Characterization of red mud derived from a combined Bayer Process and bauxite calcination method

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Abstract

Red mud can be derived from the processing of bauxite using different methods. The chemical and mineralogical composition of the combined Bayer Process and bauxite calcination red mud (BPBCRM) differs markedly from those of the pure Bayer Process red mud (PBPRM). In this study, red mud derived from a combined Bayer Process and bauxite calcination method was characterized. The results show that pH of the red mud decreased with increasing duration of storage time. Na dominated among the soluble cations, but the concentration of soluble Na decreased with increasing duration of storage time as a result of leaching. Cation exchange capacity also decreased with increasing duration of storage time, probably due to a decrease in pH causing a reduction in negatively charged sites on the red mud particles. Ca was the predominant exchangeable cation in the fresh red mud but the concentration of exchangeable Ca markedly decreased in the old red mud, which was dominated by exchangeable Na. The degree of crystallization and thermal stability of the red mud increased with increasing duration of storage. The acid neutralizing capacity of red mud obtained from this study was about $10 \, mol \, kg^{-1}$, which is much greater than the reported values for the pure Bayer Process red mud. Column filtering experiment indicates that the red mud also had a very strong capacity to remove Cu, Zn and Cd from the filtering solution. It is conservatively estimated that the simultaneous removal rates of Cd, Zn and Cu by red mud are over $22,250 \, mg \, kg^{-1}$, $22,500 \, mg \, kg^{-1}$ and $25,000 \, mg \, kg^{-1}$, respectively. The affinity of these metals to the red mud was in the following decreasing order: Cu > Zn > Cd. In general, the fresh red mud retained more heavy metals than the old red mud did.
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Keywords: Red mud; Bayer Process; Bauxite calcination; Acid neutralizing capacity; Calcite; Caustic substance

1. Introduction

Red mud is the residue of bauxite processing for alumina production. Globally, there are approximately 70 million tonnes of red mud being produced every year (http://www.redmud.org/home.html). Red mud is highly alkaline with a pH usually ranging from 10 to 13 [1–3]. Consequently, red mud is viewed as a corrosively hazardous substance requiring carefully handling. This caustic material is often contained in constructed storage facility to minimize its adverse impacts on the environments.

Utilization of red mud is of environmental and economic significance [4–6]. Red mud varies in physical, chemical and mineralogical properties due to differing ore sources and refining processes employed. The Bayer Process is the principal industrial means of producing alumina [7,8]. However, this process is not sufficiently effective for processing low grade bauxite ores containing substantial amounts of Si. Alternatively, bauxite calcining method is used for the processing of low grade bauxite ores [9,10]. In this method, bauxite ores are often mixed with limestone and sodium carbonate, and the mixture was roasted under high temperature to form the readily soluble sodium aluminate upon addition of diluted alkaline solutions or water. In China, most of the local bauxite ores contain a high proportion of Si and therefore either a bauxite calcination method or more frequently a combined Bayer Process and bauxite calcination method is used for alumina refining [10,11]. It was estimated that annually there are about 6 million tonnes of red mud being disposed of in China but only about 10% of the red mud is derived from pure Bayer Process [12].

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The alkaline nature of red mud makes it a potential acid-neutralizing agent [5,13]. Red mud has also been found to have a strong binding capacity for heavy metals and therefore can be used for remediation of heavy metal contaminated soils [2,14–16]. Bayer Process red mud and their environmental applications have received substantial research [13–25]. However, little has been done to red mud that is derived or partly derived from bauxite calcination methods. Bauxite calcination requires addition of substantial amounts of limestone and this may result in a high residual calcium oxide or calcium carbonate in the combined Bayer Process and bauxite calcination red mud (BPBCRM), which could significantly differ from pure Bayer Process red mud (PBPRM). The objective of this study was to characterize red mud derived from a combined Bayer Process and bauxite calcination method, with a particular interest in its characteristics relevant to remediation of acidic, heavy metal-enriched soil and water environments.

2. Materials and methods

Red mud samples of varying ages (fresh, 5-year-old, and 10-year-old) were collected from the red mud storage facilities of Zhengzhou Changcheng Aluminium Limited. At each sampling location, five sub-samples were collected within an area of $50 \, m^2$ to form a composite sample. In the laboratory, the red mud samples were air-dried and ground to pass a 2 mm sieve.

To determine pH, electrical conductivity (EC), water soluble and exchangeable cations, 1:5 (red mud–water) and 1:5 (red mud–1 mol L^{-1} NH$_4$Cl) extracts were prepared. The pH and EC in water extract were determined by a calibrated pH meter and a EC meter, respectively. K, Na, Ca and Mg in both water and 1 mol L^{-1} NH$_4$Cl extracts were determined by atomic absorption spectrometry (AAS); water soluble K, Na, Ca and Mg were estimated by water-extractable K, Na, Ca and Mg, respectively; exchangeable K, Na, Ca and Mg were estimated by the difference between 1 mol L^{-1} NH$_4$Cl extractable K, Na, Ca and Mg and water-extractable K, Na, Ca and Mg, respectively. Since no exchangeable acidic cations are expected to exist in alkaline conditions, the sum of exchangeable K, Na, Ca and Mg can be used as an estimate of cation exchange capacity (CEC).

Total element composition was analyzed by X-ray fluorescence spectroscopy (XRF). Mineral composition was determined by X-ray diffraction (XRD). The samples were also used for examination of micromorphological characteristics by scanning electron microscopy (SEM) and for thermogravimetric analysis (TGA).

Acid neutralizing capacity (ANC) of each red mud sample was determined by repeatedly titrating a red mud suspension (mixture of 5 g red mud with 25 mL deionized water) with an acid solution to pH 5.5. The detailed procedure was as follow: weigh 5 g of red mud sample in a 500 mL conical flask; add 25 mL of deionized water; titrate the suspension with a 0.1 mol L^{-1} HCl or 0.05 mol L^{-1} H$_2$SO$_4$ standardized solution to pH 5.5; retitrate the suspension next day when pH rises to above 5.5; repeat titration-standing cycle until pH becomes stable (in the latter stage of experiment, the interval of titration extended to more than two days due to a slow rise in pH following titration). In

this experiment, all three samples (each in duplicate) of varying age were used for ANC determinations. Both 0.1 mol L^{-1} HCl and 0.05 mol L^{-1} H$_2$SO$_4$ solutions were used to compare their effects on ANC recovery of red mud. The experiment lasted for 780 days with a total titration number of 367 times.

To test the heavy metal binding capacity of red mud, a column filtering experiment was conducted. For each red mud sample, 10 g of red mud were filled into a plastic column with an inner diameter of 20 mm. A double layer of nylon filter fabric was placed on the bottom of the column. A mixed solution containing equal concentration (50 mg L^{-1}) of three metals (Cd, Cu and Zn) was used as the filtering liquid, which was made by dissolving appropriate amounts of CdCl$_2$·2.5H$_2$O, CuSO$_4$·5H$_2$O and ZnSO$_4$·7H$_2$O in deionized water. For each filtration cycle, 50 mL of the filtering solution was passed through the red mud column at a rate of 6.25 mL per hour. In this experiment, a total number of 107 filtration cycles were performed. Selected filtrates were collected and analyzed for Cd, Cu and Zn using AAS.

3. Results and discussion

3.1. Basic chemical characteristics

It can be seen from Table 1 that pH of the red mud decreased with increasing duration of storage time. The pH of the fresh, 5-year-old and 10-year-old red mud samples was 11.58, 10.56 and 9.61, respectively. The EC of red mud also decreased with increasing duration of storage time; the EC average was over 20 dS m^{-1}, indicating that the red mud was highly saline. The molar concentrations of various soluble cations were in the following decreasing order: Na > K > Ca > Mg; soluble Na accounted for about 78% of the sum of soluble Na, K, Ca and Mg (on mean molar concentration basis). This indicates that soluble Na dominated the soluble basic cations in the red mud. However, the concentration of soluble Na decreased from 89.2 mmol(+) kg^{-1} in the fresh red mud to 27.9 mmol(+) kg^{-1} in the 10-year-old red mud, indicating that a large amount of dissolved Na was removed from the red mud during the period of storage. Soluble K also shows the same trend as soluble Na. Soluble Ca, particularly soluble Mg only took up a very small proportion of the sum of soluble Na, K, Ca and Mg; both show a trend that the molar concentration increased with increasing duration of storage time.

Unlike soluble basic cations, the mean molar concentration of exchangeable basic cations was in the following descending order: Ca > Na > K > Mg. It is interesting to note that CEC decreased with increasing duration of storage time. This may be attributed to a decrease in pH over time, which results in a reduction in negative charges on the variably charged colloids (VCC), for example:

$$[Al(OH)_6]^{3-} - [Na^+ + Ca^{2+}]^{3+} \rightarrow [Al(OH)_3]^0$$
$$+ NaOH(leaching) + Ca(OH)_2(precipitation)$$
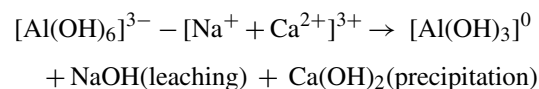
In the above equation, due to leaching of NaOH and precipitation of Ca(OH)$_2$, the originally negatively charged [Al(OH)$_6$]$^{3-}$ loses three OH$^-$ to become the zero-charged [Al(OH)$_3$]^0. This may partly explain the reduction in CEC.

Table 1
Basic chemical characteristics of red mud of different ages

Chemical parameter	Fresh	5-year-old	10-year-old	Mean
pH	11.58 ± 0.02	10.56 ± 0.03	9.61 ± 0.02	10.58 ± 0.07
EC (dS m^{-1})	28.4 ± 0.12	17.2 ± 0.08	15.4 ± 0.14	20.3 ± 0.34
Soluble K (mmol(+) kg^{-1})	19.0 ± 0.65	10.9 ± 0.38	7.9 ± 0.26	12.6 ± 1.29
Soluble Na (mmol(+) kg^{-1})	89.2 ± 1.92	52.3 ± 0.51	27.9 ± 0.83	56.5 ± 3.23
Soluble Ca (mmol(+) kg^{-1})	1.5 ± 0.02	2.5 ± 0.02	3.9 ± 0.04	2.6 ± 0.08
Soluble Mg (mmol(+) kg^{-1})	0.1 ± 0.00	0.3 ± 0.01	0.6 ± 0.01	0.3 ± 0.01
Exchangeable K (mmol(+) kg^{-1})	70.2 ± 0.59	94.6 ± 1.23	136.9 ± 1.36	100.5 ± 3.18
Exchangeable Na (mmol(+) kg^{-1})	346.6 ± 5.60	230.9 ± 3.50	190.6 ± 2.20	256.0 ± 11.32
Exchangeable Ca (mmol(+) kg^{-1})	464.5 ± 5.20	177.6 ± 2.80	145.9 ± 0.80	262.7 ± 8.83
Exchangeable Mg (mmol(+) kg^{-1})	2.3 ± 0.05	11.5 ± 0.26	23.2 ± 0.26	12.3 ± 0.57
CEC (mmol(+) kg^{-1})	883.6 ± 14.15	514.6 ± 8.80	496.6 ± 5.89	631.6 ± 28.85

3.2. Total element composition

Total content of major elements contained in the red mud samples of different ages was determined by XRF. It can be seen from Table 2 that the red mud consisted of many elements. Among these elements, O accounted for about 40%. Other major elements included Ca, Fe, Si, Al, Ti, Na, C, Mg and K. The red mud also contained some S, Cl and P. Most of the heavy metals were under detection limits and only a few heavy metals (Cr, Zn, Ba and Mn) were detected in trace amounts in the red mud.

3.3. Mineral composition

XRD analysis shows that the red mud was mainly composed of calcite, perovskite, illite, hematite and magnetite. The old red mud also contained some kassite and portlandite. In addition, there were about 20% of amorphous materials in all red mud samples (Table 3). Based on XRF results above, red mud contained about 9.41% of Si and 4.39% of Al. However, except for illite, these two elements are not parts of the above-mentioned minerals. The amounts of these two elements contained in illite were much less than those estimated by XRF analysis. Therefore, it is likely that the amorphous materials consisted mainly of Si and Al oxides.

It is also interesting to note that there is a trend that the content of magnetite decreased while the content of hematite increased with increasing storage time. This suggests that conversion of magnetite to hematite may occur during the period of red mud storage.

3.4. Micromorphological characteristics

SEM photographs (Fig. 1) show that the particles of fresh red mud was in poorly-crystallized or amorphous forms, while the 10-year-old red mud contained crystallized grains. This indicates that some mineral phase (probably mainly calcite) contained in the red mud undergoes crystallization with time after its disposal of in the storage facility.

3.5. Thermal gravimetric analysis

The TGA diagram of the fresh red mud sample shows three steps for the weight loss. The first one occurred in the range of 20–105 °C (weight loss was about 3% of the total weight), corresponding to the evaporation of physically adsorbed water; the second one occurred in the range of 105–450 °C (weight loss was about 3.6% of the total weight), corresponding to loss of

Table 2
Total element composition (%) of red mud samples of different ages

Element	Fresh	5-year-old	10-year-old	Mean
C	1.60	1.60	1.60	1.60
O	40.5	39.7	40.8	40.3
Na	3.76	1.76	1.39	2.30
Mg	1.02	0.61	1.35	0.99
Al	5.96	3.03	4.2	4.39
Si	8.74	9.31	10.2	9.41
P	0.122	0.143	0.116	0.13
S	0.413	0.141	0.362	0.31
Cl	0.0332	0.0087	0.0174	0.0197
K	0.873	0.685	0.912	0.82
Ca	22.6	29.7	26.4	26.2
Ti	3.12	4.03	4.04	3.73
Cr	0.0415	0.0545	0.0685	0.0548
Mn	0.0417	0.044	0.0455	0.0432
Fe	10.80	8.75	7.95	9.17
Zn	0.0041	0.0041	0.0126	0.0069
Rb	0.0364	0.0585	0.0512	0.0479
Sr	0.105	0.125	0.100	0.110
Zr	0.151	0.206	0.206	0.187
Nb	0.0125	0.0178	0.0153	0.0152
Ba	0.0242	0.0179	0.0217	0.0212
Ce	0.0260	0.0179	0.0220	0.0220
Pb	0.0182	0.0174	0.0198	0.0185
Th	0.00901	0.00108	0.00953	0.00655

Table 3
Mineral composition (%) of the red mud samples of different ages

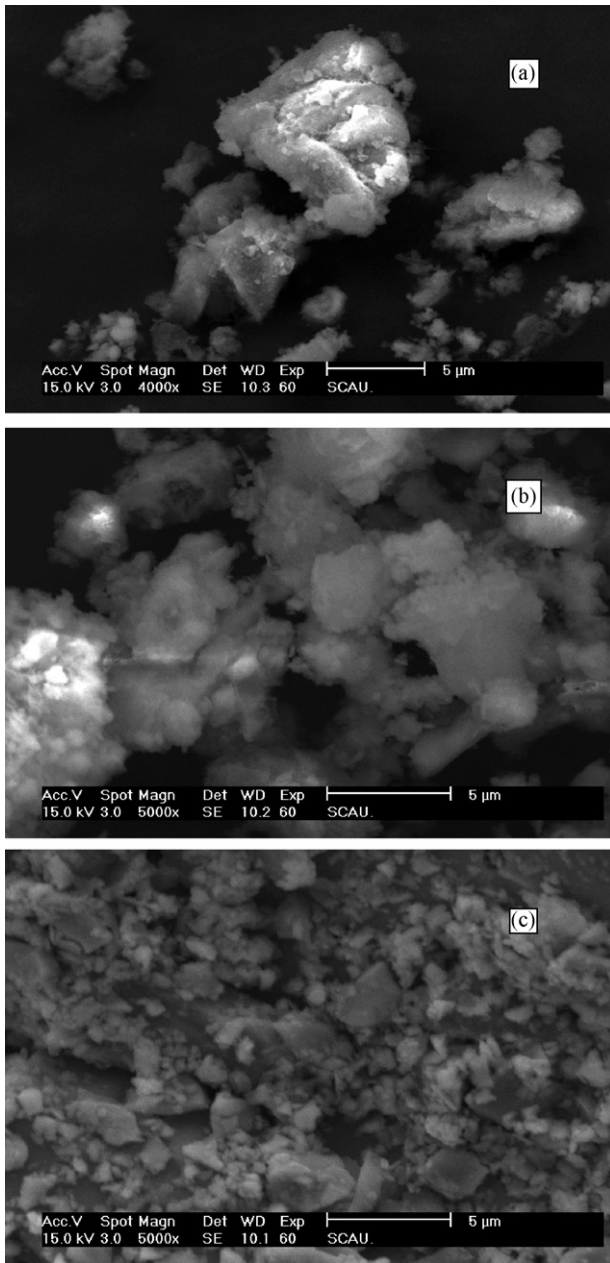| Mineral	Fresh	5-year-old	10-year-old	Mean ± S.D.
Calcite	46.8	32.8	46.3	41.9 ± 7.94
Perovskite	10.2	10.9	11.5	10.9 ± 0.65
Magnetite	8.0	7.8	0	5.27 ± 4.56
Hematite	7.4	8.2	6.7	7.43 ± 0.75
Illite	3.2	6.7	10	6.63 ± 3.40
Kassite	0	8.7	2.5	3.73 ± 4.47
Portlandite	0	0	2.3	0.77 ± 1.33
Amorphous materials	20.8	24.6	20.7	22.0 ± 2.22

Fig. 1. SEM photographs showing micromorphological characteristics of (a) fresh, (b) 5-year-old, and (c) 10-year-old red mud samples.

Fig. 2. TGA diagram showing weight loss of the red mud samples in the temperature range of 20–880 °C.

chemically adsorbed water; and the third one occurred in the range of 560–720 °C (weight loss was about 4.3% of the total weight), possibly corresponding to release of CO_2 during calcination of calcite (Fig. 2a). The TGA diagrams of the old red mud samples show similar pattern to that of the fresh red mud. However, there is a trend where weight loss in the range of 20–450 °C decreased with increasing age of the red mud (Fig. 2b and c). This indicates that dehydration of red mud has taken place since it was disposed of in the red mud impoundments. In addition, it is also clear that the temperature value corresponding to the maximum weight loss in the highest temperature range increased with increasing age of the red mud, suggesting that more energy was needed for the thermal decomposition of better crystallized calcite contained in the older red mud, compared to the poorer
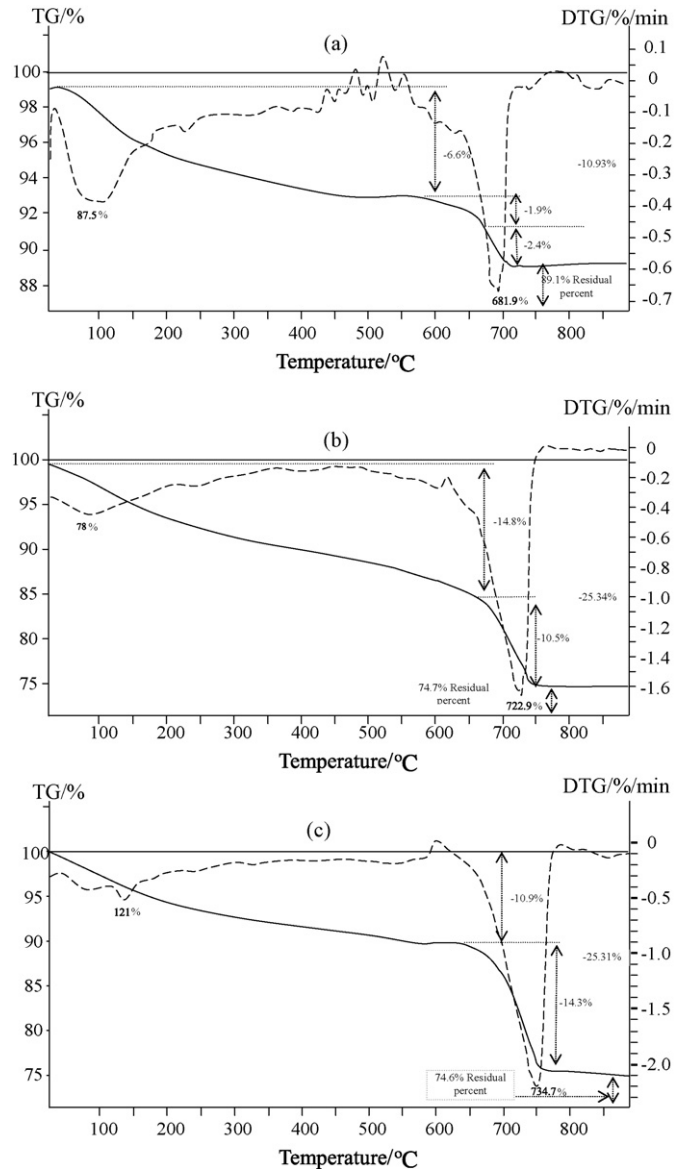
crystallized calcite in the younger red mud. The increased thermal stability of red mud over time suggests that the fresh red mud is more reactive than the old red mud. Nevertheless, even for the 10-year-old red mud, the temperature range (630–760 °C) corresponding to the decomposition of calcite occurred at a lower region, relative to that for natural limestone (850–1000 °C) [26]. This suggests that calcite in red mud may be more reactive than that in limestone when being used for neutralizing acid materials.

3.6. Acid neutralizing capacity

Slow titration of the red mud samples with either standardized H_2SO_4 or HCl solutions for a period of about 2 years indicates that the fresh red mud had an ANC of about 10 mol kg^{-1} (Fig. 3). In general, The ANC of the fresh red mud was slightly higher than that of the old red mud. This is attributable to partial removal of NaOH in the old red mud by leaching during rainfall events.
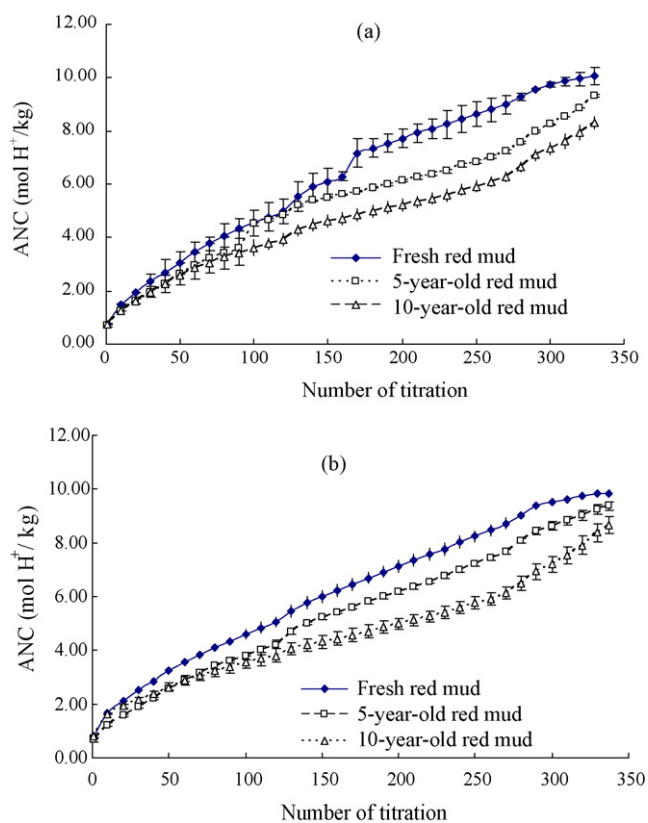
Fig. 3. Accumulative curves of ANC for red mud samples of different ages; ANC obtained by slow titration of a red mud sample with (a) standardized $0.1 \, mol \, L^{-1}$ HCl solution and (b) standardized $0.05 \, mol \, L^{-1}$ H_2SO_4 solution (35 points plotted were selected from a total of 367 data points obtained from the 2-year titration experiment).

After the first titration with HCl solution to pH 5.5, in the second day the pH of the red mud suspensions bounced back to 10.63 (0.95 unit lower than the original pH), 9.52 (1.04 unit lower than the original pH) and 8.90 (0.71 unit lower than the original pH) for the fresh, 5-year-old and 10-year-old red mud, respectively. After the second titration, the next-day pH further dropped to 9.23, 8.39 and 8.33 for the fresh, 5-year-old and 10-year-old red mud, respectively. In the first 11 days, the next-day pH sharply decreased for all the three red mud samples. After this period decrease in the next-day pH tended to be gentler during the remaining period of the experiment (data not shown). A similar pattern was observed for the $0.05 \, mol \, L^{-1}$ H_2SO_4 titration experiment (data not shown). These results suggest that under the experimental conditions described in this study, the rapid-reacting component of the red mud's ANC was consumed within 11 titrations. The total amounts of the red mud's rapid-reacting ANC accounted for less than 20% of the total ANC. This nature is preferable if the acidic materials that need to be neutralized contain both readily and slowly releasing acids such as in sulfidic soils [2,13].

3.7. Heavy metal binding capacity

Variation of heavy metal concentrations in the filtrates during the 107 filtrations can be seen from Fig. 4. Similar pattern
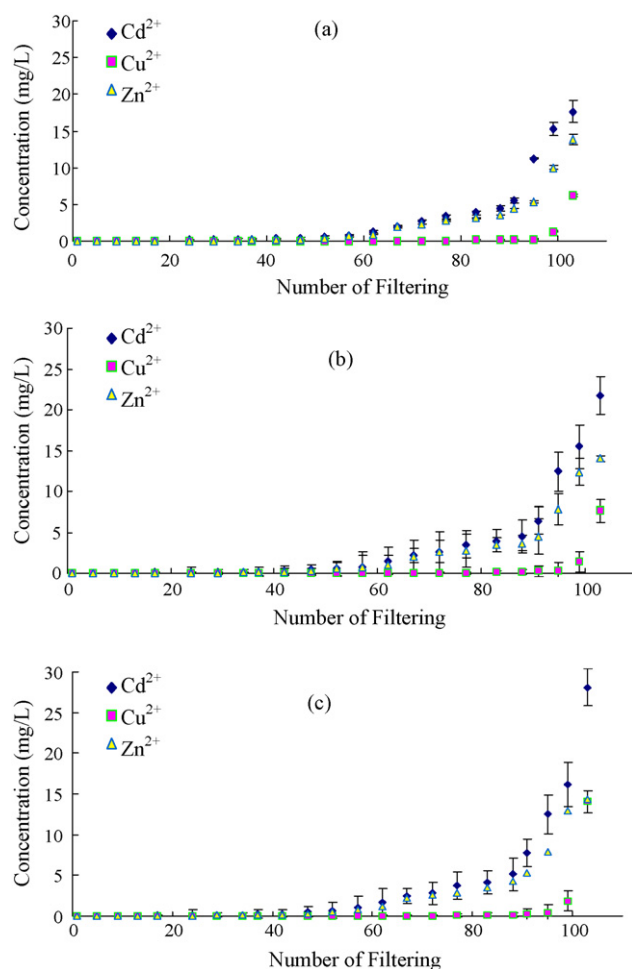

Fig. 4. Variation of filtrate Cu, Zn and Cd during the period of 107 filtrations using a mixed Cu, Zn and Cd solution as a filtering liquid, and (a) the fresh red mud, (b) the 5-year-old red mud, and (c) the 10-year-old red mud as filtering media.

is observed for all the three red mud samples of different ages. For the first 50 filtrations, Cd, Zn and Cu were not detected from the filtrates, indicating that all of the heavy metals originally contained in the filtering solutions were completely retained by the red mud samples. Concentration of Cd and Zn in the filtrates gradually increased from the 50th to the 90th filtration and then suddenly increased until the last filtration (107th filtration). However, concentration of Cu did not significantly increased until the 90th filtration. At the last filtration, the concentration of Cd, Zn and Cu in the filtrate was $17.63 \, mg \, L^{-1}$, $13.90 \, mg \, L^{-1}$ and $6.22 \, mg \, L^{-1}$ for the fresh red mud; $21.90 \, mg \, L^{-1}$, $13.99 \, mg \, L^{-1}$ and $7.64 \, mg \, L^{-1}$ for the 5-year-old red mud; and $28.11 \, mg \, L^{-1}$, $14.38 \, mg \, L^{-1}$ and $14.05 \, mg \, L^{-1}$ for the 10-year-old red mud. Based on these data, it is conservatively estimated that under the experimental conditions set in this study, the simultaneous removal rates of Cd, Zn and Cu by red mud are over $22,250 \, mg \, kg^{-1}$, $22,500 \, mg \, kg^{-1}$ and $25,000 \, mg \, kg^{-1}$, respectively. This suggests that the affinity of these three metals to red mud was in the following decreasing order: $Cu > Zn > Cd$.

Table 4
Comparison of mineralogical and chemical composition between the BPBCRM and the PBPRM

Order of abundance	BPBCRM (%)	PBPRM [27] (%)
Mineral composition		
1	Calcite (42)	Imogolite (32)
2	Perovskite (11)	Hematite (19)
3	Hematite (7)	Calcite (12)
4	Illite (7)	Ilmenite (10)
5	Magnetite (5)	Rutile (3)
	Amorphous (22)	Amorphous (22)
Chemical composition		
1	CaO (41.6)	Fe_2O_3 (26.9)
2	Fe_2O_3 (12.5)	Al_2O_3 (26.8)
3	SiO_2 (19.9)	CaO (23.5)
4	TiO_2 (6.7)	SiO_2 (13.1)
5	Na_2O (2.4)	TiO_2 (7.3)
6	CO_2 (5.9)	MgO (1.1)
7	MgO (1.0)	Cr_2O_3 (0.5)
8	K_2O (0.8)	ZrO_2 (0.5)

3.8. Comparison between the BPBCRM and the PBPRM

Table 4 gives a comparison of mineralogical and chemical composition between the BPBCRM (mean of three samples of different ages) from the Zhengzhou Alumina Refinery and the PBPRM (composite sample consisting of 3 sub-samples collected from locations receiving red mud disposed of at different times) from the Pingguo Alumina Refinery. It can be seen that there were marked differences in mineralogical and chemical composition between the two red mud types. The major minerals that these two red mud types had in common are calcite and hematite. It is clear from Table 4 that the proportion of calcite in the total red mud weight was much higher in the BPBCRM than in the PBPRM while the proportion of hematite in the total red mud weight was much lower in the BPBCRM than in the PBPRM. This is also reflected in the chemical composition between the two red mud types; the BPBCRM contained much higher CaO and much lower Fe_2O_3, compared to the PBPRM.

Previous work showed that the red mud derived from the pure Bayer Process had a ANC less than $5 \, mol \, kg^{-1}$ [2], which is much lower than that obtained here from the red mud derived from a combined Bayer Process and bauxite calcination method.

4. Conclusion

It was found that pH of the red mud decreased with increasing duration of storage time. Sodium dominated among the soluble cations, but the concentration of soluble Na decreased with increasing duration of storage time as a result of leaching. CEC, as estimated from the sum of exchangeable cations, also decreased with increasing duration of storage time, probably due to a decrease in pH causing a reduction in negatively charged sites of the red mud. Calcium was the predominant exchangeable cation in the fresh red mud but the concentration of exchangeable Ca markedly decreased in the old red mud that was dominated by exchangeable Na. XRF results show that the red mud consisted mainly of O, Ca, Fe, Si, Al, and Ti. Other important elements include Na, C, Mg and K. XRD results show that the major mineral types in the red mud were calcite, perovskite, illite, hematite and magnetite. There is a trend that the content of magnetite decreased while the content of hematite increased with increasing storage time, suggesting the conversion of magnetite to hematite. SEM observations reveal that fresh red mud sample is poorly crystallized while the 10-year-old red mud sample is better crystallized. TGA results show that the temperature at which the valley of weight loss occurred increased from 681.9 °C for fresh red mud to 722.9 °C for the 5-year-old red mud to 734.7 °C for the 10-year-old red mud. This indicates that the thermal stability of red mud increased and consequently the reactivity of red mud may decrease with increasing storage time. The BPBCRM had an ANC of about $10 \, mol \, kg^{-1}$, which is much greater than that reported for the PBPRM. Column filtering experiment results indicate that the red mud also had a very strong capacity to remove Cu, Zn and Cd from the filtering solution. The affinity of these metals to the red mud was in the following decreasing order: Cu > Cd > Zn. In general, the fresh red mud sample retained more heavy metals than the old red mud samples did.

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