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ARTICLE

Recovery of Titanium, Vanadium and Tungsten from Waste SCR Catalysts by Roasting with Waste NaCl Salts

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Abstract: Tungsten and vanadium was separated from Ti in waste selective catalytic reduction (SCR) catalyst by roasting with waste NaCl salts (WNCS). Results show that the leaching efficiency of tungsten and vanadium reaches 84.63% and 66.42%, respectively, under the optimal conditions (roasting temperature 900 °C; roasting time 3 h; mass ratio of WNCS to waste SCR catalysts 0.5; reaction temperature 80 °C; reaction time 60 min), and the loss rate of titanium is 1.3%. The waste NaCl salts and roasting temperature play a positive role in converting anatase TiO_2 to rutile TiO_2. And the rutile TiO_2 is obtained. The valence of rutile TiO_2 is quadrivalent, and the lattice oxygen and chemisorbed oxygen occupy 57.26% and 42.74%, respectively.

Key words: waste SCR catalyst; waste NaCl salts; recovery; tungsten; vanadium; rutile TiO_2

Selective catalytic reduction (SCR) method is one of the best available and popular ways to covert NO_x to N_2 from stationary source[1–3]. In NH_3-SCR system, V_2O_5-WO_3/TiO_2 catalyst is used in nearly 90% of coal-fired power plants in China for its high activity and durability to SO_2[4]. V_2O_5-WO_3/TiO_2 catalyst, composed of approximately 1%–5% of V_2O_5, 3%–10% of WO_3 and about 80% of TiO_2, occupies most of the cost in denitrification system. However, the activity of catalyst is decreased by As, Na, K, SO_2 and Ca after use for about 3 years[5–8]. The spent catalyst cannot play the role after several times of regeneration. In China, the waste SCR catalyst is classified as hazardous solid waste in 2014 and the waste catalyst is a potential hazard to animals, humans and environment[9–11]. Meanwhile, vanadium, tungsten and titanium are strategic metals which are widely used in many fields[12–14], such as the steel, aerospace, and chemical industries. Considering the limited mineral resources, it is essential to recovery vanadium (V), tungsten (W), and titanium (Ti) from the secondary resource[15–18].

NaCl is used in many industries and our daily life. At present, a large amount of by-product salt residues are produced in the production process of some industries, such as leather production, pharmaceutical industry and inorganic chemical industries[19–20]. The by-products or waste materials, containing a large amount of NaCl, are not edible. Meanwhile, kinds of impurities or metals exist in the waste salts. So the disposal problem of these industrial salts is also concerned. Some NaCl residues are disposed by a serious of processes to remove impurities, aiming to produce purer NaCl salts. Though the products may be reused in industrial production, the disposal cost is high. Large amounts of waste NaCl salts are landfilled, which cause resources wasted.

Fortunately, some metals, such as V and W, can be leached out from minerals by roasting with NaCl or other sodium salts. Many researchers have focused on leaching vanadium from stone coal by roasting with NaCl[21–23]. It is found that the structure of stone coal can be destroyed by NaCl. Meanwhile the water soluble sodium salts ($NaVO_3$, $Na_4V_2O_7$, Na_3VO_4) form[24]. And above 60% of V is leached out from stone coal.

V_2O_5 and WO_3 exist in waste SCR catalyst, and there are reactions between W, V, and NaCl. The reaction products about W and V are soluble, while most of the titanates are not soluble. So it is easy to separate W and V from Ti. Though many researchers have conducted leaching experiments on waste catalyst with pure chemicals, some scholars put forward roasting with Na_2CO_3[12,25]. The equipment, waste gas and water during roasting process cost a lot yet. Leaching vanadium from waste SCR catalyst with acid or sodium

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hydroxide is also posed[10,13], and excessive leaching agents are needed. Meanwhile, the waste water and the new solid waste need to be considered. Considering the profit during dealing of the waste catalyst, it is not advisable to solve the waste with pure chemicals. Interestingly, the polluted gas, waste water, and solid waste are also faced during the dispose process of waste NaCl salts. The two kinds of waste materials were used together in this experiment. And the pollutants were managed together. Therefore, the recovery process can reduce expenditure. The mixture of waste salts and waste catalyst was roasted in a muffle furnace. The roasted products were leached out after reaction with water, and the leaching efficiency of W and V was considered.

1 Experiment

The waste honeycomb SCR catalyst was obtained from a coal-fired power plant in China, which has been used for about 3 years. The waste catalysts as feedstock were milled and screened less than 0.149 mm for analysis and experiment. The waste NaCl salt residue was collected from a chemical company, which was mainly composed of NaCl. The waste salt was also milled below 0.149 mm. The waste salt was the industrial by-product. Pulverized feedstock was dried at 105 °C in an oven over night to remove the residual moisture. Further pre-treatment process was not conducted for the waste catalyst and waste salt. For further research, the waste SCR catalyst and the waste NaCl salt were defined as WSCR and WNCS, respectively.

The whole process of the experiments included roasting stage, reaction stage and leaching stage. The WO_3 and V_2O_5 from WSCR reacted with NaCl in WNCS. The main composition of WSCR and WNCS was analyzed by X-ray fluorescence spectrometer (XRF, ARL ADVANT XP+, Thermo Corporation, USA). The phases of WSCR and WNCS were identified by X-ray diffraction (XRD, X'Pert Pro, PANalytical B. V., Holand). The content of V and W in solution was analyzed by Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-OES, Prodigy 7, USA). The valence of Ti and O was detected by X-ray photoelectron spectroscopy (XPS, QUANTUM 2000X, Physical Electronics Company, USA). The particle morphology of product was observed by scanning electron microscope (SEM, S-4800, Hitachi, Japan). The leaching efficiency of W and V was calculated according to their content in solution and WSCR.

The mixtures of WSCR and WNCS were roasted in a muffle furnace at a certain temperature for the setting time. In the roasting process, the effect of WNCS addition, roasting time and roasting temperature on the efficiency of the recovery of vanadium and W from the spent SCR catalyst was investigated. The calcined product was milled again before the leaching experiment. The main reactions in the roasting stage can be expressed as the following equations:

$$y(V_2O_5)+2x\text{NaCl}+(x/2)O_2 \rightarrow x\text{Na}_2\text{O}\cdot yV_2O_5+x\text{Cl}_2\uparrow \quad (1)$$

$$y(V_2O_5)+2x\text{NaCl}+xH_2O \rightarrow x\text{Na}_2\text{O}\cdot yV_2O_5+2x\text{HCl}\uparrow \quad (2)$$

$$y(WO_3)+2x\text{NaCl}+(x/2)O_2 \rightarrow x\text{Na}_2\text{O}\cdot yWO_3+x\text{Cl}_2\uparrow \quad (3)$$

$$y(WO_3)+2x\text{NaCl}+ xH_2O \rightarrow x\text{Na}_2\text{O}\cdot yWO_3+2x\text{HCl}\uparrow \quad (4)$$

where V_2O_5 is the vanadium bearing oxide in WSCR. For the products of $x\text{Na}_2\text{O}\cdot yV_2O_5$ and $x\text{Na}_2\text{O}\cdot yWO_3$, the numerical values x and y are correlative with roasting condition.

The leaching experiment was conducted in a teflon beaker with a cover in the water bath. First 100 mL water was put in the teflon beaker. The mixture powder after roasting and milling was put in the teflon beaker when the settled temperature was reached. The reaction process was carried out in the water bath at a certain temperature with a magnetic rotor. When the reacting solution was cooled to room temperature after the reaction was finished, the filtration experiment was conducted to separate the solution and the residue.

2 Results and Discussion

2.1 Components and phases analysis of feedstock

According to Table 1, the WSCR used in this research consists of Ti, V, W with contents of 47.87wt%, 0.59wt%, 3.32wt%, respectively. In addition, 0.52wt% of Ca and 4.20wt% of Si are detected. The CO_3^{2} in the WNCS is identified with dilute acid, and CO_3^{2-} less than 4% is found in the WNCS. Meanwhile, about 2% organic matter is identified in the WNCS. Considering the XRF results, organic matter and CO_3^{2-}, the NaCl content in WNCS is still above 90%. And the pH of the water solution is about 8.3 after most WNCS is dissolved. The XRD patterns of WSCR and WNCS are shown in Fig. 1. It can be seen that the main crystal phase of the WSCR is anatase titanium dioxide (TiO_2). The phases of other compounds in WSCR do not appear due to their low contents. There is only NaCl phase in WNCS. So it is feasible to choose the waste salt as the roasting agent to recover V and W.

2.2 Influence factors of leaching efficiency

2.2.1 Effect of the roasting temperature on the leaching efficiency of W and V

To selectively leach W and V from the spent SCR catalyst, water leaching was conducted after roasting with WNCS. The effect of roasting temperature during the roasting process was considered at first. Fig. 2 shows the research results for the leaching rate of W and V at different roasting temperatures. The increase trends of leaching efficiency of V and W with the temperature are similar. The leaching efficiency of W is higher than that of V, which indicates that W is easier to be leached out. The leaching efficiency for V and W from 500 °C to 900 °C is increased by 26.72% and 31.33%, respectively, and it has no obvious increase from 900 °C to 1000 °C. So considering the cost of high temperature, 900 °C was selected as the roasting temperature in the following experiments.

Table 1 Main composition of WSCR and WNCS (wt%)

Material	WNCS				WSCR				
	Cl	Na	Mg	Si	Ti	W	V	Ca	Si
Content	60.29	38.95	0.20	0.15	47.87	3.32	0.59	0.52	4.20

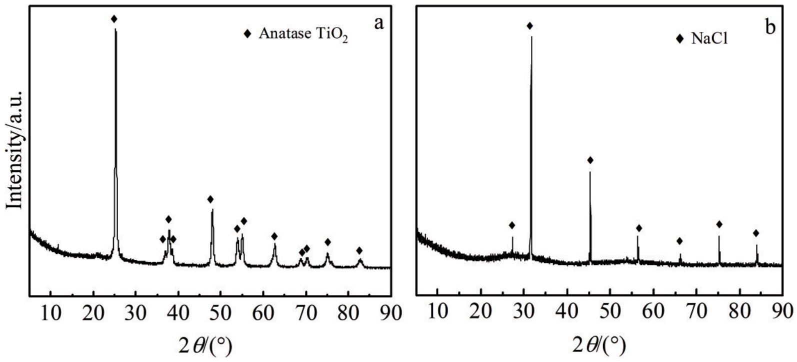

Fig.1 XRD patterns of WSCR (a) and WNCS (b)

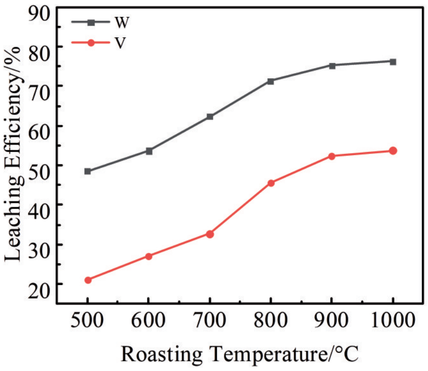

Fig.2 Effects of roasting temperature on leaching efficiency of W and V (roasting time=2 h, mass ratio of WCNS: WSCR=0.5, reaction temperature=60 °C, reaction time=60 min)

The XRD patterns of roasted products with different roasting temperatures from 600 °C to 1000 °C are shown in Fig. 3. The main phase of TiO_2 is anatase when the roasting temperature is below 900 °C. And rutile TiO_2 appears at 600 °C, which is caused by NaCl. Many researchers have found that the salts, such as NaCl, Na_2SO_4, $ZnCl_2$, can reduce the conversion temperature of anatase TiO_2 to rutile TiO_2[26–29]. When the roasting temperature reaches 900 °C, anatase TiO_2 disappears. Meanwhile, there is no obvious phase of titanate, which indicates that little TiO_2 reacts with NaCl. And the main phase after roasting is rutile TiO_2.

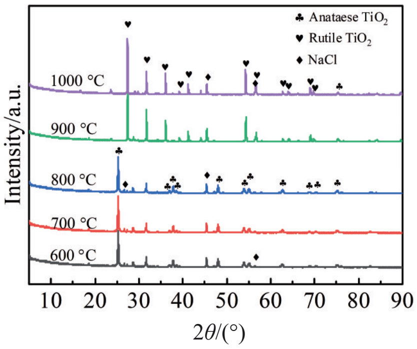

Fig.3 XRD patterns of roasting products with different roasting temperatures

2.2.2 Effect of roasting time on leaching efficiency of W and V

The influence of roasting time from 1 h to 5 h on metal leaching efficiency was studied and the results are shown in Fig.4. Both the leaching efficiency of V and W increases with the rise in roasting time from 1 h to 3 h. The leaching efficiency of W and V reaches 80.22% and 61.22%, respectively, at the roasting time of 3 h. The low valence of vanadium may exist with short roasting time[30], which needs a longer time to be oxidized. The transformation of V and W from metal oxides to soluble salt is not complete in a short roasting time. However, the leaching efficiency of W remains constant when the roasting time is above 3 h. Therefore, the completed reaction between W and NaCl needs 3 h in this experiment. Meanwhile, the leaching efficiency of V decreases a little from 4 h to 5 h. This may be due to some new complex reactions between V and other ingredients. Some new substances may have adverse influence on leaching or filtering. Above all, roasting time of 3 h is selected for roasting process. The XRD patterns is shown in Fig. 5. There are no obvious changes in the roasting products with different roasting time. The main phase of TiO_2 is rutile.

2.2.3 Effect of WNCS addition on leaching efficiency of W and V

The experiments with different WNCS addition amounts

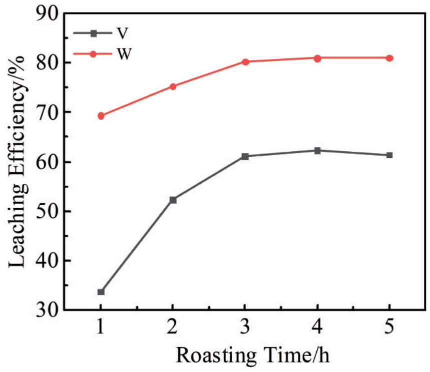

Fig.4 Effects of roasting time on leaching efficiency of W and V (roasting temperature=900 °C, mass ratio of WCNS: WSCR= 0.5, reaction temperature=60 °C, reaction time=60 min)

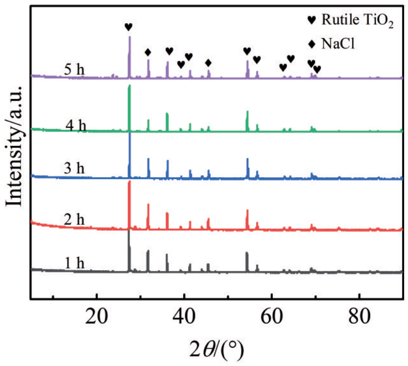

Fig.5 XRD patterns of roasting product with different roasting time

were performed during the roasting process. The leaching efficiency of V and W was examined under different mass ratios of WNCS: WSCR (0.1−0.9), as shown in Fig. 6. It demonstrates that the amount of WNCS has a positive effect on leaching efficiency of V and W. As shown in Fig. 7, the leaching efficiency of V and W is increased by 37.04% and 37.02%, respectively, from mass ratios of 0.1 to 0.5. Considering that too much NaCl in the leaching solutions may have an adverse influence on the following recovering experiments, mass ratio of 0.5 was considered to be the optimal salt addition mass ratio.

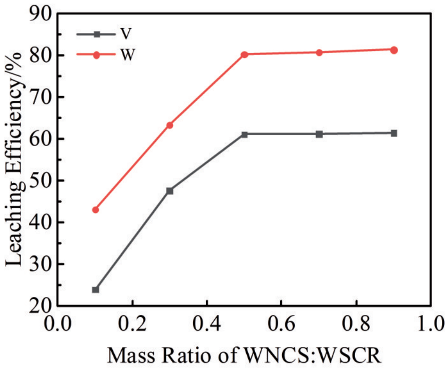

Fig.6 Effects of mass ratio of WCNS:WSCR on leaching efficiency of W and V (roasting temperature=900 °C, roasting time=3 h, reaction temperature=60 °C, reaction time=60 min)

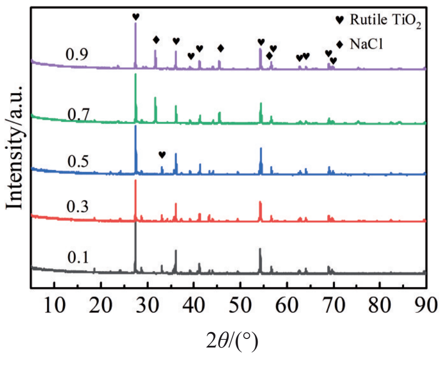

Fig.7 XRD patterns of roasting product under different mass ratios of WCNS:WSCR

The XRD patterns of roasted products with different WNCS additions are shown in Fig. 7. The main phases of TiO_2 do not change as the mass ratio of WNCS: WSCR increases from 0.1 to 0.9. NaCl is helpful to transform anatase TiO_2 to rutile TiO_2. When the mass ratio reaches 0.7, obvious NaCl phase appears. More NaCl has no effect on converting anatase TiO_2 to rutile TiO_2.

2.2.4 Effect of reaction temperature on leaching efficiency of W and V

Although roasting experiments paly the main role in transforming metal oxides into soluble substances, the reactions between the roasting products and water are also necessary for leaching metals. The reaction temperature between roasting products and water is considered in the experiment. The metal leaching efficiency at reaction temperature from 50 °C to 90 °C is shown in Fig. 8. The leaching efficiency of W and V increases with increasing reaction temperature from 50 °C to 80 °C. When the reaction temperature reaches 80 °C, the leaching rates of W and V keep constant. So the reaction temperature 80 °C is selected as the optimal reaction temperature.

2.2.5 Effect of reaction time on leaching efficiency of W and V

The reaction time from 30 min to 120 min was investigated in this part. As shown in Fig. 9, short time cannot make the soluble ions dissolve into solution. When the reaction time is above 60 min, the leaching efficiency of V and W is constant. Thus, reaction time of 60 min was considered in the following experiment. The leaching efficiency of V and W can reach 66.42% and 84.63%, respectively. A certain amount of V and W is not leached out, which may be affected by Ca and other ingredients in materials. Meanwhile the loss rate of Ti is 1.3%.

Considering the optimal conditions obtained above, the verification experiment was conducted. And the leaching efficiency of W and V is 83.96% and 66.75%, respectively, which is similar to the results above. Meanwhile, the Ti in solution was analyzed under the best leaching experiment conditions. About 98.7% of Ti remains in filter residue. The

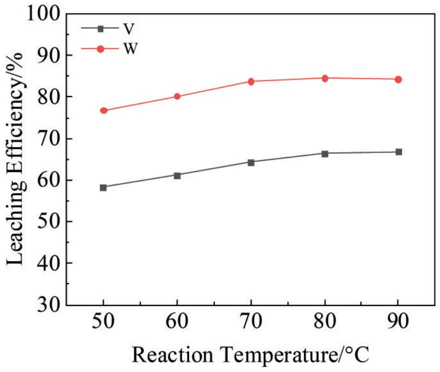

Fig.8 Effects of reaction temperature on leaching efficiency of W and V (roasting temperature=900 °C, roasting time=3 h, mass ratio of WCNS:WSCR=0.5, reaction time=60 min)

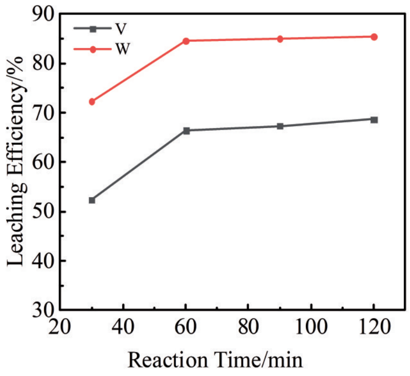

Fig.9 Effects of reaction time on leaching efficiency of W and V (roasting temperature=900 °C, roasting time=3 h, mass ratio of WCNS:WSCR=0.5, reaction temperature=80 °C)

main composition in WCNS is NaCl, so it can be inferred that WO_3 and V_2O_5 change into water-soluble salt after reaction with NaCl, as shown in Eq.(1)–Eq.(4).

2.3 Characterization analysis of leaching filter residue

2.3.1 XRD and SEM analysis of leaching filter residue

The XRD and SEM results of leaching filter residue are shown in Fig. 10 and Fig. 11, respectively. The main phase of rutile TiO_2 is obtained. And a few other ingredients, such as NaCl and $CaSiO_3$ still exist. The composition of TiO_2 occupies 88.34% in the residue, which is higher than that in waste SCR catalyst. As shown in Fig. 11, the stick structure of rutile TiO_2 is generated in the experiment. Otherwise, about 98.7%Ti remained in filter residue is rutile TiO_2. The recycled rutile TiO_2 can be used in many fields, such as photocatalytic and paint.

2.3.2 XPS analysis of leaching filter residue

The XPS analysis of leaching filter residue was conducted. As shown in Fig. 12a, only Ti^{4+} can be detected over the residue. The binding energy is assigned to Ti^{4+} $2p_{3/2}$ and Ti^{4+} $2p_{1/2}$[31–32]. It indicates that valence of Ti in the rutile TiO_2 is quadrivalent. The binding energy around 528.03 and 530.27 eV can be ascribed to the lattice oxygen (O_α) and chemisorbed oxygen (O_β), respectively[33–35]. O_α and O_β occupy about 57.26% and 42.74%, respectively.

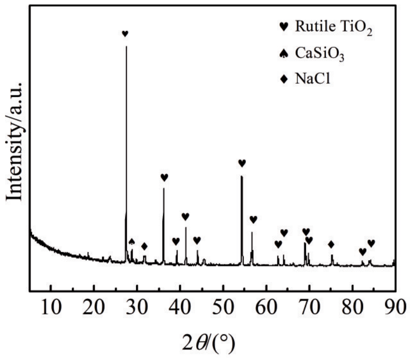

Fig.10 XRD pattern of filter residue

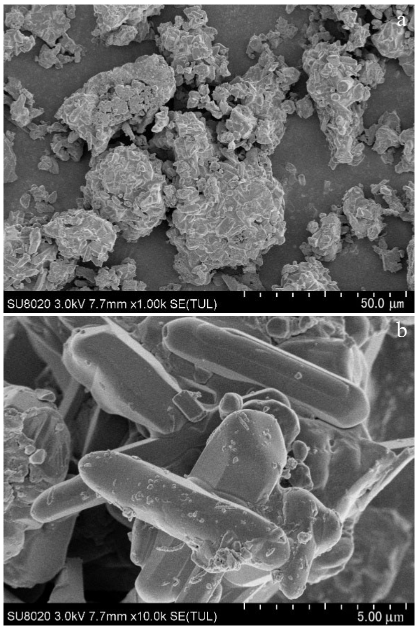

Fig.11 SEM images of rutile TiO_2

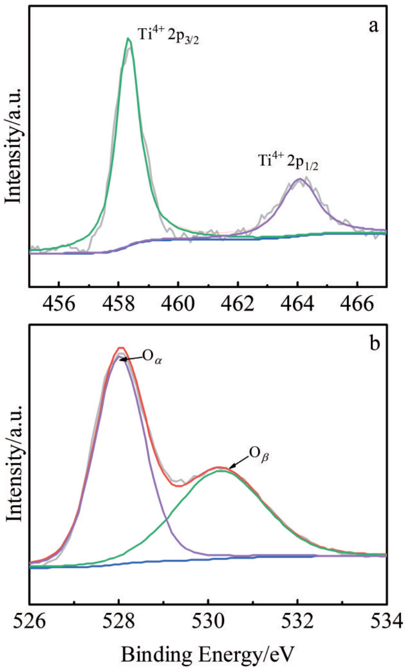

Fig.12 XPS spectra of rutile TiO_2: (a) Ti and (b) O

2.3.3 BET(Brunauer Emmett Teller) of the leaching filter residue

The specific surface area, pore structure and pore volume were considered for the leaching filter residue, which are important factors for the materials[36–37]. In this research, the value about specific surface area of leaching filter residue is about 82.44 m^2/g.

The curves about adsorption-desorption isotherms and pore distribution of leaching filter residue are shown in Fig. 13. The curve exhibits type IV adsorption-desorption hysteresis loop

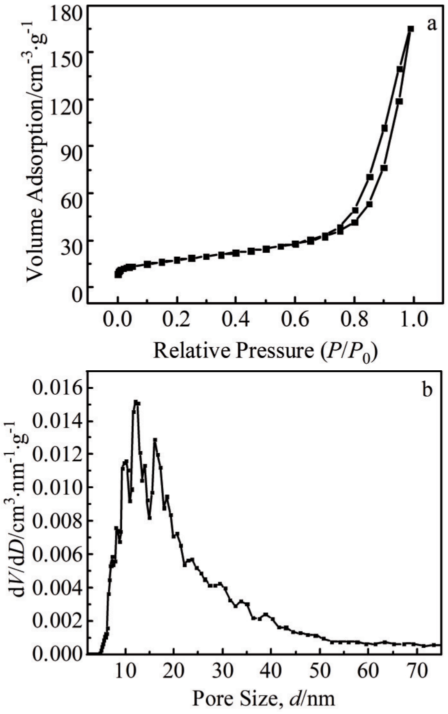

Fig. 13 Adsorption-desorption curve (a) and pore size distribution (b) of leaching filter residue

(Fig. 13a), which indicates that leaching filter residue is porous. As shown in Fig. 13b, the meso-pores peak center of leaching filter residue is at 9, 10 and 13 nm, indicating that the leaching filter residue is porous materials.

3 Conclusions

1) The method of waste selective catalytic reduction (SCR) roasted with waste NaCl salts is investigated. Water leaching after roasting stage is conducted.

2) The leaching efficiency of W and V reaches 84.63% and 66.42%, respectively, under the optimal conditions (roasting temperature 900 °C; roasting time 3 h; the mass ratio of WNCS:WSCR=0.5; reaction temperature 80 °C; reaction time 60 min), and the loss rate of Ti is 1.3%.

3) The solution containing valuable metals (W and V) and stick structure of rutile TiO_2 is obtained. The valence of rutile TiO_2 is quadrivalent, and the lattice oxygen O_α and chemisorbed oxygen O_β occupy 57.26% and 42.74%, respectively.

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废弃SCR脱硝催化剂与废NaCl盐焙烧回收催化剂中的钛、钒、钨

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摘　要：通过废弃选择性催化还原（SCR）脱硝催化剂与废NaCl盐焙烧，可以将催化剂中的钨和钒与钛分离。在最佳浸出条件下（焙烧温度900 ℃，焙烧时间3 h，废盐与废催化剂的质量比为0.5，浸出温度80 ℃，反应时间60 min），钨和钒的浸出率分别达到84.63%和66.42%，同时钛的损失率仅为1.3%。废NaCl盐和焙烧温度可以促进锐钛矿型TiO_2转化为金红石型TiO_2，反应后得到了金红石型TiO_2。金红石型TiO_2中的钛的价态为四价，晶格氧和化学吸附氧分别占57.26%和42.74%。该方法可以同时解决2种废弃物的处置问题。

关键词：废SCR催化剂；废NaCl盐；回收；钨；钒；金红石TiO_2

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