

Effect of Pretreatment of Carbon Black Derived From Pyrolysis Waste Tire on Adsorption of Ammonium Ion in Aqueous Solution

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ABSTRACT

Ammonium ion is a common pollutant which exists in municipal sewage, fertilizer plant discharge and agriculture wastewater. This study aims to investigate the potential of carbon black (from pyrolysis of tire waste) and modified carbon black on the adsorption of ammonium ion in aqueous solution. The carbon black was modified via acid treatment (HCl, CH₃COOH and H₂SO₄), and the modified sample with the highest sulphur removal was then carbonized at 500 °C for 1 h. The effects of contact time (30-150 min), pH (2-11) and adsorbent dosage (1-10 g/L) on the adsorption performance displayed by carbon black and modified carbon black were also evaluated. CB-HCl showed the most effective adsorption performance within 60 min, 3 g of adsorbent dosage at pH 11. This study indicates that shows that tire waste can be converted to carbon black for removal of ammonium ion from the wastewater.

Keywords: Ammonium-nitrogen, Adsorption, Waste tire, Carbon black

1. INTRODUCTION

Every year, a great amount of industrial wastewater is produced from various sources, sending numerous pollutants, including nitrogen-containing compounds, into the waterways [1]. Ammonium ion (NH₄⁺) is one among these substances which causes eutrophication, which leads to oxygen deprivation of aquatic organisms and algae bloom [2]. Therefore, proper treatment of industrial wastewater is vital to prevent the release of ammonium ion into the natural environment [3].

Various methods, such as biological treatment, chemical precipitation, air stripping and advanced oxidation process, have been developed for ammonium removal from wastewater. However, there are limitations or drawbacks related to these methods, including low treatment efficiency at low pollutant concentrations as well as slow and difficult removal at high concentrations (biological process) [4,5].

Adsorption is a popular strategy for ammonium removal due to the process efficiency and manoeuvrable technique according to the EPA and WHO regulations [5,6]. Various materials have been used as adsorbents in this field such as hydrogels, zeolites, biosorbents and activated carbons. For wastewater treatment purpose, these adsorbents have to be abundant, easy to process and environmentally friendly when disposed of. However, the application of adsorption

in wastewater treatment is limited by the cost of zeolites [4,5,7].

Waste tire could be an ideal carbon source for synthesis of low-cost adsorbents, which is yet to be explored extensively. Increasing number of waste tires end up in the landfills or waste dumps [8]. Irresponsible disposal of waste tires create environmental problems and breeding grounds of vectors [9]. To overcome this problem, recycling of waste tire via thermal treatment is one of the methods used. Pyrolysis is one of the proven solutions for high-efficiency material and energy recovery from the waste tire. The gasification treatment needs a higher temperature for oxidation of feedstock [10]. The products of the waste tire pyrolysis include syngas (fuel gas), oil and solid residue (carbon black) [10,11]. The solid residue contains 80-90 wt% of carbon black and 10-20 wt% of the inorganic substance used for improvement of tire properties [11].

Carbon black (CB) is an amorphous carbon material with a quasi-graphitic structure. CB has been used as activated carbon but it has low adsorption and poorer properties than commercial carbon black. CB produced from waste tire pyrolysis has lower carbon content but higher ash and sulfur content than commercial carbon black [12,13].

Acid treatment is a suitable method for removal of ash and sulphur contents. This treatment also increases carbon content and surface area of CB by removal of inorganic materials [12,14]. The acid treatment can be effectively used for improving efficiency of ammonium ion removal

from wastewater [15]. Based on a previous study, the percentage removal of ammonium by activated carbon modified with sodium dodecyl sulphate (anionic surfactant) is 60.8% [16]. Another research reports 75% removal of removal ammonium is 75 % using potassium-iron rice straw biochar [17]. However, the data related to ammonium ion removal from wastewater by carbon black is limited.

The objectives of this study are: (1) to treat CB with acid agents such as hydrochloric acid, sulfuric acid and acetic acid prior to carbonization at 500 °C for 1 hour; (2) After the acid treatment, the potential of acid-treated CB (HCl) was selected based on the decreases of sulphur content and the adsorption study of ammonium ion was performed at different contact times, pHs and adsorbent dosages.

2. EXPERIMENTAL

2.1. Materials

The carbon black (CB) sample used in this study was collected from a local waste tire recycling company in Johor, Malaysia. Hydrochloric acid (HCl), sulphuric acid (H₂SO₄), acetic acid (CH₃COOH), sodium chloride (NaCl), and sodium hydroxide (NaOH) were purchased from Qrec and used as received.

2.2. Adsorbents preparation

The CB was washed (using distilled water) and dried overnight at 80°C. The carbon black was then separately treated with three types of reagents, namely HCl, H₂SO₄ and CH₃COOH with 1 M concentration. For each acid, 5 g of sample was added into 50 mL reagent. The mixture was stirred using a mechanical stirrer (180 rpm) for 2 hours at room temperature. After that, the mixture was filtered to separate the CB from the aqueous solution. The CB was washed with distilled water to remove the chloride ions, and dried at 60 °C overnight. The raw and treated CB and CB were characterized using elemental analysis.

2.3. Carbonization and characterization

The carbon black treated with the HCl was weighted approximately 20 g were subjected to carbonization. The samples were heated at 500°C for 1 h. Next, oxidation of the sample using 100 ml of 30% hydrogen peroxide solution was performed to remove all the impurities on the adsorbent surface. The obtained adsorbent was washed with deionized water and dried in an oven (60°C for overnight). The modified adsorbent, named as CB-HCl, was stored in a desiccator prior to adsorption experiment.

The surface properties (surface area, pore volume, pore size) of the adsorbent were examined using the Brunauer-Emmett-Teller method (Micromeritics 3 Flex surface Characterization Analyzer). The functional groups of the adsorbent were identified using Fourier-Transform infrared analysis (FTIR) (IRTracer-100, Shimadzu, Japan).

The sample's point of zero charge (pH_{pzc}) was also performed. 100 mg of the sample was brought into contact with 0.1 M of NaCl solution at various initial pHs. The pH values were adjusted with 0.1 M of HCl or NaOH aqueous solution. The mixture was shaken to equilibrate for 24h at 30 rpm, before the final pH values of the solutions were measured.

2.4. Adsorption study

The adsorption study was performed to study the effects of contact time, pH and adsorbent dosage on the ammonium ion adsorption behaviour by raw and treated CB. Adsorption experiment for contact time and pH was conducted by mixing 3 g of adsorbent with 50 mL of ammonia-nitrogen solution (50 g/mL) produced via dissolution ammonium chloride (HmbG). Throughout the experiment, the contact time and pH solution was varied from 30 to 150 min and 2 to 11. The amount of adsorbent dosage ranged in 1-10 g/L with the 50 mg/L of ammonium nitrogen concentration. For the effect of pH, the pH solution was maintained constant by adding a small volume of dilute HCl and NaOH aqueous solutions. The concentration of ammonium ion was determined using COD (Standard method 5220-B) and Multiparameter Photometer (HANNA Instruments). All preparation and experiments were conducted at room temperature. The percentage removal of the ammonium nitrogen was calculated using Eq (1)

$$\% \text{ removal} = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

where C₀ is the initial concentration (mg/L), and C_e is the concentration of ammonium ion at equilibrium (mg/L).

3. RESULTS AND DISCUSSION

3.1. Adsorbent characterization

The elemental analysis results (Table 1) depict decreased sulphur content from 3.14 % (CB) to 2.29 % (CB-HCl), 2.89 % (CB-CH₃COOH) and 3.09 % (H₂SO₄) acid treatment. This result shows that sulphur was leached with a mixture of concentrated HCl and aqueous CH₃COOH. Based on the result, HCl is the best acid solution to remove inorganic components (such as sulphur). The previous studies have reported that acid treatment using an acid solution (HNO₃) onto char decreased sulphur content from 2.84 % to 0.09 % [18]. An increase of C % from 77.80 % (CB) to 83.34 % (CB-HCl), 80.79 % (CB-CH₃COOH) and 79.25 % (CB-H₂SO₄) after the acid

treatment was also observed. Such increases are related to the increased aromatization rate and addition of C atoms from the HCl molecules [17,19]. The acid treatment of CB leads to better development of the porosity [15]. The presence of sulphur content in CB could cause

environmental problem and limits the application [20]. Thus, the decreased sulphur content is one of important features which increase the viability of CB in environmental remediation. Form the result, the CB-HCl was selected to run further characterization analysis.

Table 1 Elemental analysis of acid treatment process onto CB

Samples	N (%)	C (%)	H (%)	S (%)
CB	0.43	77.80	1.04	3.14
CB-HCl	0.49	83.40	1.03	2.29
CB-CH ₃ COOH	0.46	80.79	0.97	2.89
CB-H ₂ SO ₄	0.47	79.25	0.99	3.09

Based on the elemental analysis of acid treatment onto CB, the highest removal of sulphur was selected to be further analyses of surface analysis, FTIR and point of zero charge. According to Table 2, the surface area and pore volume of CB were lower than those of CB-HCl. The low pore volume of CB indicated less sophisticated pore

development during waste tire pyrolysis [21]. Based on the result, the adsorbents are mesoporous (pore radius range of 2-50 nm) [22]. The increase of pore volume and surface area in the CB-HCl adsorbent could lead to better adsorption properties. The increases in surface area and pore volume are due to devolatilization at high temperature during the carbonization [17].

Table 2 Surface structure parameters of CB and CB-HCl

Samples	Surfaces area (m ² g ⁻¹)	Pore volume (cm ³ g ⁻¹)	Pore size (nm)	
			Pore width	Pore diameter
CB	56.73	0.45	34.60	31.44
CB-HCl	88.14	0.54	34.60	24.51

FTIR spectroscopy is used to determine the functional group in the adsorbents according to the wavelength of specific functional group [23]. In Figure 1, there is a band at 2500-3500 cm⁻¹ which is assigned to the O-H stretching mode of hydroxyl groups [24]. The bands at 1477.00 and 1476.96 cm⁻¹ appeared on both CB and CB-HCl which refer to the bending vibration of -CH₂. The bands in 1000.00-1300.00 cm⁻¹ is attributed to the C-O stretching vibration of carboxylic acids, alcohols, phenols, ethers and esters [24]. The CB-HCl has a band at 1560.15 cm⁻¹ which is attributed to the aromatic C=C and C=O stretching [25]. The results indicate the presence of functional group that helps in the adsorption process.

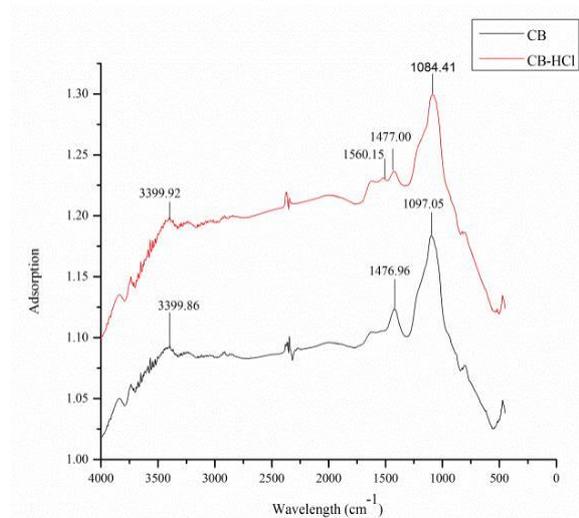


Figure 1 FTIR spectrum of CB and CB-HCl

The point of zero charge (pH_{pzc}) is a pH value where the positive charge and negative charge on the adsorbent surface is zero [27]. As shown in Fig 2, the pH_{pzc} of CB-HCl was 5.4, indicating the net negative charge on the adsorbent surface which favors the uptake of cationic molecules. When $pH < pH_{pzc}$, the adsorbent surface becomes positively charged and the high H^+ concentration hinders the uptake of cationic molecules [28,29]. Thus, the surface functional groups dissociate in response to the changes in solution pH, affecting the adsorption process. The result shows that the ammonium ion adsorption is favourable at pH above pH_{pzc} .

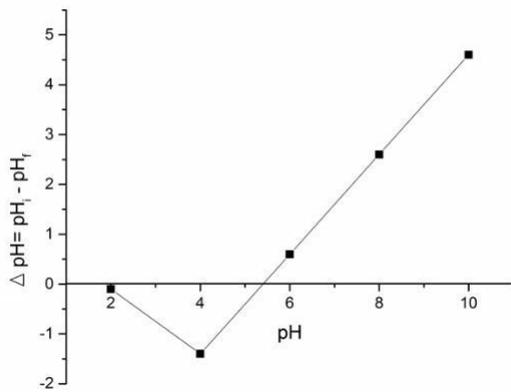


Figure 2 Point of zero charge (pH_{pzc}) of CB-HCl

3.2. Effect of contact time

Fig 3 shows the contact time of adsorbent on ammonium ion adsorption (50 mg/L). The rapid adsorption of NH_4^+ -N occurred from 30 to 60 min with percentage removal of 43.30 - 44.33 % (corresponding to adsorption capacities of 0.46 - 0.47 mg/g) for CB-HCl. Such result is related to the high number of unoccupied adsorption sites and high solute concentration gradient at the beginning of the adsorption process which promoted the rapid initial uptake [4]. After the lapse of time (90 min until 150 min) the adsorption of NH_4^+ -N was decreased from 42.53 % to 41.75 % (0.45 - 0.45 mg/g) as the remaining vacant surface sites are difficult to be occupied due to repulsive force from NH_4^+ -N [30]. On the other hand, the adsorption of NH_4^+ -N by CB was the highest at 120 min with percentage removal 33.85 % (0.30 mg/g) and decreased at 150 min with percentage removal 26.40 % (0.23 mg/g). The slower adsorption of CB indicated an ionic balance between the aqueous solution and the adsorbent [4]. The equilibrium was obtained approximately at 60 min, thus the adsorbent owns a quicker rate of adsorption and advanced adsorption capacity for ammonium ion removal. The similar result has been reported by H. Yang et al., (2018) which the rapid removal of ammonium ion is at 60

min and decreased gradually by using pine sawdust biochar and wheat straw biochar [4].

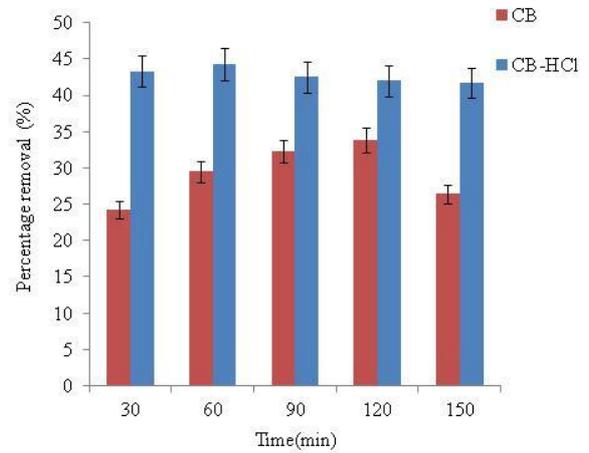


Figure 3 Effect of contact time on the percentage removal of ammonium ion by CB and CB-HCl (mass: 3 g, volume: 50 mL, concentration: 50 mg/L, pH: 7, room temperature).

3.3. Effect of pH

The ammonium ion adsorption is strongly influenced by the solution pH. As shown in Fig 4, the percentage removal of ammonium nitrogen in acidic medium ($pH=2$) was lower (CB: 0.27 mg/g, CB-HCl : 0.36 mg/g) due to the excess H^+ ions in the system which strongly competed with the NH_4^+ ions for the active adsorption sites. Furthermore, repulsion between the positively charged surface of the adsorbent and NH_4^+ ions also occurred [31] [32]. As the pH increased from 7 to 11, the competitive adsorption between NH_4^+ and H^+ weakened, therefore a significant increase in the adsorption of ammonium nitrogen was observed (CB : 0.32 to 0.67 mg/g, CB-HCl : 0.46 to 0.69 mg/g). This is due to the carboxyl and phenolic groups were deprotonated after the pH adjustment, and the negatively charged organic functional groups became the main adsorption sites [33]. This is further supported by the fact that the adsorbent surface became negatively charge when $pH \text{ solution} > pH_{pzc}$ [31]. In this study, the pH_{pzc} was 5.4, thus the adsorbent surface was negatively charged. Therefore, the influence of pH on the ammonium nitrogen adsorption was mainly caused by the variation of H^+ concentration and the surface charge of the adsorbents. This result was accordance with the previous study done by M.Li *et al.*, (2019), the adsorption of ammonium ion was increase with the increases of pH (5 to 9) used zeolite as adsorbent [34].

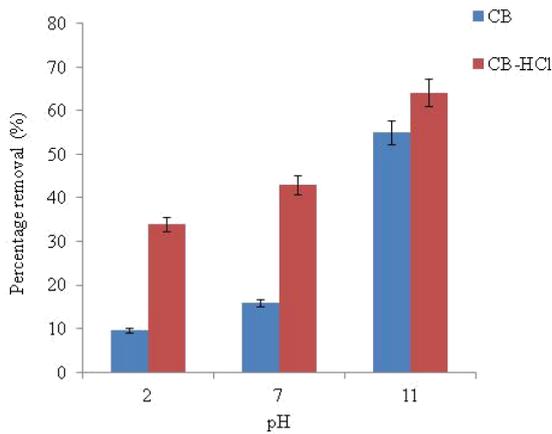


Figure 4 Effect of pH on the percentage removal of CB and CB-HCl for ammonium ion removal (mass: 3 g, volume: 50 mL, concentration: 50 mg/L, room temperature, time: 60min).

3.4. Effect of adsorbent dosage

The effect of adsorbent dosage on the ammonium nitrogen removal (Fig 5) was studied over the range of 0.5 - 7 g/L for 50 ml solution with initial concentration as 50 mg/L at room temperature. Both adsorbent showed a similar trend, where the ammonium nitrogen removal increased from 0.3 to 3 g/L. This is related to the increase in the available adsorption sites [35]. However, further increase of adsorbent dosage from 5 to 7 g/L resulted in decreased ammonium ion removal, possibly due to the aggregation of particles and shielding of active sites at large loading [35]. The other reasons of the decreases of adsorption rate is overlapping of active adsorbent sites on the surface of active adsorbent sites and decrease in NH_4^+ concentration gradient due to increased solid (adsorbent)/liquid ratio [36]. The similar result are reported by other researcher for the adsorption of ammonium onto carbon materials which when the adsorbent dosage increase, the adsorption of ammonium ion was decrease [36].

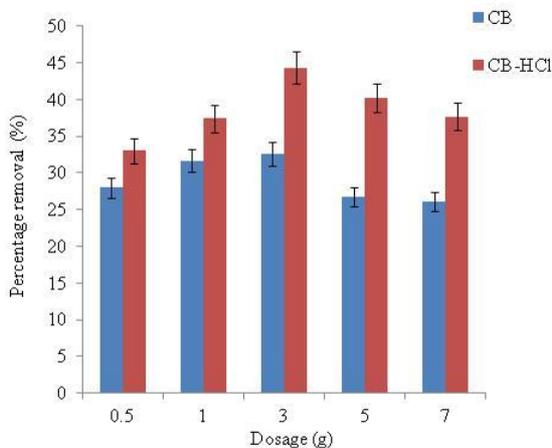


Figure 5 Effect of adsorbent dosage on the percentage removal of CB and CB-HCl for ammonium ion removal (volume: 50 mL, concentration: 50 mg/L, pH: 7, room temperature, time: 60 min).

4. CONCLUSION

The focus of this study is to improve the adsorption performance of carbon black derived from waste tires via acid treatment. Characterization results indicated decreased sulphur content after acid treatment ($HCl > CH_3COOH > H_2SO_4$). After carbonization, the surface area and pore volume of CB-HCl increased. The FTIR result indicates the contributions of carboxyl and hydroxyl groups to the adsorption of ammonium nitrogen.

The pH_{pzc} of the CB-HCl adsorbent was determine as 5.4 and this indicate the adsorbent are negatively charge and favour to uptake cationic molecules. The process conditions for optimum removal of ammonium ion removal are 60 min, pH 11 and adsorbent dosage of 3 g. This study showed that CB-HCl adsorbent can be used for wastewater treatment.

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