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The Effect of Red Mud on Sintering Processes and Minerals of Portland Cement for Roads

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Abstract: As a solid waste generated in the alumina industry, red mud poses a significant environmental hazard and a storage problem. In this study, red mud was added to road cement clinker in order to utilize it. The sintering red mud was first de-alkalized, and then mixed with fly ash, clay, limestone, and sandstone, among other materials, to make Portland cement for road clinker. The effect of the addition of red mud on the thermal decomposition characteristics of Portland cement for roads was studied. The existent states of alkali and radioactive elements in Portland cement for road clinker were investigated by XRD and SEM analysis. The research results showed that the addition of red mud in Portland cement for road raw material significantly promoted the decomposition of carbonates in raw material. The major mineral phases of Portland cement for road clinker were C_3S with a polyhedral morphology, quasi-spherical C_2S, and tubular C_4AF. A small part of the alkali combined with the silicate phase to form a solid solution, and most of the alkali combined with S to form vermiform sulfate in the intermediate phase. The radionuclide ^{226}Ra was mainly distributed in the silicate phase. ^{232}Th was mainly distributed in interstitial phases and then silicate phases, while ^{40}K was mainly distributed in the interstitial phases.

Keywords: red mud; Portland cement for road; thermal decomposition characteristics; alkali; radioactive element; existent states

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1. Introduction

Red mud is a strong alkaline solid waste that is discharged in the process of alumina production. The average discharge of red mud is 0.7–1.8 tons per ton of alumina production [1]. Red mud is considered a hazardous industrial waste due to its high alkali content, as well as its small amount of heavy metals and radioactive materials. At present, global red mud reserves are close to 3 billion tons, and the annual growth rate is 120 million tons [2]. As a large alumina production country, China produces more than 30 million tons of red mud annually, and the cumulative stock of red mud is projected to reach 600 million tons by the end of 2021. Due to the lack of economic and feasible technologies for the large-scale utilization of red mud in China, the large number of red mud deposits pose great threats to the environment. For example, on 16 September 2014, due to continuous heavy rains in Zhengzhou, a local collapse occurred in the No. 2 dam of the No. 5 Red Mud Reservoir of the Henan Branch of China Aluminum Corporation [3]. The flood of red mud damaged houses in a nearby village and polluted farmland. Therefore, the utilization of red mud in a comprehensive, efficient, and harmless manner has become urgent and could promote the healthy development of the alumina industry and be crucial for environmental protection.

Construction material industries, including wall material preparation, cement production, and concrete admixture consume a large amount of industrial waste. According to aluminum smelting methods, red mud can be divided into Bayer process red mud, sintering red mud, and joint process red mud [4]. The basic process to obtain sintering red mud is as follows: first, the bauxite raw material is mixed with a certain amount of sodium carbonate to be calcined in a kiln at high temperature to convert it into sodium aluminate, sodium ferrite, sodium orthosilicate, and calcium titanate. Then, the calcined mixture is dissolved, crystallized, and calcined to obtain alumina. Finally, the separated waste residue after dissolution is sintering red mud. Due to containing more active mineral components (such as $2CaO{\cdot}SiO_2$) in red mud, which is produced by the sintering and joint methods, most red mud is usually applied in building materials [4]. Sintering red mud contains large quantities of SiO_2, Al_2O_3, Fe_2O_3, CaO, and silicate minerals. A considerable number of research studies have been carried out on the application of red mud in cement [5–10]. However, because of the high alkali content, composition fluctuation, and high water content of red mud, alkali aggregate reactions are very likely to occur in the application of cement. The alkali in sintering red mud is mainly sodium alkali and potash alkali. K mainly exists as attached alkali, and Na mainly exists as attached alkali and in a chemically bound state. The attached alkali mainly exists as soluble chemical substances, such as Na_2CO_3, $NaAlO_2$, KOH, K_2CO_3, and $NaHCO_3$, which can be removed by washing with water. The chemically bound Na alkali mainly exists in the states of $NaAlSiO_4$, $Na_2O{\cdot}Al_2O_3{\cdot}2SiO_2$, $Na_2O{\cdot}Al_2O_3{\cdot}2SiO_2{\cdot}xH_2O$, and $Na_2O{\cdot}CaO{\cdot}SiO_2$.The alkali aggregate reaction occurring between red mud and cement is the more common and typical alkali–silica reaction. The atmospheric pressure lime method mainly refers to the removal of the bound alkali in the red mud. The typical reaction is the replacement reaction of calcium and sodium. The specific reactions are as follows [11]:

$$CaO + H_2O \rightarrow Ca(OH)_2. \tag{1}$$

$$Na_2O{\cdot}Al_2O_3 + Ca(OH)_2(aq) \rightarrow CaO{\cdot}Al_2O_3{\cdot}6H_2O + NaOH. \tag{2}$$

$$Na_2CO_3 + Ca(OH)_2(aq) \rightarrow CaCO_3 + 2NaOH. \tag{3}$$

$$Na_2SO_4 + Ca(OH)_2(aq) \rightarrow CaSO_4{\cdot}2H_2O + 2NaOH. \tag{4}$$

$$Na_2SiO_3 + Ca(OH)_2(aq) \rightarrow CaO{\cdot}SiO_2 + H_2O + 2NaOH. \tag{5}$$

$$Na_2O{\cdot}Al_2O_3{\cdot}2SiO_2{\cdot}xH_2O + Ca(OH)_2(aq) \rightarrow CaO{\cdot}Al_2O_3{\cdot}2SiO_2{\cdot}xH_2O + 2NaOH. \tag{6}$$

The radioactivity of red mud also affects its application in cement. If the dosage of red mud exceeds a critical value, the radiation intensity of its manufactured products will not meet the requirements by national standard sand cause damage to the surrounding environment and human health.

In this paper, red mud was dealkalized and used as a raw material for Portland cement. The influence of red mud on the thermal decomposition characteristics of the cement was studied by TG-DTA, SEM-EDS, γ-ray spectrometry and X-ray diffraction. Furthermore, the alkali and radioactive elements in the road cement clinkers were analyzed. This research lays a theoretical foundation for the large-scale application of red mud in cement production.

2. Materials and Experimental Methods

2.1. Materials

Red mud was taken from the red mud sintering process of the Zhengzhou Branch of China Great Wall Aluminum Industry. The XRD patterns of red mud from the sintering process are shown in Figure 1. The alkali content of the red mud was high, which limited the dosage that could be used in cement. Therefore, the sintering red mud needed to be dealkalized first. The red mud was dealkalizedusing the atmospheric pressure lime method. The dealkalizer was industrial lime. The dosage of industrial lime was 10 wt.%, and the liquid-to-solid ratio was 3:1. The dealkalizing process was performed as follows: first, the red mud, industrial lime, and water were mixed; then, the mixture was heated to 90 °C and allowed to react for 7 h; finally, the dealkalized red mud was obtained after filtration and drying.

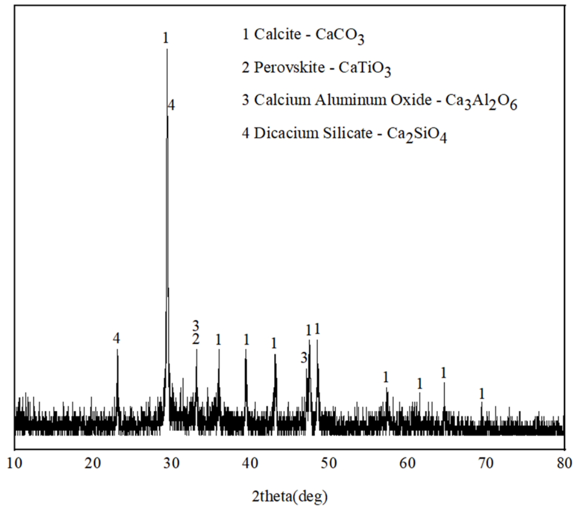

Figure 1. The XRD patterns of red mud from the sintering process.

The chemical composition of dealkalized red mud is shown in Table 1. Limestone, sandstone, and fly ash were obtained from Tianrui Group Zhengzhou Cement Co., Ltd. Their chemical compositions are also shown in Table 1. Other materials, including iron oxide and KOH, were pure reagents for chemical analysis.

Table 1. Chemical composition of raw materials (wt.%).

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	Loss on Ignition	Others
Sintering red mud	17.33	6.91	13.23	30.33	1.47	1.21	2.33	0.92	18.70	7.57
Dealkalized sintering red mud	15.95	6.34	12.50	34.35	1.76	0.50	0.30	0.86	24.50	2.94
Limestone	4.47	1.71	0.73	55.35	1.37	0.64	0.07	0.18	35.18	0.30
Sandstone	89.37	2.55	2.27	0.59	0.23	0.77	0.03	0.02	3.79	0.38
Fly ash	52.37	28.80	5.56	2.89	2.02	0.54	1.45	—	3.69	2.68
Clay	67.31	14.50	5.55	1.95	1.77	1.23	1.11	—	4.87	1.71

2.2. Preparation of Portland Cement for Road

The actual batching scheme used for Portland road cement is shown in Table 2. KBS is a kind of Portland road cement according to Chinese National Standard GB 13693-2005 (Portland Cement for Road), which was used as a blank control group without red mud. In order to study the physical and mechanical properties of Portland road cement, according to Chinese National Standard GB/T17671-1999 (Testing Method of Cement Mortar Strength), a TYE-200B cement mortar flexural and compressive machine was employed to test the 3 day and 28 day compressive and flexural strength of the test block. According to Chinese National Standard GB/T1346-2001 (Standard Test Method for Water Consumption, Setting Time, and Stability of Cement) and Chinese National Standard JC/T603-2004 (Dry Shrinkage Test Method of Cement Mortar), the water consumption at standard consistency, setting time, and dry shrinkage performance of each group of cement were tested.

Table 2. Composition of the designed clinker of Portland cement for roads.

Samples	Raw Material Content						Clinker Rate Values		
	Limestone (wt.%)	Dealkalied Red Mud (wt.%)	Sandstone (wt.%)	Clay (wt.%)	Fly Ash (wt.%)	Iron Oxide (wt.%)	KH	SM	IM
S1	68.31	23.00	6.90	0.00	1.79	0.00	0.90	1.97	0.96
S2	66.00	26.00	6.21	0.00	1.79	0.00	0.90	1.82	0.90
S3	64.41	28.00	5.80	0.00	1.79	0.00	0.90	1.73	0.87
KBS	83.41	0.00	2.90	9.50	1.79	2.40	0.90	1.95	0.96

The physical and mechanical properties of Portland cement are shown in Table 3, and the stability of each group of cement is qualified. All kinds of raw materials passed an 80 micron square-hole sieve. According to Chinese National Standard GB 13693-2005 (Portland Cement for Road), the compressive and flexural strengths of 42.5 Portland road cement aged for 3 days were 21 MPa and 4.0 MPa, respectively. Additionally, the compressive and flexural strengths of 42.5 Portland road cement aged for 28 days were 42.5 MPa and 7.0 MPa, respectively. As it can be seen from Table 3, the shrinkage decreased with the increase in red mud fraction from S1 to S3, the reason being that the C_4AF in Portland road cement increased with the increase in red mud fraction. The S3 cement had a long initial setting time and a poor compressive strength, the reason for this phenomenon being that excessive red mud content led to excessive liquid phase in the S3 clinker sintering system, which affected the formation of C_3S. The content of C_3S in S3 clinker was very small, and the content of C_2S was great. This resulted in S3 cement having a longer setting time and poor compressive strength.

Table 3. Physical and mechanical performance of Portland cement for roads.

Samples	Compressive Strength (MPa)			Flexural Strength (MPa)			Standard Consistency Water Consumption (%)	Setting Time (h:min)		28 day Dry Shrinkage (%)
	3 days	7 days	28 days	3 days	7 days	28 days		Initial Setting Time	Final Setting Time	
KBS	34.20	43.17	53.15	6.02	7.72	8.17	28.4	1:22	3:37	0.048
S1	26.80	38.30	57.50	6.17	7.95	8.78	27.4	1:32	3:20	0.043
S2	27.40	39.20	55.30	6.27	7.90	8.45	27.4	1:36	3:17	0.037
S3	18.00	29.20	39.95	4.67	6.67	7.33	27.2	2:15	3:49	0.033

2.3. X-ray Diffraction of Sintering Red Mud and Portland Cement for Roads

XRD analysis was conducted to analyze the phase of sintering red mud and Portland road cement clinker using an Uitima IV X-ray diffractometer with a Cu-Kα radiation source. The measurements range of 2θ values was 10–80°.

2.4. TG-DSC Analysis of Portland Cement for Roads

TG-DSC was performed using a PYRIS series comprehensive thermal analyzer. The heating rate was 25 °C/min. The max temperature was 1000 °C. The atmosphere was air, and the sample mass was 5 mg.

2.5. Experiments on Microstructure of Cement Clinker and Occurrence State of Alkali and Radioactive Elements Therein

In order to study the influence of radioactive elements (^{226}Ra, ^{232}Th, and ^{40}K) in red mud on the radioactivity of Portland road cement, according to the method specified in Chinese National Standard GB6566-2001 (Radionuclide Limit of Building Materials), the specific activities of ^{226}Ra, ^{232}Th, and ^{40}K in red mud road Portland cement were measured using an FP90041B low-background multichannel gamma spectrometer. The resolution of the gamma spectrometer was 2.1 keV, and the integrated background count rate was 120 min^{-1}. The safety performance of red mud road silicate cement was evaluated. Calculations referenced formulas of internal and external illumination index (Equations (1) and (2), respectively).

$$I_r = \frac{C_{Ra}}{370\ \text{Bqkg}^{-1}} + \frac{C_{Th}}{260\ \text{Bqkg}^{-1}} + \frac{C_K}{4200\ \text{Bqkg}^{-1}}, . \tag{7}$$

$$I_{Ra} = \frac{C_{Ra}}{200}, \tag{8}$$

where I_{Ra} is the internal radiation index, I_r is the external radiation index, and C_{Ra}, C_{Th}, and C_K are the specific radioactivity of radioactive elements ^{226}Ra, ^{232}Th, and ^{40}K in the sample, respectively.

In order to study the occurrence state of radioactive elements in red mud of Portland cement for road clinker, S2 clinker with a high red mud content and good physical and mechanical properties was selected. According to the principle of selective dissolution and the method proposed in [12,13], 5.0 g of S2 clinker with fineness less than 8 μm was ground to extract the silicate phase using sucrose-KOH solution. A volume of 300 mL of distilled water was added to a 500 mL beaker, heated to 95 °C, and continuously stirred. Then, 30 g of sucrose, 30 g of KOH, and 5 g of fine cement clinker were added successively. After continuous stirring at 95 °C for 30 min, the silicate mineral phase (CxS) was obtained by vacuum filtration using slow filter paper and a Brinell funnel, after which the residue was repeatedly washed with anhydrous ethanol and transferred to the surface dish. After drying to constant weight at 80 °C, the silicate mineral phase (CxS) was obtained and sealed. These operations were repeated to obtain a silicate phase weighing 200 g. The clinker and the extracted silicate phase were analyzed by XRD using an Ultima IV X-ray diffractometer. The radioactivity of the clinker and extracted silicate mineral phase were measured using anFP90041B low-background multichannel gamma spectrometer. Lastly, a JSM-7800F thermal field-emission scanning electron microscope was used for surface scanning of clinker to find the distribution of radioactive elements ^{226}Ra, ^{232}Th, and ^{40}K.

3. Results and Discussion

3.1. Effect of Red Mud on Thermal Decomposition Characteristics of Portland Cement for Use as a Raw Material in Roads

In order to study the effects of red mud on the thermal decomposition characteristics of Portland road cement raw material and the sintering process of Portland road cement clinker, the formula and blank samples of three groups of red mud were subjected to TG-DSC analysis. The decomposition temperatures of these samples were determined; the results of TG-DSC are shown in Table 4 and Figure 2.

Table 4. Thermal analysis results of Portland road cement clinker.

Sample	Decomposition Temperature (°C)			Weight Loss Rate (%)
	Initial Decomposition Temperature	Fastest Decomposition Temperature	End Decomposition Temperature	
S1	659.2	815.9	837.9	35.25
S2	653.4	804.0	827.3	35.15
S3	649.2	800.7	825.8	35.26
KBS	671.8	814.4	841.3	35.90

The results show that the initial decomposition temperature of the S1 raw material sample with 23 wt% red mud content was 659.2 °C. The temperature at which its decomposition occurred most rapidly (referred here as the fasted decomposition temperature) was 815.9 °C, and the decomposition end temperature was 837.9 °C. Compared with the blank sample of KBS, the initial decomposition temperature and the decomposition end temperature of the S1 raw material samples were lower, and the fastest decomposition temperature was similar. The decomposition temperatures of S2 raw material samples with a red mud content of 26 wt% were lower than those of S1. The decomposition temperature of the S3 raw material sample with a red mud content of 28 wt% was the lowest. In general, the addition of red mud significantly promoted the carbonate decomposition of Portland road cement raw materials. For S1, S2, and S3, with the increase in red mud content, the decomposition temperatures of raw material were significantly reduced. On the one hand, some trace components (Na, K, S, etc.) in red mud could play a role in mineralization in the sintering process of Portland road cement and promote the decomposition of carbonate in raw materials. On the other hand, the increase in red mud content could reduce the SM and IM values of the raw material. In the cement production process, it is beneficial to reduce the energy consumption of the raw material preheating process if the thermal decomposition temperature of the raw material can be reduced. Red mud is used as a raw material to prepare Portland road cement, which can reduce energy consumption during cement production and save energy.

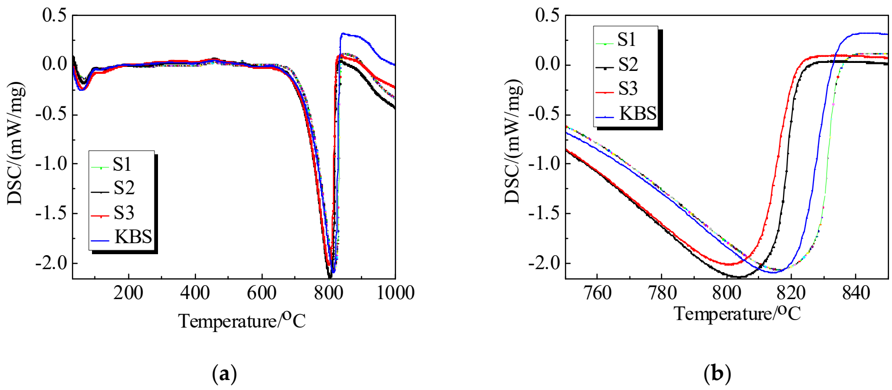

Figure 2. TG-DSC analysis curve of Portland road cement clinker: (**a**) graph over the temperature range measured; (**b**) enlarged graph over a limited temperature range.

3.2. Occurrence of Alkali in Portland Road Cement Clinker

As can be seen from Table 3, the compressive and flexural strength levels of S3 after 3, 7, and 28 days were all far lower than that of KBS. For good cement, its 28 day compressive strength should exceed 42.5 MPa; the 28 day compressive strength of S3 was 39.3 MPa. More importantly, as shown in Table 5, the external radiation index (Ir) of S3 was 1.02, which exceeded standards. Therefore, the most suitable dosage of red mud in Portland cement for roads was 26 wt.% (S2). For this reason, in order to industrialize red mud cement, the S2 sample was chosen as the object of further research.

Table 5. Results of specific radioactivity for C_XS and clinker of Portland road cement.

Sample	^{226}Ra (Bq/kg)	^{232}Th (Bq/kg)	^{40}K (Bq/kg)	I_{Ra}	I_r	Total Specific Activity (Bq/kg)
KBS	38.3	31.3	308.5	0.19	0.30	378.1
S1	108.6	94	304.8	0.54	0.73	507.4
S2	136.3	126.2	309.1	0.68	0.93	571.6
S3	151.3	138.9	311.7	0.76	1.02	601.9
C3S, C2S	311.1	91	58.1	1.6	1.2	460.2

Figure 3 shows the SEM-EDS analysis results of Portland road cement clinker in group S2. According to Chinese National Standard GB 13693-2005 (Portland Cement for Road), the content of C_4AF was more than 16 wt.% and that of C_3A was less than 5 wt.%. From the SEM photos of the clinker, it can be seen that the main mineral composition of Portland road cement clinker was C_3S, C_2S, and C_4AF.

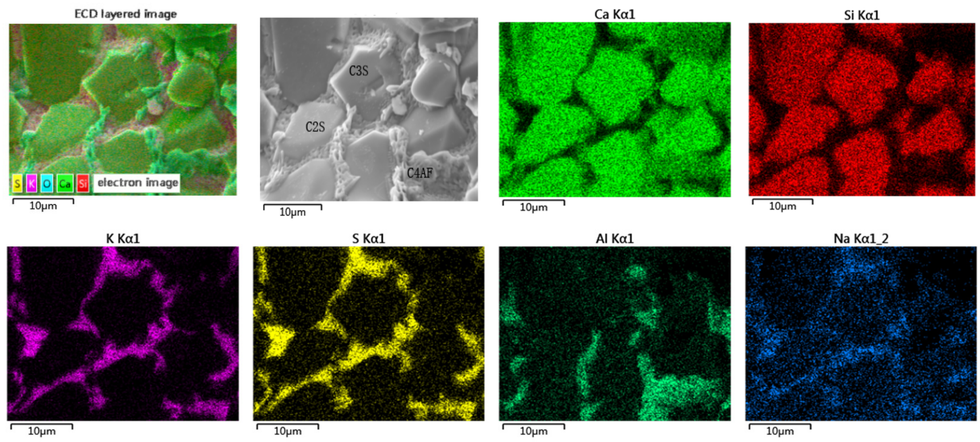

Figure 3. The SEM map scanning images of Portland road cement (×1000).

From the SEM-EDS map scanning results of Portland road cement clinker shown in Figure 3, it can be seen that the distribution shapes of the K element and S element were consistent, and that the distribution of K in other locations was very small. This indicated that K was mainly distributed in the intermediate phase in the form of sulfate formed by binding with S. The distribution of Na in the intermediate phase was greater, and it was also uniformly distributed in the silicate phase. The surface distribution image was basically consistent with that of S, indicating that more Na combined with sulfur to form sulfate, and the remaining Na existed in the silicate phase in a solid solution. In summary, potassium in red mud Portland road cement clinker mainly existed in the intermediate phase in the form of sulfate. The distribution of Na was relatively uniform. More Na existed in the intermediate phase in the form of sulfate, and the remaining Na existed in the silicate phase as a solid solution. Additionally, as can be seen in Figure 3, Al was mainly distributed in the intermediate phase in the form of C_4AF.

3.3. Occurrence of Radioactive Elementsin Portland Road Cement Clinker

Figure 4 [14] illustrates the results of the XRD analysis of Portland road cement clinker before and after extraction. The clinker in the XRD pattern was S2. The C_4AF diffraction peaks of Portland road cement clinker mineral disappeared after extraction and were only present in the silicate phase. This indicates that the main mineral composition of the extracted clinker was the silicate phase. Table 5 shows the specific activity of Portland road cement clinker and the extracted silicate phase. The extracted silicate phase was compared with S2 clinker; ^{226}Ra specific activity increased by 174.8 Bq/kg and specific activity of ^{232}Th decreased by 35.2 Bq/kg. This shows that, in Portland road cement linker, ^{226}Ra was mainly distributed in the silicate mineral phase, and ^{232}Th was present in the greatest quantity in the intermediate phase. However, ^{40} K was mainly distributed in the intermediate phase. The external radiation index (Ir) and internal radiation index (I_{Ra}) of KBS group were 0.30 and 0.19, respectively. Compared with the KBS group without red mud, the radioactivity of the clinker with red mud was much higher. From S1 to S3, the radioactivity of cement clinker increased with increasing red mud content. According to the method specified in Chinese National Standard GB6566-2001 (Radionuclide Limit of Building Materials), the radioactivities of natural radionuclides should be satisfied simultaneously, i.e., $I_{Ra} \leq 1$ and $I_r \leq 1$, in building materials. The external radiation index (I_r) of S3 was 1.02, which exceeded standards.

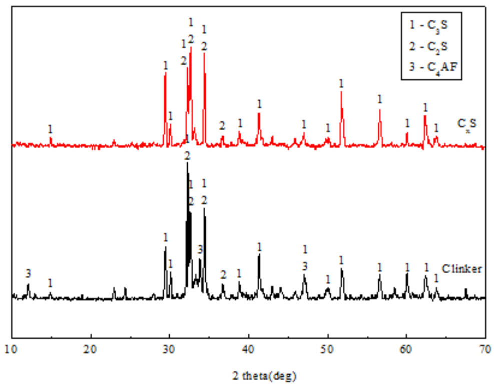

Figure 4. X-ray diffraction of Portland road cement clinker and CxS.

Ra is a typical alkaline-earth metal element, belonging to the same main group as Ca and Ba; therefore, their chemical properties are very similar. The effect of Ba ion doping on the mineral phase of cement clinker was studied in the literature [15–18], and the results showed that Ba was mainly dissolved in Belite by replacing Ca in an isomorphic manner; Ra can also be dissolved in Belite by replacing Ca as Ba did. However, Th only has a valence state of +4, which is stable even in an oxidation environment. It is difficult to incorporate in nature, but it can be incorporated via adsorption on iron oxide. The distribution of Th in the cement's mineral phase was similar to that of Fe, which was mainly distributed in the C_4AF phase, indicating that some Th will be incorporated via adsorption on iron oxide during the calcination process of the clinker and form an iron solid/melt. K mainly existed in a sulfate mineral phase, and a small amount of K may have been dissolved in silicate minerals. It can also be seen from Figure 3 that K was distributed in both a silicate mineral phase and an intermediate phase in Portland road cement clinker. K was also distributed in the intermediate phase. The distribution shapes of K and S were very similar. The results also show that most of the K and S combined to form sulfates in the intermediate phase during the calcination process of the Portland road cement, and a small amount of K was dissolved in silicate minerals. Therefore, the SEM analysis results further indicated that K was mainly distributed in the intermediate phase of Portland road cement.

4. Conclusions

The thermal decomposition characteristics and mineral constituents of Portland road cement for road presented the following characteristics:

1. The thermal decomposition temperature of each group of raw materials with red mud was lower than that of raw materials without red mud. With the increase in the red mud content, the thermal decomposition temperature of Portland road cement raw materials gradually decreased. The addition of red mud significantly promoted the carbonate decomposition process of Portland road cement raw materials.
2. Alkali in Portland road cement clinker mainly existed in the intermediate phase. The distribution of Na was relatively uniform. More Na existed in the intermediate phase in the form of sulfate, and the remaining Na existed in the silicate phase in the form of a solid solution. K was mainly distributed in the intermediate phase in the form of sulfate combined with S.
3. Under the principle of maximum red mud content, the S2 group with 26 wt% red mud content had excellent physical and mechanical properties. Therefore, the optimal red mud content in Portland road cement was determined to be 26 wt%.
4. In Portland road cement clinker, Ra was mainly distributed in the silicate mineral phase in an isomorphic manner, replacing Ca solid solution in Belite. Th and K were mainly distributed in the intermediate phase.

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