

Characterization of Leaching Feed and Residue of Mechanically Activated Sphalerite

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Article Info	Abstract
Received 1 August 2021	In this research work, the solubility of sphalerite concentrate due to mechanical
Received in Revised form 19 November 2021	activation in planetary ball mill in both the wet and dry modes is investigated, and the parameters of mean particle size, BET specific surface area, SEM, and XRD are
Accepted 12 December 2021	analyzed. The results of the particle size analysis and BET specific surface area show
Published online 12 December 2021	that the size of particles for the non-activated sample decrease from 51 to 30 microns but the BET specific surface area increase from $0.17 \text{ m}2/\text{g}$ to $1.03 \text{ m}2/\text{g}$ for the residue and feed samples. In the wet and dry mode mechanical activation, the mean particle size and BET specific surface area in the residue samples are reduced relative to the leaching feed. The results of the micro-structure characterization also show that the
DOI:10.22044/jme.2021.11054.2082	amorphization of the residue compared to the leaching feed increases in both modes
Keywords	of mechanical activation. The crystallite size and lattice strain of the activated samples
Sphalerite	in the residue increase and decrease compared to the leaching feed, respectively.
Mechanical activation	
Wet and dry modes	
Leaching residue	
Sample characterization	

1. Introduction

Sphalerite mineral ((Zn, Fe) S) is the most important source of zinc production from sulfide minerals [1], which currently produces the zinc metal by roasting-leaching-electrowinning (RLE), pressure leaching, and atmospheric leaching using suitable oxidants such as ferric sulfate. Zinc production by the pyro-metallurgical method is economical but has many environmental disadvantages due to the emission of large amounts of sulfur dioxide gas [2].

Pressure and atmospheric leaching are the two hydrometallurgical methods for the production of zinc from sphalerite. The first method has high operating costs, and the latter has a low zinc extraction rate due to the production of elemental sulfur that coats the reactive sphalerite surface. Therefore, an alternative method should be used in order to increase zinc recovery in sphalerite leaching without the above disadvantages. Since sphalerite is a resistant mineral in sulfuric acid media due to its electronic structure, destruction of its lattice structure and breaking of strong zincsulfur bonds using mechanical activation with high-energy ball mills can be an interesting option [3-5].

Some researchers have used mechanical activation pre-treatment in order to improve the hydrometallurgical process of sulfide and oxide ores. In 2003 and 2007, Hu et al. pointed to the effect of the atmosphere of the mechanical activation on the leaching of sphalerite and the mechanism of mechanical activation with a planetary ball mill for the sphalerite concentrate [6, 7]. The effect of using energetic mills such as planetary ball mill and vibrational mill on the activation of sulfide minerals and their subsequent

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leaching has been studied by Baláž et al. [8-11]. Akhgar et al. (2012) have discussed the optimization of the production of synthetic rutile nano-powder from sphalerite concentrate using planetary ball mill [12]. Ebadi (2019) has investigated the effect of mechanical activation modes using planetary ball mill on the structural changes and leaching of the ilmenite concentrate [13]. Turan (2021) has used the zinc-containing residue including the franklinite mineral using vibratory ball mill and ring mill for mechanical activation. He used the SEM and BET methods for sample characterization before and after mechanical activation, and subsequently studied the reactivity of mechanically activated samples. He did not refer to the results of micro-structural studies such as the amorphization degree, changes in the crystallite size, and lattice strain in feed and leaching residue [14].

Evaluation of the parameters such as the lattice strain, crystallite size, and amorphization degree can be interesting subjects to predict the behavior of minerals in the hydrometallurgical process [15]. In mechanically activated minerals with small crystallite size, there are a large number of grain boundaries that facilitate the reactivity of atoms Also the high degree of amorphization [16]. indicates the weakening of the atomic bond between the elements in the minerals, which increases the reactivity of mechanically activated minerals [17]. The lattice strain can be referred to atomic positions in which the distance between the atomic planes in the mineral structure changes due to the induction of mechanical stress, and increasing the lattice strain leads to an increase in the reactivity of mechanically activated minerals [18]. It has been proven that the reactivity of mechanically activated minerals can be improved by increasing the amorphization degree, increasing of the lattice strain, and reducing of the crystallite size. The most important parameter among these parameters is the percentage of amorphization degree, which absorbs the most mechanical energy induced during mechanical activation [10]. On the other hand, Xiao et al. [19] have used a thermochemical cycle in order to measure the energy stored as a result of the mechanical activation of minerals, suggesting that the extra energy in the sphalerite lattice such as dislocations and structural defects is stored as a result of the mechanical activation. It was observed that the stored energy increases with increase in the mechanical activation time, and is independent from the final specific surface area of the mineral.

Most of the references mentioned in this research work dealt with the characterization of the microstructure of mechanically activated minerals, and a few of them referred to the effect of mechanical activation on the reactivity of activated minerals but none of them mentioned the characterization of leaching residue of mechanically activated sphalerite. The main purpose of this paper is to characterize the leaching feed and residue samples of the sphalerite concentrate with and without mechanical activation in the wet and dry modes, and the results obtained are then compared with each other to investigate the effect of mechanical activation on increasing the dissolution rate of zinc in sphalerite.

2. Materials and methods 2.1. Materials

The low-grade sphalerite ore was prepared from Angouran mine, Zanjan, Iran, and then high-grade sphalerite concentrate was obtained by the flotation and high-intensity magnetic separation methods. The XRD analysis mainly shows the cubic structure of Fe-bearing sphalerite (ZnFeS) peaks that corresponds to JCPDS No. 05-0566, and also quartz (SiO₂) is the most important gangue mineral according to JCPDS reference No. 46-1045. The grade of the sphalerite concentrate is 59.46%.

2.2. Mechanical activation

Mechanical activation of the sphalerite concentrate was performed in a planetary ball mill (Mehr Alborz Ceram Industry, Iran) with a rotation speed of 450 rpm. 300 stainless steel balls with diameters of 10 mm were used for grinding with a weight ratio of ball to powder of 15:1. Mechanical activation was performed in 180 min in both the wet (70% solid) and dry modes.

2.3. Micro-structure characterization

XRD analysis was used in order to determine the diffraction pattern of non- and activated sphalerite concentrate. A standard sample of lanthanum hexaboride (LaB₆ SRM660a) was used in order to eliminate the instrument effects. X-ray diffraction pattern were prepared using a Philips X'pert MPD model diffractometer using a cobalt electrode ($\lambda = 1.799 \text{ A}^\circ$) in the range of $2\theta = 25-85$. All the recorded data were obtained using a step size of 0.02 degrees and a count time of 3 seconds per step. The mean particle size was obtained by a laser particle size analyzer model ANALYSETTE 22 NanoTec Plus made by FRITCH Germany. The specific surface area was obtained by a standard

BET surface area analyzer of NANOSORD SENSIRAN model, Iran. For the morphological investigation, SEM microscopy (TESCAN, Japan) was used.

2.4. Leaching tests

A series of leaching experiments were performed using sulfuric acid with a concentration of 2 M in order to achieve the effect of mechanical activation on the reactivity of sphalerite. For the leaching experiments, a 1000 mL glass beaker was used. For each test, a 200 mL solution was first poured into a beaker, and the desired temperature of the solution was set by a hot plate for the leaching test. The sphalerite sample was then poured into the beaker in a certain amount (5 g), and the mixture was stirred at a certain speed. At specified intervals, 3 mL of the leaching solution was removed and analyzed by atomic absorption spectrometry (AAS) in order to determine the concentration of the Zn ions.

The amount of zinc extraction was calculated using the equation presented by Demopoulos et al. [20]:

$$X_{i} = \frac{\left(V_{0} - \sum_{i=1}^{i-1} \nu_{i}\right)C_{i} + \sum_{j=1}^{i-1} \nu_{j}C_{j}}{M(\frac{C_{M}}{100})}$$
(1)

where X_i is the percentage of metal extraction in sample i, V_0 is the initial volume of the leaching solution in reactor (mL), v_i and v_j are the sample volumes i and j removed from the reactor (mL), C_i and C_j are the metal concentrations in samples i and j (mg/L), M: is the initial mass of the sphalerite concentrate added to the reactor (g), and C_M is the metal grade in the sphalerite concentrate (%).

It should be noted that the conditions of the leaching tests on all three samples (non-activated, and wet and dry modes activated) were selected in such a way that no oxygen was blown into the leaching solution and the test environment was completely closed. Therefore, as much as possible, the oxidation of ferrous to ferric ions was prevented. The special reaction conditions for sphalerite leaching were as follows solid to liquid ratio (g/L): 1 to 25, sulfuric acid concentration (mol/L): 2, stirring speed (rpm): 500, reaction time (min): 120, and the reaction temperatures (°C): 65, 80, and 95.

3. Results and Discussion 3.1. Dissolution tests

Dissolution tests were performed for the three samples of sphalerite (non-activated, and wet and

dry modes activated) at the three temperatures of 65, 80, and 95 °C. The results of zinc extraction at different times for these samples are given in Figure 1. The results obtained for the mentioned temperatures showed that the dissolution rate of zinc for the activated samples in both the wet and dry conditions had significantly increased compared to the non-activated sample. It was also observed that the maximum recovery of zinc extraction in 120 min and 95 °C in the nonactivated sphalerite was 23.7%, while in the same conditions, the recovery of zinc extraction for the activated samples for the wet and dry modes was equal to 67.12% and 85.31%, respectively. Therefore, the rate of zinc extraction in the wet and dry modes was 43.4% and 61.6% higher than the non-activated sphalerite, respectively. Also the rate of zinc extraction in the dry mode was 21% higher than the recovery of zinc in the wet mode of mechanical activation.

The semi-empirical Avrami-Jerofejev equation (Eq. 2) was used for a better comparison of the dissolution results of the sphalerite samples before and after mechanical activation.

Many researchers have used this equation to model the results of kinetic data and to compare and differentiate the effect of structural changes created by mechanical activation on increasing the mineral solubility [13, 15].

$$-\ln(1 - \alpha_{7n^{2+}}) = kt^n \tag{2}$$

where α is the part of zinc dissolved after time t, k is the kinetic rate constant, and n is the parameter that represents the order of the reaction rate control.

Table 1 shows the results obtained for the three different samples using Equation 2. Figs. 2 and 3 also show the results obtained for comparison. It is known that with increase in the leaching temperature in different modes of mechanical activation, the kinetic rate constant has increased, and the parameter n has decreased. The value of the parameter n in the non-activated sphalerite is in the range of 0.5-1, and according to the definition, the reaction control mechanism will be chemicaldiffusion control, and by reducing it to less than 0.5 in both the wet and dry modes, the reaction mechanism has changed to diffusion control. Ebadi has reported that changes in the reaction control stage from chemical to diffusion after mechanical activation of the samples relate to the high energy of the atoms involved in the reaction and the collision of the atoms with the reactants in the leaching reaction. In this case, the role of the diffusion control process in controlling the reaction rate will be more prominent [13].



Figure 1. Zinc extraction according to leaching time of sphalerite in three different samples (non-activated and activated sphalerite in the wet and dry modes) and three different temperatures (65, 80, and 95 °C).

 Table 1. Kinetic modeling results for wet and dry modes of mechanical activation at three different temperatures.

Tomporature (°C)	Non-activated		Wet mode		Dry mode	
Temperature (°C)	k (min ⁻¹)	n	k (min ⁻¹)	n	k (min ⁻¹)	n
65	0.0028	0.79	0.208	0.25	0.448	0.19
80	0.0053	0.73	0.264	0.23	0.501	0.20
95	0.0081	0.72	0.311	0.25	0.580	0.24



Figure 2. Changes in kinetic rate constant in accordance with the reaction temperature.





The Arrhenius equation (Eq. 3) is used to calculate the activation energy [2]:

$$K = A. Exp(\frac{-E_a}{RT})$$
(3)

In this equation, the parameters are as follow: k is the kinetic rate constant, A is the Arrhenius constant, E_a is the activation energy (kJ/mol), R is the global gas constant (8.314 kJ/mol.K), and T is the reaction temperature (K). Table 2 shows the calculated activation energy results for the three different sphalerite samples.

 Table 2. Activation energy for sphalerite dissolution in different modes of mechanical activation.

Activation mode	Activation energy (kJ/mol)		
Non-activation	36.70		
Wet mode	13.10		
Dry mode	8.91		

According to Equation 3 and Table 2, the values of activation energy for the three samples of nonactivated and mechanically activated sphalerite in the wet and dry modes were calculated to be 36.70 13.10 kJ/mol, and 8.91 kJ/mol, kJ/mol, respectively. These results obtained indicate the reduction of about 24 kJ/mol and 28 kJ/mol of activation energy in the wet and dry modes due to the application of high mechanical energy on the sphalerite sample. In other words, mechanical activation of the sphalerite concentrate in the wet and dry modes has reduced the leaching activation energy by 64% and 76%, respectively. The decrease in activation energy indicates that the acidic leaching of the mechanically activated sphalerite can be performed at a lower temperature than that of the non-activated sample.

Given that the value of parameter n for the nonactivated sample is in the range of 0.5-1, it can be concluded that the reaction mechanism should be a combination of chemical control-diffusion control. In this case, the reaction in the early stages will be of the chemical control and then of the diffusion control.

For the activated samples, it is observed that the value of n is less than 0.5 in all the three temperatures, and the reaction mechanism is changed to diffusion control. In other words, due to the application of mechanical energy in planetary ball mill, the effect of the reaction temperature parameter has decreased, and stirring will have a greater effect on the zinc dissolution rate in the sphalerite concentrate.

In order to confirm the results, three leaching experiments were performed on the wet and dry mode activated samples at the stirring speeds of 500 rpm, 750 rpm, and 1000 rpm at temperature of 95 °C and a 2 M sulfuric acid concentration. Figure 4 shows an increase in the zinc extraction in the activated samples with increase in the stirring speed. According to the results in this figure, it is observed that with increase in the stirring speed, the amount of zinc extraction in both samples has increased, which confirms the diffusion control of the leaching reaction.



Figure 4. Effect of stirring speed on zinc extraction in leaching of wet and dry mode activation of sphalerite samples.

3.2. Sample characterization 3.2.1. Particle size and surface area analysis

Table 3 shows the mean particle size (D_{50}) for the leaching feed and residue of the non-activated and the wet and dry mode activated samples. It can be observed that the mean particle size for the leaching residue is less than the particle size of the leaching feed in all the three samples. Since the purity of the sphalerite sample is almost high, the dissolution of the sphalerite is done uniformly from the particle surface, and the particle gradually shrinks. It can be seen that in the non-activated sample, the mean particle size has decreased from 51 to 30 microns. For the wet and dry activated samples, the mean particle size of the leaching residue decreased compared to the leaching feed, which for the dry activated sample was much more than the wet mode (79% reduction for the dry mode and 16% reduction for the wet mode).

Figure 5 shows the BET specific surface area of the leaching residue samples in the three different modes of mechanical activation. It could be demonstrated that the BET surface area for the non-activated sample increased from $0.17 \text{ m}^2/\text{g}$ to $1.03 \text{ m}^2/\text{g}$ but decreased for both the wet and dry

activated samples so that for the wet activated sample, the BET surface area decreased from 18.20 to 9.07 m²/g, and for the dry activated sample, decreased from 7.71 m²/g to 6.46 m²/g. In the non-activated sample, due to the reduction of the mean particle size during the dissolution process, the BET surface area increased by 83%. However, in the wet and dry activated samples, the BET surface area decreased by 50% and 16%, respectively, which significantly reduced the surface area in the case of the wet mode mechanical activation. The

particles after mechanical activation have a serrated and non-smooth shape on the surface, which leads to an increase in the specific surface area after mechanical activation.

After the sphalerite sample is in contact with the sulfuric acid solution, the dissolution priority will be given to the surface of the particles that have been deformed by mechanical energy. By dissolving the damaged and irregular surfaces, both the specific surface area and the mean particle size will decrease.



Table 3. Mean particle size (microns) in two modes of the wet and dry mechanical activation.

Figure 5. Changes in BET surface area of leaching feed and residue in different modes of mechanical activation.

3.2.2. SEM analysis

SEM images of the leaching feed and residue of non-activated, and wet and dry mode activated samples are given in Figure 6.

Figure 6a shows that the feed particles of sphalerite have an unequal size, irregular shape and soft surface. Figure 6b shows that the particle size of leaching residue has been reduced, and they have become more uniform, and also the particles contain pores on the surface. Comparing Figs. 6c and 6d (leaching feed and residue of the wet activated sample), it can be observed that the leaching residue of the wet activated sample contains some undissolved particles. Figures 6e and 6f show that in both the leaching feed and the residue of the dry mode activated sample, the particles in the feed sample are agglomerated.

The reason for the difference in the SEM images of the wet and dry modes of mechanical activation is in the mode of mechanical activation. Due to the less induction of mechanical energy to the particles, less disruption has occurred in the lattice structure of the wet mode but in the dry mode activation with high induction of mechanical energy to the particles and production of many defects in the crystal structure of sphalerite, the dissolution rate and zinc extraction have been found higher.



Figure 6. SEM images of non- and activated samples (a and b: feed and leaching residue of non-activated sample, c and d: feed and leaching residue of wet mode activated sample, e and f: feed and leaching residue of dry mode activated sample).

3.3. XRD Analysis

Figs. 7 and 8 show the X-ray diffraction analysis of the leaching feed and residue samples of the sphalerite concentrate, respectively. Figure 7 shows that by mechanical activation of the sphalerite, the intensities of the peaks were reduced, the peaks became broader, and some of the weak peaks were disappeared. Figure 8 also shows that the diffraction patterns in all the three residue samples have a higher peak intensity and a lower peak width compared to the leaching feed samples. Also with the dissolution of sphalerite and its reduction in the residue sample, other minimum phases in the sample such as quartz (SiO₂) and anglesite (PbSO₄) have appeared in the residue pattern.



Figure 7. XRD pattern of leaching feed samples (F-0: non-activated sphalerite, F-180W: wet mode activated sphalerite for 180 min, and F-180D: dry mode activated sphalerite for 180 min).



Figure 8. XRD pattern leaching residue samples (R-0: non-activated sphalerite, R-180W: wet mode activated sphalerite for 180 min, and R-180D: dry mode activated sphalerite for 180 min).

Ohlberg *et al.* (1962) [21] have presented an equation (4) according to which the amount of amorphization degree of the sample at different times due to mechanical activation can be calculated. This parameter characterizes the crystal irregularity, and the structural irregularity is confirmed by increasing the abundance of amorphous material (from the viewpoint of XRD concepts) by reducing the cumulative intensity of diffraction lines.

$$A = 100 - X = 100 - \left(\frac{U_0 \cdot I_x}{U_x \cdot I_0} \times 100\right)$$
(4)

where U_0 and U_x are the background intensities of the mineral before and after activation, and I_0 and I_x are the cumulative intensities of the nonactivated and activated mineral diffraction lines. A and X are the degree of amorphization and the degree of crystallization of the material, respectively.

Figure 9 shows the amorphization graph of two samples of leaching feed and residue that have been activated in both the wet and dry conditions. The amorphization degree in the wet mode activated sample for the leaching feed and residue is 75.03% and 64.79%, respectively, while in the dry mode activated sample for the leaching feed and residue is 90.35% and 73.33%, respectively. It is observed that the amorphization degree in the leaching feed samples is higher than that of the leaching residue samples, and also the value of this parameter is higher for the dry mode activated sample than that of the wet mode. The lower value of the degree of amorphization in the leaching

residues indicates that during the dissolution of sphalerite, the sphalerite concentrate with the destroyed part of the structure is in the priority of dissolution, and after the dissolution of these particles, the intact part of the sphalerite begins to dissolve. For this reason, the intensity of the peaks increases and their broadening decreases.

Figs. 10 and 11 show the changes in the crystallite size and lattice strain due to mechanical activation in the samples before and after leaching, respectively. Figure 10 shows that the crystallite size decreases from 333 nm in the leaching feed sample to 27.46 nm and 14.55 nm in the wet and dry mode activated samples of leaching feed, respectively. The same trend is true for the leaching residue samples so that the crystallite size of the non-activated, and wet activated and dry activated samples is 333 nm, 76.37 nm, and 33.70 nm, respectively. The results obtained indicate that in the case of intense mechanical activation, the crystallite size in the leaching residue sample (76.37 nm) is larger than the crystallite size of the leaching feed sample (27.46 nm). The same is true for the dry activated sample. The particles in which mechanical energy is transferred through planetary ball mill have an amorphous structure. Also the bonding strength of these particles is severely reduced so they have priority in dissolution, and the leaching residues contain particles with a lower degree of amorphization and a higher bonding strength than that of the leaching feed samples. Therefore, the crystallite size of the particles in the leaching residue is larger than that of the leaching feed.



Figure 9. Amorphization degree of wet and dry mode activated samples in the leaching feed and residue samples.



Figure 10. Crystallite size changes in three samples of leaching feed and residue.

Figure 11 shows the strain changes of the sphalerite lattice in the leaching feed and residue samples due to mechanical activation. In contrast to the crystallite size, the lattice strain in the leaching feed samples in all the three samples is higher than that of the leaching residue samples so that the lattice strain increases from 0.075% in the non-activated sample to 0.61% and 0.86% in the wet and dry modes due to the high impact of mechanical energy applied to the samples in the destruction of the lattice structure, especially in the dry mode activation.

The lattice strain in the leaching residue is less than that of the leaching feed samples. The value of lattice strain in the leaching residue of the nonactivated, and wet mode and dry mode activated samples is 0.025%, 0.40%, and 0.43%, respectively, which is significantly different from that of the leaching feed. The reason for the reduction of lattice strain in the leaching residue samples compared to that of the leaching feed samples is the preferential dissolution of the particles with destructed structure compared to the particles with a less degraded structure.



Figure 11. Lattice strain changes in three samples of leaching feed and residue.

4. Conclusions

Characterization studies were performed on the leaching feed and residue samples of nonactivated, and wet and dry mode activated samples for 180 min, and the following results were obtained:

1- The mean particle size in the leaching feed decreased from 51 microns to 30 microns for the leaching residue of the non-activated sample. The same is true for the wet and dry mode samples.

2- The BET specific surface area for the leaching residue sample of the non-activated sample increased from 0.17 m²/g to 1.03 m²/g, while for the wet and dry mode activated samples decreased from 18.20 m²/g to 9.07 m²/g and 7.71 m²/g to 6.46 m²/g, respectively.

3- The intensity and width of the peak increased and decreased in all the three modes of mechanical activation in the leaching residue samples relative to the leaching feed, respectively.

4- The degree of amorphization decreased in both modes of mechanical activation in the residue samples relative to the leaching feed.

5- The crystallite size and lattice strain in both mechanical activation modes in the leaching residue increased and decreased relative to the leaching feed, respectively.

6- The kinetic rate constants for the mechanically activated samples (dry and wet modes) were increased compared to the non-activated sample.

7- The results obtained according to the mean particle size, BET surface area, particle morphology (SEM), crystallite size, and lattice strain in both mechanical activation modes in the leaching feed and residues indicate a preferential dissolution of the sphalerite particles with destroyed structures by application of mechanical energy. In the nonactivated sphalerite, due to the natural stress in the particle structure, the value of lattice strain is much less than the two other modes, which results in a lower dissolution of the non-activated sample (23%)compared to the wet (67%) and dry (85%) mode activated samples.

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چکیدہ:

در این تحقیق، قابلیت انحلال کنسانتره اسفالریت فعالسازی شده مکانیکی در آسیای سیارهای در هر دو محیط تر و خشک مورد بررسی قرار گرفت و پارامترهای میانگین اندازه ذره، سطح ویژه BET، MBZ و XRD آنالیز شدند. نتایج آنالیز اندازه ذره و سطح ویژه BET نشان دادند که اندازه ذره برای نمونه فعالسازی نشده از ۵۱ به ۳۰ میکرون کاهش مییابد ولی سطح ویژه BET از ۱/۰۷ به ۱/۰۳ متر مربع بر گرم برای نمونههای پسماند و خوراک لیچینگ افزایش مییابد. در حالتهای تر و خشک فعالسازی مکانیکی، میانگین اندازه ذره و سطح ویژه BET در نمونههای پسماند در مقایسه با خوراک لیچینگ افزایش مییابد. در حالتهای ریز ساختاری نشان داد که درجه آمورفشدگی پسماند نسبت به خوراک لیچینگ در هر دو حالت فعالسازی مکانیکی افزایش مییابد. اندازه کریستالیت و کرنش شبکه نمونههای فعالسازی شده در پسماند نسبت به خوراک لیچینگ در هر دو حالت فعالسازی مکانیکی افزایش مییابد. اندازه کریستالیت و کرنش

کلمات کلیدی: اسفالریت، فعالسازی مکانیکی، حالتهای تر و خشک، پسماند لیچینگ و شناسایی نمونه.