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# LEACHING OF URANIUM AND THORIUM FROM RED MUD USING SEQUENTIAL EXTRACTION METHODS

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## ABSTRACT

Red mud can be generated during production of alumina from bauxite using Bayer process or sintering alumina process. The specific chemical and mineral composition of different red mud is highly dependent on the nature of the parent bauxite ore and the processing parameters. The disposal of red mud poses an issue of great importance with significant environmental concerns. As major natural radioactive elements, uranium and thorium restrict the use of red mud as building materials or additives, and threaten the environment because of their radio-toxicity and chemical toxicity. In view of the toxic nature of uranium and thorium the disposal of red mud needs prior evaluation. The concentrations and distributions of U and Th in Bayer red mud (BRM) and red mud from sintering alumina process (RMS) from Guizhou, China, were studied using sequential extraction methods. The total leachability of U in BRM and RMS was about 80% and 50%, respectively, while the leachability of Th during sequential extraction in BRM (about 15%) and RMS (about 22%) was much lower.

## KEYWORDS:

Red mud, Uranium, Thorium, Leachability, Sequential extraction

## 1 INTRODUCTION

Red mud is a solid waste by-product of bauxite processing for alumina production using Bayer process or sintering alumina process. About 90% of alumina in the world is obtained by Bayer process which is effective for processing high grade bauxite ores, whereas most local bauxite ores in China are low grade, and alternatively sintering alumina process is effectively used [1-3]. According to the two different processing methods, red mud can generally be classified as Bayer red mud (BRM) and red mud from sintering alumina process (RMS) in China.

However, large quantities of both red mud types are never applied because of high costs and transport difficulties [4-6].

There are many researches on reuse of red mud as construction and building materials, such as ceramics [7-8], cement [9], concrete [10], and clay bricks [2,11]. As major natural radioactive elements, uranium and thorium from bauxite ore restrict the use of red mud as building materials or additives. During the production of alumina, the concentration of uranium and thorium increases in red mud [12, 13]. It has been reported that more than 80% of uranium and thorium in the bauxite is concentrated in the red mud [14]. Red mud contains a significant quantity of radioactive elements (50-60 g/t uranium and 20-30 g/t thorium), so it cannot be used as a construction material without pretreatment [15]. The radionuclide concentration of natural origin in bauxites and red mud significantly exceeds the world average for building materials [16]. Red mud from Shandong, China, was reported to not meet the regulations of Chinese safety limits for radioactivity of main materials or internal materials for buildings [1]. Hungarian red mud was limited in brick production to less than 15%; otherwise, brick production would contain enhanced levels of natural radioactivity [16]. Due to the discharge of bauxitic red mud tailings, the seafloor of Antikyra bay and the basin of Gulf of Corinth are highly contaminated with natural radionuclides, where the mean activity of <sup>238</sup>U and <sup>232</sup>Th for depositional lobe is about 13 and 16 times higher than that of the world average [13].

Meanwhile, uranium is characterized by both radio-toxicity and chemical toxicity, but it is the latter which limits its exposure to humans whereas thorium is to be considered as only radiotoxic [17]. Processing of uranium- and thorium-bearing minerals lead to enhanced radiation exposures not only to the workers but also to the inhabitants. Uranium and thorium may be released to the environment in mining, ore processing and residue disposing, although concentrations are generally low. The leaching potential of toxic elements [18, 19] in red mud needs prior evaluation for environmental impacts.

The aim of the present work was to assess the environmental impacts of U and Th in red mud, and to com-

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pare the fractionation and speciation of U and Th in BRM and RMS from Guizhou, China. For this purpose a modified Tessier's sequential extraction procedure was applied to estimate the leachability and modes of occurrence of U and Th.

## 2 MATERIALS AND METHODS

### 2.1 Materials

Red mud samples used in this study, including Bayer red mud (BRM) and red mud from sintering alumina process (RMS) were obtained from Guizhou Enterprise of China Aluminum Co., Ltd. (GZHE).

### 2.2 Sequential extraction procedure

Distributions of U and Th present in BRM and RMS were evaluated by a modified sequential extraction procedure developed by Tessier et al. [20]. To Tessier's original partitioning scheme, as a first step extraction with water was added in order to perform speciation analysis of U and Th. After mechanical shaking on an elliptical orbital shaker, centrifugation and filtration, the concentrations of elements in each particular fraction were determined by ICP-MS. The solid residues after each extraction step were washed with 15 mL of water, shaken for 15 min, centrifuged at 4000 rpm for 15 min, decanted, and the washing solution discarded prior to the addition of the next extractant. After each extraction step, the leachate was separated from the residue by filtering through a 0.45  $\mu\text{m}$  Millipore filter paper using a glass Millipore vacuum filter assembly. The leachate was stored for trace element analysis. The residue on the filter paper was carried over to the next step of sequential extraction.

#### 2.2.1 Water-soluble

3.00 g of each red mud sample was weighed into a 50 mL polypropylene centrifuge tube and the sample shaken for 24 h with 30 mL of water, centrifuged, decanted and filtered.

#### 2.2.2 Exchangeable

The extract solution was 30 mL of 1 M magnesium chloride ( $\text{MgCl}_2$ ) of pH 7.0, shaken for 24 h at room temperature, centrifuged, decanted and filtered.

#### 2.2.3 Bound to carbonates

The extract solution was 30 mL of 1 M sodium acetate ( $\text{CH}_3\text{COONa}$ ) adjusted to pH 5.0 with acetic acid, shaken for 24 h at room temperature, centrifuged, decanted and filtered.

#### 2.2.4 Bound to Fe-Mn oxides

The extract solution was 30 mL of 0.04 M hydroxylamine hydrochloride ( $\text{NH}_2\text{OH}\cdot\text{HCl}$ ) in 25% (v/v) acetic acid. Extraction was carried out at  $96 \pm 1$  °C for 6 h with occasional agitation on a magnetic stirrer provided with a

heater. After cooling to room temperature, the sample was centrifuged, decanted and filtered.

#### 2.2.5 Bound to organic matter

To the residue from the previous step was added 9 mL of 0.02 M  $\text{HNO}_3$  and 15 mL of 30%  $\text{H}_2\text{O}_2$  adjusted to pH 2 with  $\text{HNO}_3$ , and the mixture was heated to  $85 \pm 1$  °C for 2 h with occasional agitation. A second 9 mL aliquot of 30%  $\text{H}_2\text{O}_2$  (adjusted to pH 2 with  $\text{HNO}_3$ ) was then added and the sample was heated again to  $85 \pm 1$  °C for 3 h with intermittent agitation. After cooling, 15 mL of 3.2 M  $\text{NH}_4\text{OAc}$  in 20% (v/v)  $\text{HNO}_3$  was added and the sample was diluted to 60 mL and agitated continuously for 30 min. After that the sample was centrifuged, decanted and filtered.

#### 2.2.6 Residual

After drying, 50 mg of the residue from the organic fraction was digested using a 1:1 HF: $\text{HNO}_3$  dissolution procedure in a microwave digester.

### 2.3 Analytical methods

The morphology of BRM and RMS samples were observed under a JEM-2000FX II Transmission Electron Microscopy (TEM). A Netzsch equipment (STA 449 C Jupiter) was applied in this study to record the differential thermal analysis (DTA) and thermogravimetric analysis (TG) curves simultaneously using 20 mg RM samples. The heating rate rose between 25-1200 °C by  $10^\circ\text{C min}^{-1}$  in flowing air.

The concentrations of U, Th and rare earth elements (REEs) in red mud samples were determined using Quadrupole Inductively Coupled Plasma Mass Spectrometer (ICP-MS, ELAN DRC-e, PerkinElmer) at State Key Laboratory of Ore Deposit Geochemistry, Institute of Geochemistry, Chinese Academy of Sciences. BRM and RMS samples and their leached residues were dried in an oven at 105 °C for 12 h, and then ground, digested using a 1:1 HF: $\text{HNO}_3$ , and analyzed by ICP-MS. In order to analyze the correlation of thorium and REEs in BRM and RMS samples, a Laser Ablation Inductively Coupled Plasma Mass Spectrometer (LA-ICP-MS) was used.

## 3 RESULTS AND DISCUSSION

### 3.1 Contrast between BRM and RMS

The specific chemical and mineral composition of red mud is highly dependent on the nature of the bauxite used. Generally, red mud derived from different refining processes or different alumina plants contains six main constituents, namely  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{CaO}$ ,  $\text{Na}_2\text{O}$ , and  $\text{TiO}_2$ . However, the amounts of these main constituents differed greatly within ranges from one region to another [21], even in the same alumina plants at different time due to the change of the parent bauxite. There are also differences in the types and amounts of mineralogical phases between BRM and RMS. BRM typically comprises residual iron

oxides, quartz, sodium aluminosilicates and sodium hydroxide [22-24], while RMS is mainly comprised of calcium silicate [25], tricalcium aluminate, perovskite, calcite, and iron-bearing minerals. Figure 1 shows typical morphological characteristics of BRM and RMS from Guizhou, China.

The results of the thermal analysis of BRM and RMS are presented in Figure 2 and 3. From the thermal curves, the combined mass loss of BRM (16.24%) was much

higher than that of RMS (8.69%) from 25 to 1200 °C. There were two main decomposition steps for the mass loss with the change in temperature in each curve. The first endothermic peak located at 240 to 300 °C may be associated to the goethite decomposition into hematite, where mass loss of BRM was 7.26% and RMS was 4.12%. The second endothermic peak starting at about 698 °C and ending at 760 °C was proposed to result from the decomposition of calcite. The results suggested that the thermal decomposition of BRM was much more complex than RMS.

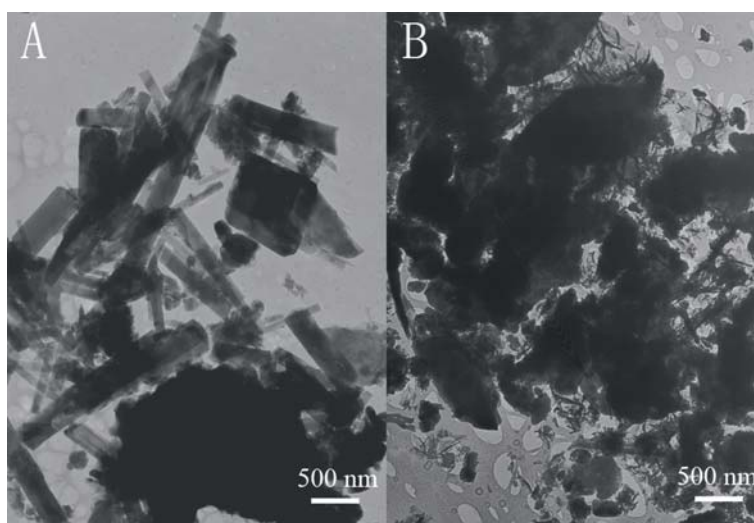


FIGURE 1 - TEM images of red mud samples from Guizhou, China. (A) BRM, Bayer red mud; (B) RMS, red mud from sintering alumina process.

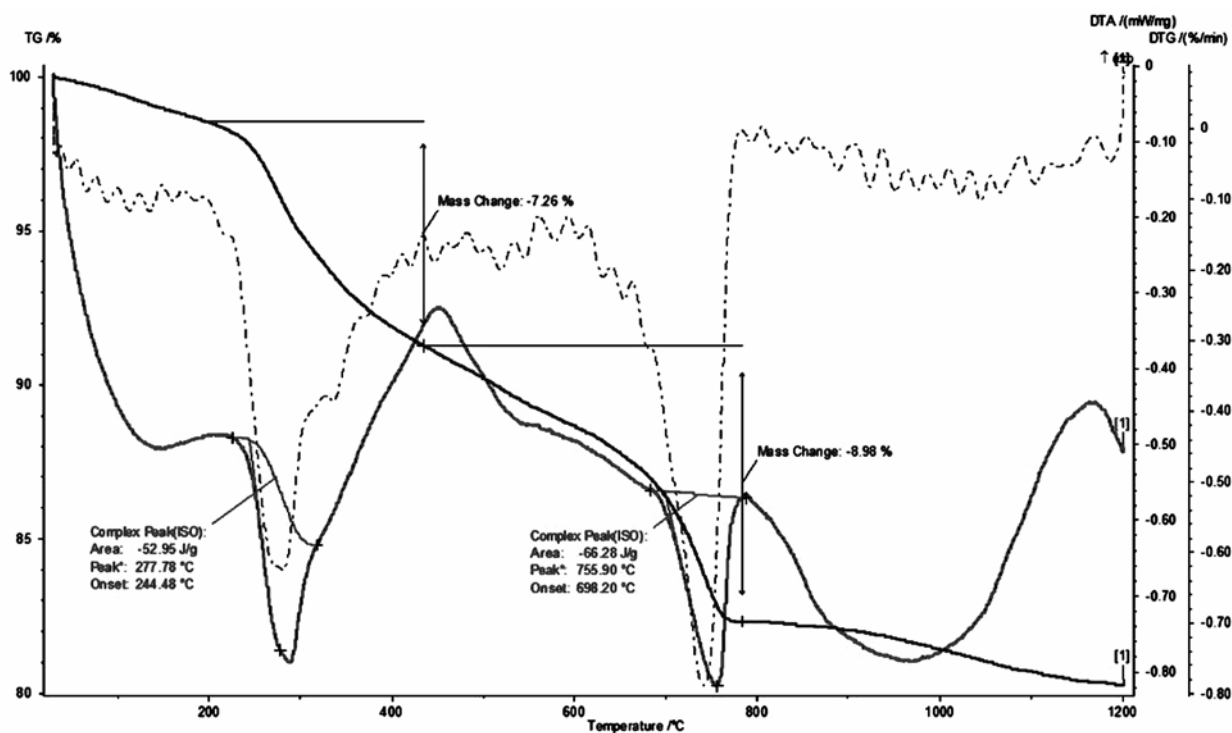


FIGURE 2 - TG and DTA curves of Bayer red mud (BRM).

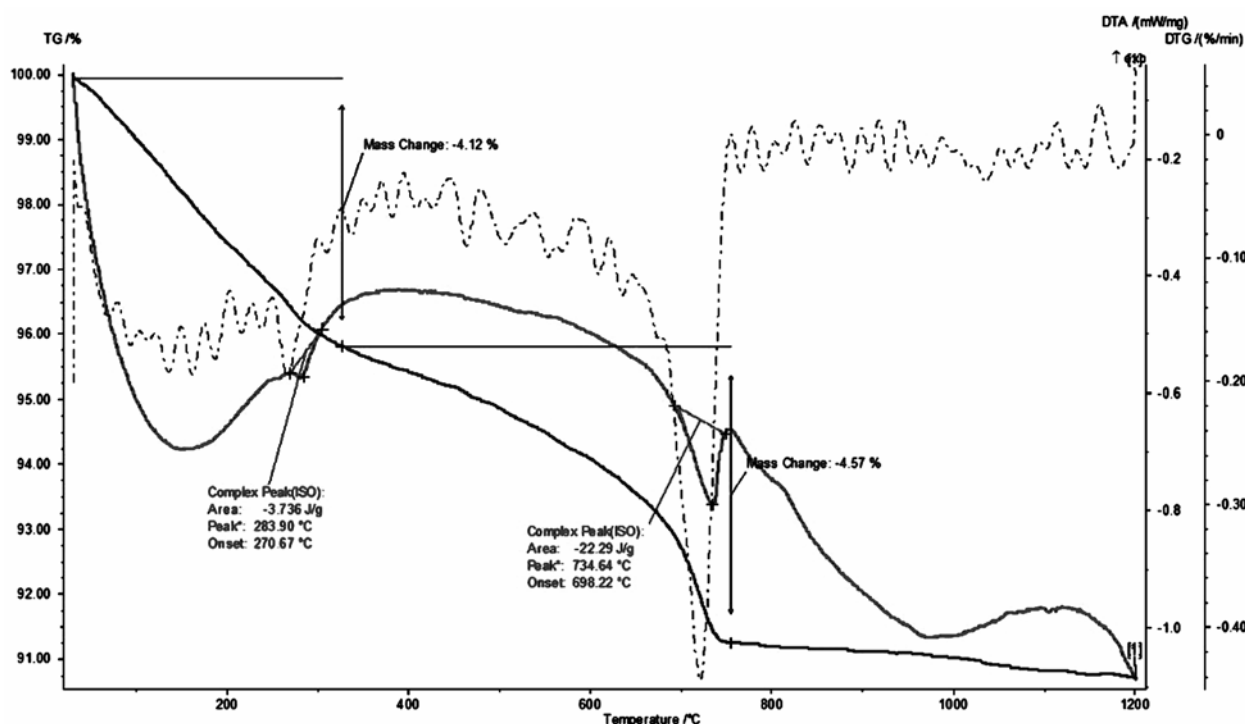


FIGURE 3 - TG and DTA curves of red mud from sintering alumina process (RMS).

### 3.2 Leachability of U in red mud

Uranium is the most common radionuclide contaminant in groundwater systems, with leaching of uranium mine tailings resulting in the contamination of soils and aquifers [26]. The mobility of uranium and its leaching is dependent on several factors [27]. Uranium present in red mud in trace concentrations (total U content in this study: BRM  $26.5 \mu\text{g g}^{-1}$  and RMS  $27.7 \mu\text{g g}^{-1}$ ) is mostly associated with the insoluble residual fraction. The mass balance of U in this study agreed within  $\pm 5\%$ , confirming the precision of the analytical work. The contents of U leached out at various steps of sequential extraction of BRM and RMS are presented in Table 1.

In contrast, total amounts of U and Th leaching out into solution during sequential extraction differed in BRM compared with RMS (Figure 4 A and B). For uranium, its leachability during sequential extraction in BRM (more

than 80%) was higher than that in RMS (more than 50%). The leaching ratios of 'water-soluble', 'exchangeable' and 'bound to organic matter' in BRM were similar in RMS. On the whole, BRM had higher concentrations and total amounts of U in 'water-soluble' and 'exchangeable' than RMS. In BRM, nearly 80% U was leached in 'bound to carbonate' (average 38.65%) and 'bound to Fe-Mn oxides' (average 39.46%), while more than 50% U in RMS was leached in 'bound to carbonate' (average 16.71%) and 'bound to Fe-Mn oxides' (average 34.54%).

### 3.3 Leachability of Th in red mud

Thorium is a relatively immobile element in the environment, but its solubility can be greatly enhanced in some acidic, sulphate- and organic-rich surface- and groundwaters [26]. It is a fact that thorium could be penalty element in the metallurgy and radiotoxic for the environment.

TABLE 1- Leachability of uranium from BRM and RMS samples at different steps of sequential extraction.

Description	Water-soluble ( $\mu\text{g L}^{-1}$ )	Exchangeable ( $\mu\text{g L}^{-1}$ )	Bound to carbonates ( $\mu\text{g L}^{-1}$ )	Bound to Fe-Mn oxides ( $\mu\text{g L}^{-1}$ )	Bound to organic matter ( $\mu\text{g L}^{-1}$ )	Residual ( $\mu\text{g g}^{-1}$ )
BRM Sam 1	12.7	25.0	739	642	6.54	12.5
BRM Sam 2	12.0	25.0	718	708	6.83	11.8
BRM Sam 3	11.9	24.3	707	713	7.78	12.4
BRM Average	12.2	24.8	721	688	7.05	12.2
RMS Sam 1	0.602	10.5	349	648	7.90	34.8
RMS Sam 2	0.740	11.1	331	614	7.09	36.2
RMS Sam 3	1.48	12.5	365	612	6.49	33.9
RMS Average	0.941	11.4	348	625	7.16	35.0



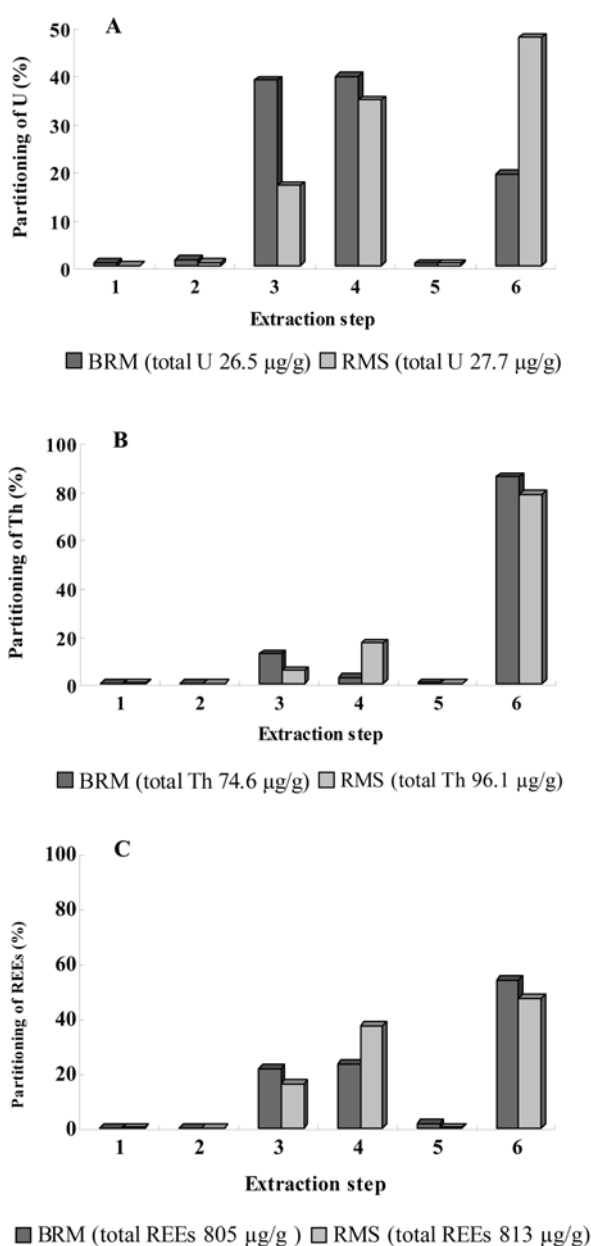


FIGURE 4 - Partitioning of Th, U, and REEs in BRM and RMS from Guizhou, China, on applying the modified Tessier extraction scheme. Extraction step: 1, water-soluble; 2, exchangeable; 3, bound to carbonates; 4, bound to Fe-Mn oxides; 5, bound to organic matter; 6, residual.

The contents of Th leached out at various steps of sequential extraction of BRM and RMS are presented in Table 2. Th present in red mud in trace concentrations (total Th content in this study: BRM  $74.6 \mu\text{g g}^{-1}$  and RMS  $96.1 \mu\text{g g}^{-1}$ ) is mostly associated with the insoluble residual fraction.

Compared with uranium, thorium was inclined to present in residual fraction in both red mud types. The total leachability of thorium during sequential extraction in BRM (about 15%) and RMS (about 22%) was much less than that of uranium. The reason for this might be that Th in red mud was usually inactive. RMS had higher concentrations and total amounts of Th than BRM as a whole, which indicated that the sintering alumina process might make some elements transferred from bauxite to red mud more active than Bayer process because of its high temperature. Meanwhile, some elements in BRM were active than in RMS, such as U, for the sintering alumina process might immobilize them in crystal lattices to form new phases. The leaching ratios of Th in ‘water-soluble’, ‘exchangeable’ and ‘bound to organic matter’ were all trace. BRM leached more Th in ‘bound to carbonate’, while RMS leached more Th in ‘bound to Fe-Mn oxides’.

In geochemical properties, thorium is most similar to the rare earth elements (REEs) [28]. Correlation analysis between Th and REEs in red mud samples was applied in this study. In both BRM and RMS types, Th correlated well with REEs (seen in Table 3), and its leaching trend was similar to total REEs (Figure 4 C). It was also reported that the amounts of Th in bauxite was positive correlated with certain accessory minerals (zircon, sphene, etc.) [29]. These accessory minerals had not been destroyed in the bauxite processing and then transferred to red mud as resistant relic minerals. In red mud, the residual minerals composition from bauxite ore remained during sequential extraction in this study. This would explain the low leachability of Th from red mud.

#### 3.4 Occurrence of U and Th in red mud

The possible modes of occurrence of U and Th in red mud in this study may be concluded. Little U in red mud formed as absorption by clay and iron minerals or association with organic matter. However, a majority of U in red mud occurred in the weak-binding form which was easily destroyed by acetate under acidic conditions. In red mud Th was rarely in the form of absorption by clay and

TABLE 2 - Leachability of thorium from BRM and RMS samples at different steps of sequential extraction.

Description	Water-soluble ( $\mu\text{g L}^{-1}$ )	Exchangeable ( $\mu\text{g L}^{-1}$ )	Bound to carbonates ( $\mu\text{g L}^{-1}$ )	Bound to Fe-Mn oxides ( $\mu\text{g L}^{-1}$ )	Bound to organic matter ( $\mu\text{g L}^{-1}$ )	Residual ( $\mu\text{g g}^{-1}$ )
BRM						
Sam 1	0.468	0.452	584	103	2.27	136
Sam 2	0.131	0.458	571	79.1	29.1	131
Sam 3	0.184	0.0650	507	119	0.397	134
Average	<b>0.261</b>	<b>0.325</b>	<b>554</b>	<b>100</b>	<b>10.6</b>	<b>134</b>
RMS						
Sam 1	0.431	0.156	207	751	0.332	133
Sam 2	0.583	0.743	233	646	0.109	139
Sam 3	5.12	0.182	287	693	0.106	128
Average	<b>2.04</b>	<b>0.360</b>	<b>242</b>	<b>697</b>	<b>0.182</b>	<b>133</b>

TABLE 3 - Contents of thorium and REEs in BRM and RMS samples using LA-ICP-MS.

		REEs ( $\mu\text{g g}^{-1}$ )														
		Th ( $\mu\text{g g}^{-1}$ )	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
BRM	1	88.8	203	460	51.0	192	28.7	5.44	25.3	3.48	21.7	3.91	12.8	1.57	10.6	2.00
	2	108	231	517	57.5	205	33.2	6.16	26.1	4.24	20.7	3.92	13.3	1.77	12.9	1.90
	3	109	248	556	65.1	215	36.5	7.15	34.1	4.54	27.6	5.34	14.8	2.33	18.6	2.57
	4	112	259	563	61.0	223	35.1	6.55	28.3	4.40	24.2	5.12	15.2	2.49	17.4	2.40
	5	116	266	594	63.7	240	36.3	6.88	31.5	4.51	24.5	4.96	16.3	2.02	17.0	2.04
	6	117	251	593	60.5	216	38.2	7.73	31.1	4.77	26.3	5.08	16.6	2.24	16.3	2.70
	7	119	259	579	67.5	236	38.2	8.13	30.4	5.07	25.4	4.86	15.0	2.26	17.0	2.27
	8	119	280	647	65.0	231	40.6	7.40	30.5	4.65	27.0	5.71	15.9	2.23	18.6	2.27
	9	120	324	655	73.7	268	45.4	8.36	31.2	4.85	25.8	5.27	17.4	2.23	19.0	2.53
	10	123	265	606	67.4	232	38.1	6.95	31.0	5.15	25.7	5.13	17.4	2.19	18.4	2.59
	11	126	295	649	71.5	247	42.4	8.38	33.4	4.48	25.0	5.35	16.1	2.23	16.9	2.63
	RMS	12	257	1089	2248	265	967	151	24.8	94.8	10.6	42.6	7.95	19.5	2.69	18.8
1		86.3	222	465	53.2	194	30.0	6.38	28.4	4.21	22.9	4.83	14.0	2.11	15.2	2.20
2		91.7	243	502	59.0	224	38.4	6.75	32.8	4.57	25.6	5.56	15.9	2.39	15.0	2.44
3		96.1	252	540	59.2	214	33.0	6.68	28.3	4.14	21.7	4.70	13.5	1.89	13.4	2.10
4		99.2	233	522	53.6	203	31.6	5.67	29.6	4.11	21.6	4.80	13.3	1.94	14.1	2.20
5		104	250	545	60.2	220	37.3	7.23	31.5	4.85	25.0	4.92	16.6	1.99	16.9	2.37
6		104	282	618	67.5	256	38.1	7.87	35.4	5.36	26.9	5.86	16.8	2.42	15.1	2.39
7		107	267	607	65.3	245	39.0	8.22	34.5	5.45	29.6	5.54	17.9	2.33	18.2	2.41
8		107	270	575	63.5	226	39.9	7.33	34.8	5.59	28.5	5.89	18.2	2.54	17.1	2.59
9		121	302	606	70.8	267	44.5	8.45	43.0	6.42	34.3	6.79	20.6	2.91	20.4	2.97
10		123	299	651	71.7	261	47.4	8.89	37.1	5.77	30.8	6.75	20.7	2.48	21.7	2.89
11		132	305	622	72.0	271	46.4	8.27	41.4	6.30	35.3	6.96	23.0	2.97	20.5	3.11
12	171	454	883	106	399	65.8	12.0	59.5	8.95	46.5	9.71	28.2	3.92	28.6	4.16	

iron minerals or association with organic matter, whereas it was mostly proposed in the form of certain accessory minerals inherited from bauxite or neo-formational minerals in bauxite processing.

Worldwide, the utilization of red mud generated from bauxite processing is limited, and most red mud is disposed in landfills or dumped at sea [4, 30, 31]. Part of U and Th in the weak-binding form in red mud pumped into pools, ponds or disposed in landfills will leach into rainwater and pollute the land, rivers, and groundwaters, especially in Guizhou, a typical Karst area in Southwest of China. As a result, the pollution of red mud may be enlarged.

#### 4 CONCLUSIONS

The results of the present investigation released that during sequential extraction and speciation analysis the leachability of U in BRM and RMS from Guizhou, China, was more than 80% and 50%, respectively. The possible forms of occurrence of U and Th in red mud in this study may be different. A large proportion of U in red mud occurred in the weak-binding form which was easily destroyed by acetate under acidic conditions. Meanwhile, in red mud Th was rarely in the form of absorption by clay and iron minerals or association with organic matter, whereas it was mostly proposed in the form of certain accessory minerals inherited from bauxite or neo-formational minerals in bauxite processing. Some of U and Th in the weak-binding form in red mud may leach into rainwater and lead to a significant impact on the environment.

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*The authors have declared no conflict of interest.*

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