Simultaneous Desulfurization and Denitrification Using La–Ce–V–Cu–ZSM-5 Catalysts in an Electrostatic Precipitator

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ABSTRACT: Different catalysts were loaded onto the collecting plate of an electrostatic precipitator to achieve the simultaneous removal of multiple pollutants from coal-fired gas. The synergistic desulfurization and denitrification effect of the catalyst and the effect of corona discharge on the activity of the catalyst were studied. The La(6%)–Ce(8%)–V(7%)–Cu(8%)–ZSM-5 catalyst prepared by successive impregnation methods had the optimum simultaneous desulfurization and denitrification efficiency at a roasting temperature of 600 °C. The desulfurization and denitrification rates reached 97.09 and 83.30%, respectively. BET and SEM characterization results showed that the loading of active components and additives improved the pore structure of the molecular sieve, which contributed to the high stability of the catalyst’s internal structure and large surface area, as well as better desulfurization and denitrification efficiency. Corona discharge can significantly improve the catalytic effect.

1. INTRODUCTION

The flue gas emitted from coal-fired power plants contains a large amount of particulate matter, SO2, and NOx, which pose a threat to human health and the environment.1–5 Traditional flue gas purification systems are made up of dust removal, desulfurization, and denitrification devices, which complicate the structure of the flue gas treatment system and have high operating and maintenance costs.6–12 With the continuous development and improvement of desulfurization and denitrification technologies, the trend of desulfurization and denitrification integration has gradually formed. In this way, the cost of operation and maintenance of the purification equipment is reduced, the equipment floor space is reduced, and the utilization rate of resources is improved.13–19

The integrated technology of desulfurization and denitrification is now widely studied in China.20 For example, an iron-based oxide desulfurization and denitrification catalyst was prepared by an equal-volume impregnation method, which can effectively remove SO2 and NOx in the tail gas of the petroleum industry. Using CO, H2, and CH4 as reducing agents can effectively remove SO2 and NOx in flue gas. Catalytic ozone was combined with NH3/(NH4)2S2O8 to complete the desulfurization and denitrification absorption of flue gas. Microwave and additives are used to help H2O2 solution perform desulfurization and denitrification simultaneously.21–24 Some researchers in other countries use sodium hypochlorite and potassium permanganate to form a new type of absorbent that has a high absorption efficiency for SO2 and NO. The oxidation reaction experiments are performed by loading V and Cu, between which a certain synergy exists.25,26 For the collaborative desulfurization and denitrification of the dust collector, some researchers established a 1D fluid model of simultaneous desulfurization and denitrification through a negative DC corona discharge, which is similar to an electrostatic precipitator (ESP). The nonthermal plasma produced by corona discharge has a significant effect on the removal of SO2 and NO in flue gas.27–29

In this study, different catalysts are loaded onto the dust collector of the electrostatic precipitator. The effects of different active components, different additive loads, different roasting temperatures, and corona discharge on the activity of catalysts were studied. At the same time, BET and SEM characterizations were performed to study the internal structure of the catalyst. This research aims to determine the best conditions for preparing a combined desulfurization and denitrification catalyst to provide a theoretical basis for the synergistic removal of multiple pollutants in an electrostatic precipitator.

2. EXPERIMENTAL MATERIALS AND METHODS

2.1. ESP Model. As shown in Figure 1, the experimental ESP model consists of three discharge electrodes and two dust collection electrodes placed in a 200 mm × 120 mm × 100 mm insulated box. The distance between the discharge electrode and the dust collection electrode is 40 mm. The discharge electrode is made of stainless steel with a diameter of...
2 mm and a length of 50 mm. It is connected to a negative high-voltage power supply. The size of the dust-collecting plate is 100 mm × 70 mm. The catalyst was supported on a dust-collecting plate by a metal glue-based two-component special modified epoxy-type adhesive to explore and evaluate the catalytic performance.

2.2. Catalyst Activity Evaluation System. The catalyst activity evaluation system is shown in Figure 2. This experiment was based on the flue gas produced by the actual combustion of boilers in thermal power plants, which was the experimental flue gas. The concentrations of SO₂, NO, CO₂, and O₂ were 1500 ppm, 600 ppm, 15%, and 6%, respectively. The experimental gas flow rate was 2 L·min⁻¹, and the experimental flue gas temperature was 110 °C.

2.3. Preparation of the Catalyst. The carrier of the experimental catalyst used the Na−ZSM-5 molecular sieve with a silicon−aluminum ratio of 25. The internal pore diameter of the ZSM-5 molecular sieve was uniform. The catalytic activity and stability were satisfactory at a low temperature.

The separate desulfurization catalyst was prepared by a one-step impregnation method, in which the active component was V and the auxiliary agent was La. V had high activity because most of the desulfurization catalysts and the auxiliary La can modify the V-based desulfurization catalyst, which can improve the dispersion degree of the active component V.

The separate denitriﬁcation catalyst was also prepared by a one-step impregnation method with Cu as the active component and Ce as the auxiliary agent. Cu−ZSM-5 had a very high efficiency for the conversion of NO. Ce can improve the thermal stability of the catalyst and promote the uniform dispersion of the active components. It can improve the activity of catalyst.

The combined desulfurization and denitriﬁcation catalyst was prepared by successive impregnation methods. Initially, we carried the load of the active component, then we added the previously weighed ammonium metavanadate, oxalic acid, copper nitrate, and deionized water into a beaker. Ammonia water was subsequently added to the solution to regulate the pH until the solution was transparent and homogeneous. A pretreated molecular sieve carrier (30 g) was added simultaneously. Afterward, the solution was stirred in a hot water bath until fully mixed and then cooled to room temperature after drying in a vacuum-drying oven. The cooled catalyst was placed in a ceramic crucible and baked in a muffle oven. The impregnated catalyst was reimmersed in the same V
and Cu solution, and the above-mentioned preparation process was repeated to obtain a semifinished catalyst for roasting after a second dip. Next, we loaded the auxiliary catalyst for the prepared semifinished catalyst. We first added lanthanum nitrate, cerium nitrate, and deionized water into the beaker. Then, we removed the roasted semifinished product and placed it in the beaker before adding deionized water for the solution to exceed the catalyst carrier. Then, we stirred the solution in a hot water bath until fully mixed. The solution was cooled to room temperature after drying in the vacuum-drying oven. The cooled catalyst was placed in a ceramic crucible and baked in the muffle oven. Thus, we prepared combined desulfurization and denitriﬁcation catalysts with different carrier loadings and different roasting temperatures.

Finally, experimental measurements were performed in the catalytic activity evaluation system to explore the activities of the different catalysts and to find the best working conditions for preparing these different catalysts.

2.4. Characterization of the Catalyst. In this experiment, the BET characterization was carried out using a QUADRASORB evo fully automatic specific surface and porosity analyzer produced by Conta. The catalyst was prepared into a sample, and BET analysis was carried out under different nitrogen subpressures.

A JEOL JSM-7800F Prime super-resolution field emission scanning electron microscope was used for SEM characterization. The prepared combined desulfurization and denitriﬁcation catalyst was prepared into a sample, and the morphology, particle size, and dispersion of the catalyst were observed by SEM characterization. We ﬁnally obtained a scanning electron microscopy image of the catalyst.

3. RESULTS AND DISCUSSION

3.1. Catalytic Desulfurization Experiment. 3.1.1. Effect of the Load of Vanadium and Lanthanum on the Desulfurization Catalyst. The metal and the carrier interact, which helps prevent the sintering of metal crystallites and the growth of grains. The interaction between the load catalyst and the carrier is strong. The load amounts of the active component V and the additive La, as well as the roasting temperature, will affect this interaction of the desulfurization catalyst. We ﬁrst explore the effects of V and La loading and roasting temperature on the desulfurization catalyst.

Figure 3 shows that when the loading of the active component V increased from 3 to 8%, the desulfurization efﬁciency increased ﬁrst and then decreased. When the loading was 7%, the desulfurization efﬁciency reached the maximum. The load of the active component V greatly improved the desulfurization efﬁciency. At the same time, each desulfurization efﬁciency line in the ﬁgure was similar, thereby indicating that the roasting temperature had an effect on desulfurization efﬁciency, but such an effect was limited.

After the optimal loading percentage of active component V was obtained, the loading of the auxiliary La was carried out. With increasing load of auxiliary La, as shown in Figure 4, the desulfurization efﬁciency increased ﬁrst and then decreased. However, the desulfurization efﬁciency was more than 90%, indicating that the addition of the auxiliary La greatly improved the desulfurization efﬁciency of the catalyst.

In summary, the desulfurization efﬁciency of La(6%)–V(7%)–ZSM-5 catalysts prepared at a roasting temperature of 600 °C was the highest. The efﬁciency reached 94.16%.

Table 1. Results of the Desulfurization Catalyst BET Analysis

| sample name | specific surface area (m²·g⁻¹) | average aperture (nm) |
|-------------|------------------------------|----------------------|
| V(7%)–ZSM-5 | 71.220                       | 3.345                |
| La(5%)–V(7%)–ZSM-5 | 73.480 | 3.350                |
| La(6%)–V(7%)–ZSM-5 | 75.992 | 3.348                |
| La(7%)–V(7%)–ZSM-5 | 72.100 | 3.340                |

Figure 3. Effect of V loading on desulfurization efﬁciency at different temperatures.

Figure 4. Effect of auxiliary La loading on desulfurization efﬁciency at 110 °C.
Thus, the internal stability of the catalysts increased. However, the continued increase in loading of the additive blocked the hole of the carrier and reduced the specific surface area of the support. Finally, the catalytic activity decreased.

3.2. Catalytic Denitrification Experiment. 3.2.1. Effects of Cu and Ce Loading on Denitrification Catalyst. The most ideal denitrification method is breaking NO$_x$ directly into N$_2$ and O$_2$. The ZSM-5 catalyst has better catalytic activity after the loading of some transition-metal oxides. Cu$^+$-ZSM-5, a copper-ion-exchanged molecular sieve, was efficient in directly decomposing NO. When the catalyst additive was CeO$_2$, the carrier’s stability improved and the uniform dispersion of the active components of the catalyst was promoted. Moreover, the overall activity of the catalyst improved. We wanted to explore the effects of Cu and Ce loading and roasting temperature on the denitrification catalyst.

Figure 5 shows that on increasing the load of the active component Cu from 6 to 9%, the desulfurization efficiency increased first and then decreased. When the loading was 8%, the desulfurization efficiency reached the maximum. These findings indicated that the loading of the active component Cu greatly improved the denitration efficiency. With increasing roasting temperature, the denitrification efficiency increased initially and then decreased rapidly. This finding was due to the low roasting temperature (250 °C). Moreover, the catalyst was not fully activated. When the temperature was high (750 °C), the internal structure of the catalyst was destroyed and the catalyst was deactivated. The catalytic efficiency of the catalyst was highest at a roasting temperature of 500 °C.

After obtaining the optimal loading percentage of active component Cu, the loading of the auxiliary Ce was carried out. After adding different Ce amounts at a roasting temperature of 500 °C, the denitrification efficiency of the catalyst did not improve significantly. As shown in Figure 6, the efficiency was highest when the loading was 7–8%, and this result was related to the roasting temperature. Therefore, two groups of catalysts, i.e., Ce(7%)–Cu(8%)–ZSM-5 and Ce(9%)–Cu(8%–ZSM-5), were roasted at 500, 600, 700, and 800 °C. Figure 7 shows that with the increasing roasting temperature, the denitrification efficiency of the catalyst improved significantly. The catalytic efficiency of Ce(8%)–Cu(8%)–ZSM-5 was highest at 600–700 °C, reaching 79.12%. This finding was due to the fact that after increasing the roasting temperature, the oxides of Ce formed a solid solution that stabilized the catalyst lattice and prevented the transformation of certain crystal types. CeO$_2$ shows satisfactory inhibition of CuO and carrier crystallization. Thus, CeO$_2$ grains were difficult to grow and maintained their fine quality. Ce–Cu(8%)–ZSM-5 showed the highest catalytic efficiency at 600 °C, reaching 79.12%.

3.2.2. BET Characterization of Denitrification Catalysts. A BET analysis of desulfurization catalysts prepared at 600 °C under different nitrogen partial pressures ($P/P_0 = 0.05–0.35$) was conducted.

Table 2 shows that the specific surface area of the denitrification catalyst continuously increased, reaching the maximum level when the loading of the additive was about 8%. A proper loading amount of auxiliary agent can help improve the pore structure of the molecular sieve and support the loading of active components, thereby increasing the internal stability of the catalysts. However, when the loading of the additive continued to increase, the hole of the carrier was...
Table 2. Results of Denitrification Catalyst BET Analysis

| sample name         | specific surface area (m²·g⁻¹) | average aperture (nm) |
|---------------------|---------------------------------|-----------------------|
| Cu(8%)−ZSM-5       | 64.365                          | 3.658                 |
| Ce(6%)−Cu(8%)−ZSM-5| 66.279                          | 3.628                 |
| Ce(7%)−Cu(8%)−ZSM-5| 69.409                          | 3.800                 |
| Ce(8%)−Cu(8%)−ZSM-5| 70.947                          | 3.715                 |

blocked and the specific surface area of the support was reduced. Ultimately, the catalytic activity was reduced.

3.3. Combined Desulfurization and Denitrification Experiments. 3.3.1. Preparation of Combined Desulfurization and Denitrification Catalysts. According to the above experiments, the removal effect of the La(6%)−V(7%)−ZSM-5 desulfurization catalyst was the best (94.16%). The Ce(8%)−Cu(8%)−ZSM-5 denitrification catalyst had the best removal efficiency of 79.12%. According to the idea of synergy, the combined desulfurization and denitrification catalyst was prepared by successive impregnation methods, and its use in the catalyst activity evaluation system was explored.

Figure 8 shows that compared to the single desulfurization and denitrification catalyst, the catalytic efficiency of the combined desulfurization and denitrification catalyst prepared by successive impregnation methods was better because the active components V and Cu had a synergistic effect on the removal of gaseous pollutants. At the same time, the combined desulfurization and denitrification catalyst still had high catalytic activity after several tests.

Some researchers have prepared iron-based oxide-supported catalysts under anaerobic reaction conditions. The desulfurization and denitrification efficiency was higher, the reaction temperature was higher, and the optimal reaction conditions were reached at 350 °C. However, when O₂ was added, the desulfurization efficiency was rapidly reduced to 40%, and the denitrification efficiency was reduced to 20%. Some researchers prepared Co−Cu−V−ZSM-5 combined catalysts. The optimal reaction conditions were reached only at a reaction temperature of 350 °C. The desulfurization efficiency was 100%, but the denitrification efficiency was only 60%. In contrast, the combined desulfurization and denitrification catalyst prepared in this experiment had a lower optimal reaction temperature. However, the desulfurization and denitrification efficiencies were 97.09 and 83.30%, respectively. Moreover, the addition of O₂ had a certain promotion effect on the catalytic efficiency. So, the combined desulfurization and denitrification catalyst in this experiment was an ideal catalyst.

3.3.2. BET Characterization of Catalysts. Table 3 shows that the combined desulfurization and denitrification catalyst

| sample name | specific surface area (m²·g⁻¹) | average aperture (nm) |
|-------------|---------------------------------|-----------------------|
| La(6%)−Ce(8%)−V(7%)−Cu(8%)−ZSM-5 | 82.331                          | 3.933                 |

prepared by successive impregnation methods had the largest specific surface area. The pore diameter of the sample was almost entirely distributed from 2 to 10 nm, which was the mesopore. The loading of active components and additives improved the pore structure of the molecular sieve, thereby contributing to the high stability of the catalyst’s internal structure.

3.3.3. SEM. Figure 9 shows the scanning electron microscopy images of ZSM-5 molecular sieve and La(6%)−Ce(8%)−V(7%)−Cu(8%)−ZSM-5 catalyst at the same multiple. The surface of the ZSM-5 molecular sieve was loose and porous and had a large specific surface area, thereby providing more active sites for better catalytic performance. Compared to ZSM-5, the size of the La(6%)−Ce(8%)−V(7%)−Cu(8%)−ZSM-5 catalyst was relatively uniform, and the shape was relatively regular without major changes.

From the SEM image (×100 000) in Figure 10, we determined that the inside of the catalyst was mostly nanoscale globular or spheroid, and many small particles were densely arranged. Many metal oxide particles were embedded on the surface, thereby causing more defects and wrinkles in the sample and increasing the specific surface area. The catalyst ultimately had better desulfurization and denitrification rates.

3.4. Effect of Corona Discharge on Denitrification Catalyst. We prepared the La(6%)−V(7%)−ZSM-5 desulfurization catalyst, the Ce(8%)−Cu(8%)−ZSM-5 denitrification catalyst, and the La(6%)−Ce(8%)−V(7%)−Cu(8%)−ZSM-5 combined desulfurization and denitrification catalyst at 600 °C. The catalysts were coated on a plate. Experiments with and without power were performed to explore the effect of corona discharge on the catalysts.

Figure 11 shows that the same desulfurization catalyst significantly improved the desulfurization efficiency when the electrostatic precipitator was energized. This finding was due to the phenomenon of corona discharge when the electrostatic precipitator was powered on. Strong oxidizing free radicals were produced to improve the catalytic activity of the catalyst, thereby increasing the rate of oxidation of SO₂ to SO₃. As for the denitrification catalyst, when the electrostatic precipitator was powered on, the increase of denitrification efficiency became more significant. The action of the electric field promoted the reduction of Ce⁴⁺ to Ce³⁺ and the release of oxygen in the catalyst lattice at the same time. Thus, Ce³⁺ was easily oxidized by CuO, thereby greatly improving the mobility of oxygen in the catalyst lattice and the regeneration ability of Cu. Thus, the catalytic activity of denitrification catalyst was significantly improved.

4. CONCLUSIONS

In the laboratory ESP model, the single desulfurization catalyst, the single denitrification catalyst, and the combined desulfurization and denitrification catalyst were prepared. The effect of
corona discharge on the performance of the catalyst was studied.

At a 600 °C roasting temperature, the La(6%)−Ce(8%)−V(7%)−Cu(8%)−ZSM-5 catalyst has the best single desulfurization efficiency. Under the influence of corona discharge, the catalytic efficiency reached 94.16%. At a 600 °C roasting temperature, the Ce(8%)−Cu(8%)−ZSM-5 catalyst had the best single denitrification efficiency. Under the influence of corona discharge, the catalytic efficiency reached 79.12%.

At a 600 °C roasting temperature, the La(6%)−Ce(8%)−V(7%)−Cu(8%)−ZSM-5 catalyst had the simultaneously optimum desulfurization and denitrification efficiencies. The desulfurization rate reached 97.09%, and the denitrification rate reached 83.30%. The active components V and Cu had synergistic effects on the removal of gaseous pollutants. Under the influence of corona discharge, the desulfurization and denitrification efficiencies were higher. This finding was due to the fact that the corona discharge can generate oxidizing groups. The action of the electric field promoted the reduction of Ce⁴⁺ to Ce³⁺ and the release of oxygen in the catalyst lattice, both of which promoted the oxidation of SO₂ to SO₃. In addition, the plasma generated by the corona discharge phenomenon treated and modified the catalyst surface, thereby increasing the dispersion on the surface of the catalyst support and the force between the active ingredient and the carrier. Ultimately, the catalytic activity was improved.

The BET and SEM characterization showed that the combined desulfurization and denitrification catalyst prepared by successive impregnation methods had the largest specific surface area, thereby contributing to the high stability of the catalyst’s internal structure. Compared to ZSM-5, the size of the La(6%)−Ce(8%)−V(7%)−Cu(8%)−ZSM-5 catalyst was relatively uniform and the shape was relatively regular without major changes. Many metal oxide particles were embedded on the surface, thereby causing more defects and wrinkles in the sample and increasing the specific surface area. Ultimately, the catalyst achieved a better desulfurization and denitrification effect.

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Notes
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