

Mineralogical and Structural Characterization of Sedimentary Rocks from Leang Panning Karst Region, Maros, Indonesia

A. Surtika Putri^{a*}, Insira Nayla Sari^a, Subhan Eka Putra Anwar^{a,b}, Subaer Subaer^a

^aDepartment of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Makassar, Makassar, 90222, Indonesia.

^bLaboratory of Natural Materials and Magnetism, Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Makassar, Makassar 90224, Indonesia

Corresponding Authors E-mail: 210104500002@student.unm.ac.id

Article Info

Article info:

Received: xx-xx-20xx

Revised: xx-xx-20xx

Accepted: xx-xx-20xx

Keywords:

Rock minerals, XRD, XRF, optical microscopy, limestone, Leang Panning

How To Cite:

xxxxxx

DOI:

xxxxxx

Abstract

This study aimed to identify the elemental composition and phases in rock minerals collected from Leang Panning Park, Mallawa, Maros Regency, South Sulawesi. The methods employed included X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), and Optical Microscopy. The XRF results indicated the presence of elements such as Ca, Si, Al, Mg, Zr, and Fe. XRD analysis revealed the presence of three main crystalline phases: Calcium Silicate (80.2%), Brownmillerite (11.9%), and Iron Oxide (7.9%). Optical microscopy and ImageJ analysis provided visual confirmation of mineral grains. The characterized sample was identified as sedimentary limestone, notable for its physical strength yet susceptibility to erosion. This study contributes to the mineralogical mapping of the region and supports the potential for further geoscientific exploration.



xxxxxxxxxx.

1. Introduction

Crystallography and mineralogy are fundamental subfields within Earth sciences that play a crucial role in understanding the structure, composition, and genesis of minerals and rocks. Crystallography specifically investigates the internal arrangement of atoms in crystalline solids, while mineralogy focuses on the physical and chemical properties of naturally occurring inorganic solids, or minerals, which compose rocks. These disciplines are essential not only for academic purposes but also for industrial applications such as mining, construction materials, ceramics, and geotechnical engineering [1].

Rocks, as aggregates of one or more minerals, are classified into three main types based on their origin: igneous, sedimentary, and metamorphic. Each rock type is formed under different geological conditions and possesses unique mineralogical compositions. For example, sedimentary rocks such as limestone are formed through the accumulation and compaction of mineral and organic particles over long periods, typically in aquatic environments. These rocks often contain high amounts of calcium carbonate and can include additional components such as quartz, clay minerals, and iron oxides depending on their depositional history [2].

Accurate identification and characterization of rock-forming minerals require sophisticated analytical tools. X-Ray Fluorescence (XRF) is widely used to determine the elemental composition of samples, while X-Ray Diffraction (XRD) provides detailed information on the crystalline phases present. Optical microscopy, complemented by digital image analysis, allows for the visualization of mineral textures and grain structures, contributing further insights into the rock's formation history [3]. These techniques enable researchers to correlate mineralogical data with geological processes and environmental conditions.

In this study, the focus was on characterizing rock samples collected from Leang Panning Park, located in Mallawa District, Maros Regency, South Sulawesi, Indonesia. This region is geologically significant due to its diverse lithological formations, particularly those associated with karst landscapes. Leang Panning Park is well-known for its limestone caves and is part of the Maros-Pangkep karst region, one of the most extensive and well-preserved karst areas in Southeast Asia. The area is not only rich in mineral resources but also holds archaeological importance, evidenced by the discovery of ancient human remains and cave art [2].



Figure 1. Leang Panning Park Mallawa, Maros.

The exploration site, situated near a clear river and surrounded by complex geomorphological structures such as caves and rocky outcrops, offers a valuable natural laboratory for mineralogical research. The presence of limestone caves, including stalactites and stalagmites, suggests the dominant role of calcium-bearing minerals in the local geology. Given these factors, Leang Panning Park was chosen for sample collection to investigate the types of minerals and elemental compositions present in the local rock formations, with the goal of contributing to both geological mapping and educational field studies.

This study aims to identify the elements and crystalline phases present in rock samples from Leang Panning Park using XRF, XRD, and optical microscopy. The findings are expected to enhance our understanding of sedimentary rock formation in karst environments and support future research on the utilization of local mineral resources.

2. Experimental Method

2.1 Sample Preparation.

This study adopted a comprehensive experimental methodology consisting of field exploration, sample preparation, and instrumental analysis to investigate the mineral composition of rocks collected from Leang Panning Park, Mallawa District, Maros Regency, South Sulawesi. The experimental workflow was structured to ensure the integrity and reliability of the data obtained throughout the characterization process.

The initial phase involved field sampling carried out on October 7–8, 2023. The site was selected due to its rich geological features, particularly its karst landscape and the presence of sedimentary rock formations exposed along riverbanks and within limestone caves. During sampling, rock specimens were carefully selected based on visible differences in color, texture, and hardness. Each sample was labeled and recorded with its location to maintain traceability and was stored in clean, sealed containers to minimize contamination during transportation.

Once in the laboratory, samples underwent a preparation process tailored to the requirements of different analytical techniques. Bulk rock samples were cut into rectangular slabs of approximately 2 cm × 2 cm with a thickness of 2 mm using a diamond wheel cutter. These slabs were used for both XRD and optical microscopy. The surfaces of these slabs were then polished using silicon carbide abrasive papers in increasing grit sizes (P400 to P1200) to obtain a smooth and reflective surface suitable for structural analysis. For elemental analysis using XRF, a portion of each sample was crushed into fine powder using a mechanical grinder, and the resulting powder was sieved to ensure a homogeneous particle size distribution below 75 microns. All samples were cleaned using an ultrasonic bath filled with distilled water for about 10 minutes and subsequently dried in ambient air.

The elemental composition of the powdered samples was analyzed using X-Ray Fluorescence (XRF) spectroscopy. This analysis was performed at the National Research and Innovation Agency (BRIN) on October 30, 2023. The XRF instrument employed an energy-dispersive detector system, and measurements were conducted under controlled conditions using standardized operating parameters such as 50 kV excitation voltage and 200 μ A current. Prior to analysis, the instrument was calibrated using certified reference materials to ensure the accuracy of the qualitative and semi-quantitative data obtained.

To determine the crystalline structure and phase composition of the rock samples, X-Ray Diffraction (XRD) analysis was conducted at the Microstructure Laboratory of the Department of Physics, Universitas Negeri Makassar, on November 6, 2023. The analysis used a PANalytical X'Pert Pro diffractometer equipped with a Cu-K α radiation source ($\lambda = 1.5406 \text{ \AA}$). Scanning was performed in a 2θ range of 10° to 70° , with a step interval of 0.02° and a scan speed of 1° per minute. The polished slab samples were mounted on a sample holder and aligned to ensure optimal

signal acquisition. Diffraction data were recorded digitally for further processing and phase identification using specialized crystallographic software.

Optical characterization was carried out using a compound light microscope at the Modern Physics Laboratory of FMIPA UNM on November 7, 2023. Prepared rock slabs were observed under magnifications of 40×, 100×, and 400×. The microscope was equipped with a digital camera to capture high-resolution images for subsequent analysis. Image processing and quantitative measurements were performed using ImageJ software, which allowed for the assessment of grain size, distribution, and surface morphology. This step was essential for examining the microstructure of the rock matrix and identifying textural features.

3. Results and discussion

This section presents the results obtained from three main characterization techniques: X-Ray Fluorescence (XRF), Optical Microscopy with image analysis, and X-Ray Diffraction (XRD). Each method contributes to understanding the chemical composition, microstructure, and crystalline phases of the rock samples collected from Leang Panning Park.

3.1 XRF Analysis

X-Ray Fluorescence (XRF) analysis was used to determine the elemental composition of the rock samples in powdered form. The results revealed that the dominant element was calcium (Ca) with an atomic percentage of 41.7%, suggesting the presence of calcium-rich minerals. Other significant elements included silicon (Si) at 1.1%, aluminum (Al) at 0.567%, magnesium (Mg), zirconium (Zr), iron (Fe), and trace amounts of potassium (K), titanium (Ti), sulfur (S), and lead (Pb), among others.

These elemental findings are consistent with the expected composition of sedimentary carbonate rocks, particularly limestone, which is typically composed of calcium carbonate (CaCO_3) with additional silicates and oxides. The presence of silicon and aluminum, albeit in lower concentrations, suggests possible impurities such as clay minerals or silicate phases within the rock matrix. The detection of iron and zirconium could be related to minor inclusions or accessory minerals, which may also influence the rock's coloration or physical weathering behavior [4].

Table 1. XRF (*X-Ray Fluorescence*)

Mineral	Mass (%)
Mg	0.304
Al	0.567
Si	1.1
S	0.0141
K	0.0553
Ti	0.0412
Mn	0.0345
Fe	0.239
Sn	0.0104
Ca	41.7
Zr	0.301

3.2 Optical Measurements

The optical microstructure of the rock was investigated using a compound light microscope, and the resulting images were further analyzed using ImageJ software. This technique provided visual evidence of the sample's grain structure and allowed quantitative measurement of particle sizes and light intensity distribution across different mineral phases.

Three major mineral components were visually identified and analyzed:

1. Calcium Silicate ($\text{Ca}(\text{Si}_2\text{O}_5)$)

Particles exhibited relatively large areas and uniform brightness under the microscope, indicating well-formed grains. The measured areas ranged between 0.06 and 0.12 mm² with intensity values between 132 and 175.

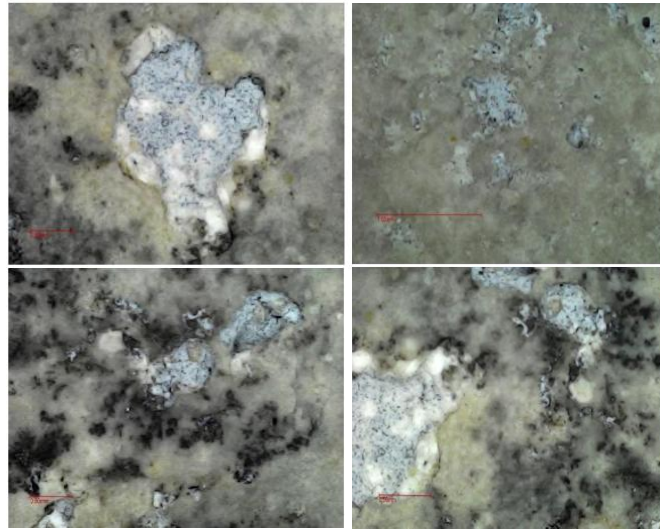


Figure 2. Optic Microscop images of Calcium Silicate

2. Brownmillerite ($\text{Ca}_2(\text{Al,Fe})_2\text{O}_5$).

These grains showed darker intensities and smaller average sizes, consistent with the orthorhombic crystal system typical of brownmillerite. Their mean intensity values ranged from 66 to 108, with smaller surface areas [3].

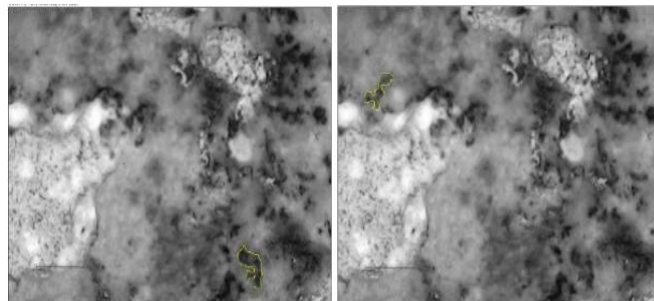


Figure 3. Optic Microscop images of Brownmillerite

3. Iron Oxide ($\text{Fe}_{0.872}\text{O}$).

The particles were distinguishable by their high reflectivity and rounded shapes. The mean intensity values ranged from 120 to 182, indicating higher light absorption compared to the silicates.

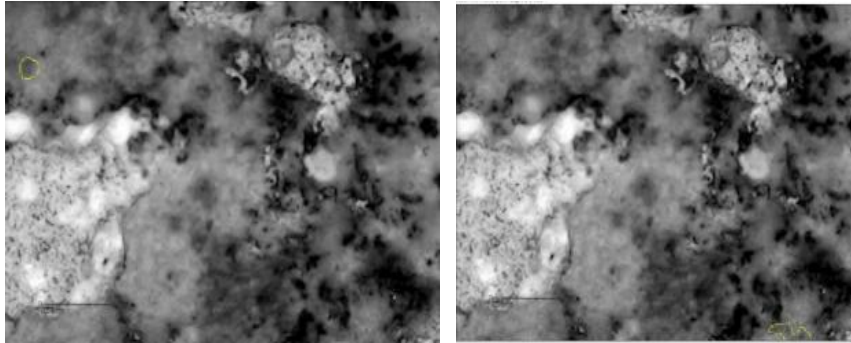


Figure 4. Optic Microscop images of Brownmillerite

The microscope analysis confirmed that the sample was polymineralic, with a matrix dominated by calcium silicate and dispersed secondary phases of brownmillerite and iron oxide. The observed grain sizes, brightness variations, and textures support the interpretation that the rock originated from sedimentary deposition followed by diagenetic alteration.

3.3 XRD Analysis

X-Ray Diffraction (XRD) was employed to identify the crystalline phases present in the rock samples. The resulting diffractogram revealed several sharp and intense peaks, indicating a highly crystalline structure. Peak matching using HighScore Plus software and the JCPDS database confirmed the presence of three primary crystalline phases: Calcium Silicate ($\text{Ca}(\text{Si}_2\text{O}_5)$), constituting approximately 80.2% of the total crystalline content, Brownmillerite ($\text{Ca}_8\text{Fe}_8\text{O}_{20}$), accounting for around 11.9%, Iron Oxide ($\text{Fe}_{0.872}\text{O}$), representing approximately 7.9% of the material [5].

The strongest diffraction peak was observed at a 2θ angle of 29.67° , corresponding to the main lattice plane of calcium silicate. Brownmillerite and iron oxide showed identifiable peaks at characteristic positions such as 23.3° , 39.7° , and 48.7° , confirming their presence as minor phases [6].

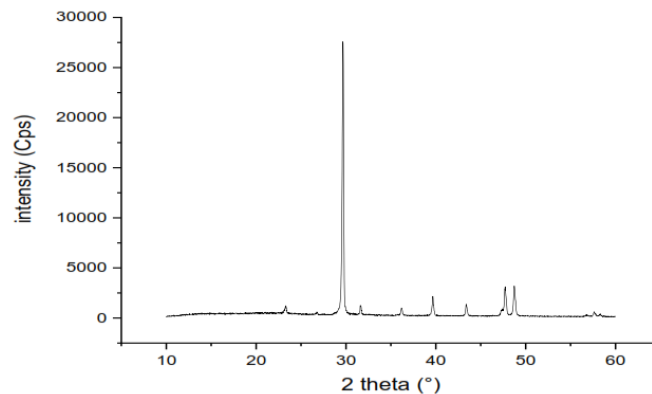


Figure 5. XRD pattern of sample

These results indicate that the rock sample is dominantly composed of calcium silicate, a typical component of cementitious materials, along with minor phases that likely contribute to the mechanical and thermal behavior of the rock. The presence of these mineral phases supports the classification of the sample as limestone, which aligns with its geological setting in a karst region [2].

4. Conclusion

Based on the comprehensive characterization of rock samples from Leang Panning Park using XRF, optical microscopy, and XRD, it can be concluded that the rocks are dominantly composed of calcium silicate, with minor phases of brownmillerite and iron oxide. The high concentration of calcium detected in the XRF analysis, the grain morphology observed under optical microscopy, and the crystalline phase identification through XRD collectively indicate that the sample belongs to the sedimentary limestone category, typical of karst environments. This study highlights the mineralogical complexity and geological significance of the Leang Panning area, providing a foundation for further research on local rock resources and their potential applications in materials science and geotechnical studies.

References

- [1] Tang W, Yang S, Cheng G, Gao Z, Yang H, Xue X, et al. Effect of TiO₂ on the Sintering Behavior of Chromium-Bearing Vanadium–Titanium Magnetite. *Minerals* 2018;8. <https://doi.org/10.3390/min8070263>.
- [2] Molahid VLM, Kusin FM, Hasan SNMS, Ramli NAA, Abdullah AM, Molahid VLM, et al. CO₂ Sequestration through Mineral Carbonation: Effect of Different Parameters on Carbonation of Fe-Rich Mine Waste Materials. *Processes* 2022;10. <https://doi.org/10.3390/pr10020432>.
- [3] Kang D, Yang Z, Zhang D, Jiao Y, Fang C, Wang K, et al. Study on the Effect of Temperature on the Crystal Transformation of Microporous Calcium Silicate Synthesized of Extraction Silicon Solution from Fly Ash. *Materials* 2023;16. <https://doi.org/10.3390/ma16062154>.
- [4] Shamsudin R, Azam F ‘Atiqah A, Hamid MAA, Ismail H, Shamsudin R, Azam F ‘Atiqah A, et al. Bioactivity and Cell Compatibility of β -Wollastonite Derived from Rice Husk Ash and Limestone. *Materials* 2017;10. <https://doi.org/10.3390/ma10101188>.
- [5] Morabito G, Marinoni N, Bais G, Cantaluppi M, Botteon A, Colombo C, et al. Synchrotron Micro-X-ray Diffraction in Transmission Geometry: A New Approach to Study Polychrome Stratigraphies in Cultural Heritage. *Minerals* 2024;14. <https://doi.org/10.3390/min14090866>.
- [6] Liang L, Bao X, Liang W, Song H, Wu X, Qin S, et al. In-Situ Single Crystal XRD and Raman Spectra Investigation of (Mg, Fe, Mn)CO₃ at Various Temperatures. *Minerals* 2023;13. <https://doi.org/10.3390/min13020207>.