



Effect of surface acidic oxides of activated carbon on adsorption of ammonia

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ABSTRACT

The influence of surface acidity of activated carbon (AC) was experimentally studied on adsorption of ammonia (NH_3). Coconut shell-based AC was modified by various acids at different concentrations. There were five different acids employed to modified AC, which included nitric acid, sulfuric acid, hydrochloric acid, phosphoric acid, and acetic acid. Acidic functional groups on the surface of ACs were determined by a Fourier transform infrared spectrograph (FTIR) and by the Boehm titration method. Specific surface area and pore volume of the ACs were measured by a nitrogen adsorption apparatus. Adsorption amounts of NH_3 onto the ACs were measured by a dynamic adsorption system at room temperature according to the principle of the ASTM standard test method. The concentration of NH_3 in the effluent stream was monitored by a gas-detecting tube technique. Experimental results showed that adsorption amounts of NH_3 on the modified ACs were all enhanced. The ammonia adsorption amounts on various activated carbons modified by different acids are in the following order: nitric acid > sulfuric acid > acetic acid \approx phosphoric acid > hydrochloric acid. It is worth to note that the breakthrough capacity of NH_3 is linearly proportional to the amount of acidic functional groups of the ACs.

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1. Introduction

Ammonia (NH_3), a colorless, pungent, and corrosive gas, is one of the most abundant nitrogen-containing compounds in the atmosphere, after nitrogen and nitrous oxide. Ammonia can be smelt at a level as low as 50 ppm in the air. Breathing levels of 50–100 ppm NH_3 can give rise to eye, throat, and nose irritation. The US Occupational Safety and Health Administration (OSHA) has set a limit of 50 ppm over an 8-h work day or 40-h work week for ammonia vapor in ambient air [1]. Prolonged contact at concentration higher than 300 ppm can cause permanent injury or death [2].

Many techniques, including catalytic decomposition, absorption by solution, reaction with another gas, adsorption by solids, and staged combustion processes have been used to eliminate NH_3 emissions. Among these techniques, the removal of NH_3 using dry adsorbents such as activated carbon is a promising approach that has attracted much attention due to its simplicity and economy in configuration and operation [3]. Activated carbons have been widely used for gas purification, solvent recovery, etc. It is well known that pore structure plays an important role in the gas adsorption on activated carbons. The chemical nature of carbon surfaces also influences their adsorptive properties.

Surface functional groups of AC are mainly divided into acidic groups and basic groups. As for the acidic groups, surface oxides such as carbonyl, carboxyl, phenolic hydroxyl, lactone and quinine groups are representative. It has been reported that the acidic surface oxides increase the adsorption capacity for polar alkaline molecule, such as NH_3 [4–10]. Tamon and Okazaki [4] studied that AC oxidized by HNO_3 at boiling temperature and showed the adsorption sites increase greatly with the surface oxides for polar molecules. Park and Kim reported in their series papers [5–7] that the ozone treatment or oxyfluorination led to an increase in NH_3 removal efficiency of ACs and activated carbon fibers, due to an increase of acidic functional groups. However, the relation between the amount of oxyacid groups of the adsorbent surface and NH_3 adsorption capacity is not still entirely explained. It is necessary to obtain more systematic adsorption data on oxidized carbons to elucidate the influence of the surface oxides on the NH_3 adsorption from the gas phase.

There are few publications in the scientific literature that reported on activated carbon from coconut shell treated by various acids for NH_3 removal. In this study, AC was modified by various acids at different concentrations. The acidic groups on the ACs surface were determined by different approaches. The NH_3 adsorption capacity was measured by an ASTM standard test method. The main objective of this study is to investigate the effect of acid treatment on enhancing the ammonia adsorption capacity of ACs. The rela-

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tion between ammonia adsorption capacity and amount of acidic groups on ACs surface was also investigated.

2. Materials and methods

2.1. Materials

Commercial coconut shell activated carbon was supplied by HAYCARB Limited (PHO.12/40). The gaseous mixture (ammonia, 10,000 ppm balanced by helium) was supplied by San-Fu Gas Company. All five different acids employed to modify AC, nitric acid (HNO₃, 65%), sulfuric acid (H₂SO₄, 98%), hydrochloric acid (HCl, 37%), phosphoric acid (H₃PO₄, 85%), acetic acid (CH₃COOH, 100%), were supplied by MERCK Corp.

As-received AC was boiled in deionized water for 2 h and then washed by deionized water. The pre-treated AC was separately modified by five different acids, nitric acid, sulfuric acid, phosphoric acid, acetic acid and hydrochloric acid, and denoted as N-AC, S-AC, P-AC, A-AC, and C-AC, respectively. All acid solutions were prepared for different concentrations as 1N, 6N, and 12N. The modification method was carried out by stirring 50 g of the AC in 0.2 l of different concentrations of acid solution at 323 K for 24 h. After filtration, the modified ACs were washed triply by large amount of deionized water with using ultrasonic cleaner and then were vacuum-dried at 378 K.

2.2. Methods

Surface functional groups on the ACs were analyzed by a Fourier transform infrared spectrometer (FTIR, PerkinElmer Spectrum One). The spectra were recorded from 4000 cm⁻¹ to 450 cm⁻¹. By comparison to the standard frequency patterns, various characteristic chemical bonds (or stretching) were determined, from which certain surface functional groups could be derived. A model Vario EL III (Elementar Analysensysteme GmbH) elemental analyzer was employed, in which the carbon, hydrogen, and nitrogen weight percentages were determined. Oxygen content was calculated by mass difference, assuming only these elements were present.

The pH of an AC sample suspension provides information about the average acidity and basicity of the surface. A sample of 1.0 g of dry AC powder was added to 50 ml deionized water and the suspension was stirred overnight to reach equilibrium. Then the solution was filtered and the pH of the filtrate was measured by a Sartorius pH Meter (PB-20). The amounts of total surface acidic groups of ACs were determined according to the method of Boehm [11]. One gram of AC sample was placed in 50 ml of 0.05N sodium hydroxide solution. The vials were sealed and shaken for 24 h and then 5 ml of the filtrate was pipetted and the excess of base was titrated with HCl. The numbers of acidic sites were calculated from the amount of NaOH that reacted with carbon.

Nitrogen adsorption isotherms were used to characterize the surface area and micropore volume. A model of Autosorb-1(Quantachrome) was used for the volumetric measurement of nitrogen (>99.99%) isotherm at 77 K. The specific surface area of AC was calculated from the nitrogen isotherms by the Brunauer–Emmett–Teller (BET) equation [12]. The mesopore and micropore volumes were calculated using the Barrett–Joyner–Halenda (BJH) method [13] and the Dubinin–Radushkevich (DR) equation, respectively. The micropore size distribution was performed by the Quantachrome software using the density functional theory (DFT) [14]. The ash content of the ACs was determined by burning off the carbon at 850 °C.

The principle of the ASTM standard test method [15] was applied to determine NH₃ breakthrough capacity of granular AC. A stain-

Table 1
Elemental analysis for activated carbons

Samples	C (wt.%)	N (wt.%)	S (wt.%)	H (wt.%)	O (wt.%) ^a
Untreated-AC	92.75	2.69	0.02	3.05	1.49
1N N-AC	86.94	2.81	0.04	2.71	7.50
12N N-AC	78.08	3.13	0.01	2.24	16.54
12N N-AC ^b	77.63	4.07	0.01	2.96	15.33
1N C-AC	92.57	2.35	0.01	2.63	2.44
12N C-AC	92.63	2.29	0.03	2.55	2.50
12N C-AC ^b	92.28	2.32	0.02	2.44	2.94

^a Calculated by difference.

^b After adsorbing NH₃.

less steel column (2.3 cm i.d. and 10 cm long) was filled with 5.0 cm height (about 11 g) of the prepared carbon particles. Before starting adsorption runs, the carbon particles were pretreated by He at 423 K for 3 h. The adsorptive gas (NH₃) was diluted by He and was introduced to the adsorption column. The gas flow rate was controlled as 800 ml/min. The temperature of thermostat was controlled at 303 K. The outlet concentration of the column was monitored by a gas-detecting tube technique (GASTEC, 3La type; detecting range 1–100 ppm). Took a sample at regular intervals for analysis and stopped the experiment when the effluent concentration of ammonia reached 50 ppm. Record experiment time to calculate the breakthrough capacity of NH₃ on the ACs.

3. Results and discussion

The contents of C, N, S, H elements of activated carbon determined by elementary analyses are listed in Table 1. The content of O element was calculated by mass balance with assumption of no ash residue. The content of C element of the untreated-AC is high as 92.75 wt.%. After substitution of C, N, S, H elements, there was only 1.49 wt.% of O element for the untreated-AC. After modification by 12N nitric acid, the proportion of C element dropped to 78.08 wt.% and the content of O element obviously increased to 16.54 wt.%. In addition, there is little rising in N and H elements after adsorbing NH₃. For the ACs modified by hydrochloric acid, the C element content did not reduce significantly comparing to that of the untreated-AC. The content of O element of the C-AC slightly increased. It is noted that treatment of the ACs with concentrated nitric acid created larger oxygen-containing functional groups while treatments with hydrochloric acid created a fewer. Wang and Lu [16] demonstrated that the effects of acid treatment, such as HNO₃ and HCl, generally increase the surface oxygen content by the XPS analysis. Chen and Wu [17] reported the HCl treatment increases the surface oxygen content by 10% based on the XPS analysis.

Table 2 lists some physical properties of ACs. The ash content of the coconut shell activated carbon is 1.18 wt.%. After treated by nitric acid and hydrochloric acid, the ash content reduced to 0.38 and 0.42 wt.%, respectively. In other words, the amounts of mineral matter removed were about the same. A similar result was obtained by Wang and Lu [16]. The specific surface area and micropore volumes of the untreated-AC are 1073 m²/g and 0.524 ml/g, respectively. From Table 2, it was found that the specific surface area of the N-ACs decreased after nitric acid modification. However, the specific surface area of the C-ACs insignificantly changed after hydrochloric acid treatment. Similar results were reported by Moreno-Castilla et al. [18]. Compare to data obtained from elementary analysis (Table 1), it was found that C element of AC could be oxidized by nitric acid, but not by hydrochloric acid. From Table 2, the mesopore volume of the N-ACs decreased after concentrated nitric acid oxidation. The micropore size distribution of the ACs computed by the DFT method is shown in Fig. 1. It is noted that

Table 2
Physical parameters for activated carbons

Samples	Ash content (wt.%)	Specific surface area (m ² /g)	V _{meso} (ml/g) by BJH method	V _{micro} (ml/g) by DR method
Untreated-AC	1.18	1073	0.226	0.53
1N N-AC	–	955	0.171	0.59
12N N-AC	0.38	926	0.151	0.51
1N C-AC	–	1041	0.223	–
12N C-AC	0.42	1123	0.216	–

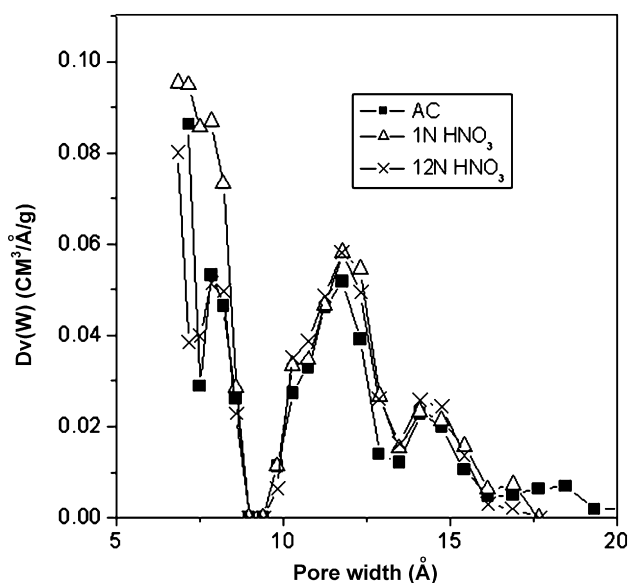


Fig. 1. Differential micropore size distribution computed by the DFT method.

local maximums of micropore exist around 6–8 Å and 10–12 Å. The micropore volume of the AC after 12N nitric acid treatment (12N-NAC) is slightly reduced. László et al. [19] studied morphology of chemically treated activated carbon by nitric acid at room temperature and at the boiling point. They found that the total porosity and the volume of the micropores of the treated-AC were slightly decreased at room temperature treatment but sharply reduced at elevated temperature. They also showed a similar illustration of micropore size distribution.

The FT-IR spectra of the ACs are shown in Fig. 2. The FT-IR spectra of all ACs displayed the following bands: 3428 cm⁻¹ free O–H stretches, 1570 cm⁻¹ C=C stretch and 1112 cm⁻¹ C–O–C stretch.

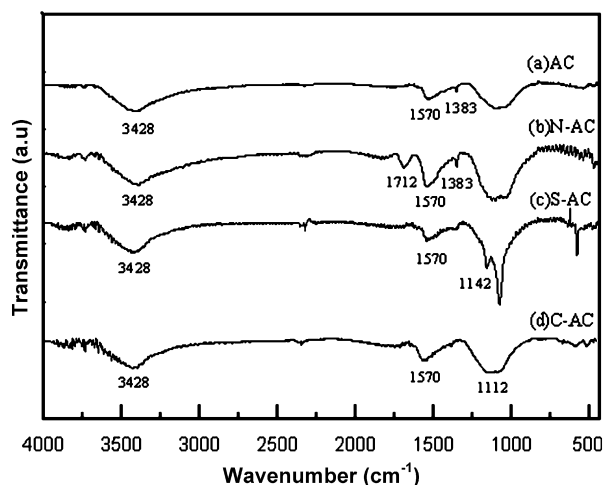


Fig. 2. FT-IR spectra of ACs: (a) untreated-AC, (b) N-AC, (c) S-AC and (d) C-AC.

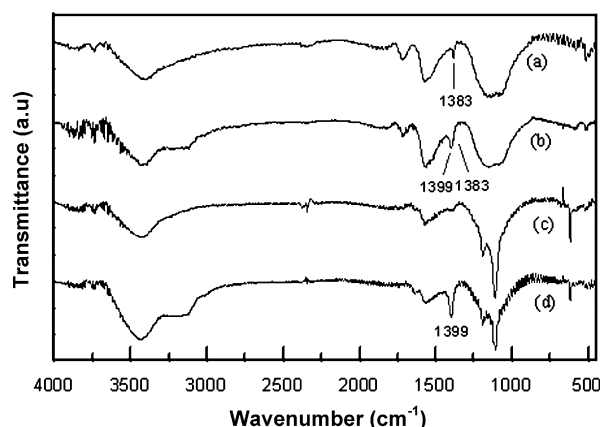


Fig. 3. Comparison of FT-IR spectra of AC before and after adsorbing NH₃: (a) N-AC, (b) N-AC adsorbed NH₃, (c) S-AC and (d) S-AC adsorbed NH₃.

From these peaks, the main surface organic functional groups presented were suggested as carboxylic anhydrides and hydroxyl groups. The FT-IR spectrum of the AC after nitric acid oxidation (Fig. 2(b)) displayed additional peaks around 1712 cm⁻¹ (C=O, stretching vibration) [18,20] and around 1383 cm⁻¹ (NO₃⁻) [21]. The slight increase in the intensity of the band around 1383 cm⁻¹ implies residual nitric acid might present in the AC. The additional peak at 1712 cm⁻¹ together with that at 3428 cm⁻¹ can be assigned to carboxyl acid groups. The FT-IR spectrum of the ACs after sulfuric acid treatment (Fig. 2(c)) displayed extra strong absorption band peaks around the 1142 cm⁻¹ (SO₂) and around 1116 cm⁻¹ (C=S, stretch) by the introduction of sulfonyl radicals. There were no significant IR peaks arose of the hydrochloric acid treated AC (Fig. 2(d)) comparing with those of the untreated-AC. However, the C-AC (treated by HCl) increases the relative concentration of the broad peak in 1000–1200 cm⁻¹. This implies a relative increase in single-bonded oxygen functional groups after HCl treatment [17]. The presence of surface acidic groups imparts a polar character to the activated carbon surface, which can affect preferential adsorption of polar alkaline adsorbates such as ammonia.

The comparison FT-IR spectra of the N-AC (HNO₃ treated) and the S-ACs (H₂SO₄ treated) before and after adsorption of NH₃ is illustrated in Fig. 3. There were new broad absorption band and peak displayed around 3050–3300 cm⁻¹ and 1399 cm⁻¹. After adsorbing NH₃, the bands between 3050 cm⁻¹ and 3300 cm⁻¹ turned into a broadband, which is difficult to distinguish and may be ascribed to the overtones of the O–H and N–H stretching vibrations [22]. The FTIR spectra of the ACs depicted that there is a peak exhibited at 1399 cm⁻¹ after the ACs adsorbed NH₃. The band at 1399 cm⁻¹ is close to the vibration frequency of NH₄⁺ chemically adsorbed on Brönsted acid sites [23,24]. The mechanism of NH₃ adsorbed on activated carbon could be described as the following. First, ammonia gas molecules diffuse into the pore of AC and are physically adsorbed at active sites on the surface. Adsorbed NH₃ molecules accept the proton from neighbor acidic groups and form ammonia complex ions (NH₄⁺) as the Brönsted acid.

Table 3

Surface pH value and amount of total acidic groups of ACs and NH₃ breakthrough capacity on the same (C₀ = 10,000 ppm)

Samples	Total acidic groups amount (mmol/g)	pH	NH ₃ breakthrough capacity (mgNH ₃ /gAC)
Untreated-AC	0.252	8.50	2.273
1N N-AC	1.025	3.77	13.945
6N N-AC	1.394	3.66	21.423
12N N-AC	2.064	3.02	41.648
1N P-AC	0.644	3.20	8.328
6N P-AC	0.747	3.01	8.662
12N P-AC	0.802	2.86	9.144
1N S-AC	0.620	3.20	5.691
6N S-AC	0.729	2.85	7.496
12N S-AC	0.747	2.74	11.245
1N A-AC	0.868	3.78	7.163
6N A-AC	0.937	3.69	8.900
12N A-AC	1.100	3.64	9.444
1N C-AC	0.379	5.09	3.819
6N C-AC	0.376	4.93	3.762
12N C-AC	0.437	4.92	3.778

The total amounts of acidic groups and pH values of the ACs are listed in Table 3. The total amounts of acidic groups of the ACs were determined by the Boehm titration method. It is revealed that the total amount of acidic groups of all acid modified ACs is larger than that of the untreated-AC. The amounts of acidic groups on surface of the ACs increase along with concentration of acid used. The order of acidic groups enhancement is as the following order: nitric acid > acetic acid > phosphoric acid ≈ sulfuric acid > hydrochloric acid. Different acidic group presents variant acid intensity. There are different kinds and amounts of acidic groups produced on the surface of AC after different acids treatment. From Table 3, it is found that the largest amount of total acidic groups produced on the AC surface after 12N nitric acid modification. The amount of total acidic groups is 2.064 mmol/g, which is 8.2 times large of that of the untreated-AC. However, there are only a few increments of total amounts of acidic groups after hydrochloric acid treatment. The hydrochloric acid treatment does not significantly oxidize the functional groups in the untreated-AC. It is note that traces of protons of acid might remain in the pores of the ACs, though, which were washed by large amount of water. A similar observation was reported by literature [4,17]. Tamon and Okazaki [4] obtained that the total acidity of the carbon oxidized by 13.2N HNO₃ is 9 times that of the original carbon. Chen and Wu [17] found that the total acidity capacity of AC after 37% HCl treatment is slightly higher than that of deionized water washed AC.

From Table 3, it is seen that the untreated activated carbon shows a basic property and the modified ACs are found to be acidic. The P-AC and S-AC have higher acidity than the others. It is known that phosphoric acid and sulfuric acid were susceptible to provide three and two protons, respectively, as compared to the other acids which can provide one proton. The pH values of the acid treated ACs decreased along with higher concentration of acid used. The pH results also indicated that acid treatment increased the acidity of the AC and more acidic groups produced [16].

Ammonia breakthrough capacity on the ACs is calculated by Eq. (1) which is proposed by the ASTM D6646 [15].

$$Q = \frac{C_0 \times F \times t \times (10^{-6}/24.86) \times (M)}{w} \quad (1)$$

C₀: NH₃ inlet concentration (ppm); F: flow rate (ml/min); t: breakthrough time as 50 ppmv of NH₃ broke (min); w: activated carbon weight (g); M: molecular weight of ammonia; Q: breakthrough capacity (mg NH₃/g AC).

The NH₃ breakthrough capacities on different ACs are also listed in Table 3. It is found that the largest increment of NH₃ break-

through capacity of ACs occurred after nitric acid modification. The breakthrough capacity of NH₃ on the 12N N-AC is 18.3 times large as that on the untreated AC. In addition, there were 6 times large for the AC modified by the dilute concentration (1N N-AC). The treatment with sulfuric acid or phosphoric acid enhanced adsorptive capacity from 2.5 to 5 folds. In addition, organic acid (acetic acid) modification can provide NH₃ adsorption capacity enhancement too. Except for hydrochloric acid, the NH₃ breakthrough capacity increases along with the concentration of acid solution employed. For AC modified by un-oxyacid solution such as hydrochloric acid, NH₃ breakthrough capacity increases slightly about 1.6 times. The increment of adsorption capacity of NH₃ on the AC modified by hydrochloric acid could attribute to residual of acid during treatment process. From Table 3, the ammonia breakthrough capacity on various activated carbon modified by different acids follows a descending order of nitric acid > sulfuric acid > acetic acid ≈ phosphoric acid > hydrochloric acid. It is inferred that the increment NH₃ breakthrough capacity of the ACs modified by acid solution can be ascribe to the enhancement of surface acidic functional group and acidity.

For obtaining the whole breakthrough curve of NH₃ dynamic adsorption, a lower concentration (1000 ppm) of NH₃ was investigated in a shorter bed adsorber. The column height reduced from 5 cm to 2 cm. The breakthrough of NH₃ was monitored using the other type of gas detecting tube (GASTEC, 3M type; detecting range 1–1000 ppm). The dynamic adsorption experiment was stopped when 80% of inlet concentration reached. A typical comparison of breakthrough curves (untreated AC and 12N N-AC) is illustrated by Fig. 4. The breakthrough curves were a sigmoidal shape which usually seen in an ordinary adsorption process. It is clear from Fig. 4 that NH₃ breakthrough the column packed with the untreated-ACs at 4 min and 80% breakthrough at 22 min. The adsorption capacity can be calculated by integration of the area above the breakthrough curves and from NH₃ concentration in the inlet gas, flow rate, breakthrough time, and mass of AC. The adsorption capacity was calculated from experimental data to be 1.06 mgNH₃/gAC. When packed by the 12N N-AC, breakthrough time was at 130 min and 80% breakthrough time was at 705 min. The NH₃ adsorption capacity on the 12N N-AC was 29.80 mgNH₃/gAC, which was 28.1 times large as that by the untreated-AC. Owing to dilute inlet concentration of NH₃ (1000 ppm), the adsorption capacity is lower than listed in Table 3 (10,000 ppm). Although, as aforementioned the specific surface area and pore volume of the 12N N-AC are less than those of the untreated-AC, the adsorption capacity of NH₃ on the 12N N-AC is about 28 times large as that on the untreated-AC. It is induced that the acidic functional groups

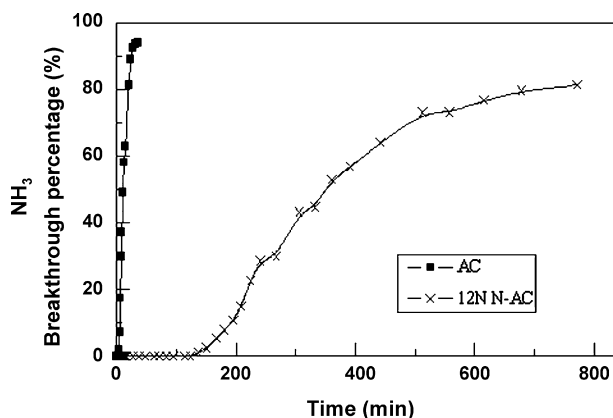


Fig. 4. Breakthrough curves of ammonia adsorbed on a fixed-bed of ACs (C₀ = 1000 ppm, 800 ml/min, 303 K).

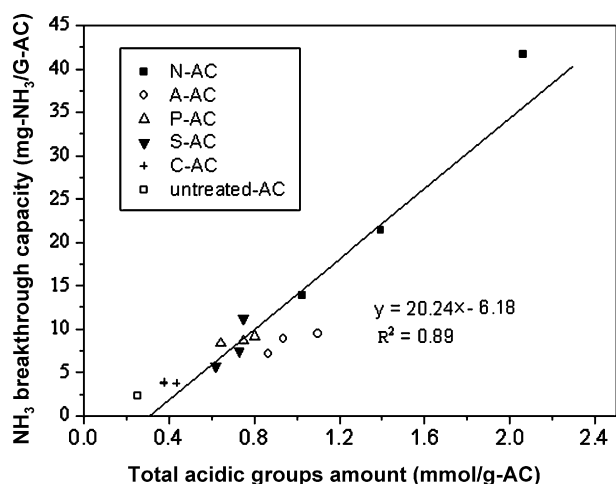


Fig. 5. Relationship between the NH₃ breakthrough capacity and the total acidic groups amount of ACs.

produced on AC surface are dominant the increase of adsorption of NH₃.

A similar observation was reported by Rodrigues et al. [25]. The adsorption capacity of 1.20 mgNH₃/gAC was obtained by feeding 1200 ppm NH₃ through a fixed bed of activated carbon. Park and Kim [6] studied the adsorption of NH₃ on activated carbon fibers (S_{BET} : 2120 m²/g; V_{micro} : 1.15 ml/g) produced by oxyfluorination. The maximum adsorption capacity of 31.29 mgNH₃/gACF was reported. They get 6 times capacity after ACF modification. Tamon and Okazaki [4] reported that the adsorption capacities of NH₃ were 1.95 mgNH₃/gAC and 33.49 mgNH₃/gAC on the untreated AC and 13.2N HNO₃ oxidized AC, respectively. This means about 16.2 times capacity increased after acid oxidation.

The relation between measured breakthrough capacity of NH₃ on ACs and the total acidic groups amount of ACs is illustrated by Fig. 5. It is evident that there is an approximate linear correlation between NH₃ breakthrough capacity and the total amount of acidic groups. By regression, a linear correlation equation is obtained as $Y = 20.24X - 6.18$, where Y denotes the NH₃ breakthrough capacity (mgNH₃/gAC), and X denotes total amount of acidic groups of ACs (mmol/gAC). It is worth to note that acidic group is the predominant factor for the adsorption capacity of NH₃ on activated carbon.

4. Conclusions

In this study, coconut shell-based activated carbons modified by five different acids were studied to enhance adsorption capacity of NH₃. The ammonia adsorption amounts on various activated carbons modified by different acids are in the following order: nitric acid > sulfuric acid > acetic acid \approx phosphoric acid > hydrochloric acid. According to the results by the Boehm titration method, it is found that the amounts of acidic group of the AC modified by acid solution increase along with the employed acid concentration. The NH₃ adsorption amount of the acid modified AC is also enhanced. However, the specific surface area and pore volume of activated carbon does not have direct influence on the adsorbing amount of ammonia. The NH₃ breakthrough capacity on the ACs, measured by the ASTM standard test method, is found to be approximately linear proportion to the total amount of acidic

groups on the surface of ACs. According to the FTIR spectra analysis, there was a peak exhibited at 1399 cm⁻¹ after the ACs adsorbing NH₃. This peak attributed to NH₃ interacted with the acidic oxygen groups of the ACs surface to form NH₄⁺ as the Brønsted acid.

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