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Influence of microwave treatment on surface roughness, hydrophobicity, and chemical composition of galena

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Abstract

The influence of microwave treatment on the surface roughness, hydrophobicity, and chemical composition of galena was studied. The pure galena specimens and purified galena concentrate were used in this work. A conventional multi-modal oven (with a frequency of 2.45 GHz and a maximum power of 900 W) was used to conduct the experiments. The results obtained from the atomic-force microscopy analysis showed that the surface roughness of galena decreased after the microwave radiation. The results also showed that the surface hydrophobicity of galena increased with increase in the duration of the microwave radiation, which was in good agreement with the micro-flotation mass recovery results. The increased surface hydrophobicity may be attributed to the decreased surface roughness by microwave radiation or formation of sulfur on the surface. The results of the SEM/EDS analyses indicated that after microwave radiation, the amount of S increased, whereas Pb decreased on the surface of galena, indicating that the average atomic number of the galena surface changed due to microwave treatment.

1. Introduction

Galena (lead sulfide, PbS) is the main ore mineral extracted by the lead industry. The production of lead, its alloys, and its compounds is important for the manufacture of accumulators, nuclear power plants, ceramic glasses and glazes, pigments, and other chemicals [1]. Some researchers have studied the microwave treatment of pure galena and have determined its heating rate due to microwave radiation [2-6]. the results of heating 40 minerals individually with the microwave energy (30 W, 2450 MHz, 3-5 min exposure time) was reported. The test results indicated that galena was heated readily upon microwave treatment with significant arcing [2]. The microwave heating of a number of minerals at 2450 MHz was studied. The results obtained showed that the temperature of 25 g of powdered galena reached 956 ℃ after 7 min of treatment [3]. The microwave heating tests (500 W of 2450 MHz) on several oxide, sulfide, and carbonate minerals was conducted. The results of these tests showed that the temperature of 50 g powdered galena rose to more than 650 ℃ after 4 min exposure [4]. the heating profile of galena in a massive sulfide ore with an applied power level of 2.6 kW and a frequency of 2.45 GHz was determined. The results obtained showed that the bulk temperature of the galena sample was 211 ℃ after 10 s radiation and reached 453 ℃ after 120 s [5]. The microwave heating on a 20 g galena sample with particle sizes of about 0.2-0.5 mm was studied. The sample was irradiated in a domestic microwave oven with an output of 900 W and at a frequency of 2.45 GHz. The results obtained showed that the temperature of the galena sample after 1 min radiation reached 741 ℃ [7].

The effect of microwave treatment on the grindabiliy of galena-sphalerite ores was studied.

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The results obtained showed that the work index of the sulfide ore was reduced from 11.22 kwhr/sht to 9.38 Kwhr/sht, 8.19 kwhr/sht, and 7.35 kwhr/sht after microwave treatment for 30, 60, and 90 s, respectively [8].

The effects of microwave treatment on the floatability and magnetic susceptibility of pyrite, chalcopyrite, galena, and sphalerite was studied. The results obtained revealed that pyrite, chalcopyrite, and galena were heated up very quickly; their surface temperatures reached ~700 °C to 900 °C, while the temperature of sphalerite reached only ~130 °C. The floatability of pyrite, chalcopyrite, and galena was decreased when the microwave energy and the duration of treatment increased, owing to the oxidation of the mineral surfaces caused by relatively high temperatures. The magnetic properties of pyrite and chalcopyrite improved as the microwave energy and the duration of treatment increased to the point where 90% of the feed was separated as a magnetic product. However, no change was observed in the magnetic properties of galena and sphalerite [9].

The influence of microwave irradiation on the heating characteristics, breakage response, mineralogy, and mechanisms of dissolution in sulfuric acid and hydrochloric acid of a sulfide ore was investigated. The ore used for this work was a complex sulfide ore consisting of silica, siderite, ferrous sphalerite, galena, pyrite, and covellite. The results obtained showed that microwave heating did not affect galena mineralogy; the slight difference in the amounts of lead dissolved was due to the slight increase in pyrite phases of the microwaved ores [10].

The variation in the dielectric properties of five powdered sulfide minerals with frequency and temperature using the cavity perturbation technique was investigated. The samples were examined at frequencies of 625 MHz, 1410 MHz, and 2210 MHz over a temperature range of 0-6500 ℃. The dielectric properties of galena (PbS) and sphalerite (ZnS) exhibited little variation with temperature, whilst pyrite (FeS_2), chalcocite (Cu_2S), and chalcopyrite (CuFeS2) showed significant variations with temperature [11].

The dependence of the real and imaginary permittivity on temperature and temperature distributions at microwave frequencies for andesite, magnesite, siderite, chalcopyrite, pyrite, and galena was studied. In the case of heating galena, the changes of permittivity were observed in the temperature range of 600-1000 ℃. The maximum value of real permittivity reached 800 ℃ where $PbSO_4.4PbO$ was created [7].

The research works conducted to date on the surface characterization of microwave-treated minerals have focused on hematite [12], ilmenite [13, 14] and chalcopyrite [15, 16]. This calls for new research works on the property changes of many other important valuable and gangue minerals exposed to microwave radiation. In this research work, the effects of microwave radiation on the contact angle, surface roughness, and surface elemental analysis of galena were studied and the relationship between these properties and their effect on the flotation recovery were estimated.

2. Experimental

2.1. Materials

Hand-picked samples were taken from the Qaleh-Zari vein copper mine located in the Southern Khorasan Province, Iran. Qaleh-Zari is a specularite-rich Cu-Pb-Ag-Au deposit with chalcopyrite and galena as the main minerals in its ores [17]. Qaleh-Zari is a vein deposit, and pure and big galena specimens can be taken by hand. A number of galena specimens were selected to provide polished sections (with a thickness of 5 mm and a surface area of 1 × 1 cm^2) for the contact angle, surface roughness, and SEM/EDS analyses. The polished sections were pure enough, examined by SEM/EDS analysis.

The rest of the materials were crushed and ground down to 100% passing 106 μm. The purified galena concentrate was produced from the ground product by several stage froth flotation. The flotation concentrate was washed for several times with distilled water and then dried.

The chemical and mineralogical analysis of the purified galena concentrate was carried out by the total X-ray fluorescence (GNR, model TX2000) and X-ray diffraction (Philips PANanalytical X'Pert XRD) methods, respectively. The results presented in Figure 1 and Table 1 indicated that the sample was pure enough to carry out the experiments. The impurities could not significantly affect the micro-flotation experiment results because compared to galena, the impurities had a low proportion.

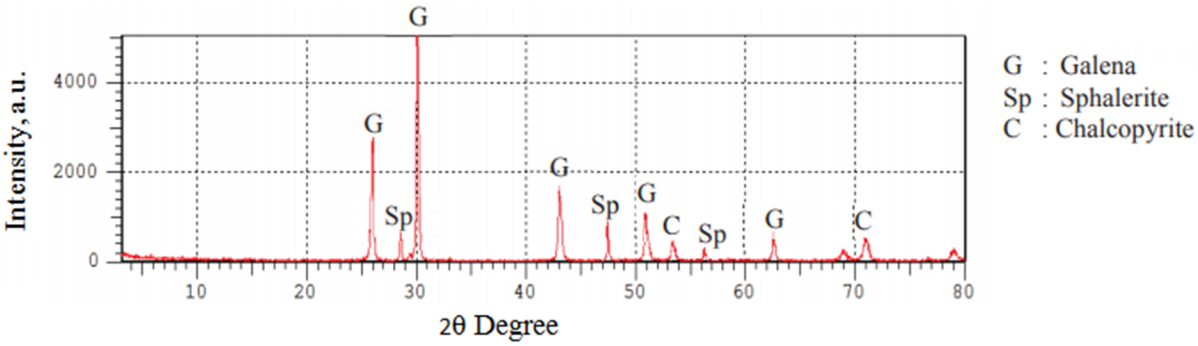


Figure 1. The XRD pattern for the galena concentrate.

Table 1. The chemical composition of the galena concentrate.

Element	Pb	S	Cu	Fe	Zn
%	66.6	15.62	7.38	2.79	0.84

2.2. Microwave treatment

The samples were treated in a 2.45 GHz microwave oven (Samsung-ST656W, with 900 W rated power and 333 × 356 × 210 cavity dimension). Table 2 shows the plan of microwave radiation experiments. For each radiation time, three polished sections and some powders of the galena concentrate with a total weight of 100 g were radiated simultaneously in a microwave transparent pyrex glass tray. The sample factors such as volume, surface area, and location in the oven are important in the magnitude and uniformity of power absorption [18]. To minimize these effects, in all the radiation experiments, the surface area and thickness of the powder spread on the tray were kept constant and the tray was located in the same central position. The radiation time was increased from 10 to 120 s in 6 steps. In all the radiation times, the microwave-rated power was kept constant at 900 W. It should be noted that for each radiation time, an individual sample (including powder and polished section) was used.

The temperature was measured by quickly inserting a K-type thermocouple into the sample after the power was turned off (in order to eliminate any interference from the microwaves, which were monitored by a digital display temperature controller). The measured temperatures are shown in Table 2. It should be noted that the measured temperature is the temperature of the powder of the particles. The sample temperature rose from 105 °C (radiation time 10 s) to 825 °C at a radiation time of 160 s. The temperature response of this sample corroborates the observations by the previous research works [2-5, 7] that the galena temperature increased with an increased microwave radiation time. The increase in ore temperature during heating could also be attributed to the presence of other minerals such as sphalerite and chalcopyrite in the sample that were easily heated by microwaves.

Table 2. Plan of microwave radiation experiments (900 W, 2450 MHz).

Sample code	Radiation time (s)	Temperature (°C)
A	10	105
B	40	220
C	70	330
D	100	500
E	130	625
F	160	925

2.3. Scanning electron microscopy examination

Each polished section was examined before and after microwave radiation by a field emission scanning electron microscope (TESCAN, model FE-SEM). The backscatter images from the sample surfaces before and after microwave radiation were recorded. Also the chemical composition changes in the surface were examined by EDS mapping analysis. EDS mapping gives the overall distribution of all the elements on the surface. The percent average of the elements inside the map was determined and reported.

2.4. Surface roughness measurement

Surface roughness is quantified by examining the deviations in the direction of the normal vector of

a real surface from its ideal form. If these deviations are large, the surface is rough; otherwise, the surface is smooth. A roughness value can be calculated on either a profile (line) or a surface (area) [19].

Surface roughness cannot be accurately characterized using a single roughness parameter. Instead, a set of surface roughness parameters are defined. The parameters that characterize surface profiles are called 2D parameters and are marked with the letter "R". These parameters are widely utilized in different applications but they do not provide full information on 3D surfaces [19]. R_a is the arithmetic average of the absolute values of the roughness profile ordinates. It is one of the most effective surface roughness measures commonly adopted in engineering practices. It gives a good general description of the height variations of the surface. R_z is calculated as a mean height value of five local maxima and local minima. R_{max} is the largest single roughness depth within the evaluation length. The units of those roughness parameters are micrometer and micro inch [19, 20].

Surface roughness can be measured using several methods and different sampling areas. An atomic force microscope is a powerful and versatile tool for measuring surface topography. Because of its ease of use and wide range of applicability, an atomic force microscope has become an increasingly important tool for the measurement of surface roughness [21]. In this research work, the roughness of the polished sections was determined by an atomic force microscope (ARA-AFM, model Full Mode) before and after microwave radiation. For each radiation time, the roughness was measured on 20 linear profiles at the surface before and after radiation. The roughness average of 20 profiles is determined and considered as the roughness surface.

2.5. Wettability measurement

Wettability can be studied by measuring the contact angle of the substrate with the given liquid. The contact angle of distilled water was measured on the polished sections before and after microwave radiation by a contact angle and interfacial tension measurement apparatus (Fars EOR Tech, model VIT-ES20) using a sessile drop method. The polished sections were cleaned in an ultrasonic bath and rinsed with distilled water. The cleaned sections were immersed in a given collector solution for 15 min at a desired pH. The polished sections were dried in an oven at 60 ℃. In the contact angle measurement, a dried polished section was placed in a visual cell and a water drop was created on the polished section using a manual pump and needle system. The angle of a sessile drop resting on a polished section was measured using a CCD camera (HD 2 megapixel) interfaced to a computer with image-analysis software in order to determine the tangent value precisely on the captured image. At least six fresh drops were placed at different locations of the polished section and the average contact angle value was reported.

2.6. Micro-flotation experiments

The aim of the micro-floatation experiments was to study the effects of the changes in the surface roughness and contact angle due to microwave radiation on the flotation behavior of galena mineral. These experiments were carried out in a 150 cm^3 Hallimond tube. In each test, 3 g of purified galena, before and after radiation, were used with a size of 100 μm. The sample was added to doubly distilled water and conditioned for 5 min. After this period, the collector (potassium amyl xanthate, 65 ppm (65 mg/kg) and frother (MIBC, 10 mg/L)) were added to the suspension, the pH was adjusted to 10.5 with an accuracy of ±0.02, and a second conditioning stage of 15 min was given to the suspension. The prepared pulp was then transferred to the Hallimond tube, where flotation was carried out for 4 min. After the flotation tests, the concentrate and tailing were filtered, dried, and weighed.

3. Results and discussion

3.1. Surface roughness

Figure 2 shows the effect of microwave treatment on the surface roughness of galena in terms of R_a, R_z, and R_{max}. In this figure, each point represents the measurements average on 20 profiles at the surface before and after radiation. It could be seen that after a short microwave radiation time (10 s), the surface roughness increased. With longer radiation periods, the surface roughness decreased considerably. The effect of increasing microwave radiation time cannot be recognized because the specimens are not the same in texture and structure. It should be noted that the measurement area of AFM is usually limited to a few square micrometers, which also limits the roughness scale that can be seen. Therefore, the measured roughness may not adequately represent the surface roughness; as a result, the roughness was measured at different points of the surface, and the mean value was reported as the surface roughness.

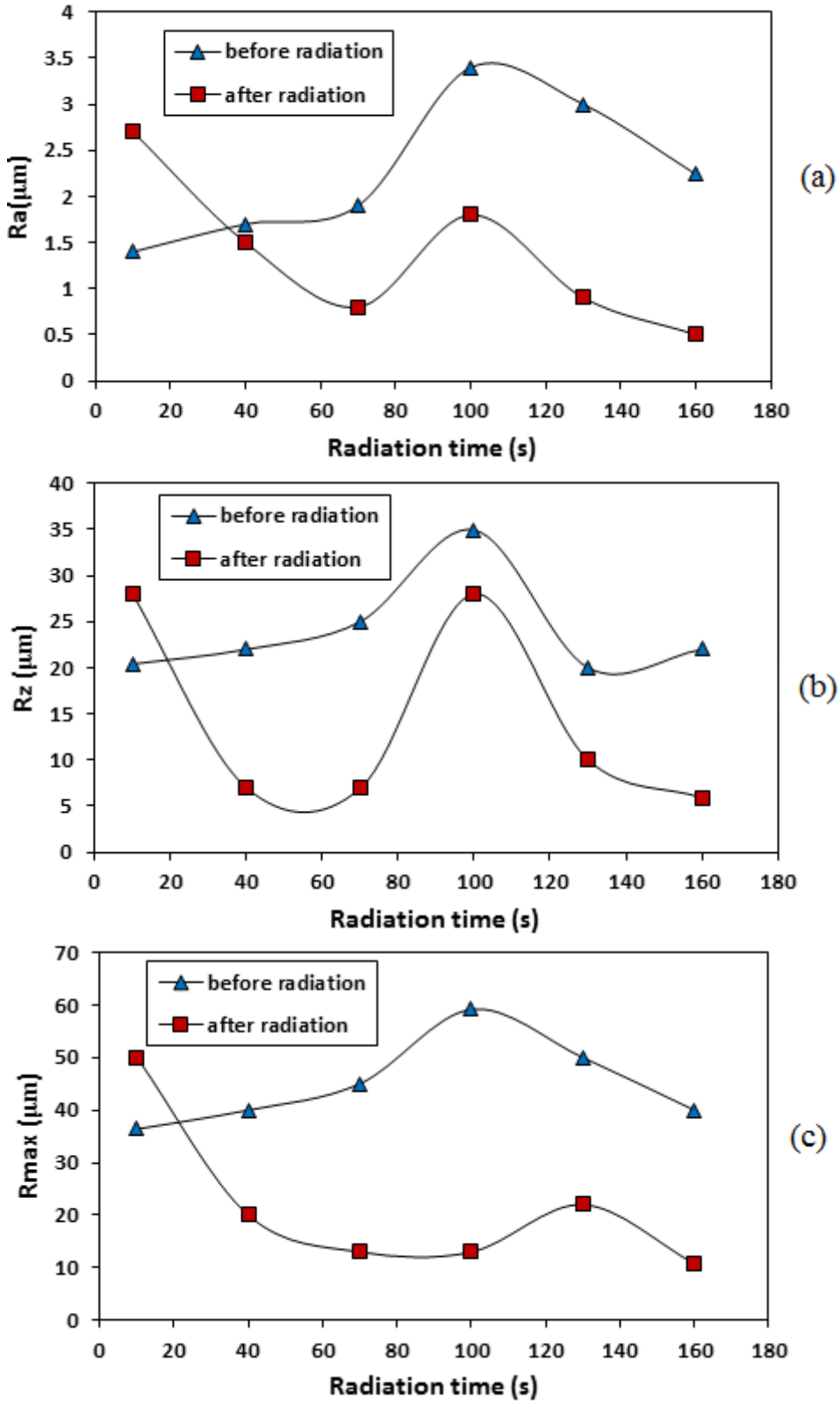
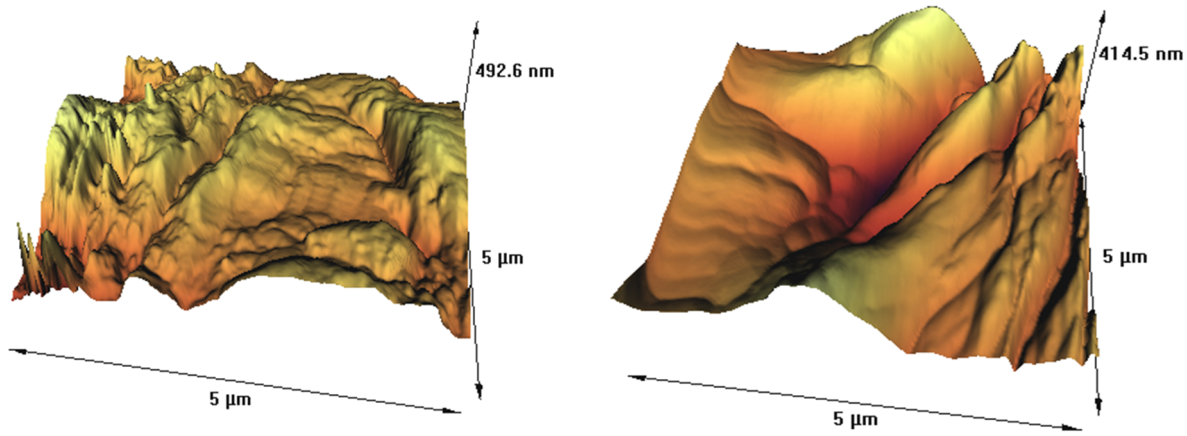


Figure 2. The effect of microwave treatment on the surface roughness of galena: (a) R_a measurements, (b) R_z measurements, (c) R_{max} measurements.

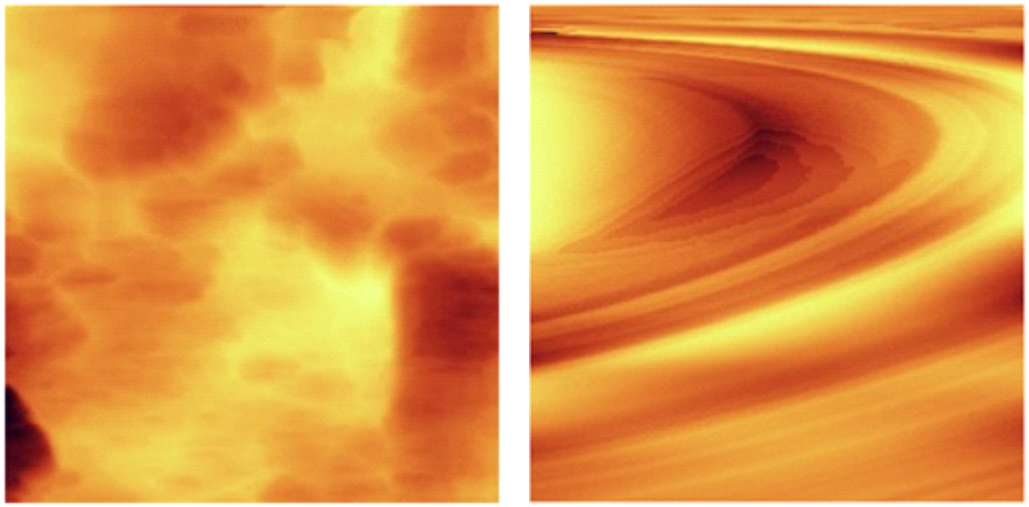
Figures 3 and 4 show the 3D and 2D AFM micro-topography images of galena before and after 40 s of microwave radiation time, respectively. The shading intensity shows the vertical profile of the surface with the light regions being the highest points and the dark regions being depressions. A comparison between the two images showed that after microwave radiation, the number and height of the picks (light points) decreased, and subsequently, the surface roughness decreased.

Microwave heating causes melting of very small particles and picks on the surface; as a result, the surface is clean and the surface roughness is reduced. Similar results have been obtained for microwave heating of other material such as alloys [22, 23] and woods [24, 25]. The results showed that the heat treatment cleaned the surface and reduced the surface roughness. Also the decrease in the surface roughness of galena after microwave radiation may be due to oxidation of surface and formation of lead sulfate and lead oxide and evolution of sulfur dioxide [1]. These compounds are deposited on the surface and fill the depression points. However, a few researchers have focused on the relationship between the heat treatment and surface roughness of materials; this issue should be further investigated.



(a) **(b)**

Figure 3. 3D AFM micro-topography images of galena surface before (a) and after (b) 130 s microwave radiation.



(a) **(b)**

Figure 4. 2D AFM micro-topography images of galena surface before (a) and after (b) 130 s microwave radiation.

3.2. Wettability and micro-flotation

Figure 5 shows the measured contact angle and flotation mass recovery as a function of microwave irradiation time for galena conditioned with 65 ppm of potassium amyl xanthate. It could be seen that after a short microwave radiation time (10 s), the contact angle and flotation mass recovery decreased. With a further increase in the

radiation time from 10 s to 130 s, the contact angle and flotation mass recovery increased slowly. This may be related to the decrease in the galena surface roughness by microwave radiation (see Figures 2-4).

The relationship between roughness and wettability was defined in 1936 by Wenzel [26], who stated that adding surface roughness would enhance the wettability caused by the chemistry of the surface. If the contact angle is greater than 90°, the contact angle will be increased when surface roughness is added, and if the contact angle is less than 90°, the contact angle will be decreased when surface roughness is added. In other words, the hydrophilic surfaces become more hydrophilic with increase in the surface roughness, while the hydrophobic surfaces become more hydrophobic with increase in the surface roughness [27, 28].

From Figure 5, The contact angle for galena surface when there is no microwave radiation is about 65°, and then the surface is a hydrophilic surface. From Figure 2, the surface roughness was decreased after microwave radiation, and then according to the Wenzel model, the contact angle should be increased and thus the galena surface will be more hydrophobic. The data in Figure 5 confirms this prediction and is in good agreement with the Wenzel model. However, quantification of these effects is difficult and somewhat controversial.

Several studies have shown that smoothness help to increase the hydrophobicity [29, 30]. However, the contact angle surface does not only depend on the surface roughness, and a quantitative description is still lacking.

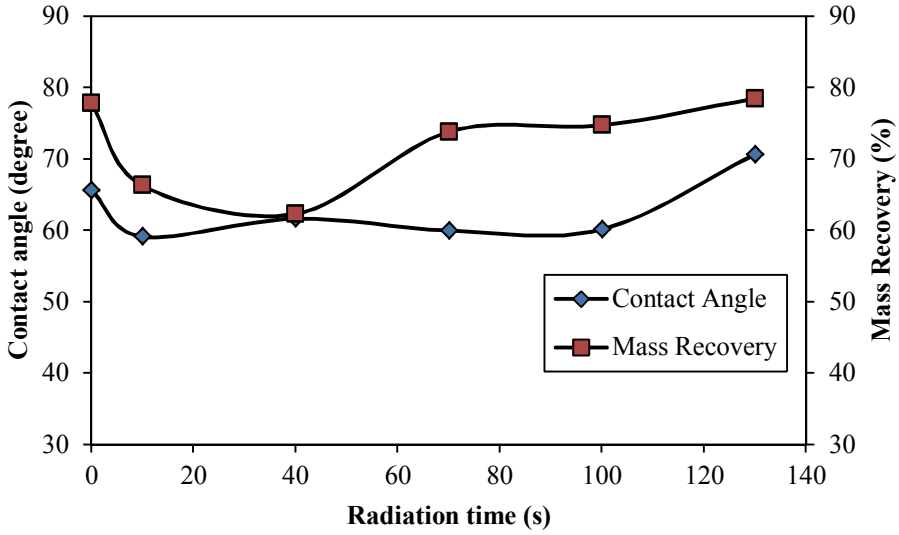


Figure 5. Effect of microwave radiation on the surface contact angle and flotation mass recovery of galena (collector potassium amyl xanthate, pH = 10).

3.3. Scanning electron microscopy

Figure 6 shows the backscatter images from the galena samples before and after 30 s and 50 s and microwave radiation time. The backscattered electron (BSE) signal can provide useful information, which is normally related to the average atomic number at the sample position. The number of BSEs reaching a BSE detector is proportional to the mean atomic number of the sample. Thus a "brighter" BSE intensity correlates with a greater average Z in the sample, and the "dark" areas have a lower average Z. The BSE images are very helpful for obtaining high-resolution compositional maps of a sample and for quickly distinguishing different phases. It could be seen in Figure 6 that after microwave radiation, the light grey color phase appeared and slightly increased at the examined surfaces. It could be concluded that the average atomic number of the galena surface decreased due to microwave treatment.

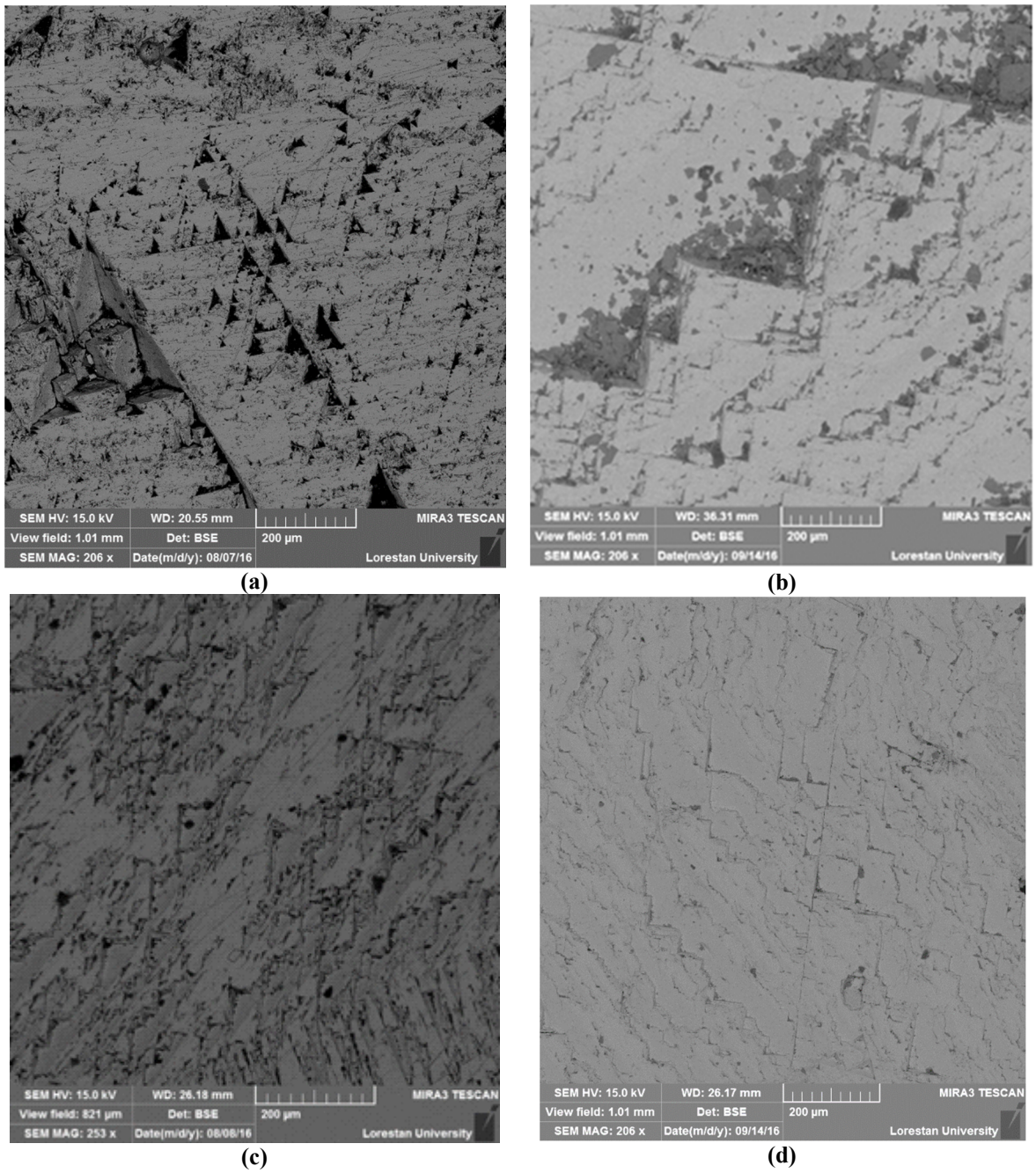


Figure 6. BSE images of galena surface before (a) and after (b) 10 s and before (c) and after (d) 70 s microwave radiation.

Figure 7 shows the EDS analysis for S and Pb before and after microwave radiation, respectively. The results obtained indicate that after microwave radiation, %S increased while %Pb decreased on the surface of galena.

The fast oxidized roasting of galena may be represented as [1, 9]:

$$2PbS + 3O_2 \rightarrow 2PbO + 2SO_2 \quad (1)$$

$$2PbO + 2SO_2 + O_2 \rightarrow 2PbSO_4 \quad (2)$$

$$2PbS + 2O_2 \rightarrow PbSO_4 \quad (3)$$

The oxidation processes that occur on the surface of galena result in the formation of lead oxide, lead sulfate, and sulfur dioxide. Most of the sulfur dioxide resulting from the oxidation of galena is consumed in the formation of lead sulfate and basic sulfates due to its reaction with lead oxide (Eq. 2). At a temperature higher than 900 °C, the sulfates decompose to lead oxide and evolution of sulfur dioxide [1]. Oxidation naturally starts from the outer layer, and it gradually deepens towards the center, forming a banded structure. At a given

microwave energy level, oxidation is a time-dependent phenomenon. Therefore, surface conditioning seems to be possible in a short time, i.e. one minute [9]. Reducing the amount of Pb and increasing the amount of S are due to deposition of sulfate and a sulfur layer (SO_2) on the surface. Also the increase in the contact angle and flotation mass recovery with increase in the radiation time (Figure 5) may be due to the formation of a sulfur layer on the surface because sulfur is a hydrophobic compound.

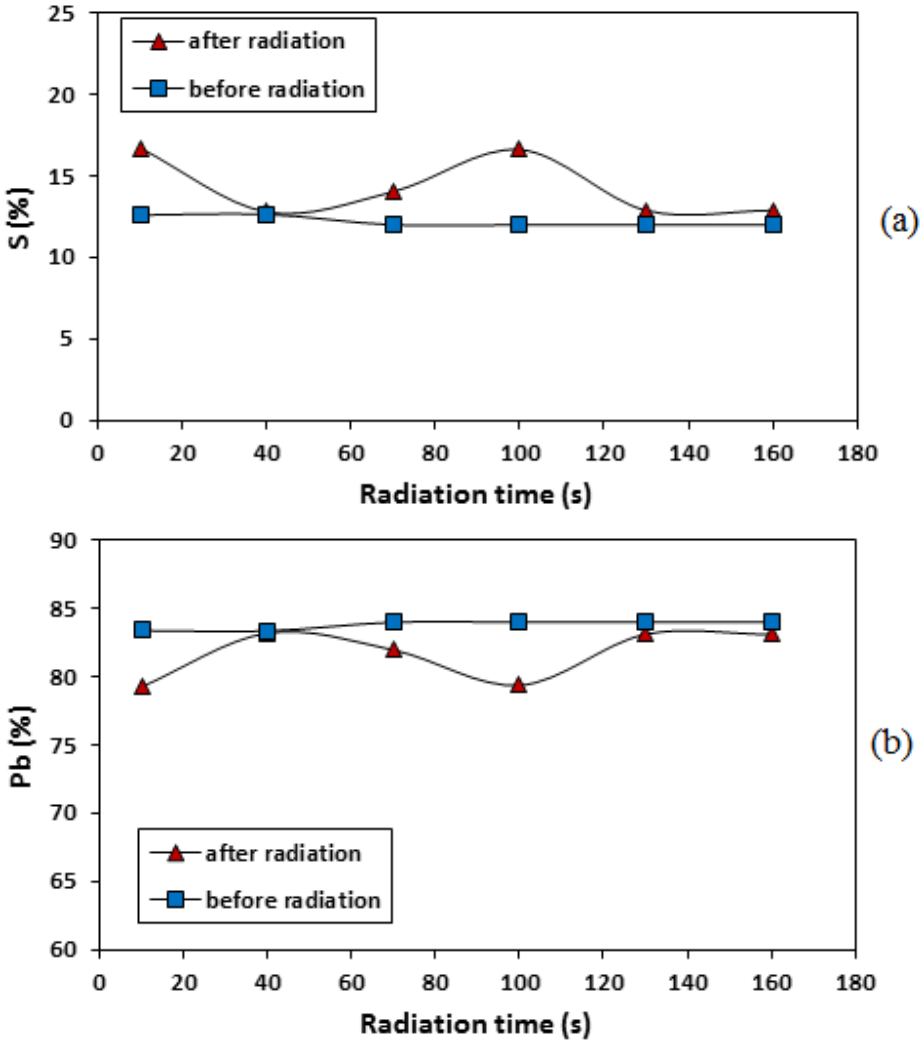


Figure 7. The EDS analysis on the surface of galena before and after microwave radiation: (a) for S, (b) for Pb.

4. Conclusions

Using atomic-force microscopy, it was shown that the surface roughness of galena in terms of Ra, Rz, and Rmax decreased after microwave radiation. The 3D and 2D AFM micro-topography images of galena before and after microwave radiation showed that after microwave radiation, the number and height of the picks decreased. The surface wettability of galena mineral was investigated using a contact angle and interfacial tension measurement apparatus. The results obtained indicated that the surface wettability decreased with increase in the microwave radiation time. The micro-flotation mass recovery increased with increase in the radiation time, indicating a good agreement with the wettability results. The decrease in the wettability of galena surface may be attributed to a decrease in the surface roughness by microwave radiation. The SEM/EDS analyses suggested that after microwave radiation, %S increased while %Pb decreased on the surface of galena so that the average atomic number of the galena surface decreased. Reducing the amount of Pb and increasing the amount of S may be explained by the deposition of SO_2 on the surface.

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تأثیر عمل‌آوری مایکروویو بر روی زبری سطح، آبرانی و ترکیب شیمیایی سطح کانی گالن

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چکیده:

تأثیر پیش عمل‌آوری حرارتی با مایکروویو بر روی زبری سطح، آبرانی و ترکیب شیمیایی سطح کانی گالن مورد بررسی قرار گرفته است. قطعات گالن خالص و پودر کنسانتره پرعیار گالن به عنوان نمونه مورد استفاده قرار گرفته است. گرمایش مایکروویو نمونه‌ها در یک آون مولتی مد (با فرکانس ۲/۴۵ گیگاهرتز و بیشینه توان مصرفی ۹۰۰ وات) انجام شده است. نتایج بررسی زبری سطح نمونه‌ها توسط میکروسکوپ نیروی اتمی نشان داد که زبری سطح گالن پس از گرمایش مایکروویو کاهش یافته است. همچنین نتایج نشان داد که آبرانی سطح گالن و بازیابی وزنی در آزمایش‌های میکروفلوتاسیون با افزایش زمان تابش مایکروویو افزایش یافته است. افزایش آبرانی سطح می‌تواند در نتیجه کاهش زبری سطح و یا تشکیل سولفور بر روی سطح گالن باشد. نتایج بررسی سطح توسط میکروسکوپ الکترونی روبشی (SEM/EDS) نشان داد که پس از تابش مایکروویو مقدار گوگرد (S) در سطح افزایش در حالی که مقدار سرب کاهش یافته است و درنتیجه میانگین عدد اتمی سطح تغییر کرده است.

کلمات کلیدی: گالن، تابش مایکروویو، زبری سطح، آبرانی سطح، فلوتاسیون.