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Denitrification performance of rare earth tailings-based catalysts

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Abstract: Rare earth tailings from the Bayan Obo mine are rich in rare earth, iron, and other catalytically active substances. In this study, Na_2CO_3 and $Ca(OH)_2$ were mixed with rare earth tailings, roasted, and the tailings modified by HCl-citric acid leaching and pickling to prepare high-performance rare earth tailings-based denitrification catalysts. Denitrification performance tests show that, in the temperature range 700°C~900°C, the alkali and acid co-processed modified tailings sample gave the best catalytic denitrification performance. XRD, SEM, and H_2-TPR analyses show that, compared with raw ore samples, Fe activity sites increased after alkali and acid co-treatment. Cracks and holes appeared on the surface of the sample, and the reduction temperature range was broadened. XPS analysis showed that Fe coexisted in the forms Fe^{2+} and Fe^{3+}, and Ce in the forms Ce^{3+} and Ce^{4+}. At a rare earth tailings microwave roasting temperature of 500°C, NO concentration of 500 ppm, CO/NO ratio 4:1, and reaction temperature of 900°C, the denitrification efficiency of the catalyst was optimal, at up to 96.2%. In this study, a relatively green and pollution-free method was used to prepare catalysts, which can provide reference for solving the problem of rare earth tailings accumulation.

Keywords: rare earth tailings; mineral modification; catalytic denitrification

1 Introduction

NOx is the main atmospheric pollutant produced during the combustion of fossil fuels. It is harmful to the environment and human health, so effective removal of NOx has always been a concern of industry and academia. At present, the catalysts commonly used for catalytic denitration mainly include alkalis and alkali metal oxides [1], metal oxides [2], and rare earth oxides [3]. These types of catalysts have effective denitrification properties. However, they also have high preparation costs, poor anti-toxicity qualities, and other problems. Therefore, the use of natural minerals [4,5] and metallurgical waste residues [6,7] as denitration catalysts have become a research hotspot.

Chinese rare earth reserves account for 23% of the Earth's total, ranking them first in the world. The Baotou Bayan Obo mine accounts for 83% of China's rare earth reserves. This mining area is rich in iron, rare earth, and niobium symbiotic deposits; rare earths are rich in light rare earth elements such as La, Ce, Sm, and Eu [8]. Rare earth tailings left after beneficiation of rare earth ores contain a variety of rare earth elements and transition metal elements [9]. Rare earth oxides and iron oxides [10] in tailings are common raw materials for the preparation of catalysts. After years of accumulation, the reserves of rare earth tailings have reached 200 million tons. However, due to the complex components of tailings and for technical reasons, these reserves have not been fully utilized, leading to serious waste of resources and environmental pollution [11,12]. Under the dual constraints of continuous utilization of energy and environmental governance, the secondary utilization of rare earth tailings is "extremely urgent", and the aim is to address this issue by "treating waste by waste".

Many scholars have studied various rare earth metal and transition metal denitrification catalysts. However, few studies have focused on the catalytic denitrification of rare earth-associated ores such as rare earth concentrate and rare earth tailings. The purpose of this study is to explore the preparation of rare earth tailings-based low-cost, high-efficiency denitration catalysts by using a relatively green and non-polluting method. Solutions will

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also be suggested for the tailings accumulation problem caused by the annual production of 8 million tons of tailings [13].

2 Experimental

2.1 Experiment material

The raw materials used in this experiment were rare earth tailings from the Bayan Obo mining area, Baotou, China. A quantity of rare earth tailings were crushed, ground, sieved, and then selected using a 200 mesh and reserved. The main components and content of rare earth tailings are shown in Table 1.

Table 1: The main components analysis of rare earth tailings.

Component	Fe_2O_3	CeO_2	MgO	Al_2O_3	SiO_2	La_2O_3
Content (%)	27.67	3.01	3.31	1.46	11.86	1.44
Component	CaO	MnO	Nd_2O_3	TiO_2	F	Others
Content (%)	27.20	1.96	1.10	1.00	8.92	11.07

2.2 Rare earth tailings and modification

The modification treatments of rare earth tailings included microwave roasting, alkali treatment, acid treatment, and co-treatment with alkali and acid, as shown in Table 2.

Sample 1 was rare earth tailings. Sample 2 was rare earth tailings microwave roasted at 500°C. Sample 3 was rare earth tailings mixed with 0.1 mol/L HCl and 0.01 mol/L citric acid solution, then magnetically stirred for 1 h, filtered, and washed with water; solid samples were obtained after drying at 100°C. Sample 4 was obtained by mixing rare earth tailings with Na_2CO_3 and $Ca(OH)_2$, then microwave baking, washing, and drying. Sample 5 was rare earth tailings co-treated with alkali and acid; that is, the tailings were first mixed with alkali, ground, calcined, washed with water, and then subjected to pickling and filtration; they were then filtered by water washing, and solid samples obtained after drying.

2.3 Catalyst activity detection

The catalyst activity detection system (Figure 1) comprised three parts: a mixed gas system, a reaction system, and an online gas measurement system. The gas distribution system was a gas mixing box produced by Nanjing Boyuntong Instrument Technology Co., Ltd. (model GXD 08-4E), with a measuring range from 0 to 500 SCCM and an accuracy of ±1.5%. The flowmeter utilized a mass flow meter. The reaction system was also produced by Nanjing Boyuntong Instrument Technology Co., Ltd. The riser furnace used in the experiment was a VTL 1600, and the basic parameters of the riser furnace were as follows: the heating rate was less than 10 K/min, the maximum rated temperature was 1600°C, and the rated power was 5.5 KW. The online gas measurement system utilized the Fourier infrared spectroscopy flue gas analyzer, GASMET-DX 4000, produced in Finland, to conduct online measurement of the flue gas composition, and used a computer data

Table 2: Preparation of rare earth tailing sample.

Sample number	Raw material	Solid-liquid ratio	Drying temperature	Calcination temperature	Roasting time	Test Methods
1	rare earth tailings	—	—	—	—	XRD/XPS/H_2-TPR / SEM/ Activity detection
2	rare earth tailings	—	—	500°C	15 min	XRD /H_2-TPR /SEM/ Activity detection
3	0.1 mol/L HCl + 0.01 mol/L $C_6H_8O_7$ + 5 g rare earth tailings + H_2O	1:10	100°C	500°C	15 min	BET/ XRD/ H_2-TPR / Activity detection
4	0.2 g Na_2CO_3 + 0.3 g $Ca(OH)_2$ + 5 g rare earth tailings	—	100°C	500°C	15 min	XRD/ H_2-TPR / Activity detection
5	0.2 g Na_2CO_3 + 0.3g $Ca(OH)_2$ + 0.1 mol/L HCl + 0.01 mol/L $C_6H_8O_7$ + 5 g rare earth tailings + H_2O	1:10	100°C	500°C	15 min	XRD/XPS/H_2-TPR / SEM/ Activity detection

acquisition system to record and save the information. The experimental principle of its activity is:

$$2NO + CO = N_2O + CO_2 \tag{1}$$

$$N_2O + CO = N_2 + CO_2 \tag{2}$$

$$NO + CO = N_2 + CO_2 \tag{3}$$

The total experimental gas flow rate was 500 mL/min, the concentration of NO was 500 ppm, and the concentration of CO was 2000 ppm. N_2 was the equilibrium gas. The vertical tube furnace was heated from room temperature at 10°C/min to 700°C, 750°C, 800°C, 850°C, and 900°C, and a Fourier infrared spectroscopy flue gas analyzer was used for online monitoring. Before the experiment, a certain amount of quartz cotton was weighed and placed in the heating section of the quartz tube to support the catalyst. Each test was conducted using samples of 0.5 g. When the temperature and atmosphere were stable, the NO value was recorded as $(NO)_{in}$. At this point, the quartz tube containing the sample was quickly placed in the furnace and sealed. The changes in CO and NO were measured online using Fourier infrared spectrometry and a computer data acquisition system, and the reaction time of each sample was not less than 20 min. The NO value was recorded as $(NO)_{out}$ when the gas concentration after the reaction tended to be stable. The denitrification rate was calculated using Eq. 4:

$$\eta = \frac{(NO)_{in} - (NO)_{out}}{(NO)_{in}} \times 100\% \tag{4}$$

where η is the conversion rate of NO, $(NO)_{in}$ is the concentration of NO detected after the NO catalyst was added to the reactor, and $(NO)_{out}$ is the concentration of NO detected after catalyst addition and after stabilization.

3 Results and discussion

3.1 Denitrification activity of rare earth tailings-based catalyst

The denitrification performance of CO in terms of reduction of NOx was tested on rare earth tailings before and after modification. The temperature range was 700°C~900°C, NO concentration was 500 ppm, and CO/NO ratio was 4:1. As shown in the graph (Figure 2), when the temperature was 700°C and 750°C, the denitrification rate was sample 5 > sample 3 > sample 2 > sample 1 > sample 4; at 800°C, the denitrification rate was sample 5 > sample 2 > sample 1 > sample 3 > sample 4; at 850°C, the denitrification rate was sample 1 > sample 5 > sample 2 > sample 4 > sample 3; and, at 900°C, the denitrification rate was sample 5 > sample 1 > sample 3 > sample 2 > sample 4. The denitrification rate was highest for the original tailings (sample 1) at 850°C, reaching 90.3%; however, the acid-base co-treated rare earth tailings had higher denitrification rates at the other four temperature points than the other four samples, and exhibited the best catalytic denitrification performance in the temperature range 700°C~900°C. Furthermore, at 900°C, the denitrification rate of acid-base co-processed

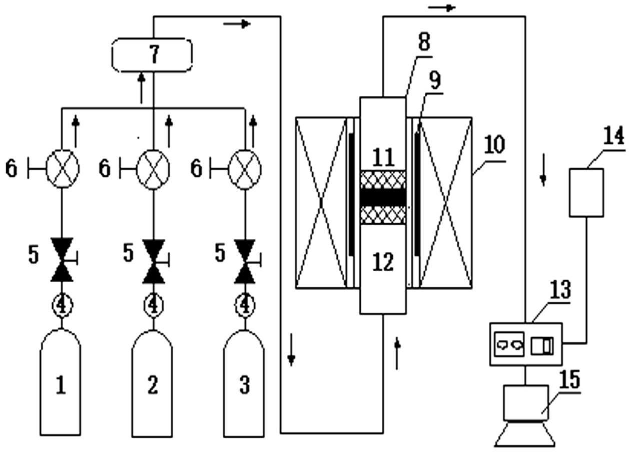


Figure 1: Reaction device schematic. 1. CO bottle; 2. NO bottle; 3. N_2 bottle; 4. Pressure gauge; 5. Pressure-reducing valve; 6. Mass flowmeter; 7. Gas mixing box; 8. Quartz tube; 9. Thermocouple; 10. Protective layer and furnace wall; 11. Catalyst; 12. Quartz cotton; 13. Fourier infrared spectrum flue gas analyzer; 14. Tail gas treatment device; 15. Computer acquisition system.

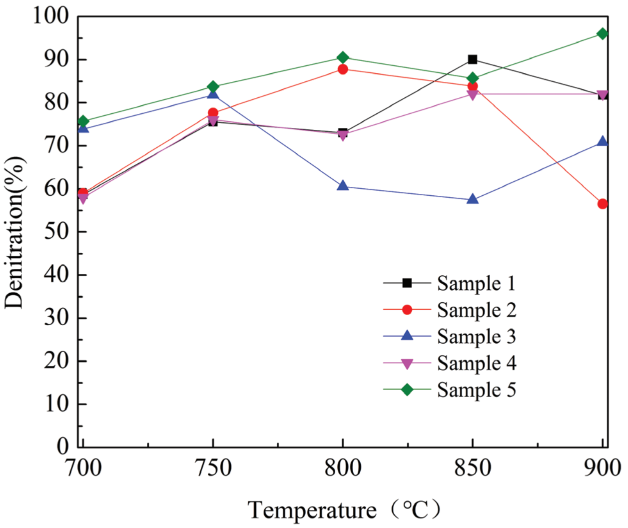


Figure 2: Denitrification efficiency before and after modification of rare earth tailings.

tailings reached 96.2%. The results show that the original tailings had a catalytic denitrification effect. And, after acid and alkali modification, the rare earth tailings catalyzed CO to reduce NOx to a greater extent.

3.2 XRD characterization of rare earth tailings-based catalysts

As can be seen from Figure 3, the main components of rare earth tailings are Fe_2O_3 and CaF_2. Among them, CaF_2 has the strongest diffraction peak intensity. This indicates that there is a certain proportion of CaF_2 in the rare earth tailings. Figure 4 shows that, after microwave roasting and acid-base treatment, the Fe_2O_3 of rare earth tailings showed new peaks, and the intensity of the diffraction peak of most Fe_2O_3 was greatly enhanced; however, the peak strength for CaF_2 and SiO_2 was greatly weakened. At the same time, rare earth tailings contain 71 different elements and 172 different minerals [14], and the components are relatively complex. XRD analysis can only analyze some of the main components of the main mineral phase, and lower content components cannot be displayed. Fe_2O_3 is an important active substance in catalytic reduction denitrification catalysts. The Fe_2O_3 content in the catalyst was increased, which is in line with the experimental design of the modified rare earth tailings as a denitrification catalyst.

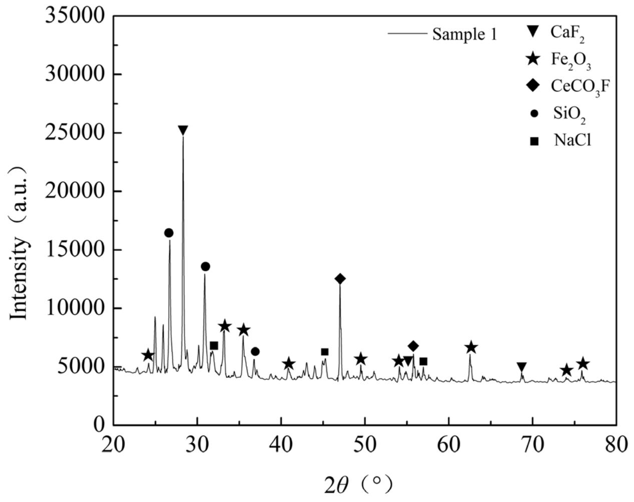


Figure 3: XRD characterization of rare earth tailings.

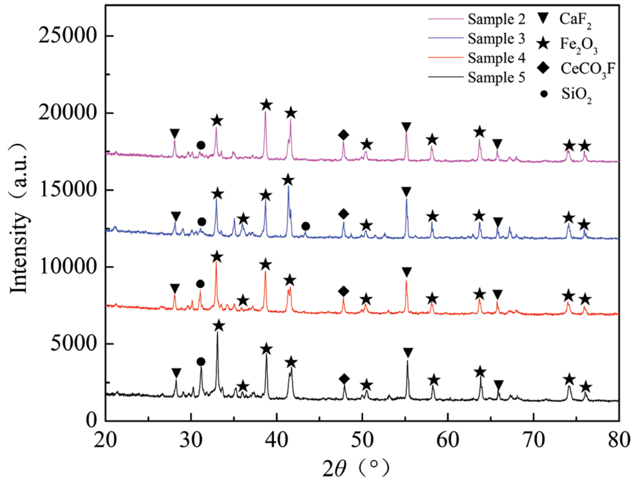


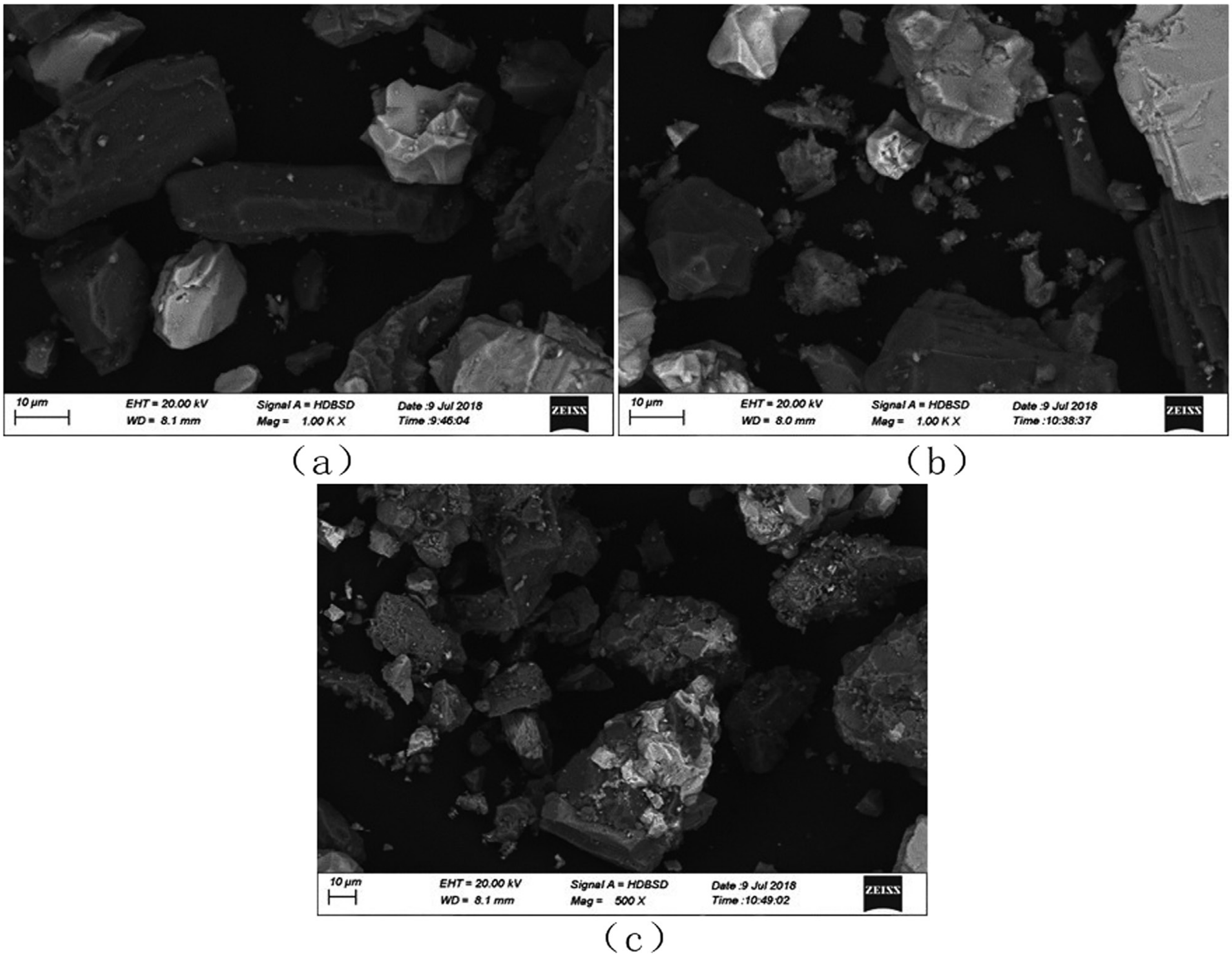
Figure 4: XRD characterization of modified rare earth tailings.

3.3 SEM characterization of rare earth tailings-based catalysts

As shown in the SEM diagram in Figure 5a, the original rare earth tailings displayed irregular blocks. The surface was smooth, and the particle size on the surface was between 4 and 45 μm. It can also be seen from the SEM diagram in Figure 5b that, after the rare earth tailings were microwave roasted at 500°C, a small number of cracks appeared on the surface, and the particle size changed slightly to between 3 and 45 μm. It can further be seen from the SEM diagram in Figure 5c that, after modification by alkali and acid, the surface was uneven, with cracks, many scrapes, and holes appearing, thus increasing the pore structure of the tailings. Microwave heating is a heating method that relies on an object absorbing microwave energy and converting it into heat energy, so heating the entire body at the same time. Materials generally absorb microwave energy to varying degrees, and the microwave energy absorbed by a material is a result of interaction between polar molecules and microwave electromagnetic fields in the material, under the action of an external alternating electromagnetic field. The polarization of polar molecules within a material is mutually frictional, thereby causing cracks in the material and increasing the pore volume. This action greatly improved the specific surface area of the tailings and, thereby, the denitrification activity of the tailings was improved.

3.4 H2-TPR analysis of rare earth tailings before and after modification

H_2-TPR experiments were carried out to investigate the ability of metal ions on the surface of modified rare earth concentrates to be reduced to low-valency metal ions,



(a) (b) (c)

Figure 5: SEM diagram of rare earth tailings. (a) Rare earth tailings; (b) Microwave-roasted tailings; (c) Alkali and acid co-treated tailings.

as well as the ability to absorb and release oxygen. The experimental conditions are: Sample of the constant temperature under 200°C for 30 min dehydration processing, and then cooled to room temperature, then, the temperature was raised from room temperature to 900°C at a heating rate of 10°C/min, and the temperature was maintained for 10 min. The results are shown in Figure 6.

As shown in Figure 6, sample 1 has two hydrogen consumption peaks at around 596°C and 859°C. For sample 2, three hydrogen consumption peaks appeared at about 714°C, 767°C and 866°C. Sample 3 showed two hydrogen consumption peaks between 500°C and 600°C, and a broad hydrogen consumption peak appeared at 856°C. Sample 4 exhibited a broad hydrogen consumption peak between 600°C and 900°C. Sample 5 showed three hydrogen consumption peaks at around 504°C, 660°C, and 859°C. It can be seen that, for the acid-base co-treated tailings, the reduction temperature range was larger than

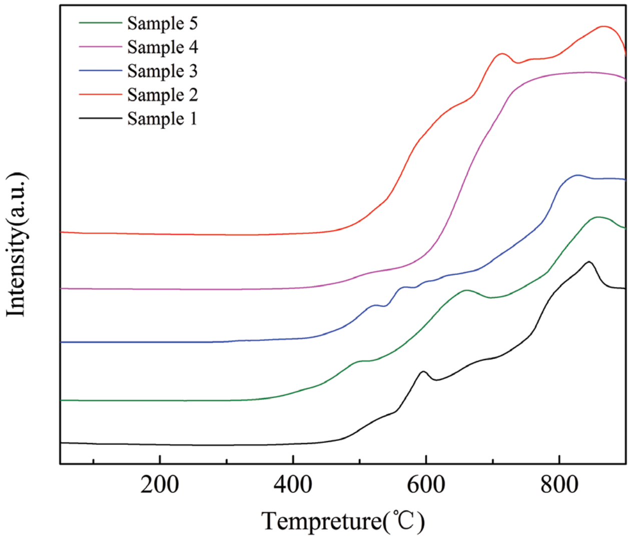


Figure 6: H_2-TPR diagram before and after modification of rare earth tailings.

for the original rare earth tailings, the roasted tailings, the acid-treated tailings, and the alkali-treated tailings. For pure CeO_2, the reduction peak appeared at 300°C~600°C, corresponding to the reduction of CeO_2 surface oxygen [15]. In summary, compared with the original tailings, after modification treatments the reduction temperature range of tailings was widened, and the reduction peak was increased. It was found that more substances in the modified tailings showed reducing ability, and the reduction temperature range of acid-base-treated tailings was the widest. This shows that modification treatment can enhance the redox capacity and oxygen storage capacity of rare earth tailings.

3.5 XPS characterization of rare earth tailings-based catalysts

XPS analysis was carried out for the elements Ce, Fe, and O on the surface of rare earth tailings and alkali and acid co-processed tailings.

Figure 7 shows the Ce 3d XPS diagram for sample 1, and Figure 8 shows the Ce 3d XPS diagram for sample 5. The Ce 3d peak was fitted according to previous reports [16]. There are two main valence states of Ce in compounds. Where v_0 (B.E. ≈ 882.2 eV), v_1 (B.E. ≈ 888.6 eV), v_2 (B.E. ≈ 898 eV), v_3 (B.E. ≈ 900.7 eV), v_4 (B.E. ≈ 907.2 eV), and v_5 (B.E. ≈ 916.15 eV), it can be seen from the standard map of Ce 3d that these six peaks correspond to the characteristic peaks of Ce^{4+}. Similarly, the four peaks of u_0 (B.E. ≈ 884.4 eV), u_1 (B.E. ≈ 880.6 eV), u_2 (B.E. ≈ 903.9 eV), and u_3 (B.E. ≈ 899.3 eV) correspond to the characteristic peaks of Ce^{3+}. The figure shows that the rare earth tailings have characteristic peaks of Ce^{3+} and Ce^{4+}, while the tailings treated with alkali and acid also have characteristic peaks of Ce^{3+} and Ce^{4+}. There is a certain number of Ce^{4+}/Ce^{3+} redox electron pairs, which are beneficial to surface oxygen storage and release, enhance the surface oxidation of the catalyst, and increase the probability of NOx adsorption on the active site.

Figure 9 shows the O 1s XPS diagram for rare earth tailings (sample 1) and the tailings treated by alkali and acid (sample 5). It can be seen from the XPS map that both the rare earth tailings and the alkali and

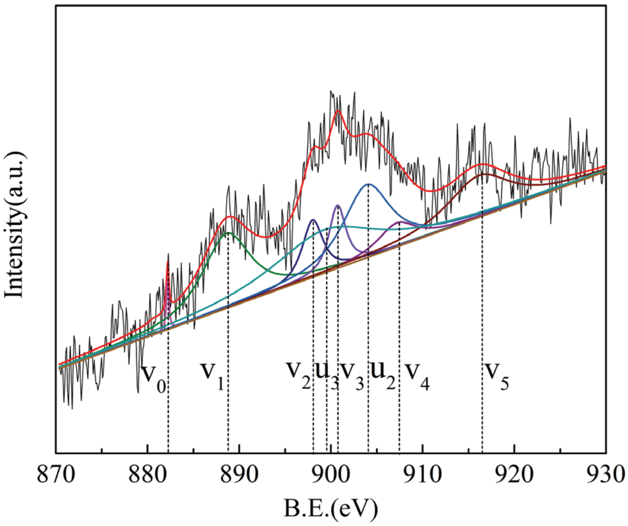


Figure 8: Ce3d XPS energy spectrum after modification of rare earth tailings.

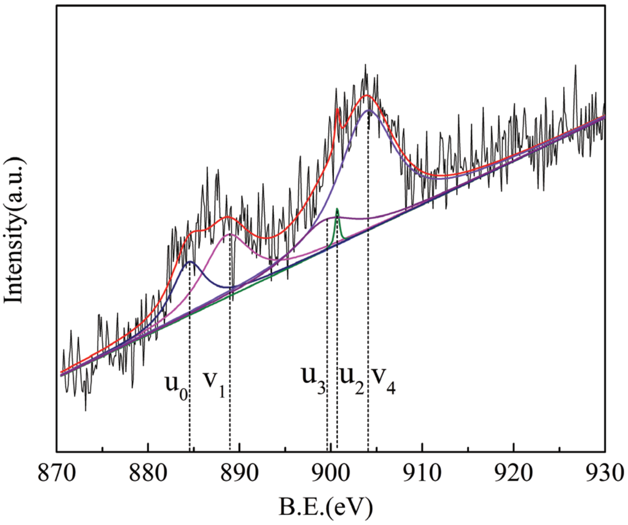


Figure 7: Ce3d XPS energy spectrum of rare earth tailings.

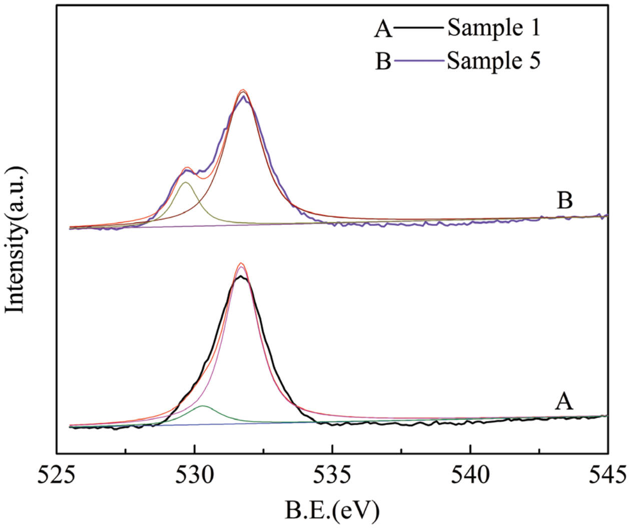


Figure 9: O 1s XPS energy spectrum before and after modification of rare earth tailings.

acid-treated rare earth tailings have two peaks. One is the peak of adsorbed oxygen at 531.5~533.0 eV, and the other is the peak of lattice oxygen appearing at 529.5~530.5 eV. The lattice oxygen content of the alkali and acid-treated rare earth tailings was significantly higher than that of the original rare earth tailings. We speculated that CeO_2 releases lattice oxygen under anoxic conditions, and the lattice oxygen is consumed to become surface-adsorbed oxygen; therefore, the Ce^{4+} portion of the CeO_2 crystal will change to Ce^{3+}, forming an O vacancy. Under oxygen-rich conditions, the oxygen adsorption becomes lattice oxygen and is stored, and Ce^{3+} once again becomes Ce^{4+}. This transformation is beneficial to surface oxygen storage and release, enhances surface oxidation of the catalyst, and increases the NOx conversion rate at the active site. This increases the probability of NOx adsorption at the active site and, therefore, the denitrification efficiency of rare earth tailings treated with alkali acid is significantly higher than that of the original rare earth tailings.

Figure 10 shows the Fe 2p XPS diagram for sample 1, and Figure 11 shows the Fe 2p XPS diagram for sample 5. For raw rare earth tailings (sample 1) and for alkali and acid-treated tailings (sample 5), two peaks appeared at binding energies of about W_1 (B.E. ≈ 711 eV) and W_2 (B.E. ≈ 725 eV); all of these peaks showed the characteristic peaks of Fe^{3+}. Sample 1 and sample 5 showed characteristic peaks at a binding energy of U (B.E. ≈ 718~721 eV); this peak represented the characteristic peak of Fe^{2+} [17]. The above results show that Fe on the surface of the catalyst coexists in the forms Fe^{2+} and Fe^{3+}, but mainly as Fe^{3+}.

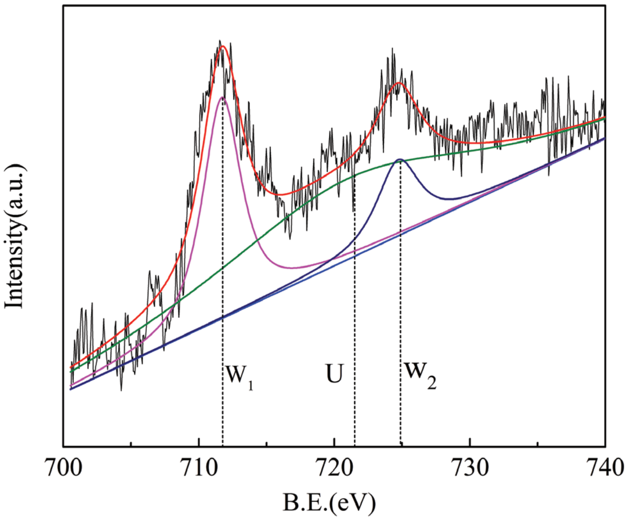


Figure 10: Fe2p XPS energy spectrum of rare earth tailings.

3.6 Analysis of catalytic process of rare earth tailings-based catalysts

As shown in Figure 12, the cyclic conversions of Fe^{3+}/Fe^{2+} and Ce^{4+}/Ce^{3+} ensure the progress of the catalytic denitrification process. Fe_2O_3 adsorbs CO molecules and reduces them to FeO. Then NO molecule then oxidizes FeO and converts it to Fe_2O_3. Meanwhile, NO is reduced to N_2. Similarly, the catalytic denitration process of cerium oxide is as follows: CeO_2 adsorbs CO molecules and reduces them to Ce_2O_3. And then, Ce_2O_3 adsorbs NO molecules and is oxidized back into CeO_2. Meanwhile, NO is reduced to N_2. Therefore, the catalytic denitration reaction can be carried out continuously.

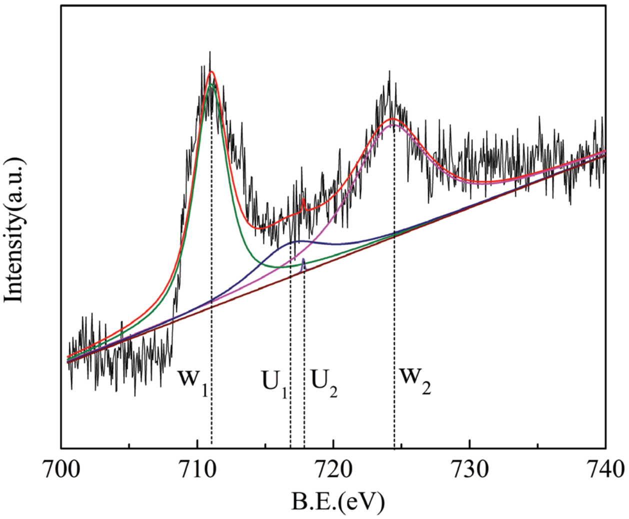


Figure 11: Fe2p XPS energy spectrum after modification of rare earth tailings.

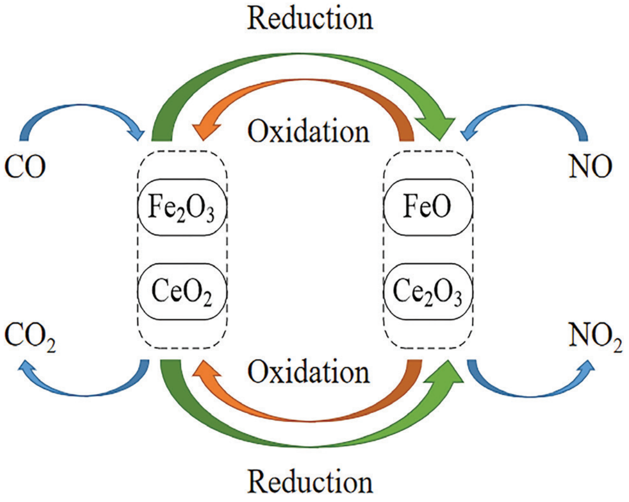


Figure 12: Schematic diagram of catalytic denitration.

4 Conclusion

In this study, the catalytic CO reduction of NO by modified rare earth tailings was investigated systematically. We can observe that the rare earth tailings have catalytic denitrification, and the rare earth tailings after alkali and acid treatment can improve the removal rate of NO to a greater extent. And the main conclusions are as follows:

(1) After alkali and acid co-modification of the tailings, when the NO concentration was 500 ppm, CO/NO ratio was 4:1, and reaction temperature was 900°C, the optimal denitrification efficiency of the catalyst treated with acid-base was 96.2%.
(2) After modification, the diffraction peak intensity of most Fe_2O_3 rare-earth tailings was enhanced, while CaF_2 and SiO_2 peak intensities were greatly weakened, and cracks and holes appeared on the surface.
(3) XPS analysis indicated that Fe content on the catalyst surface increased. In the active component, Fe coexists in the forms Fe^{2+} and Fe^{3+}, and Fe^{3+} content is higher; Ce coexists in the forms Ce^{3+} and Ce^{4+}; and lattice oxygen appeared in rare earth tailings modified by alkali and acid. Thus, oxygen vacancies carry out oxygen transfer.
(4) Modification treatment of rare earth tailings has a great influence on the redox properties of the catalyst. In general, catalysts obtained by acid-base co-treatment have the widest reduction temperature range. Furthermore, obvious reduction peaks appeared in the CeO_2 catalyst at 300°C~600°C.

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