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Process mineralogy of Bayan Obo rare earth ore by MLA

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Abstract: The maximum recovery of rare earth resource from the Bayan Obo ore deposit is a difficult task, especially without the sufficient data of mineralogy. In this paper the mineralogy of Bayan Obo ore deposit by comprehensively research with the application of mineral liberation analyzer (MLA) is reported. The MLA was applied to quantitatively analyze the complicated element/mineral compositions, the REE occurrence, the size distribution and the degree of liberation of the Bayan Obo ore. Mineralogical analysis of the rare earth ore has shown that REEs are present mainly as bastnaesite and monazite-(Ce) to a small extent as parisite-(Ce). 5.85% of the REEs, 34.99% of iron and 0.12% of niobium occur in the ore sample. There are 76.99% of iron occurred in hematite and the remaining iron is mainly distributed in magnetite and goethite. The degree of liberation of bastnaesite and monazite-(Ce) was 79.65% and 75.67% respectively when the grinding fineness was 83.57% passing 75 μm sieves. Un-liberated or partly liberated rare earth minerals are associated closely mainly with other rare earth minerals and gangues. These theoretical data could be employed to further comprehensively utilize the rare earth ore.

Keywords: process mineralogy, rare earth, Bayan Obo ore, MLA

1. Introduction

The rare earth elements (REEs) are a group of the lanthanide elements, along with scandium and yttrium. REEs have distinctive physical and chemical properties, which make them strategically important for infrastructure, technology, and modern lifestyles (Mehmood, 2018). Known as "industrial vitamins", REEs are indispensable in optics, electricity, magnetism, nuclear radiation and other fields because even a low-dose of REEs can lead to an obvious improvement of performance of the matrix materials (Cardoso et al., 2019; Wang et al., 2019).

REEs can be classified into light, medium and heavy types according to the electron configuration of each rare earth element and the solubility of their sulfates (Chi & Wang, 2014). La, Ce, Pr and Nd belong to light REEs, while Sm, Eu, Gd, Tb and Dy are medium REEs and the rest of the elements Ho, Er, Tm, Yb, Lu and Y are classified as heavy REEs (Arrambide et al., 2019; Chen et al., 2018; Kovalenko et al., 2019). There are two main types of REEs ore; mineral-type ore and weathered crust elution-deposited type ore (Zhang et al., 2018). The weathered crust elution-deposited type rare earth ore mainly provides medium and heavy rare earth, and the light REEs occur mainly in mineral-type rare earth ore such as bastnaesite and monazite (He et al., 2017; Liu et al., 2019; Wu et al., 2019; Xu et al., 2019). Bastnaesite, monazite and xenotime are the main sources of the light REEs, which account for

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about 95% of the REE currently used (Abdou et al., 2019). The rare earth resources distribute unevenly in the world and are primarily concentrated in China, Brazil, Vietnam and Russia (U.S. Geological Survey, 2020). Although China accounts for only 23% share of the global reserve, currently supplies approximately 97% of global rare earth consumption, 85% of the rare earth supplied come from mineral-type rare earth ores. More than 70% shares of the light rare earth products are provided by the Bayan Obo deposit (Weng et al., 2015; J. Zhang, 2005; Zhou et al., 2019).

Bayan Obo, world-class reserves of rare-earth elements, situated in Inner Mongolia on the northern edge of the Paleoproterozoic-Archean North China craton, which is one of the oldest cratons on Earth (Wu et al., 2018). The deposit is hosted in dolomitic marble, which forms part of a sequence of Proterozoic metasedimentary rocks (the Bayan Obo Group) dominated by sandstones and slates. As the largest rare earth deposit in the world, it contains 71 elements and more than 170 minerals (L. Z. Li & Yang, 2016; Smith et al., 2016). For dozens of years, numerous researches have been engaged on ore genesis (Hu et al., 2019; Ren et al., 2019; K. Wang et al., 2019), occurrences of REEs (Zhang et al., 2015), reagents for beneficiation (Che et al., 2004; Li et al., 2018; Wang et al., 2013), mineral processing (Chen, 2014; Yu & Chen, 1992), tailing recovery (Yu et al., 2012) and so on. However, studies on process mineralogy of Bayan Obo rare earth ore are rarely reported, which restricts the efficient exploitation of the resource.

The MLA is a scanning electron microscope (SEM) equipped with energy dispersive X-ray (EDX) spectrometers, and computer software that automates microscope operation and data acquisition for automated mineralogy (Celep et al., 2019; Schulz et al., 2019). MLA System is designed to provide quantitative analysis of mineral samples. It is an automated measurement system for rapid and statistically reliable mineralogical measurements, which can scan more than tens of thousands of particles from one sample (Fandrich et al., 2007; Fu et al., 2019). Therefore, the MLA was employed to obtain valuable mineralogical information of the Bayan Obo rare earth ore, quantify a wide range of mineral characteristics (such as mineral composition, mode of occurrences of REEs, distributions of grain size, intergrowth and dissociation), and aimed to provide the precise theoretical data for further comprehensive utilization of the ores.

2. Materials and methods

The ore sample is from Bayan Obo Main orebody. The run-of-mine ore was crushed and ground to the mineral powders with a size smaller than 4 mm using cone crushers, roll crushers and wet ball milling machines (EMERSON, Model: S60AAW-6118) successively. Then 200 kg of ore was obtained by coning and quartering method, and sent to our laboratory. Representative rare earth ore samples obtained from the 200 kg of the ore by coning and quartering method were weighed for 12 sets, taken and placed in a ball mill for wet grinding. The ore grinding time was 15, 30, 45, 60, 75, 90, 105, 120, 135, 150, 165 and 180 s, respectively. Then the mineral powder was dried after grinding and 1.0 g was taken and mixed with epoxy resin and curing agent, respectively. Next, the mineral powder was added to a plastic mold for mixing, then heated followed by a 24 h standing time to be cured to get the MLA resin sample. The samples were polished, cleaned, dried, covered with carbon and analysed by MLA.

The Type-250 automatic MLA, equipped with back-scattered Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Analysis (EDXA) and MLA software, came from FEI Company in Australia. Automated SEM-based image analysis was carried out on FEI MLA 250 system. The instrument and image acquisition was controlled by the MLA software. The detailed introduction is available in the previous literature (Xu et al., 2019). The S60AAW-6118 grinder was purchased from LOUIS company in America. The METPOL 2V ore sample polisher was obtained from MetLab Canada Company.
Graphite (G67500), epoxy resin (C105-B), polishing solution (C40-6633), and curing agent (C205-B) are of analytical grade, purchased from Fisher Scientific Canada Company.

3. Results and discussion

3.1. Component of ore sample

The average values of multi-element analysis on the twelve ore samples are listed in Table 1. Bayan Obo Mine is a deposit containing various valuable elements. The light REEs of La, Ce, Pr and Nd with a total content of 5.85% was detected in these ore samples. The content of iron and niobium elements reaches up to 34.99% and 0.12%, respectively. The radioactive element thorium accounts for a proportion of 0.16% (see Table 1). Impurities are mainly Ca, Si and Ba, the content of which is 17.35% in total. It must be noted that great attention be paid in ore dressing process that impurities have a serious adverse effect on the grade and quality of iron and rare earth concentrates.

Table 1. Element analysis results of the ore by MLA (wt%)

Element	REE*	La	Ce	Pr	Nd	Nb	Th	Fe	Ca
Content	5.85	1.40	3.09	0.29	1.07	0.12	0.16	34.99	10.53
Element	Si	Ba	Mg	Mn	Al	Ti	K	Na	Pb
Content	4.25	2.57	0.80	0.49	0.38	0.23	0.27	0.21	0.06
Element	Zn	Sn	S	P	C	Cl	F	O	H
Content	0.02	0.01	1.79	0.75	1.48	0.12	5.94	28.89	0.05

*Means the REEs of La, Ce, Pr and Nd with a total content of 5.85%

Bayan Obo rare earth ore contains mainly rare earth mineral, hematite, fluoride, barite, apatite and ankerite, of which the rare earth minerals are bastnaesite and monazite-(Ce) as well as a little parisite-(Ce) and cebaite-(Ce). The contents of bastnaesite, monazite-(Ce) and parisite-(Ce) are 4.79%, 3.36% and 1.32%, respectively. The other valuable minerals are hematite (38.82%), magnetite (3.39%), fluorite (10.93%) and niobium-bearing minerals (such as columbite-(Fe), Aeschynite-(Ce), Ilmenorutile and pyrochlore). Detailed mineral compositions and contents are listed in Table 2.

Table 2. Minerals and contents in the ore by MLA (wt%)

Mineral	Bastnasite	Monazite-(Ce)	Parisite-(Ce)	Allanite-(La)	Aeschynite-(Ce)	Cebaite-(Ce)
Content	4.79	3.36	1.32	0.38	0.41	0.17
Mineral	Hematite	Magnetite	Goethite	Pyrite	Pyrrhotite	Ankerite
Content	38.82	3.39	1.99	1.70	0.71	5.74
Mineral	Fluorite	Barite	Quartz	Apatite	Riebeckite	Calcite
Content	10.93	4.25	4.02	3.29	3.27	2.92
Mineral	Biotite	Actinolite	Manganoan Calcite	Almandine	Albite	Ilmenite -(Mn)
Content	2.67	1.92	0.85	0.76	0.49	0.35
Mineral	Ferrosilite	Muscovite	Ilmenorutile	Andradite	Sanidine	Gonyerite
Content	0.22	0.16	0.14	0.13	0.12	0.12
Mineral	Kutnohorite	Diopside	Galena	Yangzhumingite	Columbite-(Fe)	Bafertisite
Content	0.10	0.10	0.07	0.07	0.07	0.05
Mineral	Grossular	Sphalerite-(Fe)	Kaolinite	Pyrochlore	Rhodonite	Cassiterite
Content	0.05	0.05	0.04	0.04	0.02	0.02
Mineral	Zircon	Sellaite	Baotite			
Content	0.01	0.01	0.01			

3.2. Distribution of main REEs and other valuable elements in the ore

In Bayan Obo rare earth ore, Ce and La occur mainly in bastnaesite, monazite-(Ce) and parisite-(Ce); Pr occurs mainly in bastnaesite; Nd occurs mainly in bastnaesite and monazite-(Ce). The specific distribution is demonstrated in Table 3. Most of the REEs could be recovered by flotation separation. However, a few REEs can hardly be recovered ascribing that they permute in isomorphism or scatter as fine rare earth mineral included in other minerals.

Iron occurs mainly in hematite, magnetite, goethite, riebeckite, ankerite and pyrite. Most iron elements occur in hematite and magnetite (see Table 4) and could be obtained through magnetic separation. Consequently, the iron-bearing minerals could be extracted from the Bayan Obo rare earth ore by magnetic separation, then both niobium-bearing minerals and REE-bearing minerals enriched in the tailings from the magnetic separation could be recovered by flotation separation (Yang et al., 2015).

The Nb element occurs mainly in columbite-(Fe), aeschynite-(Ce), ilmenorutile, pyrochlore and other minerals, and distributes uniformly in these minerals (see Table 5). However, these minerals have large differences in magnetic and flotation separation so that they could not be concentrated at the same time, resulting in the low recovery of niobium element. The niobium element was generally extracted with pyrogenic process from niobium-rich iron concentrates ascribing that the floatability of niobium minerals approximates to that of hematite and limonite.

Table 3. Distribution of REEs in the ore by MLA (wt%)

Element	La	Ce	Pr	Nd
Bastnasite	41.72	50.78	96.67	61.97
Monazite-(Ce)	34.49	31.40	n.d.	37.07
Parisite-(Ce)	22.00	12.28	n.d.	n.d.
Allanite-(La)	1.79	0.59	3.33	0.96
Aeschynite-(Ce)	n.d.	3.68	n.d.	n.d.
Cebaite-(Ce)	n.d.	1.26	n.d.	n.d.

Table 4. Distribution of Fe in the ore by MLA (wt%)

Mineral	Hematite	Magnetite	Goethite	Riebeckite	Ankerite	Pyrite	Pyrrhotite	other
Fe (%)	76.99	7.28	3.50	2.92	2.80	2.16	1.32	3.04

Table 5. Distribution of Nb in the ore by MLA (wt%)

Mineral	Columbite-(Fe)	Aeschynite-(Ce)	Ilmenorutile	Pyrochlore	Baotite
Nb (%)	28.93	28.15	22.82	19.38	0.73

3.3. Main mineral grain size distribution

When the grinding ore with size below 75 μm accounts for 19.86% share, the sizes of several main minerals are still large (see Fig. 1A and Fig. 1B). It can be seen from the curvilinear shape that the grains of monazite, bastnasite are fine, 80% of which distributes during 10-100 μm. However, the grain sizes of hematite and magnetite are larger than other minerals. Grains larger than 100 μm of hematite and magnetite account for 60% and 50% share, respectively. The sizes of the major minerals would become smaller when the grinding ore with size below 75 μm accounts for 83.57% share (see Fig. 2A and Fig. 2B). Except that the grains of hematite, fluorite, barite and ankerite are coarse, the sizes of the other minerals are smaller than 50 μm. Several rare earth minerals and iron minerals are easy to grind due to their less hardness. Dissemination sizes of rare earth ores in Bayan Obo deposit are so small that fine grinding is required to liberate them from gangue minerals to improve the recovery of rare earth mine-

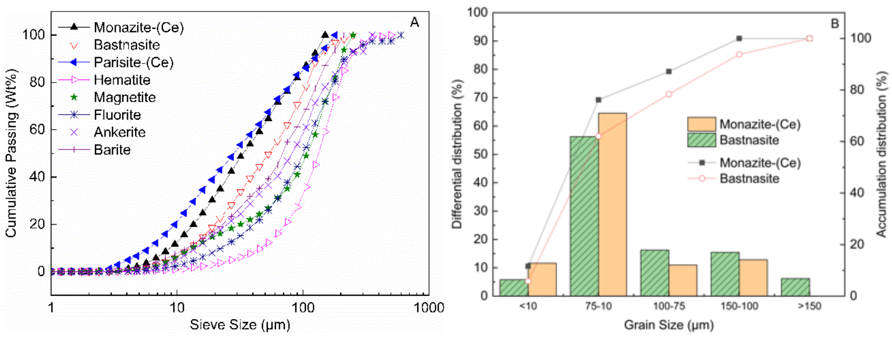

Fig. 1. Mineral grain size distribution with grinding fineness of 19.86% passing 75 μm
A: Main minerals, B: Main rare earth minerals

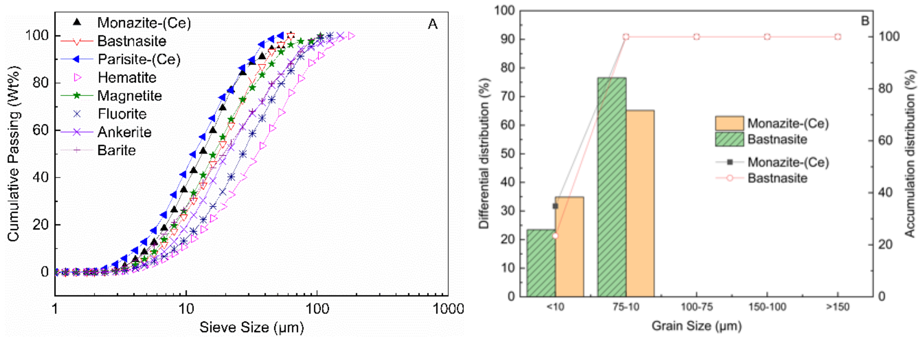

Fig. 2. Mineral grain size distribution with grinding fineness of 83.57% passing 75 μm
A: Main minerals, B: Main rare earth minerals

rals in flotation.

3.4. Mineral association

Bastnaesite associates mainly with rare earth minerals such as monazite-(Ce), parisite-(Ce), allanite-(La), and aeschynite-(Ce) and closely intergrows with hematite, fluorite, ankerite, calcite, apatite and quartz. For the embedding feature and intergrowth relation, see Table 6 and Figure 3. It can be inferred from Table 6 that the liberation degrees of monazite-(Ce) and bastnaesite are 79.65% and 75.67%, respectively when the grinding ore with size below 75 μm account for 83.57% share. Some un-liberated or partly liberated rare earth minerals accrete closely with other rare earth minerals and hematite, fluorite, ankerite, calcite, apatite, quartz, etc. There are a few monazite-(Ce) and bastnaesite scattered in other gangues, (such as albite, barite, pyrite and grossular), which is about 4%.

Monazite-(Ce) and bastnaesite occur in small lumps to disseminate in a large block of goethite (see Fig. 3A). Bastnaesite is embedded in large blocks of ankerite in dots and stripes (see Fig. 3B), hematite in small lump (see Fig. 3C), and fluorite and aeschynite-(Ce) in small lump (see Fig. 3D). Monazite-(Ce) is embedded in ankerite in lump and dots (see Fig. 3E). From the back scattered electron (BSE) imaging in Figure 3A-E, the accretion and package status of bastnaesite and monazite-(Ce) with the other rare earth minerals and the gangues could be known more intuitively, that could explain well why the Bayan Obo rare earth ores have hard dissociation and the low recovery and require fine grinding.

Table 6. Mineral association

Mineral	Liberated	Assosiation						
		Monazite-(Ce)	Bastnasite	Parisite-(Ce)	Allanite-(La)	Aeschynite-(Ce)	Cebaite-(Ce)	Hematite
Monazite-(Ce)	79.65	-	3.49	1.86	0.69	0.19	0.01	2.14
Bastnasite	75.67	3.61	-	3.77	0.83	0.35	0.01	3.15
Mineral	Goethite	Magnetite	Fluorite	Ankerite	Calcite	Apatite	Quartz	Riebeckite
Monazite-(Ce)	0.34	0.30	2.52	2.32	1.08	1.44	1.11	0.60
Bastnasite	0.32	0.40	3.23	2.32	1.30	1.65	0.74	0.44
Mineral	Barite	Biotite	Actinolite	Almandine	Andradite	Pyrrhotite	Ilmenite-(Mn)	Ilmenorutile
Monazite-(Ce)	0.26	0.49	0.48	0.11	0.11	0.05	0.16	0.06
Bastnasite	0.43	0.43	0.34	0.30	0.12	0.09	0.09	0.07
Mineral	Mangano an Calcite	Gonyerite	Grossular	Albite	Pyrochlore	Pyrite	Diopside	Others
Monazite-(Ce)	0.10	0.04	0.03	0.07	-	0.07	0.03	0.04
Bastnasite	0.06	0.06	0.04	0.05	0.03	0.02	-	0.09

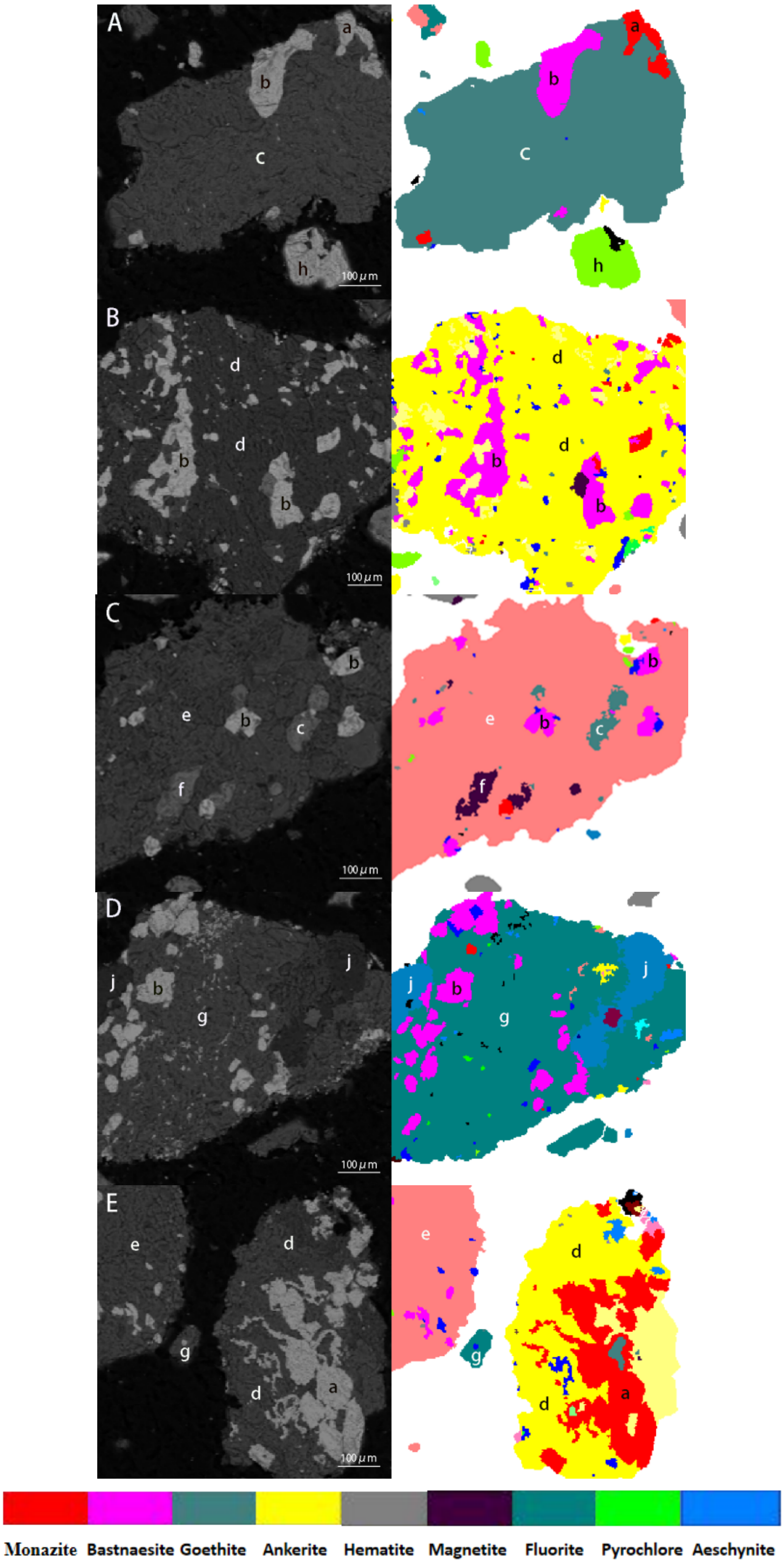

Fig. 3. BSE images and X-ray mapping with main minerals directed to false colors of Bayan Obo rare earth ores
The detailed descriptions of coexisting morphologies are listed as follows:
(A) Monazite-(Ce) and bastnaesite embeded in goethite; (B) Bastnaesite embeded in ankerite;
(C) Bastnaesite embeded in hematite; (D) Bastnaesite embeded in fluorite and aeschynite-(Ce);
(E) Monazite-(Ce) embeded in ankerite
Then, the labels from "a" to "j" stands for Monazite-(Ce) (a); Bastnaesite(b); Goethite(c); Ankerite(d); Hematite(e); Magnetite(f); Fluorite(g); Pyrochlore (h); Aeschynite-(Ce) (j)

3.5. Rare-earth mineral liberation by free surface

Mineral free surface area means the area percent of mineral not totally surrounded by other minerals in the particle, which was scanned pixel by pixel and calculated by MLA software according to the number of pixels and the area of a single pixel. The cumulative distribution of mineral in different free surface area ranges was plotted in Fig.4. With prolonging the grinding time, the mineral particles become smaller, and the liberation degree of the mineral increases gradually. The free surface area of bastnaesite increases markedly, that is, the liberation degree of monomer increases significantly with decreasing the particle size. When the grinding time is more than 105 seconds, about 70% of bastnaesite and monazite-(Ce) particles have dissociated with more than 80% of free surface area. Most iron, rare earth, and Nb minerals in Bayan Obo ore are closely associated with fine grain sizes, there are only 60% of bastnaesite and 65% of monazite-(Ce) liberated completely with 100% free surface area. Therefore, the ore was usually ground to 90–95% passing 74 μm in industrial production (Li & Yang, 2016).

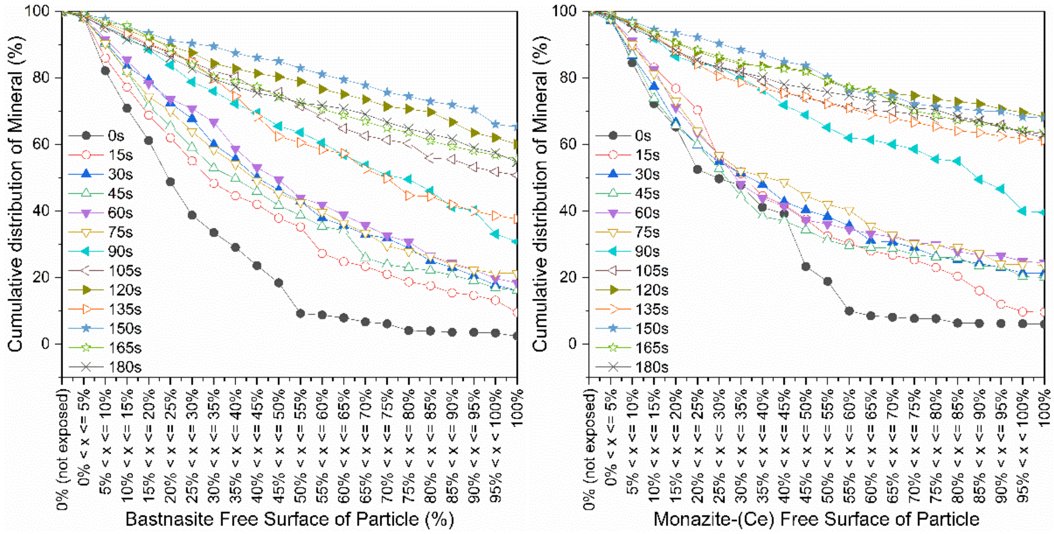

Fig. 4. Mineral liberation by free surface

4. Conclusions

Rare earth samples from Bayan Obo deposit consist mainly of La, Ce, Pr and Nd, with a total content of 5.85%. The samples bear a large amount of Fe, Nb and Th elements, which account for 34.99%, 0.12% and 0.16% respectively. The contents of three major impurity elements, Ca, Si and Ba, reach up to 17.35% in total. The contents of bastnaesite and monazite-(Ce) are 4.79% and 3.36% respectively. However, the content of valuable mineral hematite is 38.82%, and the Nb-bearing minerals also account for a large portion. Bastnaesite and monazite-(Ce) have small dissemination size, and possess complex embedding relationships with other rare earth minerals and gangues. When the grinding ore with size below 75 μm accounts for 83.57% share, the dissociation degrees of monomer from bastnaesite and monazite-(Ce) was 79.65% and 75.67%, respectively. Un-liberated or partly liberated rare earth minerals are associated closely with other rare earth minerals and gangues, which should be ground finely to achieve a high degree of liberation.

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