

Anomaly and geochemistry of rare earth elements and yttrium in the late Permian coal from the Moxinpo mine, Chongqing, southwestern China

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Abstract The rare earth elements and yttrium (REY) of the K2 coal from the Moxinpo mine, Chongqing, were determined using inductively coupled plasma mass spectrometry (ICP-MS). The results show that REY are enriched in the K2 coal, with the average content up to 462 $\mu\text{g/g}$, much higher than average values of most coals in the world. The REY distribution patterns indicate that the light REY is enriched and show a well-pronounced Eu minimum. The fractionation of individual light-REY is higher than that of the heavy-REY. The REY distribution through the K2 coal seam shows that the top and bottom portion of the coal seam have a lower content of REY than the middle portion. Goyazite and rhabdophane were identified with a scanning electron microscope equipped with an energy-dispersed X-ray spectrometer (SEM-EDX). The REY distributions through the coal seam, SEM-EDX data and the correlation analysis between ash yields and the concentrations have revealed that the REY mainly occurs in the organic matter. The K2 coal is a potential rare-metal resource due to its high REY contents, and the coal ash could be regarded as a new and promising raw material for recovery of REY as a by-product.

Keywords Rare earth elements and yttrium · Late Permian · Chongqing · Moxinpo mine

1 Introduction

Owing to their unique physical and chemical properties (Chi and Tian 2006), rare earth elements and yttrium (REY), play a key role in the fields of information, biology, new energy and material, space and ocean industry. REY consumption has shown an exponential growth in recent years and will continue to grow in the foreseeable future. China has the

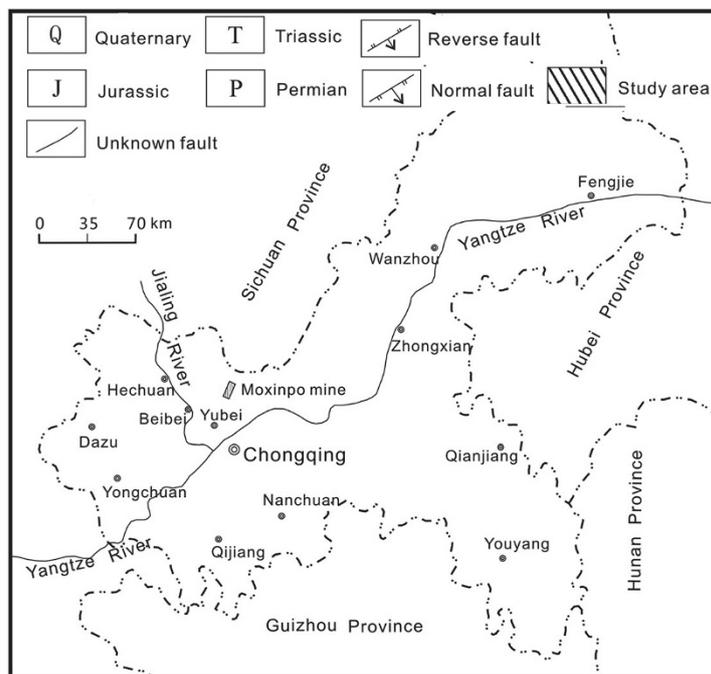
most REY reserves in the world, including the light rare earth elements (LREY) in North China and heavy rare earth elements (HREY) in South China. However, the conventional REY deposits consumption has increasingly growth with the economic development of China, so discovering new REY deposit types and improving the reserves of REY become increasingly important. The REY content in coal is usually low, but REY can concentrate in coal under some specific geologic conditions. Because of their stable chemical properties during coal combustion, such as higher fusion and boiling point, the REY may primarily accumulate more in the ash (Zhao 2002; Ren et al. 2006). There are some researches on the coal deposits being potential alternative sources and a promising raw material for extracting REY as by-product in recent years (Eskenazy 1987, 1999; Seredin 1996; Ren and Dai 2009; Dai et al. 2012a, 2013, 2014; Seredin and Dai 2012).

As REY in coal-bearing have some special geochemical properties including chemical stability and little influence from metamorphism, the REY distribution, modes of

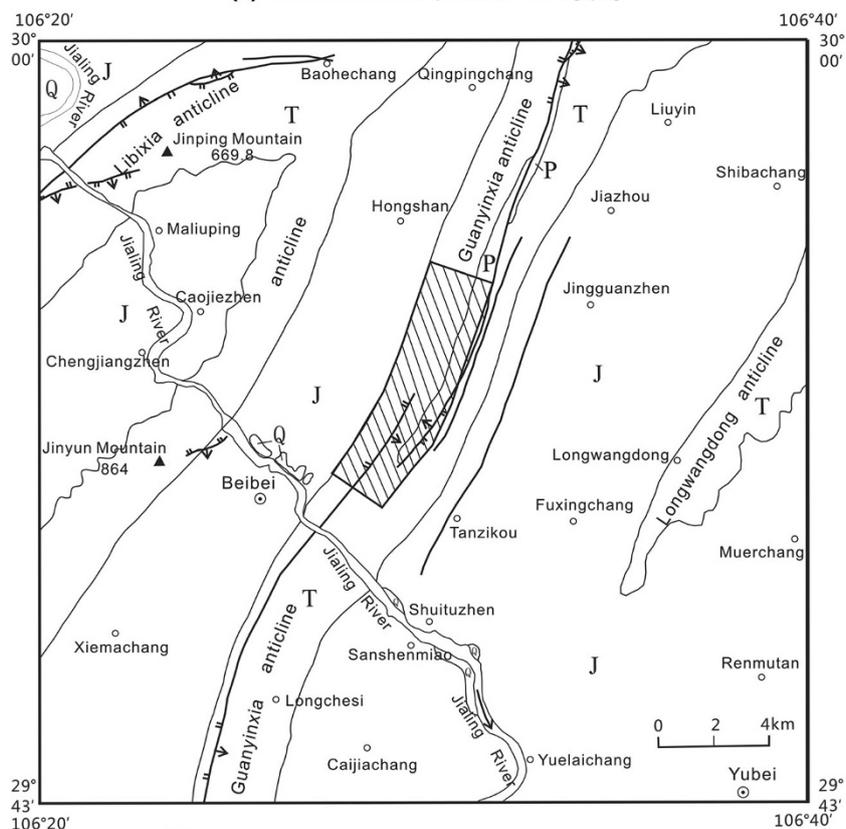
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(a) Location of Moxinpo mine, Chongqing



(b) Geological sketch map of Moxinpo mine, Chongqing

Fig. 1 Location and geological sketch map of the Moxinpo mine, Chongqing (Modified after Zhong et al. 2011)

occurrences and geochemical parameters are usually used as geochemical indicators and to illustrate the origin of coal accumulating materials (Zheng et al. 2006).

The K2 coal with high REY concentrations in the Moxinpo mine, which is comparable to some coal basins in the Far East, Russian (Seredin 1996), were studied. This

paper reports the first data of the REY concentration level, distribution patterns and modes of occurrences in the K2 coal seam, Moxinpo mine, Tianfu Coalfield, Chongqing. It also contributes to the discussion on the prospects of their use as a new source of REY.

2 Geological setting

The Moxinpo mine is located in northern Chongqing, 30 km far away from the Chongqing urban area (Fig. 1a). The coal-bearing stratum is the late Permian Longtan Formation, composed of limestone, siliceous limestone, sandstone, mudstone and coal, which is enriched in brachiopods, bivalves, dragonfly branch, cephalopods and other fossils. The Longtan Formation deposited in a confined sea carbonate tidal environment, of which the average thickness is 138.76 m. The Longtan Formation can be divided into five portions, in which the first and third are coal-bearing portions. The major coal seam in the Moxinpo mine is the K2 coal, which is located in the first portion of the Longtan Formation. The thickness of the coal seam varies from 1.54 to 5.16 m and averages 3.25 m. The Changxin Formation

conformably overlies the Longtan Formation and is mainly composed of thick layers of limestone intercalated with thin layers of mudstone and rich in marine fossils. The Maokou Formation disconformably underlies the Longtan Formation, which is consisted of thick layers of limestone and enriched in marine fossil fragments, corals, flint nodules, and locally, banded mudstone.

Tectonically, the Moxinpo mine is located in southern part of Chongqing fold bundle and west limb of the Guanyinxia anticline. Tectonic line trend orients mainly NNE-SSW. Anticlines of Xishan, Libixia, Wentangxia, Guanyinxia and Longwangdong, synclines among the anticlines are the southern extension of the Chongqing fold bundle, which is a typical interval block fold and characterized by close anticlinal folds, synclinal fold-relief. The fracture zone is developed mainly by reverse fault, shown in Fig. 1b.

3 Sampling and methods

A total of 20 bench samples were taken from the working face of the mined coal in the Moxinpo mine, following the

Table 1 Concentrations of REY in the K2 coal from the Moxinpo mine, Chongqing (in $\mu\text{g/g}$)

Samples	Lithology	A_d (%)	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Y	Ho	Er	Tm	Yb	Lu
K2-1	Coal	22.02	23.1	38.3	4.21	15.5	3.10	0.50	3.44	0.56	2.95	16.0	0.59	1.39	0.24	1.27	0.15
K2-2	Coal	15.24	10.2	14.9	1.52	4.91	0.89	0.10	0.92	0.17	0.92	5.96	0.20	0.51	0.09	0.56	0.07
K2-3	Coal	13.02	41.5	50.5	5.36	18.1	2.85	0.24	2.77	0.44	2.27	11.2	0.45	1.11	0.19	1.04	0.12
K2-4	Coal	13.44	85.8	114	11.1	35.4	5.24	0.30	5.17	0.71	3.40	36.5	0.67	1.67	0.27	1.49	0.17
K2-5	Coal	19.97	80.1	110	10.5	34.8	5.15	0.32	5.41	0.90	4.67	24.3	0.99	2.54	0.46	2.60	0.30
K2-6	Coal	11.4	87.3	151	11.5	36.3	5.40	0.29	5.27	0.85	4.75	26.6	1.02	2.71	0.51	3.14	0.37
K2-7	Coal	22.06	134	334	30.7	108	18.57	0.85	14.9	2.00	8.29	66.6	1.59	4.04	0.69	4.13	0.49
K2-8	Coal	14.79	182	365	32.7	115	19.2	0.85	14.9	1.80	7.16	98.9	1.36	3.59	0.60	3.62	0.44
K2-9	Carbonaceous mudstone	53.38	99.3	212	25.5	91.3	15.0	0.66	11.4	1.63	7.42	76.5	1.44	3.58	0.63	3.78	0.44
K2-10	Coal	21.06	91.4	250	25.3	93.5	17.2	0.78	12.0	1.60	6.99	98.7	1.38	3.60	0.63	3.92	0.45
K2-11	Coal	13.52	222	415	34.3	111.5	15.3	0.74	13.2	1.73	8.19	29.9	1.68	4.64	0.89	5.49	0.65
K2-12	Coal	11.54	84.4	172	13.5	44.5	9.88	0.67	10.9	2.08	11.3	60.8	2.31	5.71	1.03	5.95	0.72
K2-13	Coal	12.54	177	334	27.8	90.8	12.9	0.64	11.3	1.57	7.51	46.6	1.55	4.09	0.74	4.45	0.54
K2-14	Coal	11.54	50.2	99.0	12.2	47.1	8.59	0.48	6.99	1.11	5.55	30.5	1.17	3.06	0.58	3.58	0.44
K2-15	Coal	16.09	119	303	30.7	117	21.6	1.12	16.0	1.91	6.55	68.6	1.14	2.85	0.46	2.91	0.34
K2-16	Coal	19.04	51.6	111	13.6	52.6	10.2	0.60	8.51	1.44	7.48	62.8	1.55	3.96	0.72	4.32	0.54
K2-17	Coal	23.64	50.4	98.3	11.4	41.8	7.49	0.45	6.60	1.07	5.51	61.0	1.18	3.03	0.54	3.29	0.41
K2-18	Coal	20.9	95.3	164.7	19.6	73.2	12.7	0.87	10.9	1.65	7.80	39.2	1.52	3.82	0.68	4.16	0.50
K2-19	Coal	12.36	183	358	33.6	118	17.6	0.89	14.8	2.06	9.76	130	2.06	5.59	1.08	6.74	0.83
K2-20	Carbonaceous mudstone	85.58	85.6	140	14.9	45.9	9.41	0.64	9.67	1.87	10.6	42.8	2.17	5.58	1.11	6.68	0.77
WA in coal		16.34	98.2	193	18.3	64.3	10.8	0.59	9.11	1.31	6.17	50.8	1.25	3.22	0.58	3.48	0.42

A, ash yield; d, dry basis; WA, weighted average

Chinese Standard GB/T482-2008, including 18 coal benches, one parting and one floor samples. Each coal bench sample was cut over an area 10 cm wide, 10 cm deep, and 10 cm thick. All collected samples were immediately stored in plastic bags to minimize contamination and oxidation.

Samples were crushed and ground to 200 meshes for coal chemical analysis. The ash yield was conducted according to the Chinese Standard GB/T212-2008. The contents of REY were determined by inductively coupled mass spectrometry (Thermo X series II ICP-MS). A scanning electron microscope (JEOL-JSM 6610LV, accelerating voltage 20 kV) in conjunction with an energy-dispersed X-ray spectrometer (SEM-EDX) was used to study the characteristics of the minerals in the coal. The procedures of ICP-MS were: weighed 0.25 g sample in a 50 mL Teflon beaker, added 20 mL HNO₃-HClO₄-HF (volume ratio of 4:1:5) and 2 mL H₂SO₄, placed in a temperature controlled heating plate and heated to 230 °C like wet salt, then heated to 280 °C and evaporated to dryness, turned off

the heating plate to make the sample cooling for 3 min, added 8 mL concentrated aqua regia, incubated for 10 min, transferred the solution to a 25 mL plastic flask, mixed and volumed, then took 5 mL of solution to a 25 mL volumetric flask and diluted to the mark, then measured under the high resolution inductively coupled plasma mass spectrometry instrument.

4 Results and discussion

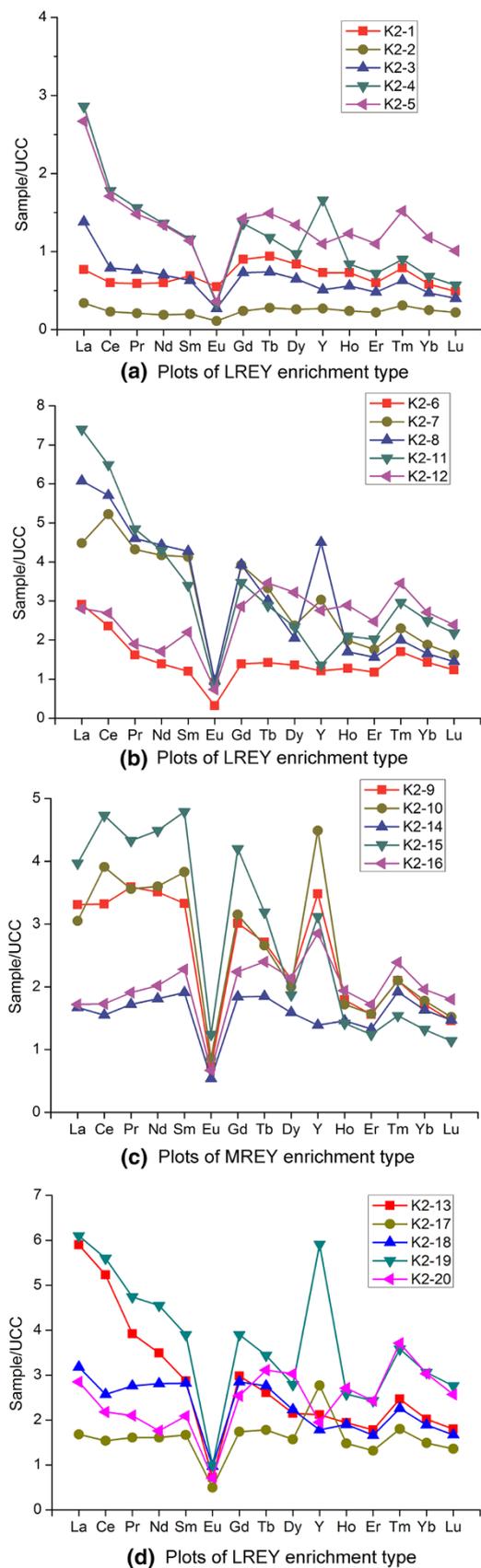
4.1 REY content in coal

The abundances and geochemical parameters of REY in the K2 coal benches, parting, and floor samples, are listed in Tables 1 and 2, respectively. According to the Moxinpo coal exploration geological report, the average vitrinite reflectance of the K2 coal is 1.55 %, indicating a coking coal (Chongqing Institute of Geology and Mineral Resources 2010). The content of total REY in the Moxinpo

Table 2 REY geochemical parameters in the K2 coal from the Moxinpo mine, Chongqing (μg/g)

Samples	ΣREY (Coal basis)	ΣREY (Ash basis)	LREY	MREY	HREY	L/M	L/H	M/H	(La/ Lu) _N	(La/ Sm) _N	(Gd/ Lu) _N	Enrichment type	δCe	δEu
K2-1	111	506	84.25	23.44	3.63	3.59	23.19	6.45	1.57	1.12	1.84	L	0.89	0.70
K2-2	41.9	275	32.37	8.07	1.42	4.01	22.72	5.66	1.56	1.72	1.11	L	0.86	0.50
K2-3	138	1,061	118.36	16.92	2.91	6.99	40.73	5.82	3.43	2.18	1.81	L	0.77	0.40
K2-4	302	2,246	251.53	46.09	4.27	5.46	58.87	10.79	4.98	2.45	2.37	L	0.84	0.27
K2-5	283	1,415	240.14	35.61	6.88	6.74	34.92	5.18	2.64	2.33	1.41	L	0.86	0.28
K2-6	337	2,955	291.32	37.76	7.75	7.71	37.57	4.87	2.34	2.42	1.12	L	1.08	0.25
K2-7	730	3,308	626.11	92.64	10.94	6.76	57.26	8.47	2.76	1.08	2.41	L	1.19	0.24
K2-8	847	5,729	714.06	123.65	9.61	5.77	74.27	12.86	4.17	1.42	2.70	L	1.08	0.23
K2-9	551	1,032	443.48	97.63	9.87	4.54	44.93	9.89	2.27	0.99	2.06	M	0.96	0.23
K2-10	608	2,886	477.81	120.03	9.98	3.98	47.86	12.02	2.01	0.80	2.08	M	1.19	0.25
K2-11	866	6,403	798.63	53.76	13.34	14.86	59.85	4.03	3.39	2.18	1.59	L	1.09	0.24
K2-12	426	3,692	324.69	85.69	15.73	3.79	20.65	5.45	1.18	1.28	1.19	L	1.16	0.30
K2-13	722	5,757	642.93	67.65	11.38	9.50	56.52	5.95	3.27	2.05	1.65	L	1.09	0.24
K2-14	271	2,345	217.11	44.64	8.82	4.86	24.62	5.06	1.14	0.88	1.25	M	0.91	0.29
K2-15	693	4,307	591.15	94.13	7.70	6.28	76.75	12.22	3.50	0.83	3.69	M	1.14	0.28
K2-16	331	1,738	238.97	80.83	11.09	2.96	21.55	7.29	0.96	0.76	1.25	M	0.96	0.30
K2-17	293	1,237	209.47	74.62	8.44	2.81	24.82	8.84	1.24	1.01	1.28	L	0.93	0.29
K2-18	437	2,089	365.46	60.37	10.68	6.05	34.21	5.65	1.90	1.13	1.71	L	0.87	0.34
K2-19	885	7,157	710.82	157.55	16.29	4.51	43.64	9.67	2.21	1.56	1.42	L	1.04	0.25
K2-20	377	441	295.25	65.59	16.30	4.50	18.11	4.02	1.11	1.36	0.99	L	0.89	0.31
WA in coal	462	3,061	385	68.0	8.94	5.92	42.22	7.57	2.46	1.51	1.77		1.00	0.31

ΣREY is the sum of La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Y, Ho, Er, Tm, Yb and Lu; LREY means the sum of La, Ce, Pr, Nd and Sm; MREY means the sum of Eu, Gd, Tb, Dy and Y; HREY means the sum of Ho, Er, Tm, Yb and Lu; L/M is the ratio of LREY and MREY; L/H is the ratio of LREY and HREY; M/H is the ratio of MREY and HREY; (La/Lu)_N is the ratio of (La)_N and (Lu)_N; (La/Sm)_N, ratio of (La)_N and (Sm)_N; (Gd/Lu)_N, ratio of (Gd)_N and (Lu)_N; δCe = Ce_N/(La_N × Pr_N)^{1/2}; δEu = Eu_N/(Sm_N × Gd_N)^{1/2}; N, REY are normalized by Upper Continental Crust (UCC) (Taylor and McLennan 1985). L, light REY enrichment; M, medium REY enrichment



◀**Fig. 2** REY distribution patterns in the K2 samples of the Moxinpo mine, Chongqing

coals varies from 41.4 to 885 $\mu\text{g/g}$, with an average of 462 $\mu\text{g/g}$, higher than the average value in common Chinese coals (136 $\mu\text{g/g}$; Dai et al. 2012b) and common world coals (68.5 $\mu\text{g/g}$; Ketris and Yudovich 2009). The content of total REY in the Moxinpo coal ashes varies from 214 to 7157 $\mu\text{g/g}$, averages 3061 $\mu\text{g/g}$, much higher than Chinese upper crust (87.2 $\mu\text{g/g}$; Li 1994) and the upper continental crust (168 $\mu\text{g/g}$; Taylor and McLennan 1985).

4.2 REY geochemistry

Yttrium is closely associated with lanthanides in nature, because its ionic radius is very similar and its ionic charge is equal to that of Ho (Seredin and Dai 2012). For this reason, yttrium is generally placed between Dy and Ho in normalized REY plots (Bao and Zhao 2008). A three-fold geochemical classification of REY was used in the present study, including light (L-REY: La, Ce, Pr, Nd, and Sm), medium (M-REY: Eu, Gd, Tb, Dy, and Y), and heavy (H-REY: Ho, Er, Tm, Yb, and Lu) REY (Seredin and Dai 2012). Accordingly, three enrichment types are identified, L-type (light REY; $\text{La}_N/\text{Lu}_N > 1$), M-type (medium REY; $\text{La}_N/\text{Sm}_N < 1$, $\text{Gd}_N/\text{Lu}_N > 1$), and H-type (heavy REY; $\text{La}_N/\text{Lu}_N < 1$), in comparison with the upper continental crust (UCC) (Seredin and Dai 2012). Table 2 and Fig. 2 illustrate that the K2 coal in the Moxinpo mine are mainly enriched in light REY, only the samples of K2-9, K2-10, K2-15 and K2-16 are enriched in medium REY. From top to bottom, the REY enrichment type of the K2 coal can be divided into five sections, in which Sect. 1 (from K2-1 to K2-8), Sect. 2 (from K2-11 to K2-13) and Sect. 3 (from K2-17 to K2-20) are L-type enrichment, Sect. 4 (from K2-9 to K2-10) and Sect. 5 (from K2-14 to K2-16) are M-type enrichment. The L-type coals of the Moxinpo mine appear a broad V-shape curve (Fig. 2), the La-Sm portion and Gd-Lu portion curves are steep and smooth, respectively, demonstrating that the fractionation of light REY is high, and that of the heavy REY is low. The δCe values vary from 0.77 to 1.14, with an average of 0.99, indicating a slight Ce positive anomaly. The δEu varying from 0.23 to 0.77, with an average of 0.31, shows a well-pronounced Eu minimum.

4.3 REY distribution through the K2 coal seam

The REY content variations through the K2 coal in the Moxinpo mine shows that the top and bottom portion of the coal seam have relatively lower contents than the middle portion (Fig. 3), indicating a certain REY fractionation. In addition, samples K2-9 and K2-20, the lithology of which

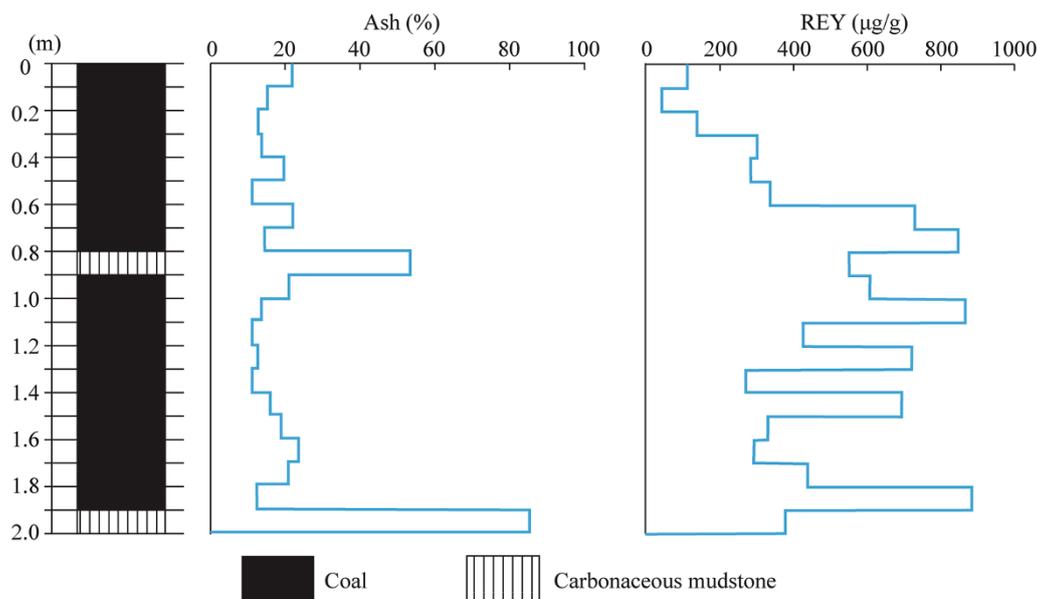


Fig. 3 Ash and REY variations through the K2 coal seam of the Moxinpo mine, Chongqing

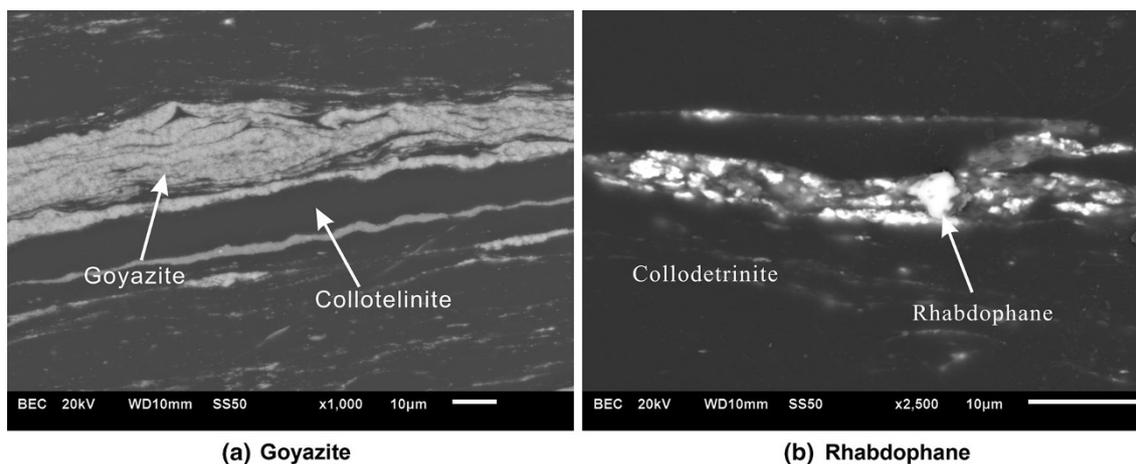


Fig. 4 Goyazite and rhabdophane in the K2 coal from the Moxinpo mine, Chongqing (SEM)

is carbonaceous mudstone, are parting and floor of the K2 coal, respectively. The REY contents of samples K2-9 and K2-20 are comparable to those of coal samples of K2 reflect that lithology differences have a weak impact on REY content through the K2 coal seam, similar to the REY distribution of the Pavlovsk coals in Russian Far East (Seredin 1996).

4.4 Discovery of REY-bearing mineral in the K2 coal

Finely-dispersed REY-bearing minerals of the K2 coal in the Moxinpo mine, including goyazite (Fig. 4a) and rhabdophane (Fig. 4b) were identified using SEM-EDX.

Goyazite is a relatively common mineral in coal (Ward 2002). Aluminum phosphate containing high Sr content was recognized as goyazite under SEM. The goyazite in the K2 coal of the Moxinpo mine distributes along the bedding planes, suggesting a syngenetical origin. Dai et al. (2005) found goyazite occurring as fillings along with boehmite in fusinite cavities in the No. 6 coal seam in the Junger Coalfield. Dai et al. (2013) also found goyazite in the partings of the late Permian coals in the Fusui Coalfield, which was thought to be formed by the reaction between P released from organic matter in peat swamp and Al-rich solution and Sr leached from limestone. Rhabdophane is a light REY-bearing mineral, the Ce of which can

be replaced by La, Nd, Y and Th (Wang et al. 1987). Rhabdophane in the K2 coal of the Moxinpo mine occurred in the collodetrinite unevenly, indicating an authigenic origin. Dai et al. (2013) found rhabdophane in the partings of late Permian coal from the Fusui Coalfield. However, the medium- and heavy-REY minerals were not identified in the coals present in this study.

4.5 Occurrences of REY and its relationship with ash

No correlation (correlation coefficient of -0.06 ; Fig. 3) was found between the REY content and the ash yield of the K2 coal in the Moxinpo mine. With increasing REY atomic number, the correlation coefficient between REY and ash becomes gradually high (Fig. 5). However, overall, REY contents and ash yield are irrelevant or weakly correlated, indicating that the mode of occurrence of REY may be not only associated with minerals, but also with organic matter in the coal. Generally, REY in coal mainly originated from terrigenous material, in conjunction with the aluminosilicate minerals, which are generally major components of coal ash (Zhao 2002). Some studies have shown that the REY abundance increases with the increasing ash yield of the coals (Wang et al. 1989; Zou et al. 2013). However, the average content of REY in the Moxinpo coal and coal ash is up to $462 \mu\text{g/g}$ and $3061 \mu\text{g/g}$, respectively, much higher than the general mudstone REY content (typically $200\text{--}300 \mu\text{g/g}$; Zhao 2002), indicating that elevated contents of REY in the K2 coal of the Moxinpo is not provided by terrigenous materials alone, but organic matter plays an important role in the accumulation of REY in the coal. In addition, the content of REY of K2-19 higher than K2-20 (floor sample) and no correlation between REY and ash indicated that REY of the K2 coal in the Moxinpo mine is mainly associated with organic matter, other than minerals, which are similar to

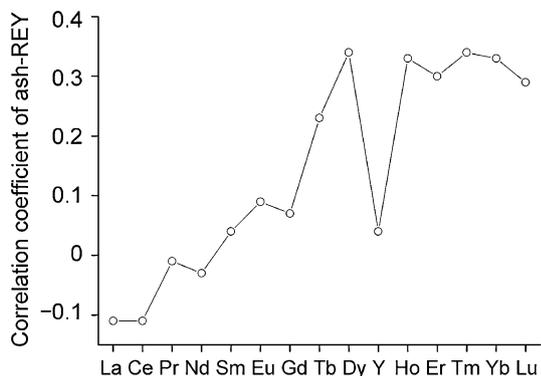


Fig. 5 Relationship between REY–ash correlation coefficients and REY atomic number

the results studied by Seredin (1996). Eskenazy (1987) found that organic compounds accounted for a large proportion of REY in the low-ash coal in Bulgaria and enriched more HREY, which may be a result of the interaction between the dissolved REY and the products of disintegration and decay of organic substances, mainly the humic acids. Seredin (1996) found that organic matter played a dominant role to the REY of coal in the Russian Far East, while fine-grained authigenic minerals and forms sorbed by the clay particles are of subordinate significance.

4.6 Preliminary evaluation of coal ash as a potential REY deposit

The average content of REY in coal ash in the Moxinpo mine is up to $3061 \mu\text{g/g}$, much higher than the cut-off grade of REY in coal ash (REO-0.1 %) proposed by Seredin and Dai (Seredin and Dai 2012). The K2 coal distribution in the Moxinpo is stable and the average thickness of the K2 coal is about 2 m; thus it can be used as a potential REY resource and deserves further study of the extraction technology of REY from coal ash.

5 Conclusions

- (1) The average contents of REY in the K2 coal and ash of the Moxinpo mine are up to $462 \mu\text{g/g}$ and $3061 \mu\text{g/g}$, respectively, higher than the cut-off grade of REY in coal ash (REO-0.1 %) proposed by Seredin and Dai (Seredin and Dai 2012), indicating a potential REY deposits.
- (2) The L-REY is the main REY type of the K2 coal in the Moxinpo mine, followed by the MREY. The REY distribution through the K2 coal shows that the top and bottom portion of the coal seam has a lower content of REY than that in the middle portion.
- (3) The occurrence of REY in the K2 coal of the Moxinpo mine may be associated mainly with organic matter and a small proportion occurs in minerals.
- (4) The K2 coal in the Moxinpo mine can be considered as a potential resource of REY and coal ash is a promising raw material for extracting REY as a by-product.

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