CHINA'S RARE EARTH ORE DEPOSITS AND BENEFICIATION TECHNIQUES

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Abstract

China possesses the largest resource of rare earth elements (REE) in the world and the reserve of REE in China takes up over 40% of the total global reserve. China has dominated the world REE production since 1980s. Bayan Obo REE-Nb-Fe deposit situated in Inner Mongolia is the world's largest known REE ore deposit with the reserve of 48 Mt REO and the largest REE production resource. Other two major in production REE deposits are Mianning REE deposit in Sichuan and Weishan REE deposit in Shandong. The weathered crust elution –deposited REE ore (ion adsorption REE deposit), as the world unique REE deposit found in China, is currently the second largest REE production resource containing high contents of heavy REE. In the paper, the ore properties of these REE deposits are introduced and the renovation and progress on the beneficiation techniques including flowsheets, flotation and leaching reagents and technological indexes on these ores are discussed.

Introduction

China has the largest resource of rare earth elements (REE) in the world. According to Mineral Commodity Summaries in 2001 the reserves and reserve bases of REE in the world in 2000 are shown in Table 1. It is indicated that the reserve of REE in China takes up over 40% of the world reserve. The newest Mineral Commodity Summaries 2014 shows the similar results.

The resources of rare earth elements (REE) in China are mainly divided into four types of deposits which consist of 95% total REE resources of China. They are the Bayan Obo REE−Fe−Nb Ore Deposit in Inner Mongolia, the Mianning REE Ore Deposit in Sichuan Province, the Weishan REE Ore Deposit in Shandong Province and the Weathered Crust Elution−deposited Rare Earth Ore (The Ion Adsorption Type Rare Earth Ore) mainly in Jianxi Province, but also distributed in Guandong, Fujian and Guanxi Provinces.

The Bayan Obo REE−Fe−Nb Ore Deposit

Mineralogy

The Bayan Obo REE-Nb-Fe deposit is situated in Inner Mongolia on the northern edge of the North China Kraton, 135 km northwest of Baotou (110°E, 41°47'N). It is a giant polymetallic rare earth element (REE)-Fe-Nb ore deposit of hydrothermal origin. It was first discovered as a Fe deposit by Prof. Ding Daoheng in 1927. The estimated total reserves of Fe (average grade Fe 35 wt%), Nb (average grade of 0.13 wt%) and REE (average grade REO 6 wt%) are 1.5 Bt, 1

Mt and 48 Mt. It is the world's largest known REE ore deposit. The Bayan Obo mining area is shown in Figure 1.

Table 1: the reserves and reserve bases of REE in the world in 2000 (REO, 10^4 t)

Country	reserve	Reserve base	%	Country	reserve	Reserve base	%
China	**4300**	**4800**	**43**	South Africa	39	40	0.4
Russia	1900	2100	19	Brazil	28	31	0.3
USA	1300	1400	13	Malaysia	3.0	3.5	0
Australia	520	580	5.2	Sri Lanka	1.2	1.3	0
India	110	130	1.1	Other	1704.8	1814.2	17
Canada	94	100	0.9	Total	10000	11000	100

The distributions of REE in the China's REE ore deposits are shown in Table 2. It is observed that high percentages of three main light rare earth elements La, Ce and Nd are found in Bayan Obo REE−Fe−Nb Ore Deposit, Sichuan Mianning REE Ore Deposit and Shandong Weishan REE Ore Deposit. However, in the Ion Adsorption Type Rare Earth Ore (especially type B) high contents of Y and other heavy REE are contained.

Table 2: the distributions of REE in the China's REE ore deposits

Content of REO	Bayan Obo REE−Fe−Nb Ore	Sichuan Mianning REE Ore	Shandong Weishan REE Ore	Ion Adsorption Type Rare Earth Ore	
				Type A	**Type B**
La_2O_3	24 – 26	28 – 30	32	31 – 40	2 – 5
CeO_2	50	45 – 50	48 – 50	3 – 7	1 – 2
Pr_6O_{11}	3 – 5	5	4	7 – 11	1 – 2
Nd_2O_3	16 – 18	12– 14	11 – 12	26 – 35	3 – 5
Sm_2O_3	1.5	1.5 – 2	1.0 – 1.8	4 – 6	2 – 4
Eu_2O_3	0.2	0.4	0.1 – 0.2	0.5	0.12
Gd_2O_3	0.4	0.8 – 1.0	0.3 – 0.5	4	6
$(Tb – Lu)_2O_3$	0.2 – 0.3	1	0.4 – 0.6	4 – 5	15 – 20
Y_2O_3	0.3	0.76	0.1 – 0.3	9 – 11	>60

The ore composition in the Bayan Obo is very complex, 71 elements and 170 minerals are found, one element could exist in several or more than ten different minerals, mineral symbiosis relationship is close and complicated, and dissemination size is fine. More than 90% rare earth elements in ore are existed in independent minerals, and about 4% ~ 7% of REE dispersed in iron minerals and fluorite. Total 15 kinds of rare earth minerals are found but the principal ones are bastnaesite [(Ce,La,Nd)(CO_3)F] and monazite [(Ce,La,Nd)PO4] with the ratio of 7:3 or 6:4, whereas magnetite and hematite are the dominant Fe-ore minerals. Meanwhile, several kinds of niobium minerals are contained in the ore such as columbite ($FeNb_2O_6$), aeschynite ($(Y,Ca,Fe)(Ti,Nb)_2(O,OH)_6$) and fersmite ($(Ca,Ce,Na)(Nb,Ta,Ti)_2(O,OH,F)_6$) etc. The main gangue minerals are quartz, apatite, dolomite and fluorite etc. These minerals consist of both primary and secondary metamorphic minerals of the host rocks and the epigenetic ore and gangue minerals introduced by hydrothermal solutions. The minerals of the Bayan Obo ore deposit are listed in Table 3.

Most of the Bayan Obo minerals, particularly in the banded ore, are very fine to extremely fine grained. Most Fe−REE−Nb minerals are closely associated with fine grain sizes. The grain sizes of REE minerals are in the range of 0.01 – 0.074 mm and the amount of less than 0.04 mm is accounted for 70% ~ 80%.

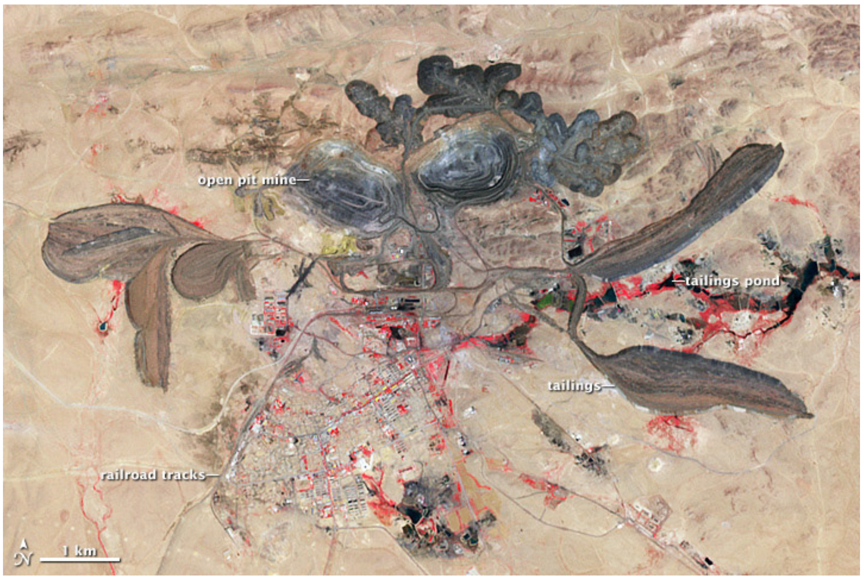

Figure 1: Satellite image of Bayan Obo mining area (modified from google)

Table 3: principal minerals of the Bayan Obo deposit

[REE's, rare earth elements]

Mineral category	Principal minerals
Host rock minerals	ferroan dolomite Sr- and Mn-calcite quartz illite biotite apatite albite
REE ore minerals	monazite, $(Ce,La,Nd)PO_4$ bastnaesite, $(Ce,La,Nd)(CO_3)F$ huanghoite, $Ba(Ce,La,Nd)(CO_3)_2F$ parisite, $(Ce,La,Nd)_2Ca(CO_3)_3F_2$ cebaite, $Ba_3Ce_2(CO_3)_5F_2$
Nb (± REE's) minerals.	columbite, $FeNb_2O_6$ aeschynite, $(Ce,Ca,Fe,Th)(Ti,Nb)_2(O,OH)_6$ fergusonite, $YNbO_4$ fersmite, $(Ca,Ce,Na)(Nb,Ti)_2(O,OH,F)_6$ pyrochlore, $(Ca,Na)_2Nb_2O_6(OH,F)$ Nb-rutile
Fe ore minerals	magnetite hematite martite goethite ilmenite
Other carbonate minerals.	benstonite, $(Ca,Sr)_6Ba_6Mg(CO_3)_{13}$ norsethite, $BaMg(CO_3)_2$ barytocalcite, $BaCa(CO_3)_2$ strontianite, $SrCO_3$
Common gangue and other minerals.	fluorite barite aegirine magnesio-arfvedsonite magnesio-riebeckite apatite phlogopite microcline albite bafertisite, $Ba(Fe,Mn)_2TiSi_2O_7(O,OH)_2$ cerianite, $(Ce,Th)O_2$
Sulfide gangue minerals.	pyrite pyrrhotite sphalerite galena chalcopyrite

Beneficiation techniques

Compared with domestic and abroad single bastnaesite ores, the Bayan Obo ores are much more difficult to upgrade due to its similar physical and chemical properties with iron minerals and gangue minerals associated closely, a lot of experimental studies by many research institutes in China on the rational exploitation of the Bayan Obo REE deposits had been conducted since the late 1950s and more than 20 beneficiation techniques have been reported. A significant progress on REE minerals processing had been made in the early 1990s and the mixed and separate REE concentrates with high-grade and high recovery were obtained from the iron ores.

The flowsheet of low intensity magnetic separation (LIMS) – high intensity magnetic separation (HIMS) – flotation (Figure 2) was considered the most successful one to be used in industrial plants which was developed by Changsha Metallurgical Research Institute in 1990.

As shown in Figure 2, after crushing the ore is ground to 90 – 95% passing 0.074 mm. By LIMS (rougher and cleaner) magnetite is recovered in the LIMS concentrate. The tailings of LIMS is processed through HIMS rougher at the magnetic field strength of 1.4T to recover another Fe mineral hematite and most REE minerals. And hematite and REE minerals contained in the HIMS rougher concentrate are separated by HIMS cleaner at the magnetic field strength of 0.6T. Combined LIMS and HIMS concentrates reported to the Fe reverse flotation to get the final Fe concentrate and the HIMS cleaner tailings containing most REE minerals is processed by flotation to get REE concentrates.

At the REE flotation circuit, the feed grade of REO is 9.78 −12% (HIMS cleaner tailings). The flotation is performed at low alkaline condition (pH9) and the flotation reagents contained naphthyl hydroxamic acid as the collector of REE minerals, sodium silicate as the depressant of silicates and J_{10} as the frother. The feed solids is 35-45% wt. One stage of rougher flotation, plus one scavenger flotation and two cleaner flotation stages produced an REO concentrate at a grade of 55% and a secondary REO concentrate at a grade of 34% with the combined recovery of 72−75%. However, these concentrates are the mixture of REE minerals mainly bastnaesite and monazite. The separation of bastnaesite and monazite has been reached by further flotation using phthalic acid or Benzoic acid as the collector of bastnaesite and alum as the depressant of monazite.

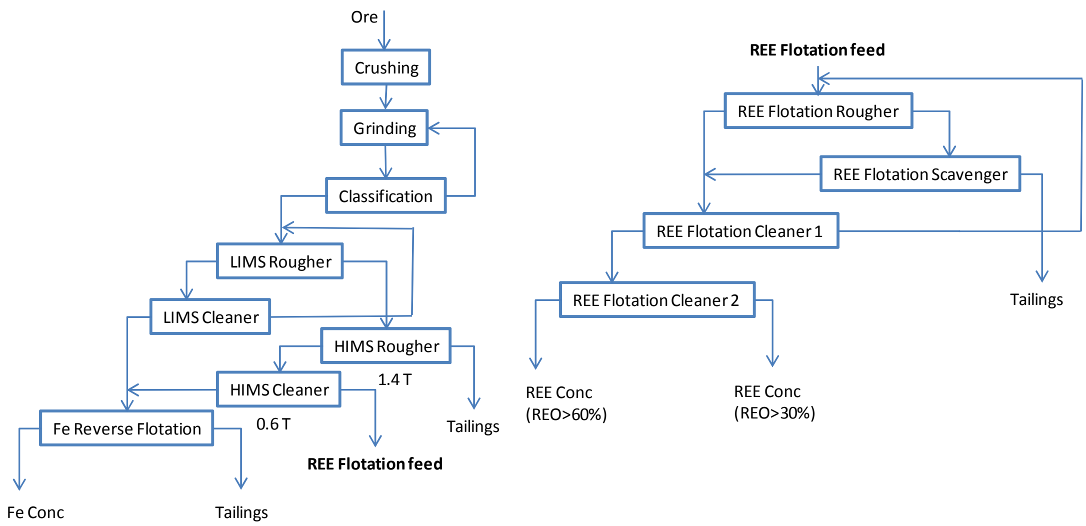

Figure 2: beneficiation flowsheet of the Bayan Obo deposit ore

Different flotation reagents had been tested. Only a rare earth concentrate with grade 15%~20% and the low recovery could be gotten by using fatty acids, such as oxidized paraffin and oleic acid, as the collector in rare earth mineral processing in earliest. C5 ~ 9 hydroxamic acid by saponification was primarily used for rare earth mineral flotation from 1970s and 1980s, then the cyclic alkyl hydroxamic acid synthesized by naphthenic acid was used as collector because the synthesis materials of fatty acids C5 ~ 9 in short supply. A miracle result of up to 60% REO flotation concentrate was obtained by processing previous concentrate of REO 25% ~ 30% from gravity separation concentrator when the major adjustments of the compatible depressants were made. In 1976, the plant test was succeeded, and the commercial production began in 1978. By application of the reagents system, the rare earth concentrate of REO> 60%, flotation recoveries of 60% to 65% were obtained. Since then, high-grade commercial rare earth concentrates were produced.

Because the selectivity of cyclic alkyl hydroxamic acid and alkyl hydroxamic acids was found poor the collector of H205, as the representative of aromatic hydroxamic acid, was successfully developed in 1986 by using naphthalene as raw material through sulfonated - hydrolysis - alkali fusion hydroxylation for preparation of intermediates naphthol, and then acylation reaction and condensation system with hydroxylamine (-NH-OH), to make naphthyl isobutyl group hydroxamic acid. The rare earth grade and recovery could significantly be improved by using new generation of rare earth mineral collector H205 with only water glass and the activator sodium fluoride in the flotation process was not needed. After the 1990s, a hydroxamic acid with dual activating group, the No.8 flotation oil, improved from H205, has been used in the Bayan Obo rare earth plant as a collector.

By the end of 2012, the Bayan Obo rare earth flotation concentrator had been developed into an annual output of 250,000 t of REE concentrates (contained 50% REO).

The Sichuan Mianning REE Deposit

Mineralogy

The Sichuan Mianning REE Ore Deposit was found between 1985–1986. It is an alkaline pegmatite carbonate type rare earth deposit. The industrial reserve is 1×10^6 t with the average grade of REO 3.7%. It is rich in light REE and heavy REE as well such as europium, yttrium etc. The chemical analysis results are shown in Table 4.

Bastnaesite is the main rare earth mineral and chevkinite and parisite are found in the ore. Other associated minerals are barite ($BaSO_4$), fluorite (CaF_2), iron and manganese minerals and small amount of galena. The ore is divided into granule and powder types. The granule ore has coarse grain size usually >1 mm. The grain size of bastnaesite is between 1 to 5 mm. The powder ore is the weathered product of original ore and takes up about 20% of total ore weight. The grade is about REO 3−7% and the grain size is 80% passing 325 mesh.

Table 4: Chemical analysis results of Mianning REE ore

Element	Wt %	Element	Wt %	Element	Wt %
REO	3.70	F	5.50	Na_2O	1.39
TFe	1.12	CaO	9.62	MnO	0.73
SiO_2	31.00	MgO	1.10	BaO	21.97
Al_2O_3	4.17	S	5.33	K_2O	1.31
FeO	0.43	P	0.24		

Beneficiation techniques

Three types of flowsheets are industrially used: the gravity concentration flowsheet, the magnetic – gravity concentration flowsheet and the gravity – flotation flowsheet.
The coexistence of massive and powdered black sludge, Fe-Mn weathered amorphous aggregates slime greatly influences the floatability of REE minerals.

Gravity separation

The ore was ground to 62% passing 200 mesh and hydro classified into the four size fractions. The shaking tables were used to process the fractions separately. Three different grade bastnaesite concentrates were obtained with the grades of 30%, 50% and 60%. The overall recovery was 75%.

Magnetic –gravity separation

After grinding the ore was concentrated by LIMS and HIMS separation and the magnetic concentrate with the grade of 5.64% was obtained. The recovery of magnetic circuit was 74.2% and yield 42%. And then the magnetic concentrate was classified into four size fractions and processed separately by shaking tables. The final concentrate with the grade of REO 52.3% was obtained. The overall recovery of RE was around 55%.

Gravity separation-flotation

As shown in Figure 3 the ore was primarily ground to 50% passing 200 mesh and hydro classified into four size fractions. The classified fractions were concentrated by the shaking tables separately. The grade of overall gravity concentrate was REO 30% with the RE recovery of 74.5%. The gravity concentrate was reground to 70% passing 200 mesh for flotation. C_{5-9} hydroximic acid (H205) and phthalate in the ratio of 1:1, sodium carbonate, sodium silicate were used as the flotation reagents at pH8-9. By one rougher, one cleaner and one scavenger flotation the concentrate with the grade of REO 50−60% was acquired at the RE recovery of 50−60%.

The Shandong Weishan REE Deposit

Mineralogy

The ore deposit was first found in 1958 and the exploration was finished in 1975. The reserve of RE was about 2.55×10^6 t and the average geological grade was 3.13%.
It is a quartz−barite−carbonate type RE ore deposit. The main RE minerals are bastnaesite and parisite and the main associated minerals are barite, calcite, quartz and fluorite etc. The grain sizes of RE minerals are coarse in the range of 0.04 −0.5 mm. The chemical compositions are shown in Table 5.

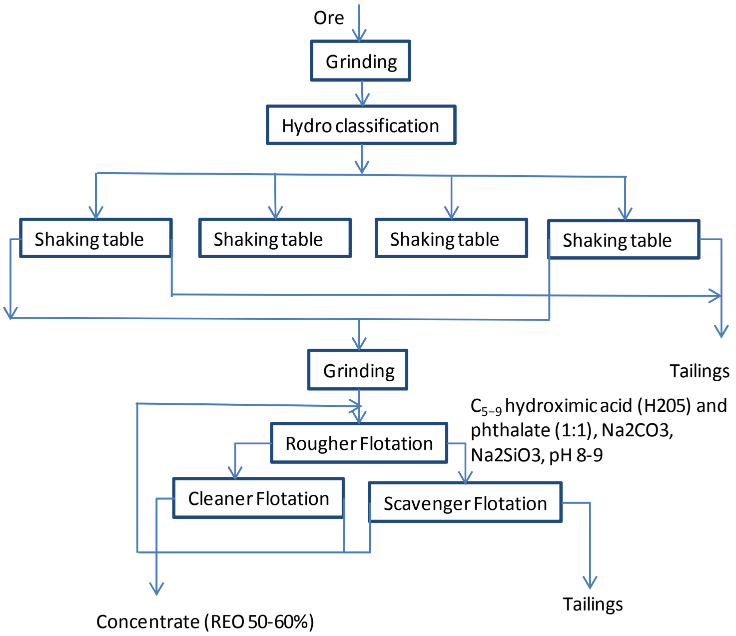

Figure 3: Gravity separation-flotation process at Mianning REE min

Table 5: Chemical compositions of Shandong Weishan REE Ore

Element	Wt %	Element	Wt %	Element	Wt %
REO	3.71	F	0.698	Na_2O	3.53
TFe	2.81	CaO	1.18	Th	0.002
SiO_2	47.92	MgO	1.18	BaO	11.99
Al_2O_3	22.48	S	2.1	K_2O	1.85

Beneficiation techniques

The Weishan REE flotation plant was built in 1982. The ore was ground to 65−75% passing 200 mesh and REE minerals were floated with once rougher, three scavengers and three cleaners. In 1980s oleic acid and kerosene were used as the collectors of RE minerals at acidic condition of pH5 using sulfuric acid. After 1991 with the ore grade dropped to 3-4%, a specific collector with formula $C_6H_4OHCONHOH$ was used with the addition of sodium silicate and frother L_{101}. The flotation was run at the weak alkaline condition of pH8−8.5. The RE concentrate with the grade of REO >60% at the recovery of 60−70% was obtained. Meanwhile, a secondary RE concentrate with the grade of REO 32% at the recovery of 10−15% was acquired. According to market demand, REE concentrate with the grade of 45~50% REO at the recovery of 80~85% has been produced and barite has also been recovered by flotation from REE flotation tailings.

The Weathered Crust Elution-deposited REE Ore (The Ion Adsorption REE Deposit)

Mineralogy

China's weathered crust elution−deposit rare earth ore or called ion adsorption rare earth ore is the unique REE deposit in the world. It was found in 1969 in Jianxi Province containing two types of this deposit, light REE type and heavy REE type, and was also found in other provinces Fujian, Hunan, Guandong and Guanxi. The known reserve of weathered crust elution−deposit rare earth ore in China is over 1×10^6t (REO) and the heavy RE reserve in China occupies over 80% of the world total heavy REE reserve. Now China produces about 1×10^4 t concentrate (REO >60%) from this ore annually.

The deposit was considered to form by many years weathering of granite and effusive rocks. It has the characteristics of shallow ore body coverage, soft ore and very fine grain size. The 80% −90% REE in the state of positive hydrated ions are adsorbed on the surface of clay minerals such as kaolinite, halloysite and illite. The ore is relatively low-grade, generally only 0.05% to 0.5% REO, with high heavy REE.

Extraction Techniques

Because the REE in the ore mainly occur in the state of hydrated ions adsorbing on the surface of clay minerals they can't be effectively concentrated by conventionally physical methods but extracted by the ion−exchange method. The adsorbed REE ions are not dissolved in water or ethyl alcohol but they are dissolved in the electrolytes solutions of NaCl, $(NH_4)_2SO_4$ and NH_4Cl by ion–exchange. The percentages of REE in different phases are shown in Table 6. It is indicated that over 80% REE are ion exchangeable.

Table 6: REE concentration in different phases (%, mass fraction)

Rare Earth Ore	Aqueous Soluble	Ion Exchangeable	Colloidal Sediment	Mineral
LN	6.15×10^{-3}	80.62	5.30	13.36
XF	8.14×10^{-3}	83.58	3.23	12.63
NH	1.47×10^{-3}	84.94	4.98	9.09

In 1970s as a major extraction method NaCl was used as the leaching solvent to dissolve REE by ion exchange. But high solvent concentration and long leaching time were required. And the solvent consumption and processing cost were high. Meanwhile, the grade of RE concentrate can only reach 70% REO. Since 1980s $(NH_4)_2SO_4$ has been used to replace NaCl as the solvent. A high grade (REO 92%) of RE concentrate has been achieved.

The process is usually run in a cement bath with the volume of about 10−20 m^3. When the bath is filled with the ore to the height of about 1.5 m the solvent of $(NH_4)_2SO_4$ solution with the concentration of 1−4% is poured through the ore for leaching. The pregnant solvent is obtained at the bottom of the bath. Oxalic acid is used as the precipitant to get mid product of oxalic acid−rare earths and further processed by burning to get final rare earths product (>REO 92%). Ammonium bicarbonate also has been used as a competitive precipitant. The flowsheet of the extraction technique is shown in Figure 4.

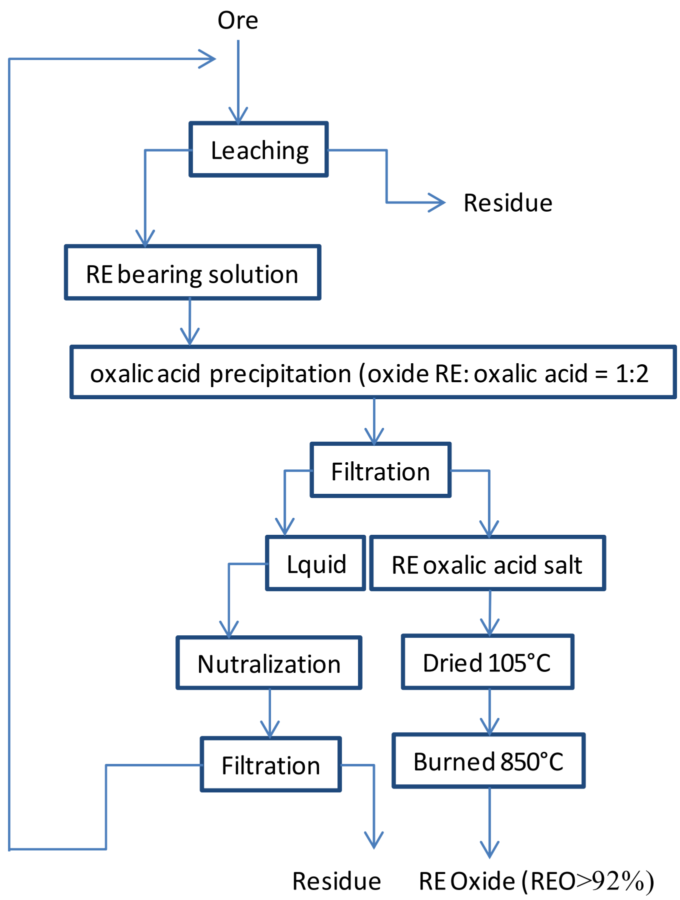

Figure 4: REE extraction technique for weathered crust elution–deposit REE ore

The in-situ leaching process also called the solution mining was studied from 1980s to resolve the ecological environmental problems in the exploitation by the bath leaching technology. The wells are drilled in the natural orebody and the leaching regent is injected. REE ions were selectively leached. Collected leachate is precipitated by the solution of oxalic acid or ammonium bicarbonate to obtain rare earth oxide products. The water is recycled. It was reported that two key technical problems must be solved during the in-situ leaching process. One is how to avoid the leaching reagent solution spreading around the wells to cause the environmental pollution and the other is how to recover the leachate so as optimize the leaching rate. The technology has been utilized in Wenfeng, Jiangxi province. More than 200 t of REE are extracted annually at the REE recovery of 70%.

Conclusions

China possesses the largest resource of rare earth elements (REE) in the world and has dominated the world REE production since 1980s.The resources of rare REE are mainly divided into four deposits which consist of 95% total REE resources of China. They are the Bayan Obo REE–Fe–Nb Ore Deposit, the Sichuan Mianning REE Ore Deposit, the Shandong Weishan REE Ore Deposit and the Weathered Crust Elution–deposited Rare Earth Ore.

The Bayan Obo REE-Nb-Fe deposit situated in Inner Mongolia on the northern edge of the North China is a giant polymetallic rare earth element (REE)-Fe-Nb ore deposit of hydrothermal origin. It is the world's largest known REE ore deposit with REO 48 Mt with average grade REO 6 wt%. The ore composition in the Bayan Obo was very complex and the principal REE minerals are bastnaesite and monazite. Most Fe−REE−Nb minerals are closely associated with fine grain sizes. The flowsheet of low intensity magnetic separation (LIMS) – high intensity magnetic separation (HIMS) – flotation was considered the most successful one to be used in industrial plants which was developed in 1990s.

The Sichuan Mianning REE Ore Deposit was found between 1985 – 1986. It is a alkaline pegmatite carbonate type rare earth deposit. The industrial reserve is 1×10^6 t with the average grade of REO 3.7%. Bastnaesite is the main rare earth mineral. Three processes have been used industrially, magnetic – gravity separation process, gravity separation process and gravity separation – flotation process.

The Shandong Weishan REE Deposit was first found in 1958 and the exploration was finished in 1975. The reserve of RE was about 2.55×106 t and the average geological grade of 3.13%. It is a quartz−barite−carbonate type RE ore deposit. The main RE minerals are bastnaesite and parasite. The flotation plant was built in1982.

Chinese weathered crust elution−deposit rare earth ore or called ion adsorption rare earth ore is the unique REE deposit in the world. The 80% −90% REE in the state of positive hydrated ions are adsorbed on the surface of clay minerals which can't be effectively concentrated by conventionally physical methods and usually are extracted by the ion−exchange method using NaCl, $(NH_4)_2SO_4$ or NH_4Cl as leaching solvents. The process is usually run in a cement bath and the in-situ leaching process has been also used.

References

1. U.S. Geological Survey, Mineral Commodity Summaries, February 2014.
2. Ruan Chi and Jun Tian, Comments on Weathered Crust Elution-deposited Rare Earth Ores, Journal of The Chinese Rare Earth Society, Vol.25, No.6, Dec.2007. p.641−649.
3. Ruan Chi and Jun Tian, Weathered Crust Elution-deposited Rare Earth Ores, Nova Science Publishers, Inc., New York 2008.
4. Yu Yongfu, Development on beneficiation techniques of Chinese REE deposit ores, West China Exploration Engineering, series No.63, Mar.2000. p.1−4.
5. Jörg Neßler, Giant Bayan Obo REE-Fe-Nb Ore Deposit of Inner Mongolia, China, January 2007
6. Jun Ren et al., Selective flotation of bastnaesite from monazite in rare earth concentrates using potassium alum as depressant, Int. J. Miner. Process. Vol.59, 2000. 237–245.
7. Cao Yongdan, Cao Zhao, Li Jie and Qu Qilong et al, Current Study Situation and Development on Flotation of rare earth in Baiyunebo Mine, *Mining Machinery*, Vol.41, No.1,2013, 93-96.
8. Xiao Shenming, Research on Method of Rare Earth Ore Processing, World Non-ferrous Metals, No.5, 2011, 48-49.
9. Li Yong, Zuo jicheng, Liu Yanhui, Application and Research of Hydroximic Acid as Collectors on Flotation of Rare Earth Ores, *Non-ferrous Mining and Metallurgy*, Vol.23, No.3, 2007, 30-33.
10. Luo Jiake, Ren Jun, Tang Fangqong, Zhou Gaoyun, Development on Flotation Reagents of Rare Earth Minerals in China, *Journal of the Chinese Rare Earth Society*, Vol.20, No.5, 2002, 385-391.
11. Yu Yongfu, Dressing Technology of REO Ore and Its Development in China, *Journal of China University of Mining & Technology*, Vol.30, No.6, 537-542.
12. Yu Yongfu, Zhou Chaoying, Advance in Baotou's Rare Earth Ore Beneficiation Technology, *Metal Mine*, Series No.281, 1999, 18-22.

13. Ci Ru-an, Wang Dianzuo, A new Process of Ion Type REO, *Non-ferrous Mine*, No.5, 1990, 47-51.
14. Personal Letters with Technical Staff of Baotou Iron and Steel Group Corporation, March 2014.