

Characterization of Moganite Obtained From Natural Zeolite By Ball Milling

Musa Sarikaya¹, Aysegul Yucel^{1*}, Selda Sezer², Sukru Uckun¹, Tolga Depci³

¹(Department of Mining Engineering, Inonu University, Malatya, Türkiye)

²(Department of Chemistry, Inonu University, Malatya, Türkiye)

³(Department of Engineering Science, Iskenderun Technical University, Hatay, Türkiye)

*Corresponding Author: Musa Sarikaya

ABSTRACT: The aim of this study is to investigate the effect of mechanical activation on the phase change of the zeolite composition of the Hekimhan region which is located in the northern of the Malatya Basin in Eastern Anatolia. Planetary mill was used for this study at different speeds and times. The laboratory experiments had shown that the microcrystalline moganite was formed at 450 rpm and 180 min and 540 rpm and 120 min, depending on the time and milling speed. Characterization studies of the zeolite and moganite (450 rpm/180min and as well 540 rpm/120 min) have done by XRD(X – Ray Diffraction), SEM (Scanning Electron Microscopy) images and FT – IR (Fourier – Transforms Infrared Spectroscopy).

Keywords : Mechanical Activation, Moganite, Natural Zeolite, Silica

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I. INTRODUCTION

There are 50 different types of zeolites in the nature, such as clinoptilolite, chabazite and mordenite, which have different physical and chemical properties [1, 2]. Especially zeolite depositions occur from volcanic tuffs, low grade metamorphosed sediments or formed as crystals in cavities of basaltic rock [2, 3]. The main structure of the Hekimhan zeolite consist of clinoptilolite – (Cs) and calcit. The sources of zeolite deposits are marine tuff and tuff of the volcanic rocks interfingering with deposited in deep sea [2]. Zeolites are microporous and aluminasilicate minerals. Zeolites have a wide use of the industry due to their unique and versatile properties such as ion exchange, catalysis, adsorption and separation [1, 4 – 6]. Mechanical activation is a multi-step process, with changes in energetic parameters at each step and the amount of energy that accumulates. Four processes, namely accumulation of defects, amorphization, formation of highly stable polymorphic forms and chemical reaction, are combined with mechanical activation [7]. Zeolites can be grinded to increased. The external surface area depending on the amorphization and studies are being made to use it in catalytic and adsorption activation by changing the crystal structure [7, 8]. Structural changes, zeta potential, and rheological behavior, the material structure of zeolite has been changed by mechanical activation. At the end of the 360 minute milling period, material with a size of 200 nm and a surface area of 18 m²/g has been obtained [6]. When the effect of mechanical activation on the properties of natural zeolites in Tokaj Mountain is examined, the specific surface area of zeolite powders increased in the first 60 minutes and decreased thereafter. The crystal structure of this natural zeolite was found to be amorphized up to 52% [9]. Mordenite nanoparticles form low-cost natural zeolite has been produced by recrystallization with high-energy ball mill. The crystallinity of natural zeolites decreased by about 65%. According to this result, the zeolite structure approaches the amorphous structure. [10].

II. MATERIALS AND METHODS

The natural zeolite samples used in this study were taken from Malatya Hekimhan region. The main composition of the zeolite consists of SiO₂ and Al₂O₃. In addition there is CaCO₃ between the zeolite layers. The zeolite samples were crushed, ground and sorted in laboratory. Subsequently the zeolite sample were milled by ball milling (Retsch PM 100 Planetary Mill). The aim was to obtain moganite, a type of microcrystalline silica mineral. The mill was operated at various speeds. The samples obtained by grinding at 450 rpm and 540 rpm with 1 – 15 – 30 – 45 – 60 – 120 – 180 minutes were selected for characterization purposes. Characterization of

the samples were done by XRD (Rigaku Miniflex 600 with Cu K α , 40kV, 15 mA, $\lambda=1.54 \text{ \AA}$), SEM (Leo – Evo 40xVP scanning electron microscope) and FT – IR (Perkin Elmer Spectrum Two). The purpose of XRD analysis was to identify the crystalline phases for zeolite, moganite that obtained at 450 rpm in 180 min and moganite 540 rpm in 120 min. SEM images have provided us with information about surface topography and composition of the samples. FTIR analysis has given information on the molecular structure of the material by energy absorbance at various wavelengths while absorbing the sample light.

III. RESULTS AND DISCUSSION

The crystallinity and chemical structure of the natural zeolite and moganite obtained at 450 rpm and 540 rpm were determined by XRD and diffractions are given in Figure 1 and 2. The zeolite peaks showed that this structure consist of zeolite D (Cs) (ICDD PDF – 4 Minerals 2015 RDB Card No: 00 – 039 – 0131) and calcite (ICDD PDF – 4 Minerals 2015 RDB Card No: 01 – 085 – 0849). The zeolite D (Cs) chemical formula is $\text{CsAlSiO}_4 \cdot \text{H}_2\text{O}$ and the calcite chemical formula $\text{Ca}(\text{CO}_3)$. All intense reflections match with the powder diffraction data of the structure has a certain crystal order. The natural zeolite, which is exposed to mechanical activation for 180 minutes at a grinding speed of 450 rpm and 120 minutes at a grinding of 540 rpm, was converted to the moganite which is the chemical formula SiO_2 (ICDD PDF – 4 Minerals 2015 RDB Card No: 00 – 052 – 1425). As a result, while some structures were moving away, some structure remained trace amount. The average particle size decreased to about 60 nm to about 30 nm for 450 rpm and below 30 nm for 540 rpm.

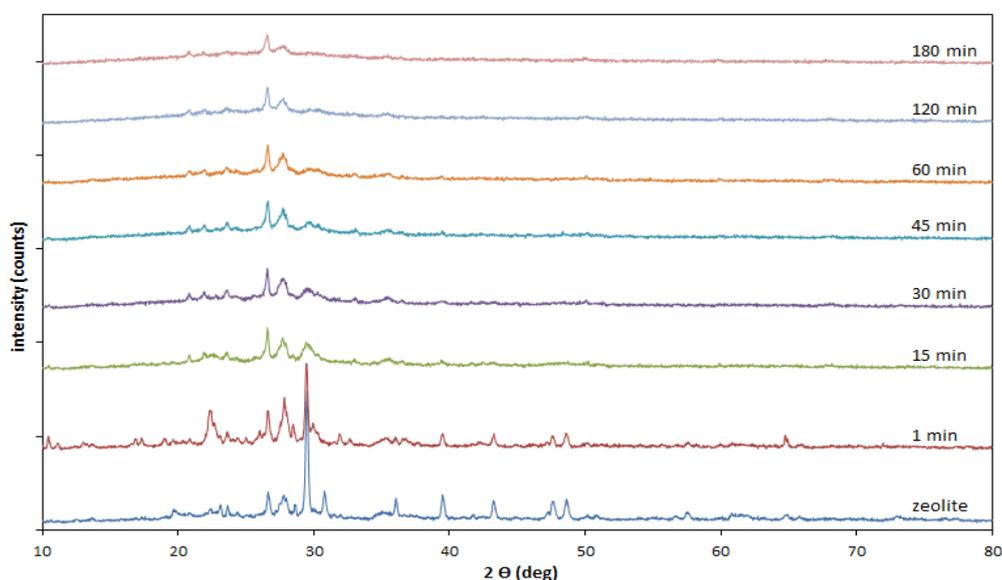


Figure 1: XRD phase analysis of obtaining moganite from 450 rpm zeolite.

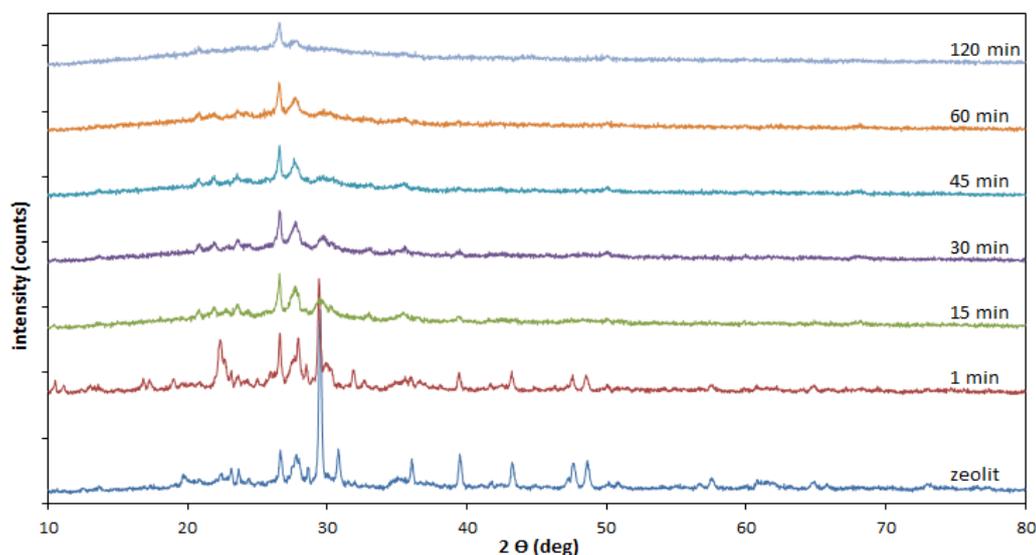


Figure 2: XRD phase analysis of obtaining moganite from 540 rpm zeolite.

Figure 3 shows the FT – IR spectrum of the zeolite and moganite particles which was obtained in the range of 400 – 4000 cm^{-1} . The FT – IR peaks of zeolite, the moganites were used for explicate of moganite structure. The FT – IR peaks of zeolite, around 3600 cm^{-1} is appears to the H – O – H stretching vibrations and the stretching mode of vibration of C=O is observed around 1428 cm^{-1} . Zeolite bands, creating a broad intense asymmetric band between 1600 – 1300 cm^{-1} as the special feature. Furthermore, the band at 1000 cm^{-1} and the peak at 513 cm^{-1} are related to zeolite. Bands at 1000 cm^{-1} were shown to Si – O bonds in the SiO_2 molecules. In addition, the appearance of the band at 787 cm^{-1} indicated the presence of Si – O – Al units, consistent with the presence of tetrahedral and octahedral layers within the zeolite. The band for zeolite around 500 cm^{-1} is due to vibration of the ring of tetrahedral structure[11-13].

The moganite characteristic peak, the moganite removes zeolite bands, leaving a broad intense asymmetric band around 1000 cm^{-1} as the main special. The stretching mode of vibration of C=O has been observed at 1420 cm^{-1} . Bands at 1000 cm^{-1} were seen to Si – O bonds in the SiO_2 molecules. FT – IR spectra of moganite shows absorption bands at around 580 cm^{-1} which is based to Si, trace amount Al – O bond, and those at 1000 and 700 cm^{-1} are, respectively based to asymmetric and symmetric stretches of the moganite. The bands at 700 cm^{-1} and 620 cm^{-1} observed Si – O symmetric stretching.

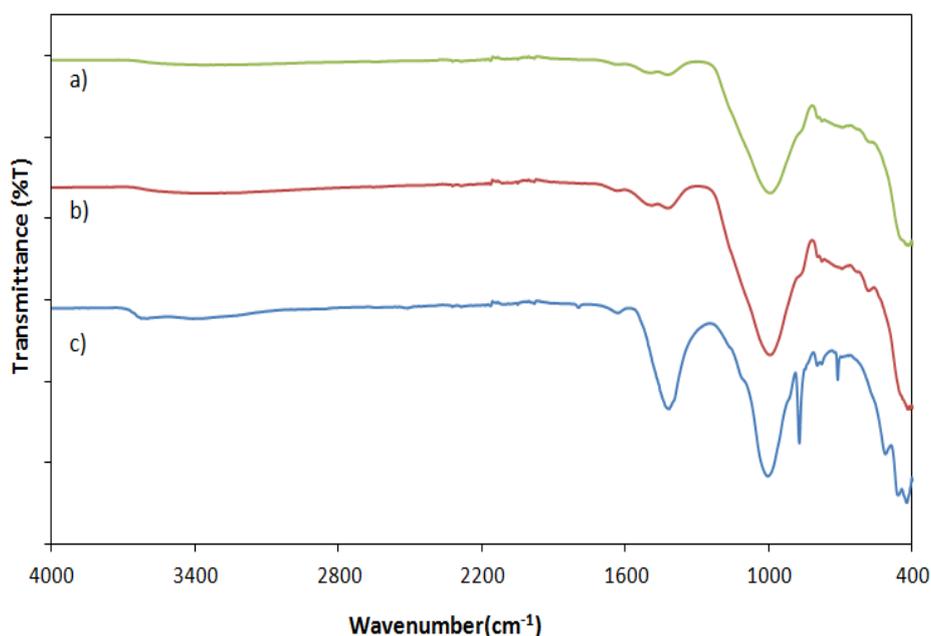


Figure 3: FTIR spectra of zeolite (c), obtained moganite from 450 rpm 180 min zeolite (b) and obtained moganite from 540 rpm 120 min zeolite (a)

The morphology and structure of mechanical product was characterized by SEM. It is obvious that the zeolite consists of layers as can be seen from the SEM images (Figure 4. a, b). Figure 4 (c, d), (e, f) represents the scanning electron micrographs of moganite obtained from zeolite grounded at 450 rpm at 180 minutes and 540 rpm at 120 minutes respectively.

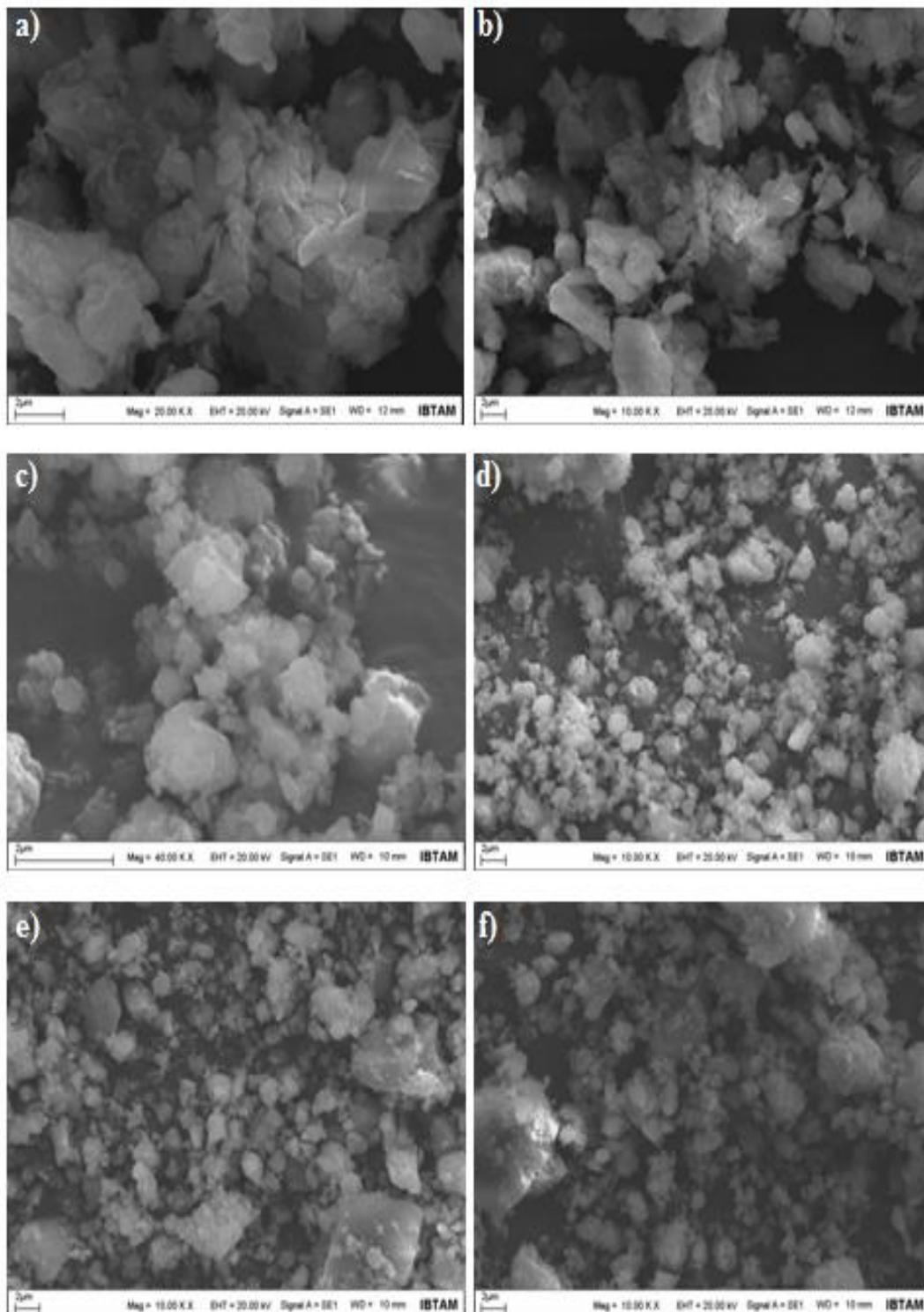


Figure 4: Different magnification SEM images of zeolite and moganite (a-b SEM images for zeolite, c-d SEM image for moganite 180 min 450 rpm, e-f SEM image for moganite 120 min 540 rpm)

IV. CONCLUSION

The zeolite structure has been changed by milling at various speeds and times and converted to moganite after 180 minutes at 450 rpm and 120 minutes at 540 rpm. Particles of original zeolites converted into spherical shape with an average particle size of 30 nm at 450 rpm after 180 minutes grinding and 540 rpm after 120 minutes grinding. The moganite removes zeolites bands, leaving a broad intense asymmetric band around 1000 cm^{-1} as the main special. The results will give a reference about future studies.

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