

Effects of mechanical activation on the structure of talc

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This paper presents the results of research on mechanical activation of raw talc in planetary mill. Raw talc was mechanically activated in a planetary mill for five different grinding times as 15 min, 30 min, 45 min, 60 min, 90 min. Non-activated and activated talc powders were characterized by using particle size analysis, X-Ray diffraction (XRD), scanning electron microscopy (SEM), the Fourier transform infrared spectroscopy (FTIR) and differential thermal analysis (DTA). Mechanical activation reduced the particles size, increased the specific area and affected the loss of structural water.

Key words: Talc, Mechanical activation, Planetary mill, Microstructure.

Introduction

Talc ($\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$) is a natural material widely used as one of the most important magnesium silicates in the form of a fine powder in several industrial applications. The main characteristics of talc which make it attractive for applications are whiteness, softness, low thermal and electrical conductivity and adsorption capacity of organic substances. Talc has found extensive application in many industries with its thermal endurance, lubricity and chemical stability [1, 2]. The general applications of talc are found in the production of paints, paper, rubber, pharmaceutical and cosmetic products, high-voltage insulators and also in ceramic industry for the production of refractories [3].

Mechanical activation of starting material is a promising way in precursor preparation. Particle size reduction, which increases the contact surface between particles is the direct consequence of milling. Mechanical activation is used to change the structure, increase the reactivity of raw materials. [4]. Also, mechanical activation decreases in the reaction temperature due to increasing the energy of the system [5]. Mechanical activation of talc provides loss in crystallinity (amorphization), a formation of active surfaces and increasing reactivity [6, 7].

Thermal decomposition product of talc, enstatite (MgSiO_3), is the main component of steatite ceramics. The chemical formula of steatite ceramics is MgSiO_3 which have four polymorphic forms such as enstatite, protoenstatite, clinoenstatite and high temperature clinoenstatite. Steatite ceramics are usually available as an enstatite phase whose density is 3.21 g/cm^3 , melting point is 1557°C and crystal lattice is orthorhombic.

Due to steatite's attractive mechanical properties, low power losses in addition to high dielectric constant at ambient and high temperatures, steatite has been widely used applications as insulator for high frequency systems [6, 8].

In this study, the effects of mechanical activation on the structural properties of talc were investigated with X-ray diffraction (XRD), particle size analysis, scanning electron microscopy (SEM) and the Fourier transform infrared spectroscopy (FTIR).

Experimental Details

Talc from Kalemaden Company, Turkey was used as raw material. The chemical composition of the talc was as follows (wt %): 63% SiO_2 , 0.40% Al_2O_3 , 0.25% Fe_2O_3 , 0.40% CaO , 0.04% K_2O , 0.08% Na_2O and 30% MgO with a 4.83% loss on ignition. Mechanical activation treatment was performed for different times (15, 30, 45, 60, 90 min respectively) in a planetary ball mill (Fristch Mono Mill Pulverisette 6) at 600 rpm at room temperature with batches of 10 g samples and 10 mm diameter of the tungsten carbide (WC) balls at ball to sample weight ratio of 20 : 1.

The crystallinity of the samples was determined by X-ray diffraction analysis (XRD) using Rigaku Ultima X-ray diffractometer and Cu K_α radiation in the 2θ range of $2-120^\circ$ with 0.02° steps at a rate of $1^\circ/\text{min}$. The chemical composition was measured by X-ray fluorescence using a Rigaku RIX spectrometer. Shimadzu FTIR spectroscopy was used for FTIR analysis of non-activated and activated talc samples. The morphology of the non-activated and activated talc samples was observed by using Joel 6060 LV scanning electron microscope (SEM). Mikrotrac S3500 was used for particle size analysis. The degree of amorphization (%A) in the talc structure after mechanical activation was calculated by Eq.1 [9];

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$$\%A = \left[1 - \frac{B_0 \times I_x}{B_x \times I_0} \right] \times 100, \quad \text{Eq.1}$$

where I_0 is the integral intensity of the diffraction peak for non-activated talc, B_0 is the background of the diffraction peak for non-activated talc and I_x and B_x are equivalent values for mechanically activated talc.

Results and Discussion

XRD analysis

The XRD patterns of talc are shown in Fig. 1 before and after activated for different times. As one can see, with the increase of grinding time, peak broadening occurred and the intensities of the diffraction peaks decreased gradually. This fact is the result of amorphization and structural disordering in the talc structure. This might be explained as the formation of a metastable ‘amorphous phase’, due to line broadening and disappearance of diffraction peaks occurring on X-ray patterns after high-energy milling [10]. According to Pourghahramani *et al.* [11], the occurrence of the amorphous phase is generally inferred by observing the presence of less intensive peaks in the XRD pattern. After activated for 90 min, some of the peaks disappear and the others become smaller. It shows that the mechanical activation causes to decrease powder crystallization and surface activation increases gradually [12].

On the other hand, because of the contact between powder-ball and attrition between powder-ball-bowl, local temperatures might be increased for higher rpm [13]. Talc structure contains –OH group and dehydroxylation of talc occurs as temperature increases. Owing to dehydroxylation of talc results in enstatite and SiO_2 formation. It shows that mechanical activation caused dehydration, deformation and crystal amorphization and these three processes are related with phase transformation mechanism [14].

D_{10} , D_{50} and D_{90} sizes corresponding to the particle sizes at the 10, 50 and 90% points on the cumulative distribution analysis for non-activated and activated

Table 1. Particle size analysis of non-activated and activated talc samples.

Milling duration [min]	d_{10} [μm]	d_{50} [μm]	d_{90} [μm]
0	2.339	9.97	32.30
15	0.734	9.02	45.01
30	0.719	4.77	34.54
45	0.723	4.38	53.42
60	0.714	3.44	33.91
90	0.734	4.22	39.14

Table 2. The degree of amorphization in the talc structure as a function of the milling time.

Milling duration [min]	% A
15	99.294
30	99.665
45	97.788
60	99.029
90	99.128

talc samples are given in Table 1. The degree of amorphization in the talc structure as a function of the milling time is calculated by Eq.1 and is shown in Table 2. for 15, 30, 45, 60 and 90 min. of mechanical activation. As a result, the degree of amorphization in the talc structure increases while milling duration increases. But, there is a little reduction of degree of amorphization for 45 min of activation. It is related about increment of intensity of (001) peak.

Since the particle size of the powder becomes smaller, the surface area and surface energy are much higher with the increase of milling duration due to the crushing and forming new surfaces become more reactive. Therefore, particle agglomeration is observed with crystallinity loss while specific surface area increases in the early stages of the mechanical activation, diminishing afterwards [6]. The effect of particle agglomeration during activation is observable, agglomerates are greater than the initial particle size. After 45 min of mechanical activation, agglomerates decayed into smaller again.

Microstructure analysis

The scanning electron micrographs (SEM) of the non-activated and activated talc samples are shown in Fig. 2. The non-activated talc sample has micro-size grains with well-defined faces and edges. It is obvious that particle size decreases and particle shape becomes rounded particles while milling duration increases. After 30 min of mechanical activation, a reagglomeration of the fine reactive particles is demonstrated. Also, after 90 min of mechanical activation, agglomerates consist clusters of large and rounded particles.

Thermal analysis

DTA and TG analysis of non-activated and activated

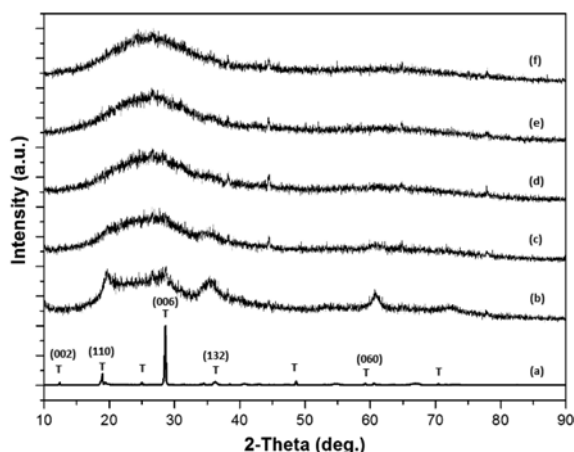


Fig. 1. XRD patterns of non-activated (a) and activated (b) for 15 min, (c) 30 min, (d) 45 min, (e) 60 min and (f) 90 min talc samples [T: Talc].

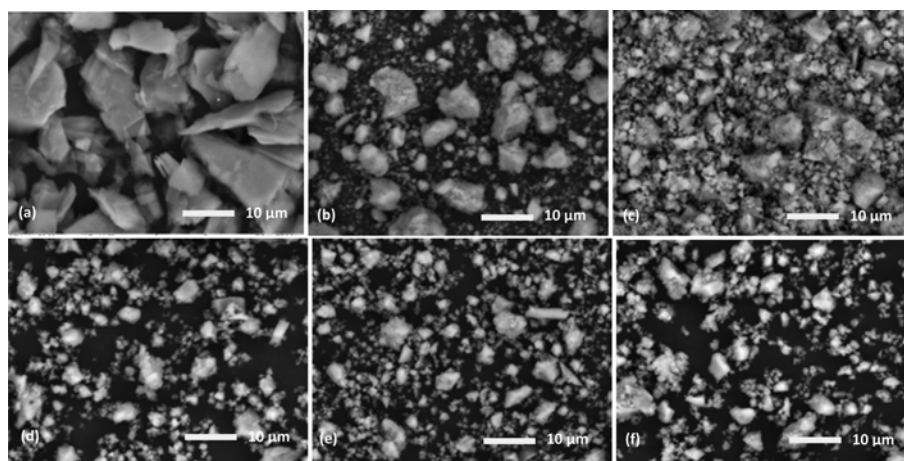


Fig. 2. SEM micrographs of talc: (a) non-activated, (b) activated for 15 min, (c) activated for 30 min, (d) activated for 45 min, (e) activated for 60 min, (f) activated for 90 min.

talc samples are shown in Fig. 3. The weight loss occurred in two stages: the first stage from 550 to 685 °C and the second stage from 858 to 996 °C for non-activated talc in this study. Liao and Senna [1] reported that the increase in the weight loss at the first stage is related to the desorption of the adsorbed water molecules due to mechanical activation. In case, the weight loss at the second stage is owing to the loss of the hydroxyl groups still bound to the bulk solid. It can clearly be seen that the total thermal weight loss of talc decreases with the increasing of milling time as a result of partial dehydration during mechanical activation [1].

TG curves of activated talc samples exhibit the weight loss at low temperatures (30-150 °C) with respect to non-activated talc samples, which increases as milling time increases. This loss is related to the release of adsorbed water with particle size reduction and new surface area evolution of activated talc sample [15, 16]. In the DTA analysis of non-activated talc sample, an endothermic peak at around 938 °C corresponds to the formation of enstatite (MgSiO_3) and silica [6]. This is confirmed by the weight loss of non-activated talc sample in range 858-996 °C. This endothermic peak is not present in the activated talc samples, which is probably due to the occurrence of dehydration of talc during mechanical activation. However, an exothermic peak appears at around 801 °C after 60 min of mechanical activation.

DTA results stated that mechanical activation of talc enhances the formation of high temperature phases of talc. The exothermic peak at 801-831 °C for different milling time is related to the crystallization of non-crystalline phases, formed by mechanical activation of talc sample. The metastable non-crystalline phase crystallizes to form orthorhombic enstatite at this temperature [6].

FTIR analysis

FTIR spectra of the raw and activated talc samples for different times are given in Fig. 4. Infrared

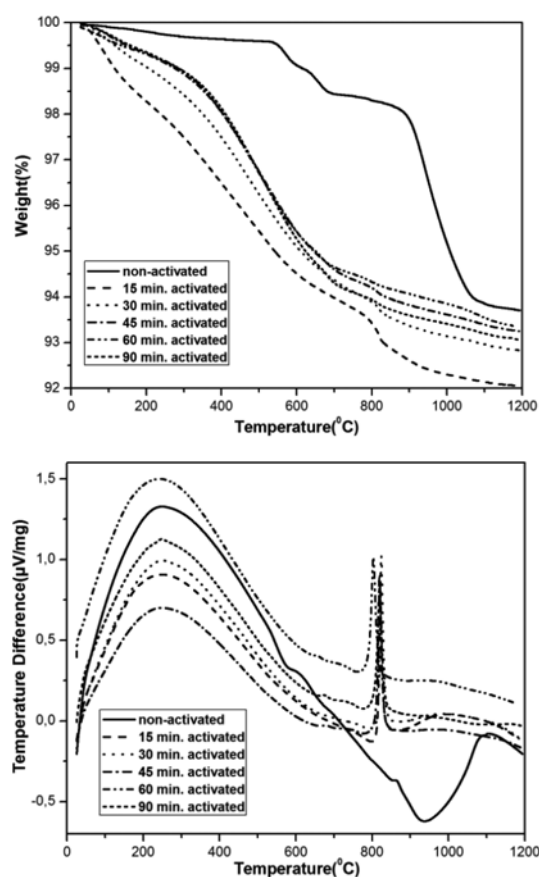


Fig. 3. DTA and TG analysis of non-activated and activated talc samples.

spectroscopy is used to evaluate the effects of mechanical activation on the structure. The changes in infrared spectra due to fine grinding effect on talc structure such as decrease in peak intensity, broadening of band, shifting of vibration band and disappearance or appearance of new band in the spectrum are estimated. The OH-stretching frequency of the octahedral Mg-O-H unit in talc structure occurs at 3672 cm^{-1} wavelength [17]. The

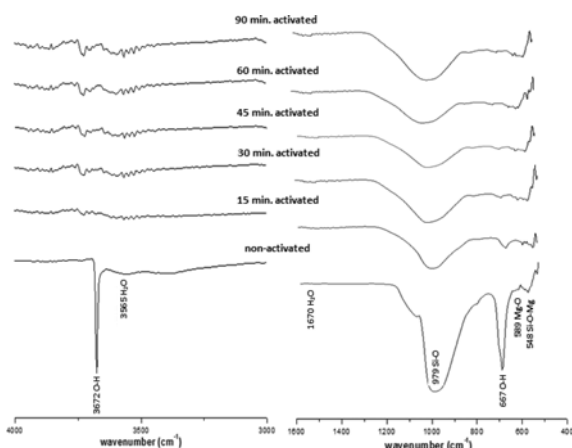


Fig. 4. FTIR analysis of non-activated and activated talc samples for different times.

band at 3672 cm^{-1} increases to 3699 cm^{-1} after grinding for 90 min.

An increase of adsorbed water is also detected. It is related to the increase of specific surface area because of mechanical activation [2]. At 1670 cm^{-1} and 3565 cm^{-1} centered bands belongs to the OH- deformation of absorbed water. The in plane stretching vibration of Si-O at 979 cm^{-1} shows broadening effect due to fine grinding. Otherwise, the band centre could have shifted to the higher wavenumber with increasing milling duration.

Mechanical activation has mostly affected the Mg-OH bonds within the octahedral sheet. Therefore, the intensity of band centres at 667 cm^{-1} and 3672 cm^{-1} become weaker by destroying Mg-OH bands with increasing milling duration [7] [12]. The bands of around 548 cm^{-1} are attributed to the deformation of Mg-O-Si [18]. Smaller changes are observed for the Si-O-Mg and Si-O bonds.

Heat energy is occurred during mechanical activation and leads to dehydration of talc. The band centres shift to higher wavenumber with increasing temperature. Shifting of band centre to higher wavenumber with increasing milling duration means that strength of bonds decreased. Hence, shifting of band centre to lower wavenumber is related to tighter bonding [14].

Conclusions

The mechanical activation of talc by planetary ball milling at room temperature induced amorphization and structural disordering in talc. X-ray diffraction and FTIR analyses supported those changes in talc structure.

The effect of particle agglomeration during mechanical activation is appreciable. The dehydration reaction in talc occurred during milling. The endothermic DTA effect at around $938\text{ }^{\circ}\text{C}$ is related to the decomposition of talc into enstatite and SiO_2 . This effect disappears after mechanical activation of talc and it is replaced by an exothermic peak at $801\text{--}831\text{ }^{\circ}\text{C}$ for different milling time due to the crystallization of a noncrystalline phase formed by amorphization of talc into enstatite.

Acknowledgments

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