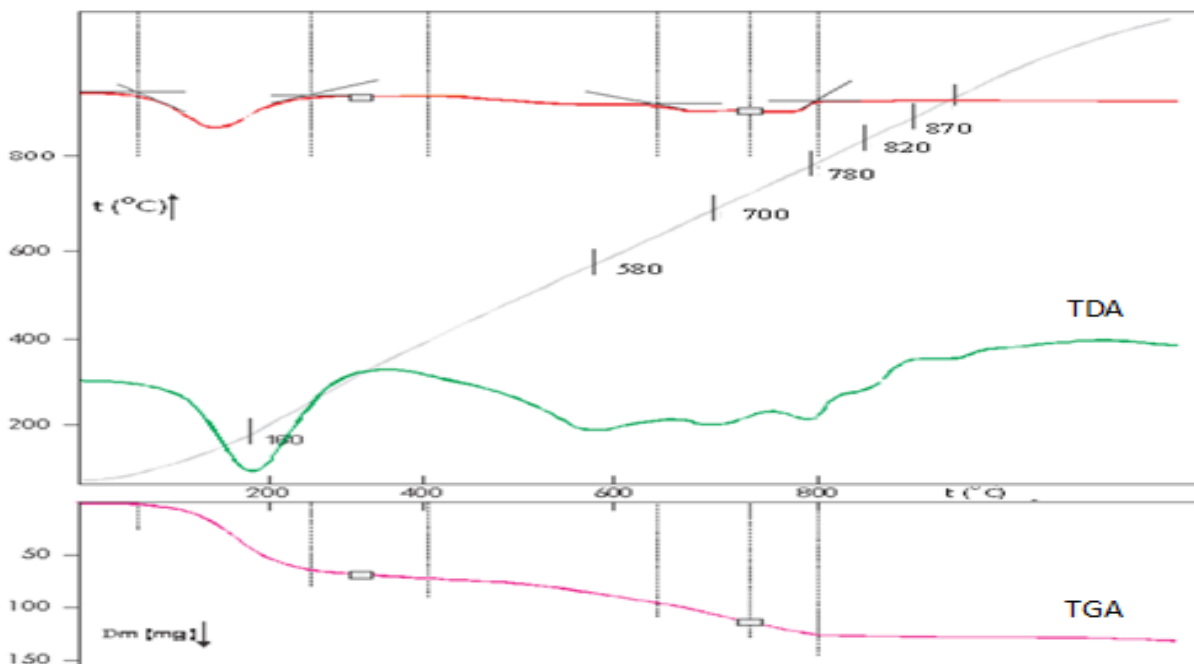


Fig.-5: DTA and TGA analysis of a natural sample

From the diffractometric analysis (Figures-1 to 4 and Table-1), it is seen that montmorillonite is the main component of bentonite for all the tested samples, illite is presented as minor components, while calcite, dolomite, quartz, and feldspar appear as components in the trace.

With the diffractometric analysis of natural bentonites, it has been observed that natural bentonite fractions with a particle size smaller than  $2\ \mu\text{m}$  are characterized by increased montmorillonite content and reduction of quartz content.

From Fig.-5 and Table-2, for the natural bentonite sample, it is seen that the minimum dehydration temperature is reached at  $160^\circ\text{C}$ , in the low-temperature zone ( $<300^\circ\text{C}$ ).

The minimum temperature of dehydroxylation and transformation of  $\beta$ -quartz in  $\alpha$ -quartz occurs at  $580^\circ\text{C}$  (dehydroxylation zone  $400$ - $750^\circ\text{C}$ ), the minimum calcite decomposition temperature is  $700^\circ\text{C}$ , while the maximum dehydration temperature is  $780^\circ\text{C}$ . The minimum digestion temperature occurs at  $870^\circ\text{C}$ , water contained in the pore and between the layers is  $8.14\%$ , while the constitutive water is  $3.14\%$ .

From figure 6 and table 2 for the activated bentonite sample, it is seen that the minimum dehydration temperature is reached at  $140^\circ\text{C}$ , in the low-temperature zone ( $<300^\circ\text{C}$ ), the minimum dehydroxylation temperature and the transformation of  $\beta$ -quartz in  $\alpha$ -quartz occur at temperature of  $570^\circ\text{C}$  (dehydroxylation  $400$ - $750^\circ\text{C}$ ). The minimum temperature of calcite decomposition is  $700^\circ\text{C}$ , while the maximum dehydration temperature is reached at  $780^\circ\text{C}$ , in the high-temperature zone ( $>800^\circ\text{C}$ ). The minimum temperature of decomposition occurs at  $850^\circ\text{C}$ , the water contained in the pore and between the layers is  $7.06\%$ , while the constitutive water is  $4.25\%$ .

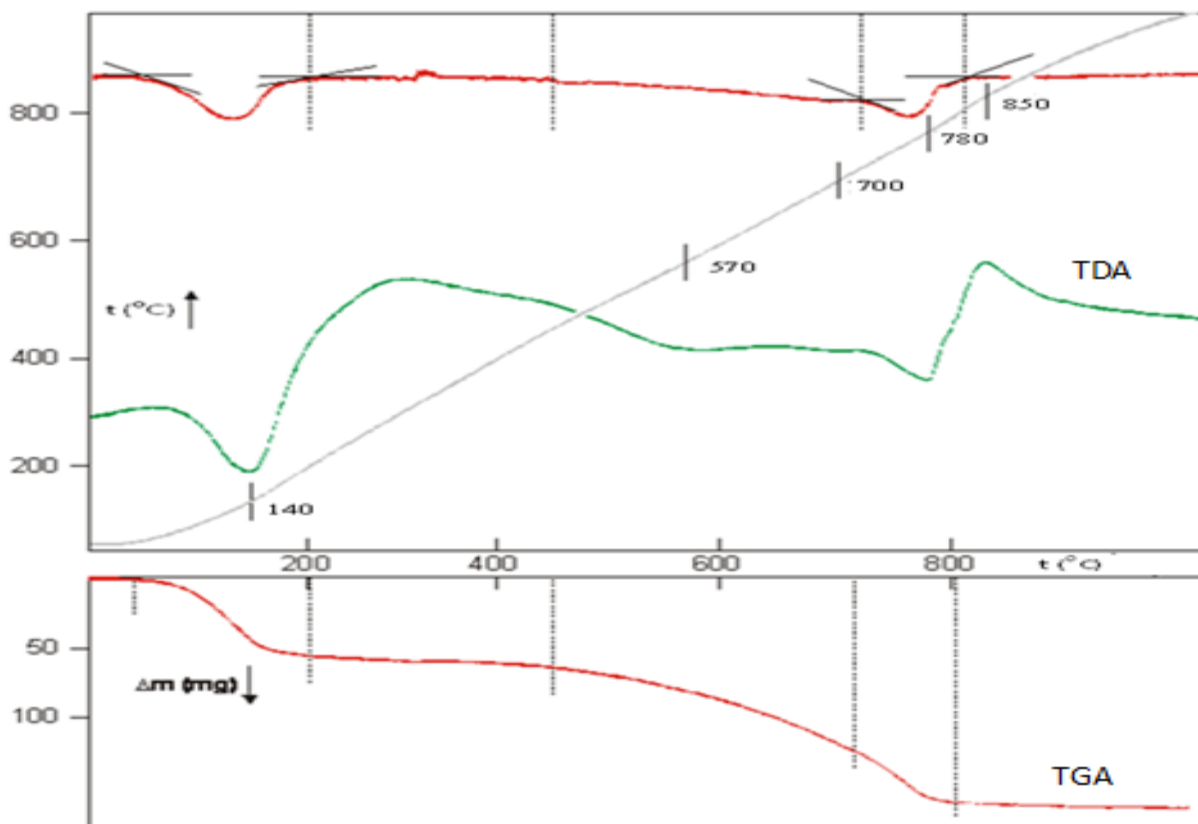


Fig.-6: DTA and TGA analysis of sample activated with 3%  $\text{Na}_2\text{CO}_3$ .

## CONCLUSION

Based on the results obtained from experimental research, can conclude:

- The mineralogical composition of Karaçevabentonite is characterized by the large presence of montmorillonite in it.
- Acid activation has changed the structural and mineralogical properties of Karaçevabentonite.
- Acidification of bentonite with 10%  $\text{H}_2\text{SO}_4$  causes the removal of calcite from bentonite.
- By diffractometric analysis, in the researched samples of natural and activated bentonites, it was found that Karaçevabentonite contains montmorillonite as the main mineral, accompanied by other minerals, illit, quartz, feldspar, dollomite and calcite.
- Relevant diffractometric studies have shown that the basic activation of these bentonites has not resulted in any change in their mineral composition.
- Based on the results of diffractometric and thermogravimetric measurements, the Karaçevabentonite contains very little calcite and dollomite.

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