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Rare Earth Elements (REE) Extraction from The Stones Hill at Padang Area by Precipitation

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Abstract: Rare Earth Elements (REE) are a very important element in modern life such as electronics, materials engineering, and even medicine. Gadolinium (Gd) is one of the REEs that is used as a basic material for implants as well as being used for other basic materials such as a corrosion inhibitor, control rods for nuclear reactors, and others. In this case, the elements are found along hills in the Padang area which have the potential to be produced in large quantities from six rock minerals such as granite, basalt, clay, silica, tuff, and limestone.). Based on the results of identification using the XRF spectrometer test tool, it was found that the six mineral rocks had an abundance of rare earth elements. The results of testing the extraction of Gadolinium Oxide (Gd2O3) using XRF were successfully carried out, Gd2O3 can be extracted by 0.06% - 1.84% in basalt rock, 0.47% - 0.95% in clay rock, and 0% - 0.88% in silica rock. This proves that the main rock minerals from stones have the Rare Earth Elements (REE) and can be extracted with using the Precipitation Extraction method.

Keywords: Basalt; Clay; Extraction; Gadolinium; Granite; Limestone; Rare Earth Metal (REM); Silica; Tuff; XRF Spectrometer.

Introduction

The use of rare-earth elements triggers the development of new material technologies (Klinger, 2018). The utilization of rare earth elements is needed in the large various fields from the electronics industry to the modern transportation industry and also the development of material processing technology, potentially in high technology industries (Filho et al., 2023). REE also plays a very important role in the needs of modern production materials such as in the world of superconductors, lasers, electronic optics, LED and iPad applications, glass, and ceramics (Henderson, 1984)(Aide, 2023). In the future, it is estimated that the use of rare earths will expand, especially single rare earth elements, such as neodymium, samarium, europium, gadolinium, and yttrium. (Randive et al., 2014; Aldila et al., 2024; Sapulete et al., 2023)

Rare earth Elements (REE) are found in complex compounds. The content of REE is found in granite because carbonatic deposits are also followed by the formation of granite rocks (Muecke et al., 1979; Humphris & E, 1984). Other mineral rocks such as basalt rock, tuff rock (tuffaceous mineral), silica mineral rock, limestone (limestone), and clay rock (clay) are also indicated to contain REE (Lee & Tanaka, 2021; Arsyad et al., 2022). The REE has found potential in Sumatera, which is part of Indonesia, especially in hilly areas. So far, the production of rare earth elements in Sumatra has been exploited in Bangka Belitung. And then the REEs are processed by extracting and refining because their chemical properties are similar and difficult to separate from each other (Syaeful et al., 2014; Tirono, 2022; Efendi et al., 2023). This process involves sophisticated chemical separation technologies (Anggara et al., 2018; Mulyono et al., 2023). Recently, the need for REE has increased, especially since that is used as degradable implant Material. Gd is one of the REEs which is used as a material to resist the corrosion rate of the alloys for an implant. Therefore, the exploitation of Gd had become a production target due to the need for implant materials (Susanti & Harjanto, 2019; Lu et al., 2022; Bian et al., 2018;

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Tong et al., 2022). Gadolonium is one element that has the potential to be used as an implant material. This element is combined with Mg which aims to slow down the corrosion of the Mg element. This element comes from rocks that are processed through filtering to get pure Gd (Rahayu et al., 2023)

Gadolinium

Gadolinium is found in several other minerals, including monazite and bastnasite (Kanazawa & Kamitani, 2006; Balaram, 2019). Gadolinium is a chemical element in the periodic table of elements having the symbol Gd and atomic number 64, which is a very strong paramagnetic element that is an important requirement for contrast compounds and has a very high absorption capacity of neutrons. Gadolinium is a rare earth element that significantly improves the mechanical properties and creeps resistance of magnesium through processing at high temperatures and also improves solid solution strengthening and precipitation hardening (Gao et al., 2009; Kong, 2017). Gadolinium can contribute to recrystallization (Mouhib et al., 2024). As a material for implants, a ductile material that is easily formed is required to produce a variety of components according to the damaged bone tissue. The addition of gadolinium to magnesium greatly affects grain size and texture (Susanti et al., 2020). Gadolinium can enhance the performance of iron, chromium, and related alloys, as well as increase resistance to oxidation at concentrations of <1% (Affi; et al., 2023; Susanti & Harjanto, 2019).

Mineral Rocks of the Hills at Padang Area

Along with the Hills Padang area has six main mineral rocks, namely granite, basalt, clay, silica, tuff, and limestone (Rahmi & Helendra, 2018). Rock Granite is a coarse-textured acid igneous rock Granite is part of the granitoid rock group and includes acid igneous rocks with characteristics composed of crystals of coarse size and phaneritic-porphyritic granularity. The S-type granite rock in the northern part of Bangka Island (Kabat Granite) is one type of granite rock that is rich in REM content (Zulfikar et al., 2020). Based on data from the Mining Bureau of PT. Semen Padang, granite rock contains a chemical composition of 72.6% SiO_2, 18.1% Al_2O_3, 1.3% CaO, and 0.6% MgO. Basalt is a volcanic igneous rock that generally has a smooth (aphanitic) and holocrystalline texture. Based on data from the Mining Bureau of PT. Semen Padang basalt rock contains 62.0% SiO_2, 19.9% Al_2O_3, 3.6% CaO, and 0.7% MgO. Clay rocks are also called clay stones. Clay minerals are a group of minerals that make up sedimentary rocks. Granitoid rocks contain completely weathered REM deposits and almost all REM is concentrated in the form of a clay layer on the weathered crust (Anshori, 2007).

Based on data from the Mining Bureau of PT. Semen Padang, clay rock contains a chemical composition of 54.17% SiO_2, 21.94% Al_2O_3, 0.26% CaO, and 0.51% MgO. Silica in nature comes from igneous rocks and metamorphic rocks which are destroyed by weathering processes, and undergo transportation and deposition. Based on data from the Mining Bureau of PT. Semen Padang, silica rock contains a chemical composition of 67.6% SiO_2, 19.0% Al_2O_3, 0.2% CaO, and 0.2% MgO. Tuff rock stone is divided into two types, namely white tuff and red tuff. Granitic instrumentations pierce the mapped tuffs. Mineralization in mineral-rich tuff deposits that carry REM Zr-Nb. Based on data from the Mining Bureau of PT. Semen Padang, tuff rock contains a chemical composition of 79.13% SiO_2, 6.49% Al_2O_3, 5.56% CaO, and 1.27% MgO. Limestone or limestone, also known as limestone, is an important ingredient in cement production. Limestone Karts mineral deposits have monazite mineral density. Based on the latest data from the Mining Bureau of PT. Semen Padang, limestone contains a chemical composition of 2.2% SiO_2, 0.5% Al_2O_3, 53.7% CaO, and 0.5% MgO.

REE Processing

Rare earth elements processing from rare earth carrier minerals can be carried out using extraction, such as H_2SO_4 solvent precipitation extraction, REM-Oxalate feed extraction, and n-hexane solvent extraction. REM processing can also be done through the beneficiation process of Rare Earth Metal (REM) carrier minerals(Thomas et al., 2024; Trisnawati et al., 2020; Talens-Peiró & Villalba Méndez, 2013).

REM Extraction

Extraction is the process of separating materials from the mixture using a suitable solvent. After the extraction process, the solvent is separated from the sample by filtration (Nascimento et al., 2020). Extraction in general is a process of separating the active substance from a solid or a liquid using a solvent (Zhang et al., 2018; Patel et al., 2019). The choice of solvent is needed in the extraction process because the ssolvent used must be able to separate or extract the desired substance without dissolving other unwanted substances (Dulanlebit & Hernani, 2023). The refining process of REE can be carried out in various ways, namely extraction, ion exchange resin, and precipitation.(El-Ouardi et al., 2023) The digestion process is carried out using concentrated H_2SO_4 which breaks down the structure of the sample and precipitates refractory metals such as iron, aluminum, copper, and chromium. Destruction results in the form of sediment and filtrate (Balaram & Subramanyam, 2022). The addition of

hydrogen peroxide to the digestive filtrate aims to optimize the solubility of REM. The filtrate is added with NH_4OH (ammonia) reagent. The commonly used precipitation reagents are NH_4OH and NaOH. The advantages of using NH_4OH are easy handling and cheaper price (Nadiatul & Zainal, 2017; Susanti et al., 2015; Zakiyuddin et al., 2020).

REE Beneficiation

The beneficiation process is also called the process of physically increasing the levels of oxide compounds (Rodliyah et al., 2021). The REE beneficiation process is an increase in the levels of REE oxide compounds in rare earth metal carrier minerals (Jordens et al., 2013). The shaking table is a material separation device by flowing thin water (Flowing Film Concentration) to the mineral carrying REE. On a rocking table equipped with a refile (barrier). Factors that can affect the performance of the Shaking Table are the size of the feed, Operational (roughing/cleaning), and the specific gravity differences. Mineral concentration carried out by gravity (Gravity Concentration) is the separation of minerals that depend on the difference in density (density), the shape and size of the density difference with the estimated mineral concentration criteria (Nadiatul & Zainal, 2017; Borisov, 2019).

Method

Materials and Equipment

The materials used in the research were granite, basalt, clay, silica, tuff, and limestone 120 mesh, concentrated H_2SO_4 (98%), 20% H_2SO_4, 25% NH_4OH, 3% H_2O_2, and Aquades (Suparno & Simamora, 2023). The type of chemical used is chemical Pro Analysis (PA). Samples and extraction results were tested using the PAN analytical Epsilon 3 Spectrometer XRF device. The tools used were the Pulverisette 6 Classic Line Fritsch Planetary Mono Mill Ball mill, Analytical scales, measuring cups, reflux flasks, heating mantles, 125 mm Whatman Ashless filter paper, Magnetic stirrer, oven, as well as research support tools such as drop pipettes, stirring rods, glass funnels, and others.

Collection and Sample Preparation

The Gd extraction process can be seen in Figure 1. Rock samples were taken at one of the Hills Padang areas. A sampling of granite, basalt, clay, silica, tuff, and limestone was carried out three times at different points in the Padang area mining area. After the sample is taken the sample is crushed into small gravel, then dried under the sun, after drying the sample is ground using a ball mill at a speed of 200 rpm for 15 minutes per sample and repeated until a fine sample is obtained which is sufficient for extraction. Extracted fine samples are filtered using a 120-mesh sieve until a fine 120 mesh sample is obtained which is ready to be tested at extraction stages.

Extraction (Separation) of REE

A total of 20 grams of 120 mesh fine rock powder samples from each rock mineral was put into a reflux flask, 40 ml of concentrated H_2SO_4 slowly added, and stirred until well blended. To the mixture, add 100 ml of Aquades and 5 ml of 20% H_2SO_4 slowly, stirring until evenly distributed and allowed to cool. After chilling, the mixture was heated to boiling (± 130°C-150°C), then allowed to cool in a fume hood, after cooling the solution was filtered using 125 mm Whatman Ashless paper. The results of filtering produce filtrate and sediment. The filtrate was added with 50 ml of 3% H_2O_2, and 70 ml of 25% NH_4OH, then stirred using a Magnetic Stirrer ± 30 minutes at a speed of 240 rpm. Furthermore, the filtrate is allowed to stand in a fume hood for ± 3 days until REE crystal oxide precipitate is formed. The precipitate was filtered using Whatman Ashless paper and dried in an oven at 100°C for 1 hour. The dry crystalline precipitate is allowed to stand and mashed. The final result is the fine REE oxide analyzed using an XRF spectrometer. (Dulanlebit & Hernani, 2023; S, 2022)

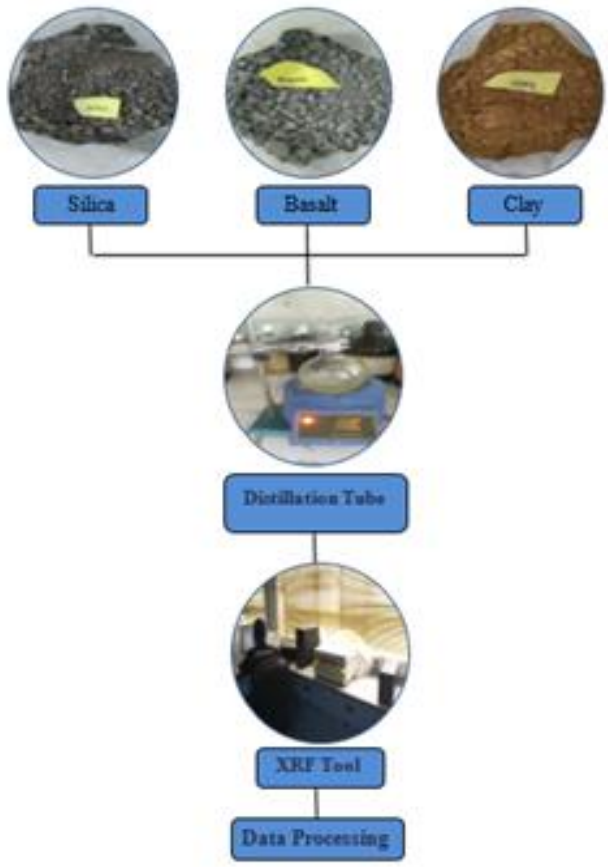

Figure 1. Processing of REE filter

Results and Discussion

The extraction method was carried out on the six rock mineral powders of Granite, Basalt, Clay, Silica, Tuff, and Limestone with a fine powder of 120 mesh. The extraction was carried out three times per rock mineral sample according to the sampling. Based on the results of the extraction of 20 grams of rock samples, it was found that the extraction method of deposition succeeded in forming REE oxide crystals on three mineral rocks, namely basalt, clay, and silica. Meanwhile, REE crystal oxide deposits in granite, tuff, and limestone rock minerals are not formed. Gadolinium has been successfully extracted from basalt, clay, and silica minerals. The extracted REM oxide deposits do not form in granite, tuff, and limestone minerals. Based on (Arianto et al., 2020)this can be caused by the unsuitable H2SO4 solvent so that it cannot digest REM minerals. Based on the extraction data, from 100% of the total rare earth in the final REE oxide deposition analyzed after separation or extraction, it can be seen that the percentage of Gadolinium increases compared to the initial identification data before extraction. The largest percentage of Gadolinium obtained was 1.835% in basalt rock mineral sample 2, while the smallest was found in sample 1 silica at 0%. On the other hand, the elements that are also effectively attracted are Terbium (Tb) and Europium (Eu). The XRF test results of the extracted REE crystal oxide precipitate can be seen in Table 1.

Table 1. The Extracted REE Precipitated Oxide

REM Oxides	Basalt			*Clay*			Silica		
	Sample (%)			Sample (%)			Sample (%)		
Y_2O_3	0	0.16	0.15	0.46	0.29	0.26	0	0.27	0.33
La_2O_3	0	0	0	0	0	0.48	0.99	0	0
Nd_2O_3	1.14	1.45	1.47	0	0	0	2.2	0.57	2.82
Eu_2O_3	0.97	0.73	0.73	0.78	0.49	0.51	1.39	0.69	1.55
Gd_2O_3	0.06	1.83	1.82	0.47	0.94	0.68	0	0.87	0.35
Tb_4O_7	97.3	95.81	95.8	97.66	98.21	9.98	94.32	97.44	94.86
Lu_2O_3	0.25	0	0	0.611	0	0	1.07	0	0

The precipitation extraction method using concentrated sulfuric acid solvent can extract the elements Yttrium (Y), Lanthanum (La), Neodymium (Nd), Europium (Eu), Gadolinium (Gd), Terbium (Tb), and Lutetium (Lu). Comparison of the extraction yield of Poboya gold tailings with mineral rock from one of the hills at Padang Area such as Karang Putih Mine of PT. Semen Padang can be seen in Table 2.

Table 2. Comparison of the Extraction Result of Precipitation Method

REM	Poboya Gold Tailings	Mineral Rocks Karang Putih Mining		
		Basalt	Clay	Silica
	Average (%)			Average (%)
La	13.49	0	0.16	0.33
Nd	0.02	1.44	0	1.86
Eu	4.84	0.81	0.59	1.21
Gd	0.02	1.24	0.69	0.41
Tb	78.98	9.630	9.79	95.54

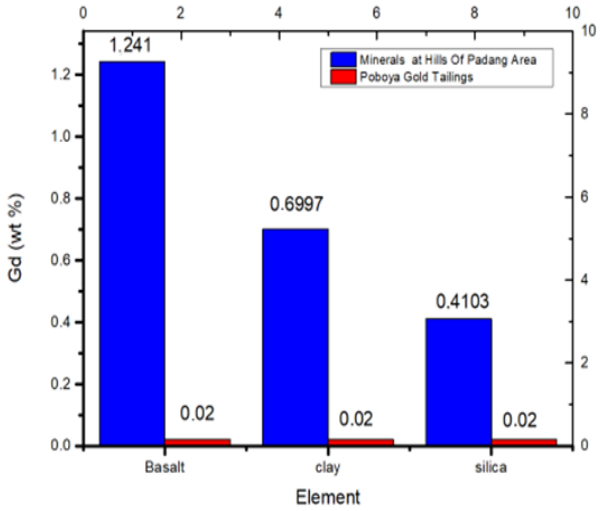

Figure 2. Gadolinium Extraction Result Diagram

Based on (Arianto et al., 2020) using the precipitation extraction method, the concentration of Gadolinium after extraction is 0.02% and Terbium can be extracted from 77.82% - 80.06%. Compared with the extraction results, the researchers obtained that the concentration of Gadolinium after extraction was 1.241% for basalt rock minerals, 0.69% for clay rock minerals, and 0.4103% for silica rock minerals, and Terbium (Tb), which is 94.33%. - 98.22%. Based on the results obtained by the researchers, this proves that the precipitation extraction method is more effective and suitable for basalt, clay, and silica rock minerals. On the other hand, the precipitation extraction method is also very effective for extracting the Terbium (Tb) element, which is 94.33% - 98.22% and the Europium (Eu) element is 0.49% - 1.55%. This proves that the precipitation extraction method with concentrated sulfuric acid solvent is suitable for basalt rock, clay, and silica minerals for the extraction of the elements Terbium (Tb), Europium (Eu),

and Gadolinium (Gd). A diagram of the results of Gadolinium extraction from basalt, clay, and silica can be seen in Figure 2.

Conclusion

Gadolinium was successfully extracted using precipitation extraction methods in basalt, clay, and silica minerals. The results of testing the extraction of Gadolinium Oxide (Gd2O3) using XRF were successfully carried out, Gd2O3 can be extracted by 0.06% - 1.84% in basalt rock, 0.47% - 0.95% in clay rock, and 0% - 0.88% in silica rock. This proves that the main rock minerals from stones have the Rare Earth Elements (REE) and can be extracted with using the Precipitation Extraction method.

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Author Contributions
Conceptualizing research ideas, O. S; research methodology, O. S, Z. A; data analysis, O. S; securing funding, O. S; writing original drafts, O. S; Z. A; management responsibilities, O. S; coordination for research planning and implementation of activities, O. S, Z. A.

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Conflicts of Interest
The authors declare no conflict of interest, financial or otherwise.

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