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Surface chemical properties and pore structure of the activated coke and their effects on the denitrification activity of selective catalytic reduction

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Abstract In order to study the mechanism of selective catalytic reduction of activated coke to remove NO in low-temperature flue gas and provide some theoretical basis for the development of related technologies. The pore size distribution and BET specific surface area of AC were obtain by data analyzing of N_2 adsorption/desorption isotherm at − 196 °C and carbon matrix and surface chemistry of virgin activated coke samples were characterized by acid–base titration and XPS. The process of selective catalytic reduction of activated coke (AC) samples with NH_3 as reducing agent was studied in a fixed bed reactor at 150 °C. The result shows that pore size distribution or BET specific surface of activated cokes have not correlation with denitrification activity for SCR. The NO reduction activities of the activated cokes are apparent to increase with their surface oxygen element content and total amount of acidic sites. Obviously there is good linear relationship between the NH_3 adsorption capacity and activity for SCR with linear correlation coefficient 0.943. It has been presented that adsorption of NH_3 on acidic functional groups in the edge of large polycyclic aromatic ring of activated coke is key rate controlling step in the SCR heterogeneous catalytic reaction.

Keywords Activated coke · SCR · Pore structure · Surface chemical properties · Denitrification

1 Introduction

A large amount of Nitrogen oxides (NO_x) is emitted into the atmosphere from the process of high temperature combustion of fossil fuels, mineral sintering and chemical industry. NO_x can cause the acid precipitation and photochemical smog which are considered as serious pollutants. The recently studies have revealed that secondary nitrates aerosols originating from NO_x play important roles in the forming of regional haze and fog pollution (Zhang et al. 2018; Yuan et al. 2015). Although modification in combustion mode technologies reduction of NO_x formation in furnace to some extent (Liu et al. 2018), but the introduction of ultra-low emission standard for atmospheric pollutants in China force the application of flue gas denitrification ($DeNO_x$) technologies (Yuan et al. 2017). Currently the most widely employed flue gas $DeNO_x$ technologies is selective catalytic reduction (SCR) which has been successful used in the power plant (Xu et al. 2018).

There is undoubted that catalyst is the key for SCR technologies, the common employed catalysts include noble mental, mental oxide, which have an optimal temperature range of 200–350 °C (Chen et al. 2018). The exhaust temperature of sintering machine is usually lower than 200 °C (Zhou et al. 2017) and reducing flue gas

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temperature is beneficial for boiler to improve thermal efficiency (Yu et al. 2018). Consequently, when using these catalysts for denitrification in boiler and sintering flue gas require reheating the flue gas, which means a lot of energy consumption.

The adsorbent and catalytic properties of porous carbonaceous materials are usually relying on their porosity and surface chemistry. In another aspect, the presence of carbon matrix makes porous carbonaceous materials to denitrification at low temperature (120–200 °C) (Xie et al. 2018a, b). Many high-end carbon materiel products have been prepared from coal, such as graphite or porous grapheme (Xing et al. 2018, 2019). Activated coke as a mature commercial product of porous carbonaceous materials prepared from coal with characteristics of high mechanical strength has been successfully applied in dry DeNO$_x$ process of moving bed (Xie 2017). The catalytic reduction of NO to H_2O and N_2 with NH_3 over activated coke in flue gas is heterogeneous reaction. Mochida et al. first proposed the key reaction of mechanism is NH_3 on acidic site on the surface of activated coke (Mochida et al. 1983). However, Gomez et al. revealed that BET surface area has a decisive role in SCR process with samples covering wide rang of surface area of activated carbons (Illan-Gomez et al. 1993). Zhang et al. also proposed that NO filling process in micropore of activated carbon is an important step (Zhang et al. 2008). On the contrary, some research proposed that catalytic activities of activated coke can be significantly improved by chemical modified that introduction of oxygen or nitrogen groups on carbon surface (Abdulrasheed et al. 2018). These conclusions were draw using porous carbonaceous material as catalyst by modification, thus properties of activated coke were not appropriate estimated and introduction of lots of groups by modification may reverse SCR reaction path. Moreover, chemical modification technical will cause production cost rising, so these technologies can not widely used.

The unmodified commercial activated cokes were used for SCR experiments at low temperature. The target of the research is to clarify the influence of pore structure and surface chemistry of the activated cokes and offer same quantitative analysis about the SCR mechanism.

2 Experimental

2.1 Samples

Five activated coke samples were used in this research (labeled A1–A5) which was prepared from coals with different degrees of metamorphism. The elemental analysis of activated coke samples are listed in Table 1.

Table 1 Elemental analysis of activated coke samples (wt%, daf)

C	H	O*	N
97.12	1.05	1.37	0.46
96.49	0.85	2.04	0.62
96.43	1.43	1.59	0.55
97.15	1.34	0.79	0.72
96.64	0.85	1.84	0.67

daf, dry and ash-free basis; * by difference

The granular of 0.35–0.5 mm were obtained after milled and sieved from columnar activated coke (i. d. 9 mm).

2.2 Activity measurements for NO reduction

The NO reduction experiments were operated in the fixed-bed experiment bench shown in Fig. 1. The experiment bench consists of a quartz tube (14 mm diameter) as fixed bed reactor, situated in the resistance furnace center jointing gas pipe line and mass flow controllers. The fixed-bed reactor exit gases are monitored by mass spectrometer (GAM 200, Germany).

An amount of 1 g of activated coke was used at each experiment. The reactor was previously heated and sample in N_2 atmosphere until 150 °C. Then, the SCR experiment was initiated by substituting N_2 by 150 ml/min with a composition of 1000×10^{-6} NO, 1100×10^{-6} NH_3, 10%CO_2, 8% H_2O, 6%O_2 and the balance gas, N_2.

The NO conversion is used for evaluating the denitrification catalytic activity of the samples, it was calculated as follows:

$$\eta = (C_{in,NO} - C_{out,NO}) \times 100\% / C_{in,NO} \quad (1)$$

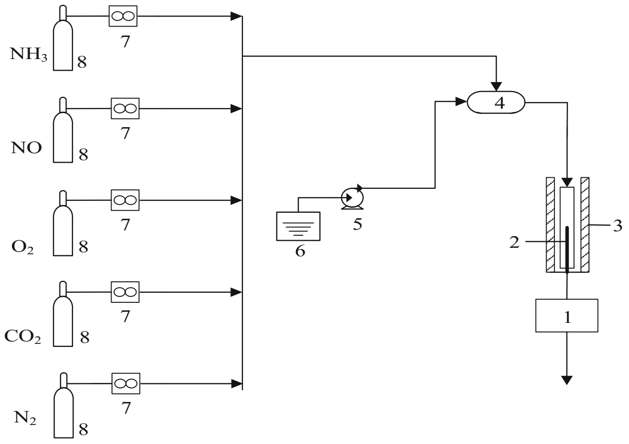

Fig. 1 Experimental system used for SCR. 1. Mass spectrometer; 2. Quartz tube; 3. Resistance furnace; 4. Vaporizer; 5. Gas mixing tank; 6. Water tank; 7. Mass flow meter; 8. High Pressure steel cylinders

where, $C_{in,NO}$ represent the NO inlet concentrations and $C_{out,NO}$ correspond to its outlet concentrations.

2.3 NH_3 adsorption capacity

The NH_3 capacities of activated coke sample are different from each other according their porosity or surface chemical. The NH_3 adsorption capacity in per unit mass of activated coke was obtained by integrating of penetration curve versus time. During the experiments, the 1000×10^{-6} NH_3 and N_2 passes through the activated coke layer and NH_3 exit concentration gradually increase until steady. Thereby, integrating the penetration curve of NH_3 of activated coke samples according to formula (2) can obtain their NH_3 adsorption capacity.

$$G = F * \frac{M}{W} \int_0^t (1 - Y_{out}/Y_{in})dt \tag{2}$$

where, F is the total gas flow, w is the weight of activated coke, M is the molar mass of NH_3, and Y_{out}, Y_{in} are the outlet and inlet volume concentration of NH_3. The t is the NH_3 adsorption time when NH_3 outlet concentration reaches steady.

2.4 Samples characterization

2.4.1 *Characterization of pore structure*

Pore structure parameters of the virgin activated cokes were determined by N_2 adsorption/desorption isotherm at − 196 °C over the relative pressure range 0.002–0.15 Pa (Quantchrome Autosorb-1). The Density Functional Theory (DFT) and Brunauer Emmett Teller (BET) equation can be used for calculating pore size distribution (PSD) and specific surface areas, respectively. The mesopore volume can be acquired through total pore volume subtraction of micropore volume which integrating differential pore volume in pore size distribution versus pore diameter. If pores are regard as cylindrical and have no intersection, the average pore diameter can be obtained according to pore volume and surface area.

2.4.2 *Characterization of surface chemical properties*

2.4.2.1 Acid and base re-titration Surface concentrations of acidic and basic sites of activated coke samples can obtain according usual Re-titration (Barton et al. 1997). Weighed amounts 0.5 g of powder carbon sample of 200 mesh were placed in 25 mL of about 0.05 mol/L NaOH solution in conical flask, then sealed and 24 h agitated gently at home temperature for chemical equilibrate. The liquid was then separated by filter paper. Measuring of 10 ml NaOH supernatant was depleted with a 0.05 mol/L HCl solution until pH value of the titrant solution was up to 7. The concentration of surface acidic sites was calculated by:

$$C_{H^+} = \frac{n \cdot m(\mathrm{OH}^-) - \Delta n \cdot m(\mathrm{H}^+)}{W} \tag{3}$$

where, n and m (OH^-) are the volume and concentration of the NaOH solution respectively; Δn is the titration volume of HCL solution; m (H^+) is the concentration of HCl; and w is the weight of activated coke samples. The similar operation was used to determine the concentrations of basic sites with HCl as impregnating solution and using NaOH as titration solution.

2.4.2.2 X-ray photoelectron spectroscopy X-ray photoelectron spectroscopy (XPS) can be used to characterize the surface chemical properties of activated cokes. The XPS model used in the experiment is Thermo ESCALAB 250 with Al Kα radiation (hν = 1486.6 eV). The C1s peak (E_b (C1s) = 284.6 eV) was taken as an internal standard. Data processing was executed using software of XPSPeak 4.1 with least square fitting algorithm and a Shirley baseline. The C1s high resolution peaks were fitted using Lorentzian–Gaussian equation.

3 Result and discussion

3.1 Physical characteristics of the activated cokes

The BET specific surface areas of the activated cokes are given in Table 2. Pore structures characteristics confirm a micro porous character of all samples, with the pore structure in the pore width of 0.35–2 nm constituting the most specific surface areas. The microporous BET specific surface area and total BET specific surface area of A1 reached 459.3 and 471.8 m^2/g, respectively, which was the highest among all samples.

Figure 2 shows the pore size distributions (PSD) of activated coke samples. In Fig. 2, all samples have almost

Table 2 The pore structure of the activated cokes

Sample	BET surface area (m^2/g)	
	Total pore	Micro pore (< 2 nm)
A1	471.8	459.3
A2	355.6	346.2
A3	389.6	386.3
A4	220.6	210.8
A5	259.9	245.6

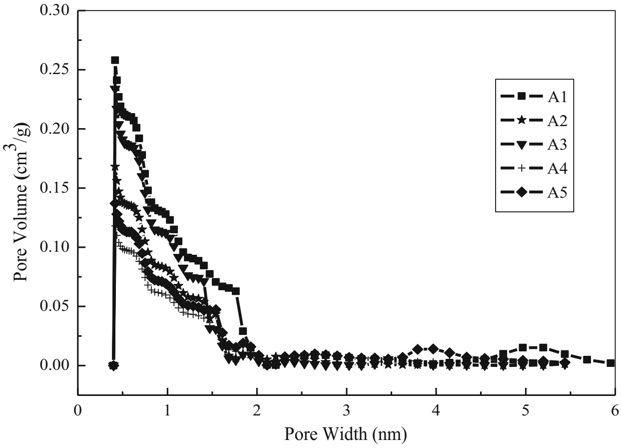

Fig. 2 Pore size distribution of the activate coke samples

same range of pore size distribution, but the region and intensity of distribution for those samples are different. The distribution intensity of A4 is low for pore volume at all of pore width range, which shows that pore structure of A4 is not well developed. A1 has a high intensity of micro pores compared with the other samples, which shows that A1 has a high micro pore volume and the BET specific surface area parameters can be mutually verified.

3.2 Surface chemical properties

The results of concentration of surface acidic and basic sites in the samples determined by acid–base Re-titration method is shown in Table 3.

The concentration of surface basic sites in the samples was significantly lower than that of surface acidic sites. The concentration both basic and acidic sites in A4 and A5 were more than those in A1 and A2. Considering the surface areas for A4 and A5 were 220.6 m^2/g and 259.9 m^2/g respectively, the intensity of surface and basic in A4 and A5 were higher than others samples.

The XPS can detect the carbon surface to a maximum depth of 10 nm (Sousa et al. 2013). Thus these results indicate not only oxygen and nitrogen functional groups but also structure of carbon layer. The elemental contents of the activated coke surface analyzed by XPS survey spectra are showed in Table 4. The activated coke surface oxygen element taken up during activation process whereas hetero atoms nitrogen may originate raw (László et al. 2001). The graphitic carbon layers are easily attacked by oxygen or activating agent at their periphery or at defect, hence the surface oxygen groups are bound to the edges of the carbon layers (Boehm 1994).

Table 3 Concentration of surface acidic and basic sites of the activated cokes

Concentration (mmol/g)	A1	A2	A3	A4	A5
Concentration of acidic sites	1.089	1.057	1.151	1.258	1.342
Concentration of basic sites	0.410	0.439	0.728	1.149	1.001

Table 4 Elemental contents of the samples surface measured by XPS (%)

Sample	N	O	C
A1	0.41	10.32	89.24
A2	0.62	10.07	89.28
A3	0.66	13.71	85.62
A4	0.78	14.02	85.17
A5	0.67	14.34	84.96

The C1s deconvolution peaks obtained from XPS spectrums could be ascribed to the carbon matrix and/or bonded different functional groups. According to the literature (Ju et al. 2008), mainly 5 peaks are usually have been fitted. Figures 3 show the high resolution deconvolution of C1s of A1 as an example. The optimization deconvolution was achieved for C1s spectrum: graphitic carbon layer C1s1 (BE = 284.0–285.1 eV), C=N groups or ether, phenolic, alcohol C1s2 (BE = 285.3–286.3 eV), quinone or carbonyl groups C1s3 (BE = 286.8–288.1 eV), ester or carboxyl groups C1s4 (BE = 288.5–290.0 eV), and π–π* transitions in large polycyclic aromatic ring systems C1s5 (BE = 290.2–291.1 eV).

The results of five different forms of peaks calculated by integral for area are shown in Table 5.

The material basis of carbon matrix mainly constitutes large polyaromatic ring structure. The π–π* transitions in carbon matrix of activated coke surface can bind protons and thus reflects basic property (Boehm 1994). The

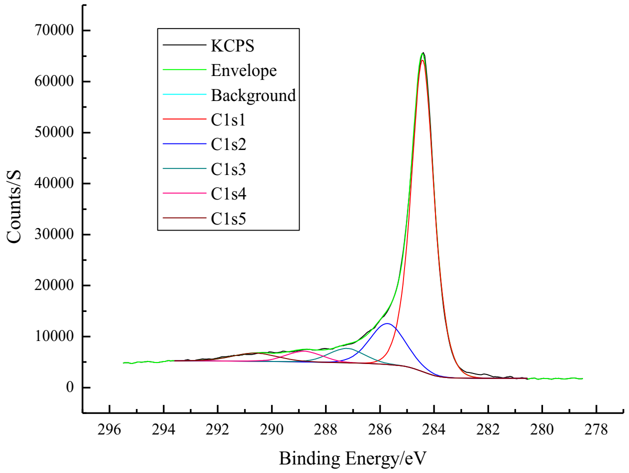

Fig. 3 High resolution of C1s spectrum of the A1

Table 5 Distribution of the five forms of the samples

Content (%)	A1	A2	A3	A4	A5
C1s1	65.56	63.41	57.55	57.74	57.61
C1s2	12.74	13.47	15.05	14.35	14.59
C1s3	4.48	5.21	5.87	5.60	5.41
C1s4	3.36	3.81	3.85	3.73	3.71
C1s5	3.16	3.34	3.26	3.71	3.63

concentration of basic sites order of A4 > A5 > A3 > A2 > A1 is different from π–π*content order of A4 > A5 > A2 > A3 > A1. It may be that the presence of basic oxygen-containing or nitrogen-containing functional groups on the surface affects the total amount of acid and base on the surface (Grzyb et al. 2009).

Acidity property of the surface of the activated coke comes from the fact that acidic functional groups that can give protons to other substances. The richer the surface oxygen element content, the dispersion is the more acidic (Xie et al. 2014). Carbon with low oxygen element content shows anion exchange behavior and basic surface property (Boehm 1994). The oxygen element content of the activated coke surface probed by XPS follows a same order to that concentration of acidic sites measured by Re-titration. Equivalently to Re-titration results, the highest and lowest of oxygen element content are A5 and A2, respectively. The concentration of acidic sites of the activated coke samples have an rank of A5 > A4 > A3 > A1 > A2.

3.3 NO reduction activities of the activated cokes

The variations of NO conversion for the reaction of SCR by different activated cokes at 150 °C were shown in Fig. 4. These curves indicate a pattern in which NO conversion decreases remarkably in the initial period of the denitrification and then remain steady-state lasting more than 200 min. Some studies have reported that NO can be physical adsorbed directly by carbon materials at low temperature (Zhu et al. 2000; Teng et al. 2001). It is deduced that NO physical adsorbed as well as reduction of SCR lead to higher denitrification efficiency, during the initial stage of the NO conversion. Hence, it is concluded that stabilized NO conversion ascribed activated coke reach saturation for NO physical adsorption and NO reduction be totally controlled by SCR reaction.

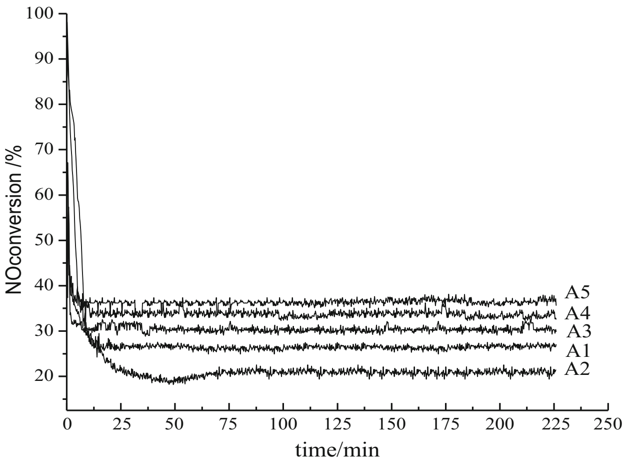

Fig. 4 Reaction profiles of NO conversion over the activated cokes

The calculated results of stabilized NO conversion reflected the SCR activities of samples are summarized in Table 6.

It is well known that micropore and specific area of carbonaceous materials is very useful for gas physical adsorption. Previous investigation showed that the optimum adsorption pore size of activated carbon is 1.7–3 times to that of adsorbate (Xie et al. 2005). However, it has been found that the weak correlation between the most probable apertures with the molecular diameter for 0.317 nm of NO or 0.38 nm of NH_3. In other hand, Zhang et al. proposed that NO filling process in micropore of activated carbon is an important step for NO oxidation to NO_2 (Zhang et al. 2008). Koebel purposed that the process of catalytic oxidation of NO to NO_2 is an important path of SCR mechanism and equimolar amount of NO and NO_2 can facilitate SCR reaction (Koebel et al. 2002). However, there are no correlations between stabilized NO conversions with the data of pore structure or BET surface area. Although, A1 and A2 have high micropore volume and thus high contribution to BET surface area, they do not exhibit high conversion of NO reduction, thus the correlation cannot be made.

It is the difficult to correlate stabilized NO conversions of the samples with any carbon functional groups fitted high resolution deconvolution of C1s. The nitrogen functional groups are mostly basic, such as pyridine and pyrrole (Xie et al. 2018a, b), and its content is significantly lower than that of oxygen functional groups on the unmodified activated coke surface. Nevertheless, the stabilized NO conversion could be correlated with total content of surface oxygen element analyzed XPS survey spectra or concentration of acidic sites by re-titration. In Figs. 5 and 6 the stabilized NO conversion are plotted against the oxygen

Table 6 The stabilized NO conversions of the activated coke samples (%)

A1	A2	A3	A4	A5
26.42	20.69	30.32	33.72	36.34

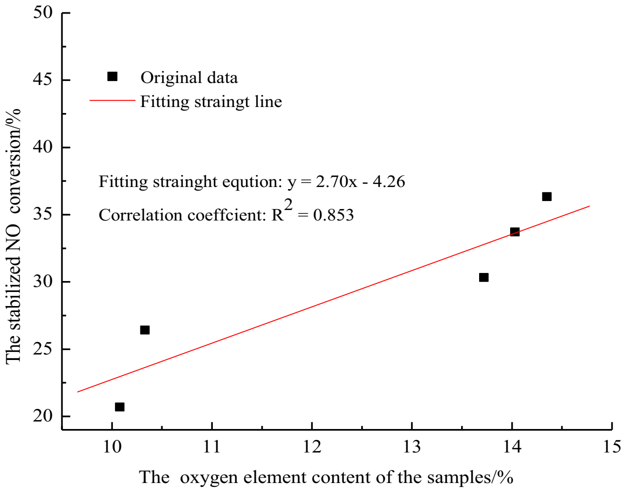

Fig. 5 Relationship between oxygen element content and stabilized NO conversion

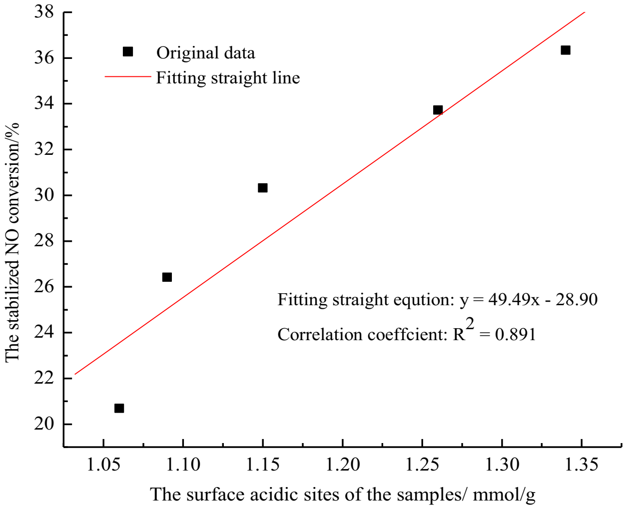

Fig. 6 Relationship between acidic sites and stabilized NO conversion

element content and acidic sites respectively, and the fitting straight equation was obtained by the least square method fitting of the original data. The linear correlation coefficient ($R^2 = 0.891$) between acid sites and stabilized NO conversion rates was significantly higher than that ($R^2 = 0.853$) between oxygen element and NO conversion.

The present work has demonstrated that the stabilized NO conversion increase with the surface acidic properties of activated coke. Previous studied ascribed the acidic properties of carbonaceous materials to its surface oxygen groups. Boehm summarized the carboxyl; carboxylic anhydride or lactons that in the edge graphene layer is acidic oxygen functional groups (Boehm 1994). The basic properties of carbonaceous materials originate from the π electrons within the graphite planes, but is has been shown that the surface presence of oxygen structures is also sufficiently basic. Boehm considered that the basic oxygen structures to chromene-like, ether-type or xanthene-type is very difficult to detect (Boehm 1994). The surface properties of the activated cokes by re-titration showed relatively precise surface acidity than oxygen element content by XPS and they had strong affinity for basic NH_3 molecules through comparing correlation coefficient.

Two SCR mechanisms proposed of metal oxides are used to describe the SCR process of activated coke (Zhu et al. 2000): (1) adsorbed NH_3 with gaseous phase NO to form an active intermediate species which is then decomposed into H_2O and N_2; (2) the adsorbed state NO at sites in the ortho position adsorbed NH_3, subsequently reaction between them to form the final products. Although, there is a common agreement for SCR mechanism that NH_3 adsorption on activated carbon/coke surface is an important step whereas it is hard to prove the SCR activity for NO removal with in the presence of NH_3. Figure 7 presents the stabilized NO conversion versus NH_3 adsorption capacity.

A good correlation between stabilized NO conversion and NH_3 adsorption capacity of the activated cokes has been found. The fitting straight equation was also acquired with least square method based on the original data. There is evident that the linear correlation coefficient ($R^2 = 0.943$) of NH_3 adsorption capacity and stabilized NO conversion is higher than those linear correlation coefficient related to surface properties. It is deduced that NH_3 adsorption capacity directly reflects the binding capacity of active cokes to NH_3, which should be a combination of surface chemical properties, pore structure or other influencing factors. It was thus suggested that the reduction of NO with NH_3 over activated coke be controlled by the adsorption of NH_3 rather than step between NO with

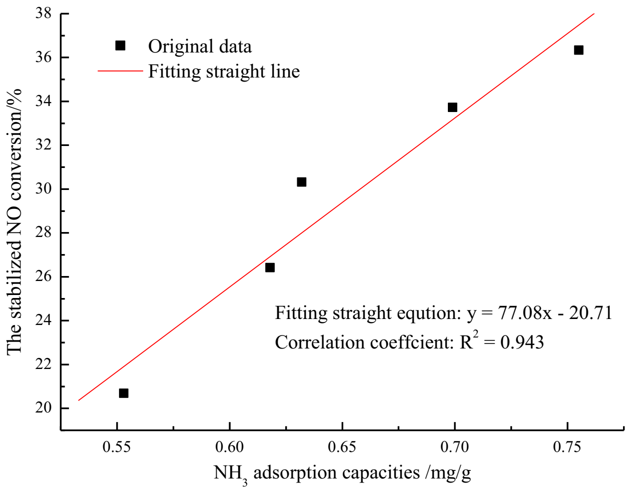

Fig. 7 Relationship between stabilized NO conversions and NH_3 adsorption capacities by the samples

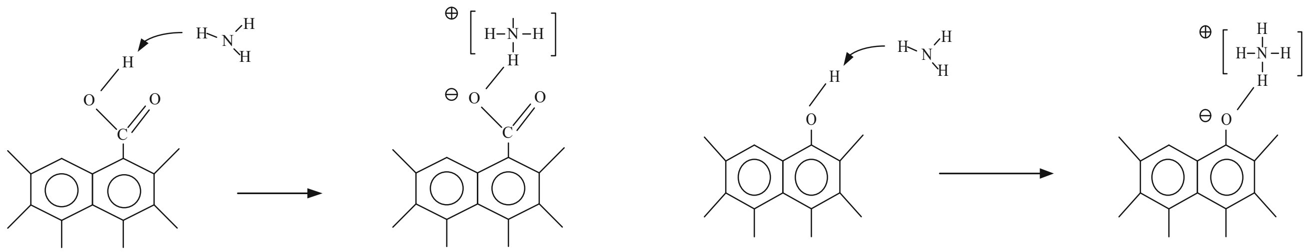

Fig. 8 Reaction step for the formation of surface $[NH_4]^+$ complexes during SCR

adsorbed NH_3.The surface chemical properties of activated coke is undoubtedly the most important factor for process of NH_3 adsorption. Therefore, the key step of SCR is the reaction of NH_3 with the acidic sites, such as carboxylic and phenolic groups, as shown in Fig. 8.

It is acknowledge that carboxyl and phenolic hydroxyl on the activated coke surface could react with basic NH_3. The carbon matrix of activated coke is compose large polycyclic aromatic ring and branched chain-like carbon structure (Zhu et al. 2017). The carboxyl and phenolic hydroxyl are usually in the edge of large polycyclic aromatic ring (Boehm 1994). The reaction step involve the interaction of NH_3 with proton part of carboxyl groups to form $CO^-(NH_4^+)$ and phenolic groups to form $O^-(NH_4^+)$. The formation of $CO^-(NH_4^+)$ and $O^-(NH_4^+)$ instead of carboxyl and phenolic groups, are very critical steps for NO reduction on activated coke surface.

As shown above, pore structure properties presented by pore size distribution and BET surface hardly affects the catalytic activity of the activated cokes for SCR. NH_3 and NO kinetic diameter are 0.26 nm and 0.317 nm respectively (Poling et al. 2001), they all in the micropore diameter range. In this sense, it can infer that physical properties mainly exert the accessibility of reactants towards the active centers. A good correlation between stabilized NO conversion and NH_3 adsorption capacity of the activated cokes indicates that acidic sites are active centers for SCR catalytic reaction. The adsorption of NH_3 on acidic functional groups of the activated coke is key rate controlling step in the SCR heterogeneous catalytic reaction.

4 Conclusion

At present study, pore structure and surface chemical properties of activated coke over adequate samples having a variety were analyzed and denitrification activities of samples for SCR were measured in fixed bed at 150 °C.

(1) It is difficult to correlate the denitrification activity for SCR of the activated cokes with their pore structure characteristic. The NO reduction activities of the activated cokes are apparent to increase with their surface oxygen element content and total amount of acidic sites.

(2) Obviously there is good linear relationship between the NH_3 adsorption capacity and activity for SCR with linear correlation coefficient 0.943.

(3) It has been presented that adsorption of NH_3 on acidic functional groups in the edge of large polycyclic aromatic ring of activated coke is key rate controlling step in the SCR heterogeneous catalytic reaction.

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