



# Characterization of Dunggilata Gold Tailings and Treatment Process Recommendations

Ninasafitri<sup>1\*</sup>, Vivi Dia Afrianti Sangkota<sup>2</sup>, Yuyu Indriati Arifin<sup>1</sup>

<sup>1</sup>Geological Engineering Study Program, Faculty of Sciences and Mathematics, Universitas Negeri Gorontalo, Gorontalo, Indonesia.

<sup>2</sup>Department of Chemistry, Faculty of Sciences and Mathematics, Universitas Negeri, Gorontalo, Indonesia.

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Corresponding Author:

Ninasafitri

[ninasafitri14@gmail.com](mailto:ninasafitri14@gmail.com)

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**Abstract:** Rare earth elements (REEs) are a group of seventeen metallic elements consisting of scandium (Sc), yttrium (Y), and fifteen other lanthanide elements. Rare earth metals have an important role in various technologies, so the analysis and separation of these elements from their minerals is very important to do. This study aims to characterize the tailings through X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF), and Inductively Coupled Plasma Mass Spectrometry (ICP-MS) techniques. The results of XRD analysis show that the mineral composition of tailings is dominated by quartz (SiO<sub>2</sub>) with a percentage of 88%, followed by albite (NaAlSi<sub>3</sub>O<sub>8</sub>) with a percentage of 11%. In addition, the results of XRF analysis show that the main elemental composition in the tailings consists of SiO<sub>2</sub> (69.294%), Al<sub>2</sub>O<sub>3</sub> (20.20%), and Fe<sub>2</sub>O<sub>3</sub> (4.681%). In addition, heavy metal concentrations in the tailings indicate a potentially serious risk of environmental pollution, with arsenic (As) reaching 100 ppm and mercury (Hg) more than 5 ppm. In addition, rare earth elements identified in the tailings include cerium (Ce) at 14,578 ppm, lanthanum (La) at 7,850 ppm, and neodymium (Nd) at 6,897 ppm. This method is expected to help tailings management, reduce environmental impacts, and support sustainable mineral utilization.

**Keywords:** Mineral characterization; Rare earth element; Tailings waste

## Introduction

Gorontalo Province has abundant gold reserves, making it one of the areas with high community mining activities. These artisanal mines are scattered in various regions and are a source of livelihood for the local community (Idrus, 2021). However, despite the economic benefits, these activities also generate large amounts of tailings, which are often disposed of without further treatment. These tailings have the potential to pollute the environment, especially as they contain heavy metals such as arsenic and mercury that are harmful to ecosystems and human health (Sari, 2019). Although considered as waste, tailings from the gold mining process actually still contain various valuable elements, one of which is rare earth elements (REE).

Rare earth metals are a group of 17 metallic elements that have unique properties, making them

widely used in advanced technologies (Singh et al., 2024). As the global demand for rare earth metals increases, especially to support environmentally friendly technologies, the utilization of these resources is becoming increasingly important (Kusmita et al., 2022). Rare Earth Metals are a form of associated minerals from major minerals such as gold, silver, tin, copper, etc (Hendriana et al., 2023). In addition, their use extends to various sectors such as healthcare, aviation, defense, modern transportation, and future technologies such as superconductors. The demand for rare earth metals is expected to increase along with the development of the electric vehicle trend (Kusmita et al., 2022; Chung et al., 2022). Rare Earth Metals consist of 17 elements, with 15 elements belonging to the lanthanide group, as well as two other elements, namely Scandium and Itrium. These elements include lanthanum (La), cerium (Ce), praseodymium (Pr), neodymium (Nd),

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prometium (Pm), samarium (Sm), europium (Eu), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), erbium (Er), tulium (Tm), erythorbium (Yb), lutetium (Lu), yttrium (Y), and scandium (Sc) (Hendriana et al., 2023). Broadly speaking, Rare Earth Metals are divided into three types, namely light rare earth metals (La, Ce, Pr, Nd, Pm, Sm, Eu), medium rare earth metals (Gd, Tb, Dy, Ho), and heavy rare earth metals (Er, Tu, Yb, Lu) (Idrus, 2021; Sulista, 2019). Rare earth metal elements are rarely found in pure metal form, but are more often found in the form of minerals such as carbonates, phosphates, and halides. To obtain rare earth metal elements in pure form, a gradual separation and purification process is required (Musyaffa et al., 2024).

In an effort to utilize natural resources sustainably, Indonesia is currently committed to reducing the negative impact of industrial activities on the environment by applying the concept of sustainable development. One approach that is increasingly being applied is zero waste, which aims to minimize waste through the reduction, recycling and reuse of materials that still have value (Zhou et al., 2023). In this case, it can be done by reprocessing the tailings so that they can be utilized optimally, including in extracting the rare earth metals that are still contained in them.

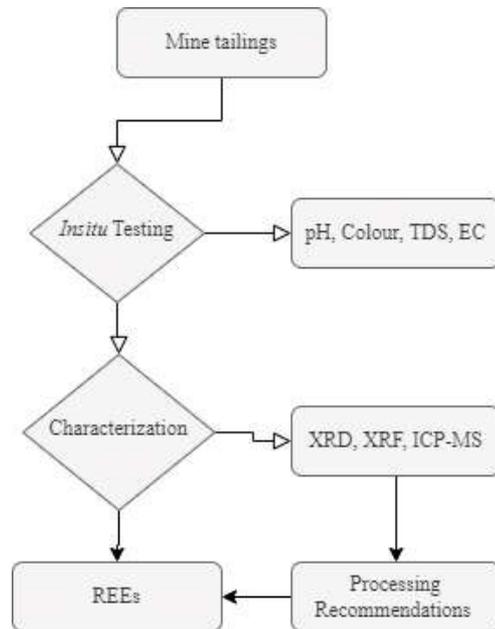
The area of Dunggilata Village, in Bulawa Sub-district, Bone Bolango Regency, Gorontalo, has enormous gold mineral potential, with gold mining activities carried out by the local community since 1991. The gold processing process produces tailings that contain not only gold but also other minerals, including rare earth metals, which are very abundant but have never been utilized as a potential source. Therefore, further research is needed to identify through characterization of tailings from artisanal gold mining in Dunggilata to determine the mineral composition, especially the content of rare earth metals. With a clear understanding of the characteristics of this waste, the most effective treatment process to recover rare earth metals from tailings can be recommended. These treatment process recommendations are expected to not only lead to the utilization of valuable rare earth metals, but also as an innovative step in supporting the zero waste concept, with significant economic impact and contribution to environmental restoration.

**Method**

*Materials and Instruments*

Tailings sampling was conducted randomly from the settling ponds. In situ testing in the form of color, temperature, pH, TDS and electrical conductivity tests were carried out directly in the field to obtain real-time

and accurate data. Furthermore, the samples were characterized using XRF (EDX-720), XRD (PANanalytical Empyrean) and then analyzed to determine the concentration of Rare earth Element using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (SUPEC 7000) conducted at PT Indo Mineral Research and PT Geoservicesdefined.



**Figure 1.** Mine tailings processing flowchart

*Sample Collection and Preparation*

The sample preparation process was carried out by drying in an oven at 105°C until dry conditions were achieved, then grinding until smooth using a mortar and pestle. Next, the milled samples were sieved with a standard mesh sieve to obtain particles of uniform size.



**Figure 2.** (a) Tailings in the settling pond; (b) Dry tailings

*Tailings Sample Characterization Technique*

*X-Ray Diffraction (XRD)*

This analysis was carried out to determine the crystalline phase present in the sample, thus providing information on its chemical composition through examination of its crystal structure (Yaseen & Scholz, 2017; Dordevic, 2019). Phase discrimination is achieved by comparing the acquired data with the information stored in the reference database (Ali et al., 2022).

*X-Ray Fluorescence (XRF)*

This analysis was conducted to determine the elemental composition of the tailings. XRF analysis provides information on the chemical elements contained in the samples so that the potential environmental impact and economic value of the tailings can be assessed. This continuous analysis is very important to determine the characteristics of tailings so that good mineral resource management can be determined from the results of the analysis obtained (Idrus, 2021; Sulista, 2019).

*Chemical Composition*

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is a highly effective analytical technique for determining the elemental composition of tailings, which are the waste products of mineral extraction processes. The technique has high sensitivity, allowing the detection of elements at very low concentrations, even down to parts per trillion (ppt) (Bilqis et al., 2022). In tailings samples, hazardous constituents such as arsenic (As), lead (Pb) and cadmium (Cd) are often found by this analysis, so the use of ICP-MS is essential for monitoring and evaluating potential environmental and health risks (Mpanza et al., 2020).

**Result and Discussion**

Tailings from several settling ponds were tested in situ to determine the physical characteristics of the samples. Tailings testing data is shown in Table 1.

**Table 1.** Physical Characteristics of Tailings

Settling Pond	pH	TDS	EC T(°C)	Color	Tailings Texture
I	8.96	233	4.64 31.9	Grey	Fine Grain
II	9.59	130	2.77 29.00	Green	Fine Grain
III	8.81	123	2.47 26.40	Brown	Fine Grain
IV	8.11	151	3.01 27.50	Brown	Fine Grain
V	8.60	133	2.67 31.30	Brown	Fine Grain
VI	8.94	158	3.17 28.30	Brown	Fine Grain

Based on Table 1, the pH of the samples ranged from 8.11 to 9.59, indicating that the tailings are alkaline, which can affect the solubility and mobility of harmful elements in the environment (Soomro et al., 2022). TDS varied from 123 - 233 mg/L, with the highest value in pond I, indicating the potential for pollution if not properly managed (Parvin & Tareq, 2021). EC ranged from 2.47-4.64 mS/cm, indicating the presence of dissolved ion concentrations in the tailings (Edwin, 2022). Tailings temperature varies from 26.4°C to 31.9°C, which can affect chemical and biogeochemical reactions in the pond (Leong et al., 2018). The color of the tailings varies from ash to brown, as well as the fine texture,

indicating the potential to disperse in water, increasing the risk of further pollution (Grande et al., 2016).

Based on the physical characteristics of the tailings above, samples from Pond I were selected for further analysis using XRD, XRF, and ICP-MS. Pond I has a pH of 8.96; TDS of 233 mg/L, and EC of 4.64 mS/cm, indicating a potentially high concentration of dissolved ions, as well as an ash color that may provide relevant mineralogical information (Dordevic, 2019).

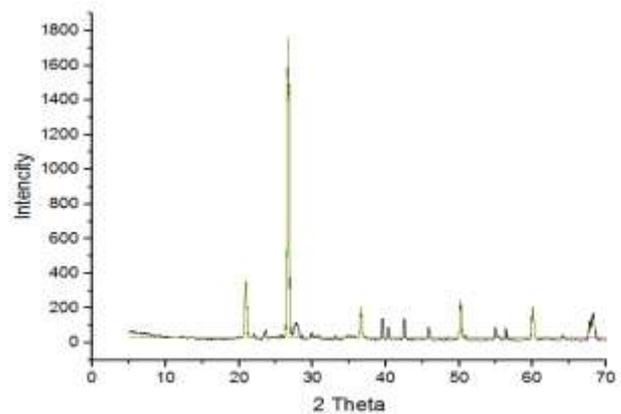
*Tailings Characteristics*

*XRD Analysis*

The results of the analysis are presented in Table 2.

**Table 2.** XRD Analysis Results of Tailings Samples

Sample	Crystal size (nm)	Total (%)	Phase	Crystal form
Tailing	26.43	88	quartz	trigonal
	26.18	11.20	albite	triclinic



**Figure 3.** XRD diffraction pattern of tailings sample

X-ray Diffraction (XRD) analysis of the tailings samples in table 2 shows that the main mineral composition consists of quartz (SiO<sub>2</sub>) with a crystal size of 26.43 nm which dominates as much as 88% of the total composition, as well as albite (NaAlSi<sub>3</sub>O<sub>8</sub>) which has a crystal size of 26.18 nm and contributes 11.2%. Quartz, which has a trigonal crystal structure, is a common mineral found in tailings and can provide information about the geochemical processes that occur during mineral processing (Yaseen & Scholz, 2017). Meanwhile, albite, which has a triclinic crystal structure, shows the presence of manganese and iron elements (Ali et al., 2022). The 13% of the mineral composition that was not identified indicates the presence of mineral phases that may be more complex or in such small quantities that they were not detected by this analysis.

Quantum dots (QDs) with small diameters, the XRD peaks will tend to be wider and the intensity decreases as the QD size decreases, indicating that small size may indicate lower crystal quality (Kim et al., 2020). This is in line with the findings of Alam et al., who stated

that the smaller the particle size, the wider the width of the resulting XRD peak, indicating that smaller crystal sizes are associated with lower crystal quality (Alam et al., 2011). Thus it can be concluded that SiO<sub>2</sub> with larger crystal size tends to show better crystal quality and higher stability compared to Manganoneptunite having smaller crystal size, which can have more defects and lower quality.

*XRF Analysis*

The data obtained provides information on the main oxide compounds and the elemental concentrations contained therein. The results of the analysis are presented in Table 3 and Table 4.

**Table 3.** Tailings Characterization Data Using XRF

Tailings Sample	
Compound	% Weight
SiO <sub>2</sub>	69.294
Al <sub>2</sub> O <sub>3</sub>	20.20
Fe <sub>2</sub> O <sub>3</sub>	4.681
K <sub>2</sub> O	2.351
SO <sub>3</sub>	1.521
CaO	1.440
TiO <sub>2</sub>	0.268
MnO	0.092
BaO	0.081
SrO	0.019
HgO	0.019
MoO <sub>3</sub>	0.015
ZnO	0.008
ZrO <sub>2</sub>	0.008
Br	0.003

**Table 4.** Elemental Composition Based on the Results of XRF Analysis of Tailings

Tailings Sample	
Element	Conc (%)
Si	32.294
Al	5.347
Fe	1.638
K	1.951
S	0.60
Ca	1.028
Ti	0.161
Mn	0.071
Ba	0.072
Sr	0.016
Hg	0.017
Mo	0.01
Zn	0.803
Zr	0.006
Br	0.003

Based on X-Ray Fluorescence (XRF) analysis of the tailings samples (Table 3), the chemical composition varies, with the dominance of SiO<sub>2</sub> compounds reaching 69.294%, followed by Al<sub>2</sub>O<sub>3</sub> (20.20%) and Fe<sub>2</sub>O<sub>3</sub> (4.681%).

The high SiO<sub>2</sub> content indicates that the tailings are mostly composed of silicate minerals, which are commonly found in geological materials and can affect the physical properties and stability of the tailings. In addition, the presence of Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> suggests that alumina and iron oxides also contribute to the tailings composition, which could have implications for potential interactions with other elements in the environment (Giannoni et al., 2015). Other compounds such as K<sub>2</sub>O (2.351%) and SO<sub>3</sub> (1.521%) indicate the presence of potassium and sulfur, which may play a role in biogeochemical processes and the potential for sulfuric acid formation under certain conditions (Vrdoljak et al., 2021). Although HgO and MoO<sub>3</sub> were detected in very small amounts, the presence of these compounds is still important to note as they may pose an environmental pollution risk (Christensen et al., 2011).

*Heavy Metal Analysis*

Heavy metal analysis of tailings samples was conducted using the ICP-MS method to identify the content of hazardous elements such as arsenic (As), cadmium (Cd), chromium (Cr), mercury (Hg), and lead (Pb). The results of the analysis can be seen in Table 5.

**Table 5.** Heavy Metal Analysis Results

Sample	As (ppm)	Cd (ppm)	Cr (ppm)	Hg (ppm)	Pb (ppm)	Hg (ppm)
Detection Limit	2	0.50	2	5	5	5
ST 2A	12	<0.50	15	0	8	0.276
ST 2B	100	2.40	6	107	59	> 5

Based on the Inductively Coupled Plasma Mass Spectrometry (ICP-MS) analysis of the tailings samples in Table 5, the ST 2A sample showed an arsenic (As) concentration of 12 ppm, which is above the detection limit, while cadmium (Cd) was not detected (<0.5 ppm) and chromium (Cr) was detected at 15 ppm. On the other hand, sample ST 2B showed much higher concentrations, with As reaching 100 ppm, Cd at 2.4 ppm, and lower Cr at 6 ppm. More strikingly, sample ST 2B contained mercury (Hg) at 107 ppm and lead (Pb) at 59 ppm, with very high concentrations of Hg (>5 ppm), indicating the potential for serious pollution at the settling pond site (Kumari et al., 2024; Mazarakioti et al., 2022). High concentrations of heavy metals, especially As and Hg, may indicate negative impacts on local ecosystems and public health, requiring immediate attention and mitigation measures to reduce the risk of further pollution (Garcia et al., 2022; Lai et al., 2021).

### Rare Earth Element Analysis

Table 6 provides an overview of the presence and distribution of rare earth element in samples from 2 different locations.

**Table 6.** Rare Earth Elements Analysis Results

REEs	ST01 (ppm)	ST02 (ppm)
Ce	14.253	14.578
Dy	0.827	0.845
Er	0.400	0.399
Eu	1.484	0.752
Gd	0.575	0.534
Ho	0.179	0.224
La	7.257	7.850
Lu	0.062	0.056
Nd	7.209	6.897
Pr	1.894	1.768
Sc	< 0.001	< 0.001
Sm	1.413	1.422
Tb	0.097	0.108
Tm	0.068	0.098
Y	5.388	4.970
Yb	0.457	0.392

Analysis of rare earth element using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) on the samples showed varying concentrations for each element. Sample ST 01 contained Cerium (Ce) at 14.253 ppm, supported by Neodymium (Nd) and Lanthanum (La) detected at 7.209 ppm and 7.257 ppm respectively, indicating the potential for a significant rare earth metal resource at the site. Meanwhile, sample ST 02 showed a slightly higher Ce concentration of 14,578 ppm, with La also increasing to 7,850 ppm, but Nd decreasing to 6,897 ppm. Other elements such as Dysprosium (Dy) and Europium (Eu) were detected at lower concentrations of 0.827 ppm and 0.752 ppm for ST 01 and ST 02, respectively.

Rare earth element have wide applications in modern technology due to their chemical, catalytic, electrical, magnetic, and optical properties (Hendriana et al., 2023). Rare earth Element, specifically Cerium (Ce), Neodymium (Nd), and Lanthanum (La), have a variety of important benefits in modern industry and technology; Cerium is used in automotive catalysts and as an abrasive material, Neodymium is used in powerful permanent magnets for electric motors and generators, while Lanthanum is used in the production of optical glass and as a catalyst in petroleum processes (Anggraini et al., 2019).

The analytical results obtained show that samples ST 01 and ST 02 contain significant amounts of Rare earth Element Cerium (Ce), Neodymium (Nd) and Lanthanum (La). Therefore, it is necessary to carry out good and effective treatment of tailings waste to minimize the potential for environmental pollution and

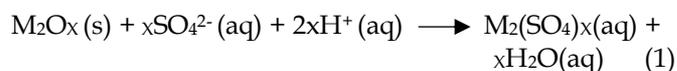
utilize the rare earth metal elements contained therein, as revealed in research on the kinetics of leaching rare earth metal elements from phosphate rock which shows that proper processing can increase the efficiency of recovering these elements (Zhili Li et al., 2021; Fedeli et al., 2024; Bobaker et al., 2019).

### Rare Earth Element Processing Process Recommendations

The characterization results of tailings waste show that Cerium (Ce) has the highest concentration among the rare earth elements found in tailings. Therefore, further processing is needed through the reuse of tailings waste to obtain Cerium optimally. The hydrometallurgical approach has proven to be a sustainable method, with low waste discharge, low emissions, efficient energy consumption, and economic and environmentally friendly value (Abadi et al., 2024). Most studies use chemical and/or bioleaching processes on secondary waste, followed by solvent extraction to separate clean rare earth elements (REE) (Zaiyu Li et al., 2024). Furthermore, rare earth compounds are recovered through a precipitation process. Solvent extraction (liquid-liquid extraction) is one of the critical stages in the overall process, because REEs (including light and heavy rare earths) show a low tendency to separate when extracted using various solvent reagents from a suitable aqueous medium (Jyothi et al., 2020).

### Bioleaching Stage

The processing of the rare earth metal cerium (Ce) begins with extraction by dissolving the tailings using hydrochloric acid (HCl) or sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) as solvents to release the rare earth metal (REM) (Hendriana et al., 2023). The separation process using dilute sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) aims to dissolve rare earth elements from insoluble minerals such as CeO<sub>2</sub> (Wang et al., 2023). Sulfur (S) is used as a reducing agent to convert insoluble CeO<sub>2</sub> into Ce<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> (Ghaly et al., 2024). This process improves the efficiency of cerium extraction from tailings (Zhou et al., 2021). The reaction that occurs in the process of dissolving sulfuric acid with the metals contained in the tailings is shown in the following equation:



(Hosseini et al., 2022)

### Precipitation Stage

The next step after separation is the precipitation of rare earth elements. Rare earth elements are precipitated in an acidic solution by adding oxalic acid. Oxalic acid is a precipitating agent that functions to precipitate rare earth metal elements from the solution. This process can involve multistage precipitation to increase separation

efficiency (Meilinda et al., 2021). The efficiency of oxalic acid precipitation reaches 98.68% (Wang et al., 2021). Furthermore, the oxalate precipitate is filtered and dried at a temperature of 80 °C for 12 hours. Furthermore, the dried product is calcined in a furnace at a temperature of 850°C for 120 minutes to produce rare earth oxides as the final product (Ricardo et al., 2023; Tilp et al., 2024).

#### Purification Stage

After precipitation, further separation steps are taken to increase the purity of the precipitated Rare Earth Elements. Techniques such as filtration, gradual precipitation, or the use of ion exchange resins can be applied to remove any remaining contaminants and ensure that the resulting metals meet the quality standards required for industrial applications (Fatimah et al., 2021; Novriyanisti et al., 2021). Solvent extraction is used to separate rare earth elements from other contaminants, while the use of ion exchange resins can also selectively bind rare earth elements (He et al., 2023). Both techniques are effectively used to purify rare earth elements. By following these steps, it is possible to effectively process Rare Earth Elements from tailings, utilize existing resources sustainably, and reduce the environmental impact resulting from mining and processing activities (Gholami et al., 2025).

#### Conclusion

The characterization method has been successfully applied to determine the mineral composition, chemical elements and the presence of Rare Earth Elements from tailings using XRD, XRF and ICP-MS techniques. XRD analysis shows that the tailings sample is dominated by quartz (SiO<sub>2</sub>) minerals at 88% and albite (NaAl<sub>3</sub>SiO<sub>8</sub>) at 11.2%. XRF analysis shows that the tailings contain chemical elements with a chemical composition of SiO<sub>2</sub> reaching 69.294%, Al<sub>2</sub>O<sub>3</sub> 20.20%, and Fe<sub>2</sub>O<sub>3</sub> 4.681%. Rare earth elements are found in tailings with cerium (Ce) content of 14.578 ppm, lanthanum (La) of 7.850 ppm, and neodymium (Nd) of 6.897 ppm.

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#### Author Contributions

Investigation, NN and Y.A; formal analysis, NN, V.D.A.S and Y.A; resources, NN and Y.A; data curation, NN, V.D.A.S and Y.A; writing—original draft preparation, NN, V.D.A.S and Y.A; writing—review and editing, NN, V.D.A.S and Y.A; visualization, NN, V.D.A.S and Y.A; supervision, NN, V.D.A.S and Y.A; project administration, NN; funding acquisition, NN, V.D.A.S and Y.A. All authors have read and agreed to the published version of the manuscript.

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#### Conflicts of Interest

There are no conflicts of interest among any of the authors.

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