

Selective Precipitation of Neodymium oxide (Nd₂O₃) from Monazite

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Abstract—To extract Neodymium oxide (Nd₂O₃) from Myanmar Moemeik Myitsone heavy sand is the objective of this paper. Monazite concentrate can be separated from heavy sand by using mineral processing methods (sizing, high intensity magnetic separation, high tension separation and dry and wet tabling). Rare earth hydroxide is extracted from monazite by alkali digestion, hydrochloric acid leaching, filtration and selective precipitation by using ammonium hydroxide. After then, need to remove the cerium from rare earth hydroxide by oxidation and precipitation with KMnO₄ and Na₂CO₃. By calcination, the rare earth oxalate can get the rare earth oxide with free cerium. Finally, neodymium oxide is extracted from rare earth oxide by leaching with nitric acid, selective precipitation with ammonium hydroxide solution and calcination. The final result of the research, the Neodymium oxide (Nd₂O₃) was extracted 1.79% from the heavy sand and 10.57% from the monazite concentrate.

Keywords—extraction, rare earth, monazite, neodymium oxide, precipitation.

1) INTRODUCTION

Heavy sand is generally found in rivers and beaches. Heavy sand is the generic name for mineral sand with a higher specific gravity. Mineral content includes magnetite, ilmenite, monazite, zircon, rutile, titanite, dravite and garnet.

Monazite is a reddish-brown phosphate, containing rare earth metal and the important source of thorium, lanthanum and cerium. Monazite is discovered locally as heavy sand in Myitsone area; Moemeik Township, Homelain area; Sagaing Division, Thabeikyin Township and Kanbauk and Heinze area; Tanintharyi Division in the Myanmar.

The rare earth group contains 17 elements namely scandium, yttrium, and lanthanides (15 elements in the periodic table with atomic numbers 57 to 71). There are three groups of rare earth metals. They are Cerium group (LRE), Terbium group (MRE) and Yttrium group (HRE). In Cerium group, light rare earth metals are Lanthanum (La), Cerium (Ce), Praseodymium (Pr), Neodymium (Nd), Promethium (Pm) and Samarium(Sm). In Teribium group, medium rare earth metals are Europium (Eu), Gadolinium (Gd) and Terbium (Tb). In Yttrium group, heavy rare earth metals are Yttrium(Y), Dysprosium (Dy), Holmium (Ho), Erbium(Er), Thulium(Tm), Ytterbium (Yb) and lutetium (Lu).

95% Monazite was extracted from Myit Sone heavy sand by the methods of sizing, magnetic separation, electrostatic separation and tabling. After that, rare earth hydroxide was separated from monazite by methods of caustic soda digestion, hydrochloric dissolution and selective precipitation. And then rare earth oxide was precipitate from rare earth hydroxide with the leaching, oxidation,

precipitation and calcination method. By the method of nitric acid leaching and selective precipitation, neodymium oxide was separated from rare earth oxide.

Neodymium (atomic number 60, symbol Nd) is a soft and silvery rare earth metal with a bright, metallic luster. This metal is present in the ore minerals bastnasite and monazite and is not found unmixed with other lanthanides or in metallic form.

Neodymium is the second most abundant of the rare earth elements (after cerium) and is almost as abundant as copper. Neodymium was discovered by Baron Carl Auer von Welsbach, an Austrian chemist, in Vienna in 1885. The main areas are Brazil, China, USA, India, Sri Lanka and Australia. Reserves of neodymium are estimated to be 8 million tons; world production of neodymium oxide is about 7.000 tons a year.

Neodymium oxide is the chemical compound composed of neodymium and oxygen with the formula Nd₂O₃. The rare earth mixture didymium, previously to be an element, partially consists of neodymium oxide.

Neodymium has different compounds, including hydroxide, sulfides, halides, and carbide. They remain a popular additive to glass and are used in high power applications and in lasers which emit infrared light. The element itself is used in neodymium magnets for computer hard disks, in-ear headphones, professional loudspeakers, and microphones for which strong magnetic field and low magnet volume and mass are required. Neodymium magnets are stronger, lighter, and cheaper than samarium-cobalt magnets.

2) EXPERIMENTAL PROCEDURE

The main steps involved in the experimental work were,

- Separation of the monazite from heavy sand by magnetic separation and high tension separation.
- Extraction of the rare earth hydroxide from monazite.
- Separation of rare earth oxide from rare earth hydroxide.
- Precipitation of neodymium oxide from rare earth oxide.

Separation of the Monazite

By using the sieve shaker was selected the size of 65#, 100#, -100# heavy sand. And then magnetic elements (Ilmenite, Iron oxide) and non-magnetic elements (Monazite, Zircon, Rutile and Cassiterite) were separated from heavy sand by using magnetic separation 1.0Amp and 1.5Amp, after that magnetic elements (monazite 65-75%) and non-magnetic element(Zircon, Rutile and Tin) were separated from non- magnetic element(Monazite, Zircon, Rutile and Cassiterite) by using magnetic separation

(2.0Amp, 2.5Amp and 3.0 Amp). By high tension separation, the monazite (65-75%) was upgraded to monazite (95%). Figure 1 shows flow diagram for separation of monazite concentrate from heavy sand and table 1 shows mineralogy study of Myit Sone heavy sand.

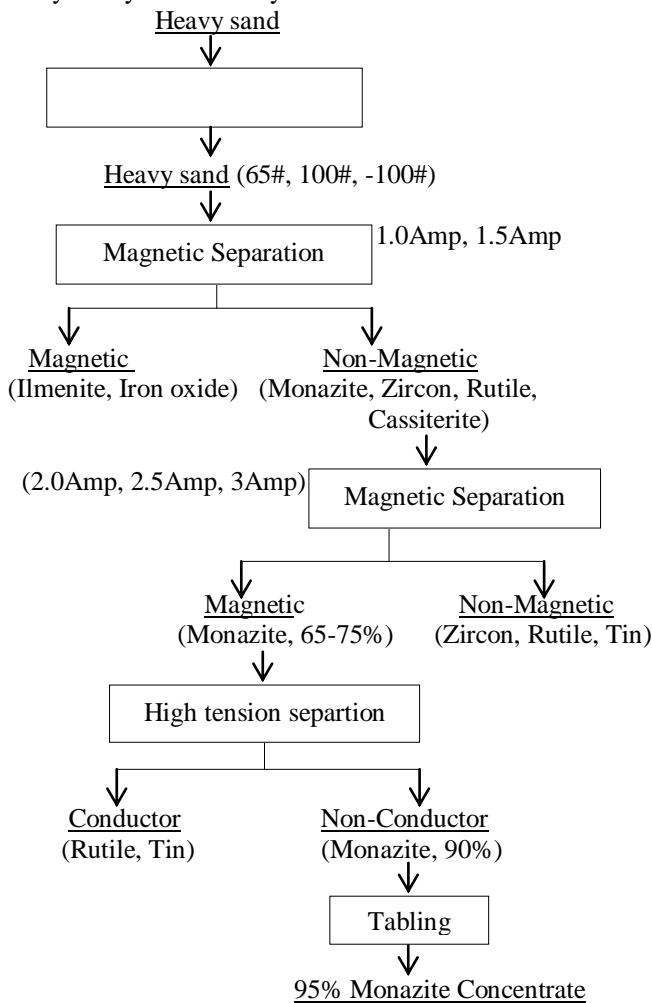


Figure.1. Flow diagram for separation of Monazite Concentrate from heavy sand

TABLE I

MINERALOGY STUDY OF MYIT SONE HEAVY SAND

No.	Name of mineral	Chemical formula	Content %
1.	Ilmenite	FeTiO ₃	42%
2.	Zircon	ZrSiO ₄	27.5%
3.	Monazite	(RE,Th)PO ₄	18.58%
4.	Rutile	TiO ₂	2.87%
5.	Cassiterite	SnO ₂	2.25%
6.	Light Mineral	Quartz, Feldspar, Mica	6.8%

Extraction of Rare Earth hydroxide

The following steps were included in the extraction of rare earth hydroxide.

- Caustic soda digestion.
- Hydrochloric acid Dissolution
- Dilution
- Selective precipitation

Caustic soda digestion

By using the ball mill reduce the size of 95% monazite to -325 mesh. Required amount of commercial caustic soda and

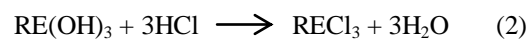
the small amount of distilled water were put into the conical flask and place on a sand bath. 100 gram of monazite was used for each experiment. When all the caustic soda dissolved monazite was slowly added to the solution and used the ratio 1:2.5 of monazite and caustic soda. Temperature 140°C and leaching time in three hours are used to heat and leach the mixture. By adding small amount of distilled water into the mixture controlled the temperature.

After heating and leaching, dilution step was continued at leaching temperature 105°C, for leaching time in one hour. After that, the mixture was filtrated and separated of the filtrate (NaOH, Na₃PO₄) and hydrous metal oxide cake (Th, U, RE). Then the metal oxide cake was washed with hot water until all the tri-sodium phosphate solution and free caustic soda had been removed from the metal oxide cake.



Hydrochloric Acid Dissolution

Hydrous metal oxide cake from caustic soda digestion was dried in the drying oven with the temperature 150°C and time in one hour. After drying, the ratio of 2:1 HCl (ml) and hydrous metal oxide cake (g) put into the 500 ml conical flask and place on the sand bed furnace with temperature 80 °C and time in one hour. In HCl dissolution process, uranium, thorium and rare earth hydroxide were dissolved in hydrochloric acid solution and undissolved impurities were left as residue. The reaction of hydrous metal cake and HCl may be represented as follow;



Dilution

After hydrochloric acid leaching, the slurry was diluted with hot water (volume 10 time of slurry) and dilution time was one hour. And then the filtrate and residue were separated by filtration. In the filtrate contain uranium, thorium and rare earth hydroxide.

Selective Precipitation

The Uranium, Thorium and rare earth hydroxide were selectively precipitated with 10% ammonium hydroxide solution at pH-5.8. Thorium, Uranium hydroxide were precipitated. Thorium, Uranium hydroxide and filtrate were separated by filtration. By pouring the excess amount of 10% ammonia into the filtrate, rare earth hydroxide was precipitate at pH-11 and filtrate was discarded. Extraction of rare earth hydroxide from monazite is shown in figure 2.

Separation of Rare Earth Oxide

100 gram of rare earth hydroxide was slowly mixed with 200ml of nitric acid and agitated in the beaker. And then leach in temperature and time are 90°C and 30 minutes by using the sand bed furnace. After 30 minutes of leaching time added distilled water up to 2 liters dilute and heating was continued. Cerium hydroxide was precipitated. The mixture was filtered with filtering bottle when cerium hydroxide precipitation was completed. In the filtration, cerium hydroxide and filtrate were separated. 16gm of potassium permanganate (KMnO₄) and 100 ml of H₂O were added to the filtrate for oxidation. And then 4 liters of water was added

to this solution for dilution. By adjusting the pH to 3.6 with sodium carbonate solution (Na₂CO₃) for cerium carbonate precipitation. After precipitation, the cerium carbonate as cake and rare earth solution was separated by filtration.

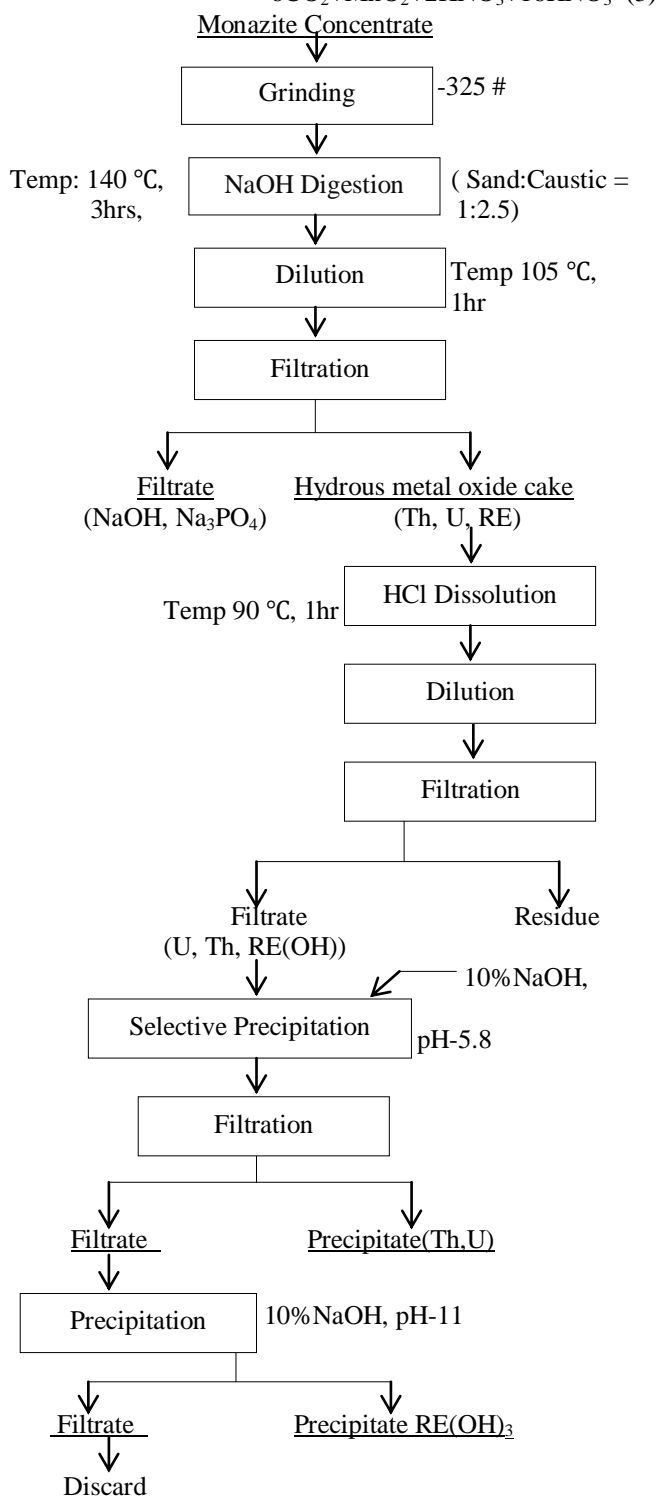
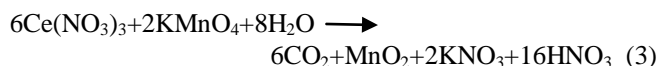


Figure.2. Extraction of rare earth hydroxide from monazite

After filtration 10% oxalic acid was added to the rare earth solution (filtrate) for precipitation of rare earth oxalate until free of particles. Cerium was removed as residue to obtain rare earth oxide at the temperature of 1000 °C and time in one hour by calcination of the rare earth oxalate. Flow diagram of separation of rare earth oxide is shown in Figure 3.

Selective Precipitation of Neodymium Oxide

After Cerium removing, Neodymium oxide can extract from rare earth oxide by leaching, dilution, selective precipitation and calcination. Firstly, 20ml of nitric acid in 1000 ml beaker was heated at 90 °C.

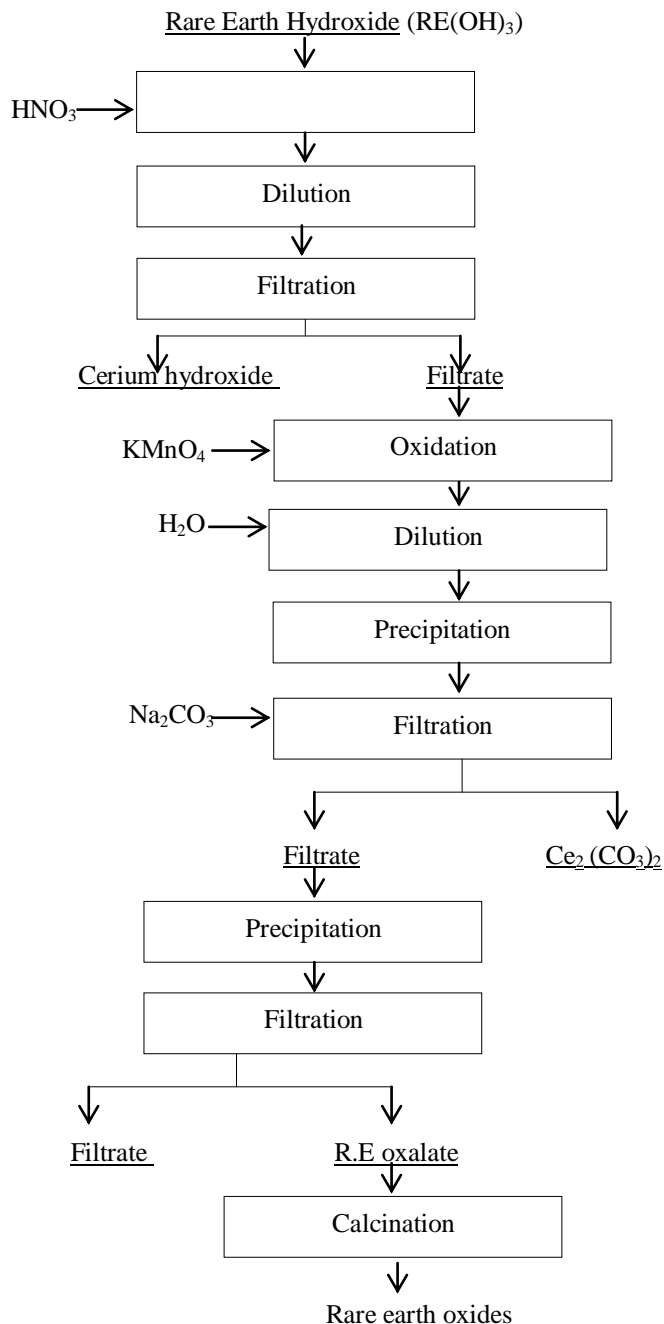


Figure.3. Flow diagram of the separation of rare earth oxide

To the nitric acid solution, 10gm of rare earth oxide was slowly added. The rare earth oxide was dissolve in nitric acid in 30 min. And then dilution was made up to 1000ml in 3ml beaker by adding the distilled water to the rare earth oxide solution. Heavy rare earth (HRE) was precipitated by first time adding 10% ammonia to the rare earth oxide solution at pH-5 and pH-6. Medium rare earth (MRE) was also precipitated by second time adding the 10% ammonia to the filtrate (1) at pH-6 to pH-6.9. Neodymium hydroxide and light rare earth (LRE) was separated by third time adding the 10% ammonia to the filtrate (2) with pH-7 to pH-8. Finally, by the calcination of neodymium hydroxide at temperature of 1000 °C and time in one hour was extracted the neodymium

oxide. Flow diagram of precipitation of the neodymium oxide is shown in Figure 4.

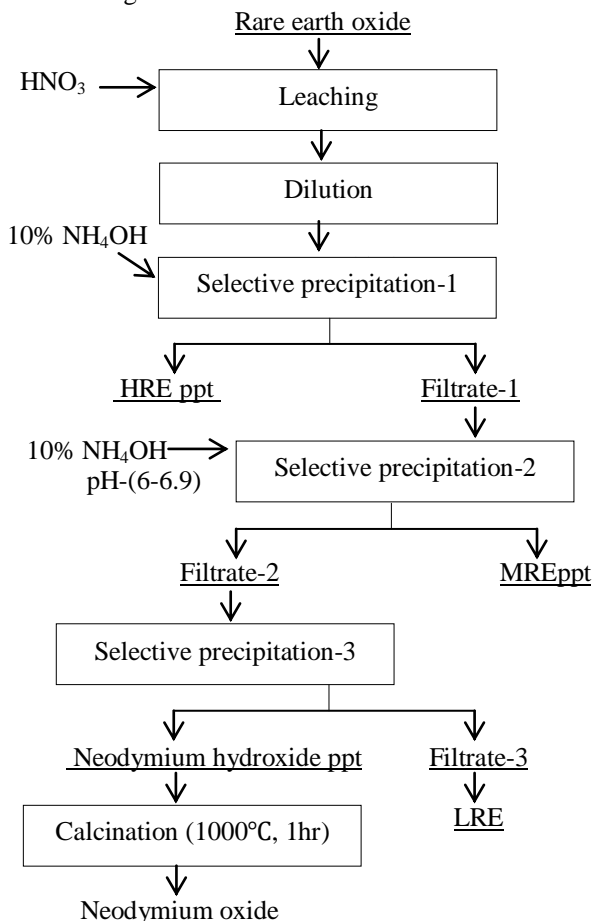


Figure4. Flow diagram for precipitation of Neodymium oxide

3) RESULTS

After separation of the minerals, the results were obtained as shown in table 2. It was observed that the maximum weight was obtained at pH-7.2. Rare earth was precipitated at pH value of 2.6, 2.8, 3.0 to 8.0. Between 2.6 and 6.5 were not precipitate and precipitation was started at pH-6.6. The weight of the neodymium hydroxide in different pH value at chloride condition is shown in table 3.

By studying the experimental data with the X-Ray Diffractometer(XRD) method, the maximum weight and intensity were obtained at pH value 7.8. The intensity value of neodymium oxide with the different of pH values are shown in table 4.

TABLE II
ANALYTICAL RESULTS OF MONAZITE FROM MOEMEIK MYIT SONE AREA

No.	Mineral	% in Heavy Sand	% in Monazite
1	La ₂ O ₃	2.97	12.5
2	Nd ₂ O ₃	1.79	10.57
3	Y ₂ O ₃	0.27	0.91
4	ThO ₂	2.23	9.55
5	Pr ₆ O ₁₁	1.19	1.52
6	Sm ₂ O ₃	1.09	1.28
7	CeO ₂	5.84	26.98
8	Others rare earth minerals	78.79	35.16

TABLE III
WEIGHT OF RARE EARE EARTH HYDROXIDE IN DIFFERENT pH VALUE

No.	pH	Weight of RE(OH)	Type of Rare of Oxide	Intensity
1	6.4	1.2	CeO ₂	-
2	6.6	2.3	Nd ₂ O ₃	860
3	7.0	3.3	Nd ₂ O ₃	1210
4	7.2	3.6	Nd ₂ O ₃	2860
5	7.4	0.8	Nd ₂ O ₃	860
6	7.6	3.0	Nd ₂ O ₃	-
7	7.8	0.78	La ₂ O ₃	-
8	8.0	1.3	La ₂ O ₃	-

TABLE IV
WEIGHT OF NEODYMIUM OXIDE IN DIFFERENT pH VALUES

Rare Earth (gm)	Nitric Acid(ml)	pH value	Nd(OH) ₃ gm	Intensity
100	200	7.4	1.5	1210
100	200	7.8	3.3	2440
100	200	8.2	3.2	2390

4) CONCLUSIONS

95% monazite concentrate was obtained from heavy sand by using magnetic separation, high tension separation machine and shaking table. Neodymium oxide was extracted from Moemeik Myitsone heavy sand, which contains about 1.79 % neodymium oxide and monazite contains 10.57% neodymium oxide. Monazite concentrate was digested with caustic soda. In caustic soda digestion process, monazite to caustic ratio of 1:2.5, digestion time is 3 hours and digestion temperature is 140°C were tested. Rare earth concentrate from monazite was contained with many rare earth elements. Firstly cerium was removed from rare earth concentrate. In Neodymium oxide precipitation test, HNO₃, 10% NH₄OH and vary pH values were selected. At the pH value, 5 to 6 rare earth elements were separated HRE. At the pH value, 6 to 6.9 rare earth element was separated MRE and LRE. Neodymium oxide was precipitated from LRE group at the pH value 7 to 8.

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