Synthesis and characterization of pyrite mineral from rock deposits in Bantimurung Maros Regency of South Sulawesi

N Ichzan¹,*, A Agussalim¹, R Syam¹, I Rahman¹

¹Physics Research Group, Department of Physics, Universitas Muslim Maros DR. Ratulangi No. 62 Street, Maros City 90511, South Sulawesi, Indonesia

*nuritchzan1@umma.ac.id

Abstract. The study has successfully obtained the mineral pyrite (iron pyrite persulfide) and Hematite (Fe₂O₃) of the natural resources in the Bantimurung District, Maros, South Sulawesi. The method used in this purification is the hydrothermal method. Based on the characterization results, it was obtained pyrite mineral with high purity (more than 90%). Based on the analysis, this material has a cubic structure \( P a \) with lattice parameters \( a = 5.4167 \) Å and a space structure for the hematite group \( -R 32 c \) with lattice parameters \( a = 5.048 \) Å, \( b = 5.048 \) Å, and \( c = 13.762 \) Å. These results are consistent with the surface morphology characterized using SEM with various magnifications showing uneven particle distribution with a particle size of 5 µm-100 nm. The results of characterization and analysis of XRF and XRD show that the degree of crystallization and homogeneity of natural material deposits in Bantimurung are rich in iron, magnesium (Mg), sulfur (S), which appear with a high percentage, and high value minerals such as molybdenum (Mo) and neodymium with a low percentage. With this composition, the materials in Bantimurung have the potential to be used as photovoltaic coatings in solar cell applications, production of sulfur dioxide, paper industry, manufacture of sulfuric acid, semiconductor materials, and applications in sodium and lithium batteries.

1. Introduction

South Sulawesi is one of the provinces in Indonesia which is rich in natural resources, both agricultural and plantation products as well as the potential for mineral wealth in the form of mining materials. One of the areas that is rich in rocks and minerals is Maros, Sulawesi Selatan. Bantimurung is one of the areas in Maros which is predicted to contain minerals such as iron, sulfur, silicon, molybdenum, sodium, aluminum and pyrite. Pyrite can be utilized as a photovoltaic coating in solar cell applications, production of sulfur dioxide, paper industry, the manufacture of sulfuric acid, semiconductor materials, as well as sodium and lithium battery applications. This area is predicted to be rich in pyrite minerals.

The mineral pyrite is an iron sulfide compound \(^{[3,6]}\). Pyrite has the same chemical formula as marcasite, FeS\(_2\), but what distinguishes it is its structure, symmetry and crystal shape \(^{[1,2,4]}\). Its chemical composition is 46.6% Fe and 53.4% S, often containing (in small amounts) the elements Co, Ni, As, Sb, sometimes Cu, Au, Ag \(^{[2]}\). The crystal structure of pyrite is face-centered cubic (FCC) with Fe\(^{2+}\) and S\(^{2-}\) \(^{[3]}\). Pyrite generally have a pale gold color so it’s known as "Fool's Gold" \(^{[6]}\). In addition, pyrite has a hardness characteristic of 6 - 6.5 mohs. The properties of the material must be known to use and take the advantage of the material, because natural materials usually have a complex composition. Natural materials also have a phase formation.
that are difficult to predict that further studies with more comprehensive characterization needed to dig deeper. Various methods of synthesis have been used including methods of thermal reaction \[9\], method of hydrothermal, methods of mechanical milling \[8\], method solvothermal \[9\], and solid reaction method. In this study, researcher used a hydrothermal method. The reasons for selecting this method is produced pyrite in this study is single crystals \[3,5\], the calcination temperature is low (Wu et al., 2004), the particles are distributed in small size, the ability to create a composite such as an organic and inorganic mixture \[4\], and the ability to make materials that have high vapor pressure.

This article discusses the results of research on methods, properties and characteristics of pyrite from rock deposits in Bantimurung area in order to obtain all details information about this material. The aim is to optimize process parameters that can be used optimally for various applications in the technology field.

2. Methods

The research is designed to explore the deposits of natural material in Bantimurung area that can be used as advanced materials that have added value to these materials. The experimental design in this study consisted of three stages: (1) Material processing including selecting, material taking and sample preparation, then sample characterization consisting of microstructural characterization (XRF, XRD, SEM) and data analysis including qualitative, quantitative and microstructure analysis.

The samples of natural materials derived from rock minerals in the Bantimurung, Maros Regency. The process of assessing this material begins with sample preparation from deposits of natural material in powder and bulk form so that the materials characterization/testing can be carried out more easily, the formation of samples in bulk form using the solid-state reaction method or synthesis by melting through several treatments such as grinding, heating (calcination, sintering and annealing) at high temperatures, and applying pressure. Furthermore, for sample characterization that will be done through:

2.1. Characterization of chemical properties

In analysis of chemical properties, data of chemical composition of materials will be obtained through XRF analysis, pH analysis of the material using glass electron method, which is a standard method for determining the pH of water, density (\( \rho \)) using the equation \( \rho = m/V \). The acidity level is obtained from the ratio of acidic oxides such as SiO\(_2\) to basic oxides such as CaO and MgO after XRF analysis, and the solubility test was performed using the Toxicity characteristic Leaching Procedure (TCLP). The results of TCLP were analyzed to determine the concentration of elements (Fe and S) using the atomic absorption value with an atomic absorption spectrophotometer.

2.2. Characterization of physical properties

In analysis of physical properties, data of grain size distribution and homogeneity will be obtained through SEM image analysis, composition of mineral compounds with EDAX, crystallography and phase analysis of the dominant compounds in the material will be obtained so that the implications of the application can be determined. Diffraction data were analyzed using a single peak pattern (single line) and a whole pattern. Data analysis was also performed using the Rietveld method which used the peak-shape function pseudo-Voigt to obtain parameters U, V, and W. The initial step in this analysis was matching (refinement) between measured data and calculated data using Rietica software.

3. Results and discussion

Bantimurung material deposits are rich in various minerals with economic value, for example Pyrite (FeS\(_2\)) and Hematite (Fe\(_2\)O\(_3\)). This research analyzes in depth the potential of Bantimurung’s natural minerals to be materials that have economic value so that extraction process is possible. The material deposits are shuttered and crushed to obtain a fine powder sample. Deposit processing procedures of natural material into powder form begins with the preparation of the basic material to be processed
followed by pulverizing using a hammer up to facilitate the processing of the material deposit. Material deposits in the form of small pebble-sized rocks are rinsed with water and then soaked with 96% alcohol to minimize contamination of impurities. Then the sample was dried using a furnace at 150°C temperature for 19 hours followed by grinding. The result of this process is a powdered sample ready for SEM and XRD characterization. In this characterization and analysis process, five samples were selected based on the differences in color obtained. Following are the results of characterization and analysis.

![Figure 1. Bantimurung Maros regency area maps](image1.jpg)

![Figure 2. Material deposits from Bantimurung](image2.jpg)

![Figure 3. Powder sample of material deposits](image3.jpg)

3.1. Ingredients and composition

To determine the elemental composition, characterization was carried out using XRF. The results of XRF characterization of some samples are shown in the picture on the side. The following figure provides information on the main elemental composition of the sample, that is Sulfur (S) and Iron (Fe) which has a 97.95% mass percentage with 66.57% Sulfur (S) and 31.38% Iron (Fe) of total mass of the sample. The sample was characterized by a shiny bright yellow color while the other samples had a
dark yellow color and slightly black color. The main elemental compositions are Fe and S which have a mass percentage of 98.26%, with 66.34% Fe and 31.92% S of the total mass. Based on the data in the Figure 4-7, it can be seen that the fundamental differences of the samples are characterized by the color and content of the dominant elements.

Figure 4. Result of XRF from sample B
Figure 5. Result of XRF from sample B

Figure 6. Result of XRF from sample C
Figure 7. Result of XRF from sample D

Characterization was continued by using XRD to determine the phase formation of each sample. Furthermore, the XRD characterization results are presented as shown in the figure below. Based on the results, it appears that for all samples of the Bantimurung natural rock deposit shows main compositions are Iron and Sulfur with varying total fractions of Iron and Sulfur characterized by differences in color of the material deposits.

Figure 8. Diffractogram of sample A
Figure 9. Diffractogram of sample B

Based on the EDS results for the natural deposit samples, it can be concluded that the main compositions of natural material deposit samples are S and Fe. The composition of Fe is around 79% and S is around 21%. With such a large composition, the Bantimurung material deposits is further processed to extract FeS₂ (Pyrite).
The next extraction process is carried out by processing the deposit sample which has a shiny golden yellow color according to the characteristics of the mineral pyrite. Then the sample was washed using distilled water and soaked in 96% alcohol then dried and crushed until powder form and lastly characterized using XRD. From the results of XRD characterization and analysis using the Match2 application, shown in the picture, a single phase of Iron Persulfides Pyrite has been obtained with a space group structure \( \text{P a\ IRA} \) in the cubic form with lattice parameters \( a = 5.4167 \text{ Å} \).
Figure 12. Diffractogram of sample after extraction

3.2. Microstructure of pyrite mineral

These results are consistent with the surface morphology characterized using SEM as shown in Figures 13-16 at various magnifications. The morphology of the natural material deposits in Bantimurung shown in Figure 13-16 respectively with a magnification of 500, 1000, 3000, and 5000 times its original size. Figure 13-16 shows the surface of natural deposit powder in solids with various sizes of non-uniform cubic form with grain sizes of 5 μm - 100 nm.

Figure 13. SEM image of 500x magnification

Figure 14. SEM image of 1000x magnification.
The XRD and XRF diffracogram patterns for the sample before purification showed a relatively low pyrite content. However, the diffracogram pattern for the samples after purification showed a high pyrite content. This is because the particles or impurities have been reduced in the sample so that the diffracogram pattern is clearly formed \([1,2,3]\). The same result is also shown by the SEM image display that the pyrite morphology obtained in Bantimurung sub-district is in accordance with the morphological form obtained in the results of the study with the average size of the particles in the form of plates with an average size of \(\pm 5 \mu m-100 \text{ nm}\) \([2,9]\). The results obtained further prove that the province of South Sulawesi contains quite high pyrite minerals, as in the previous research \([1]\) in Bontocani Bone district of South Sulawesi province.

### 4. Conclusion
It has been successfully obtained high purity pyrite (Iron persulfide Pyrite) and Hematite \((\text{Fe}_2\text{O}_3)\) from natural material in Bantimurung District, with a space group structure \(\text{P a -3}\) in cubic shape with lattice parameters \(a = 5.4167 \text{ Å}\) and space group structure Hematite \(-\text{R 3 2 c}\) with lattice parameters \(a = 5.048 \text{ Å}, b = 5.048 \text{ Å}, \text{ and } c = 13.762 \text{ Å}\). These results are consistent with the surface morphology characterized using SEM in various magnifications. The results of characterization and analysis of XRF, XRD and SEM show that the degree of crystallization and homogeneity of natural material deposits in Bantimurung District Maros Regency is rich in iron, magnesium, sulfur, and high-value minerals such as Molybdenum (Mo), Neodymium on a minor scale, all of which have the potential combined with other materials either as raw material or additive material to produce a new alloy material that has a value of more extensive benefits, both for industrial applications and economic benefits for society welfare.

### References
\[1\] Haris, A., Amin, B.D., Momang, A., Nurhasmi, N., 2015. Sintesis dan Karakterisasi Pyrite \((\text{FeS}_2)\) Dari Deposit Mineral Kecamatan Bontocani, Kabupaten Bone, Sulawesi. J. Sains Dan Pendidik. Fis. 10.
\[2\] Hsiao, S.-C., Hsu, C.-M., Chen, S.-Y., Perng, Y.-H., Chueh, Y.-L., Chen, L.-J., Chou, L.-H., 2012. Facile synthesis and characterization of high temperature phase \(\text{FeS}_2\) pyrite
8

nanocrystals. Mater. Lett. 75, 152–154. doi:10.1016/j.matlet.2012.02.033

[3] Huang, L.Y., Meng, L., 2010. Crystallographic behavior of FeS2 films formed on different substrates. Mater. Chem. Phys. 124, 413–416. doi:10.1016/j.matchemphys.2010.06.056

[4] Kim, H.T., Nguyen, T.P.N., Kim, C., Park, C., 2014. Formation mechanisms of pyrite (FeS2) nano-crystals synthesized by colloidal route in sulfur abundant environment. Mater. Chem. Phys. 148, 1095–1098. doi:10.1016/j.matchemphys.2014.09.024

[5] Liu, L., Yuan, Z., Qiu, C., Liu, J., 2013. A novel FeS2/CNT micro-spherical cathode material with enhanced electrochemical characteristics for lithium-ion batteries. Solid State Ion. 241, 25–29.

[6] Muscat, J., Hung, A., Russo, S., Yarovsky, I., 2002. First-principles studies of the structural and electronic properties of pyrite FeS2. Phys. Rev. B 65, 054107.

[7] Pelé, V., Flamary, F., Bourgeois, L., Pecquenard, B., Le Cras, F., 2015. Perfect reversibility of the lithium insertion in FeS2: The combined effects of all-solid- state and thin film cell configurations. Electrochem. Commun. 51, 81–84. doi:10.1016/j.elecom.2014.12.009

[8] Puthusserry, J., Seefeld, S., Berry, N., Gibbs, M., Law, M., 2010. Colloidal iron pyrite (FeS2) nanocrystal inks for thin-film photovoltaics. J. Am. Chem. Soc. 133, 716–719.

[9] Zhang, D., Tu, J.P., Mai, Y.J., Zhang, J., Qiao, Y.Q., Wang, X.L., 2012. Preparation and characterization of FeS2/polyaniline composite electrode in lithium-ion battery. J. Aust. Ceram. Soc. 48, 189–193.