Preparation and Characterization of Bio-Nitrogen-Doped Activated Carbon

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Abstract

Blood (livestock blood) is a cheap and readily available biomass material with a relatively high protein content. In this study, bio-nitrogen doped activated carbon (BN-AC) was prepared by chemical activation method with nitrogen-rich pig blood as raw material and magnesium chloride as activator. The specific surface area of BN-AC is $283.719 \text{ m}^2/\text{g}$, and the pore volume is $0.128 \text{ cm}^3/\text{g}$. The optimum conditions for the preparation of BN-AC were as follows: the mass impregnation ratio of activator to blood powder was 2:1, the impregnation time was 12 h, and the activation temperature was 600°C . The forms of biological nitrogen in activated carbon were studied by elemental analysis, Boehm titration, FTIR and XPS. The results showed that the total basicity of 0.720 mmol/g , and acidity of 0.313 mmol/g of the BN-AC. The surface of the precursor has only one Pyrrolic N, and the surface of BN-AC contains Pyridinic N, Pyrrolic N and Graphitic N, the N content of the precursor was successfully preserved. BN-AC has higher methylene blue and iodine adsorption values than ordinary activated carbon.

Introduction

Activated carbon (AC) is an important and unique functional carbon material with a high degree of microporosity, high surface area and pore structure (Lv et al. 2020; Li et al. 2020; Hernández-Barreto et al. 2020). AC is often used mainly as adsorbent of gases, vapors, and water-dissolved chemical substances (Gómez-Serrano et al. 2020) and also as catalyst and catalyst support (Mohammed et al. 2018). Traditional AC materials include wood (Nikolas et al. 2020), nut shells (Huang et al. 2014), and different types of coal (Song et al. 2020). These materials considered as waste fractions from industry or agricultural products and residues (Davide et al. 2018). Therefore, many efforts have recently been made to prepare AC from renewable, abundant, and low-cost biomass (Liu et al. 2020). There are mainly two strategies that can be used for preparing nitrogen-containing activated carbons (N-ACs). One uses nitrogen-containing carbon precursors to directly synthesize N-ACs, and these precursors include peony pollen (Liu et al. 2020), LSW (Han et al. 2020), etc. The other strategy involves the addition of nitrogen containing chemicals, melamine (Yang et al. 2020), polyimine (Alabadi et al. 2016), etc. during the carbonization process. Generally, N-ACs prepared by the former strategy have high nitrogen contents and can be economically employed in large-scale production by using cheap raw materials (Han et al. 2020).

Livestock blood is not only a serious waste, but also pollutes the environment. Due to the high rate of meat production in the European Union, especially for pork, a large amount of porcine blood can result theoretically resulting in 777,000 tons (Christian et al. 2019). Most blood is wasted and largely unexploited. Similar to other biomass materials, Pig blood is widely available in nature and has a high protein content, meaning more nitrogen content, and can be used as raw material for the preparation of nitrogen-containing activated carbon.

In this study, firstly, bio-nitrogen-doped activated carbon (BN-AC) with ideal performance was synthesized by direct carbonization and activation of magnesium chloride from waste animal blood with high protein content. Secondly, BN-AC was characterized by Boehm titration, FTIR, XPS, XRD and SEM. Finally, BN-AC

was compared with ordinary activated carbon to do methylene blue and iodine adsorption experiments. Bio-nitrogen-doped activated carbon is of great significance to environmental protection, waste utilization and preparation of special activated carbon.

Materials And Methods

Raw material and Reagents

The blood powders were obtained from Nanning City in Guangxi Province in China. It was dried in oven at 100°C for 24 h. The samples ready for carbonization. The commercial activated carbon without containing nitrogen was supplied by provided by Tianjin Damao Chemical Reagents. Magnesium chloride, bromocresol green, sodium bicarbonate, phenolphthalein, potassium hydrogen phthalate, anhydrous sodium carbonate, potassium dihydrogen phosphate, potassium iodide, soluble starch, sodium thiosulfate (all the above reagents are analytical pure, purchased from Tianjin Damao Chemical Reagent Factory, China); Ethanol, phenol, sodium hydroxide (analytical pure, purchased from Tianjin Obokai Chemical Co. Ltd., China); Methylene blue (biological dye, purchased from Beijing Chemical Works, China); Sodium dihydrogen phosphate (analytical pure, purchased from Beijing Hongxing Chemical Plant); Iodine (analytically pure, purchased from Shanghai Guangnuo Chemical Technology Co. Ltd., China).

Preparation of BN-AC

The blood powders were activated at a moderate temperature of 600°C for 5 h. The activation process involved adding a solution of 65 wt% Magnesium chloride (MgCl_2) to carbon in the ratio of 2:1 (blood powder/activator) to form a paste, impregnate for 24 hours. The content was heated until complete evaporation was achieved. A muffle furnace was used to conduct the treatment at a temperature of 600°C in the air atmosphere. The dried paste of the activated carbon was rinsed with distilled water at room temperature. The cleaned BN-AC particles were further dried at 105°C for 24 h to ensure that the particles were completely dry. The powdered bio-nitrogen doped activated carbon was obtained by grinding.

Characterizations of BN-AC

SEM is mainly composed of scanning electron microscope (JSM-5610LV) from Nippon Electronics Co. LTD, the amplification factor was 800–2200, Gold spraying under vacuum condition was observed. Specific surface area was measured by the Brunauer-Emmett-Teller (BET) method. Elementary Vario EL CUBE (Vario EL III) was used to analyze the elementary compositions of samples. Boehm titration method was used to further determine oxygen functional groups as follows; 200 mg of each BN-AC sample was mixed in 25 mL of one of the four reactants of 0.1 mol/L concentration (NaHCO_3 , Na_2CO_3 , NaOH or HCl). The mixtures were sonicated for 24 h, then filtered to remove the carbon. The excess of base and acid in solution was titrated with 0.1 mol/L HCl solution of 0.1 mol/L NaOH . The number of acidic and basic sites were calculated on the basis that NaOH neutralizes carboxylic, phenolic and

lactone groups, NaHCO_3 neutralizes only carboxylic, Na_2CO_3 neutralizes carboxyl and lactone and HCl neutralizes all the basic sites. The Infrared spectra of the blood powders and activated carbon samples were determined with the Fourier Transform Infrared Spectroscopy (FTIR, Llantrisant, UK) Dry samples of 0.071mm were sampled by potassium bromide tablet method. The scanning wave number ranged from 4000 to 400 cm^{-1} . X-ray photoelectron spectroscopy (XPS) was performed on an ESCALAB 250Xi (Thermo Scientific, USA) with $\text{Al K}\alpha$ X-ray (1486.6 eV) as an excitation source. The base pressure in the analysis room was about 3×10^{-10} mbar. Iodine and methylene blue adsorption capacity of activated carbons were determined according to the Chinese GB/T 12496.8–2015 and GB/T 12496.10–1999 standards (for activated carbon), respectively.

Results And Discussion

By Elemental analysis

More than 80% of swine blood waste is protein. mainly containing C, N, O, H and other elements. The elemental analysis of precursor and BN-AC is shown in Table 1. According to Table 1, the carbon, nitrogen, oxygen and hydrogen content in the precursor were 49.443%, 11.846%, 26.014% and 12.697% respectively. The protein was unstable at high temperature. After the BN-AC was prepared from blood powder, the element content changed, and the proportion of nitrogen element retained by BN-AC was proportionately high. The presence of nitrogen content indicated that N was successfully doped in BN-AC sample. Due to the high nitrogen content in the protein of blood powder, a part of nitrogen was retained during the pyrolysis process, indicating that there may be some nitrogen-containing functional groups on the surface of BN-AC. There is a small amount of magnesium because BN-AC is prepared by impregnation 2:1 with magnesium chloride as the activator. Some magnesium ions enter into the structure of BN-AC in the activation process, which is not easy to clean, so they are deposited on the surface of BN-AC or in the pores.

Table 1
Elemental Analysis of precursor and BN-AC

Sample	C/dw%	N/dw%	O/dw%	H/dw%	Mg/dw%
precursor	49.443	11.846	26.014	12.697	-
BN-AC	54.625	18.244	15.833	9.282	2.016
From the elemental analysis of precursor: - indicates that the ingredient is not detected					

By Boehm titration analysis

The surface chemistry of the functional groups in this study, which determines the acidity and basicity of the surface oxygen groups of the BN-AC. The Boehm titration results of BN-AC is shown in Table 2. Total basicity of 0.720 mmol/g, and acidity of 0.313 mmol/g of the BN-AC was obtained from the analysis. Thus, there was 2.3 times the number of basic groups as acidic groups. The content of acid and base

indicated that the surface of BN-AC contained acidic functional groups containing oxygen and alkaline functional groups containing nitrogen. With alkaline functional groups such as C = N, NH, amino, cycloamide, nitrile and pyrrole groups (Huang et al. 2020; Thue et al. 2017). The existence of basic functional groups is due to the fact that the precursor containing a large number of proteins as raw materials retains more nitrogen content.

Table 2

Boehm titration results of BN-AC

Sample	Total amount of acid/mmol g ⁻¹	The total amount of alkali/mmol g ⁻¹	Carboxyl/ mmol g ⁻¹	Ester base carbonyl/ mmol g ⁻¹
BN-AC	0.313	0.720	0.357	0.175

By FTIR spectroscopy

Infrared spectra are generated by the transition of vibrational energy levels (accompanied by rotational energy levels) of molecules. It can be widely used in the characterization of the type and number of surface functional groups on activated carbon. The surface chemical properties of activated carbon are mainly determined by the type and number of surface functional groups, while the functional groups on the microporous surface of activated carbon are mostly oxygen-containing functional groups, such as carboxyl, phenolic hydroxyl, carbonyl, ester, ketone, ether, etc (Nina et al. 2020; Yousif Mohammed et al. 2018.).

For more investigation on the adsorbents surface properties, Fig. 2 shows the infrared spectrum curve of precursor and BN-AC. The precursor and BN-AC display a number of spectral features in the range of wavenumber between 4000 – 400 cm⁻¹. The BN-AC show that the characteristic broad band at ca 3415 cm⁻¹ can be assigned to N-H and/or O-H stretching vibration. The weak band at 1624 cm⁻¹ is attributed to the distinctive absorbance of C-H bonds of benzene rings as well as the C = N bonds from the carbon framework (Alabadi et al. 2016). The peaks at 1233 cm⁻¹ suggest the presence of the BN-AC stretching vibration. The broad peak of 1099 cm⁻¹ is associated with C-N stretching vibration. The FTIR analysis, therefore, confirms the existence of N-H and C-N species in the carbon samples. It still has some nitrogen-containing groups. -NO₂ symmetric stretching vibration at wave number 1387 cm⁻¹ of precursor (Tadepalli et al. 2021). The precursor and BN-AC had similar peaks, with significant differences at the peaks of 3415 cm⁻¹ and 1624 cm⁻¹, related to the O-H, N-H, NH₂, C = C, C = N, C-N (Wang et al. 2018) which is caused by the gradual increase of carbon net and the appearance of carbon-containing functional groups under the catalysis of magnesium chloride at the end of activation of precursor.

By XPS analysis

X-ray photoelectron spectroscopy (XPS) is an effective method for detecting surface chemical structure. XPS was used to qualitatively analyze the functional groups on the surface of activated carbon (Liu et al. 2020). Figure 3 (a) is the wide full XPS spectra of precursor and BN-AC. As seen from the graphs, characteristic peaks of C1s, O1s, N1s are found in precursor and BN-AC, witnessing that N have been successfully retain on BN-AC. The peaks located in 285.56, 399.26 and 532.35 eV are corresponding with C1s, N1s and O1s, respectively. The Mg1s peak appeared in raw material at 1 305.52 eV. The Mg1s peak appears because BN-AC is activated by magnesium chloride with a concentration of 65% as the activator. During the activation process, magnesium ions enter into the structure of BN-AC and are not easy to be cleaned, so they are deposited on the surface or pores of BN-AC. This is because at relatively high temperatures, the aromatic rings of nitrogen are partially stable at the edges of the graphite layer of the carbon material. With the further increase of temperature, nitrogen gradually enters into the skeleton of BN-AC and plays a dominant role (Yao et al. 2020).

As shown in Fig. 3 (c), there is only peaks that locate at 399.48 eV (pyrrolic N) in precursor. The N1s XPS spectra of BN-AC could be deconvoluted into three types of N-containing compounds, and results are depicted in Fig. 3 (b). The peaks of N1s located in 398.709 (pyridinic N), 399.45 (pyrrolic N) and 400.15 (graphitic N) (Yang et al. 2019), respectively in BN-AC. Compared with the N1s XPS spectra of precursor and BN-AC, it was found that the preparation of BN-AC from blood containing protein would increase the types and number of nitrogen-containing functional groups in BN-AC. Pyrrolic N is converted into three nitrogen-containing groups, namely 398.709 eV (pyridinic N), 399.45 eV (pyrrolic N) and 400.15 eV (graphitic N). The O1s XPS spectrum of the BN-AC shown exhibits three peaks in Fig. 3 (d). at 530.44 eV, 531.539 eV and 533.04 eV, corresponding to (C = O), (C-O) and (C-O-C). Figure 3 (e) shows high-resolution C1s XPS spectrum of BN-AC, which can be separated into two peaks at 284.662 eV(C-C) and 286.739 eV(C = N) (Oluwatosin et al. 2019).

Table 3 shows the binding energy and specific area of precursor and BN-AC. From Table 3, in the specific functional groups in BN-AC, the proportion of nitrogen-containing groups increased significantly. The N contents of pyrrolic N, pyridinic N and graphitic N increased by 42.5%, 31.03%, 5.14%. Using waste blood (containing a lot of protein) as raw material to prepare activated carbon will increase the types and quantity of nitrogen-containing functional groups in activated carbon. In the presence of precursor, which can act as a nitrogen source, in turn cause nitrogen content to increase. For this reason, the N content of the precursor was successfully preserved.

Table 3
Binding energy and proportion area of BN-AC and precursor functional groups

Functional groups	Precursor Binding Energy/eV	Precursor Accounted for area/%	BN-AC Binding Energy/eV	BN-AC Accounted for are /%
C-C	285.75	56.15	284.662	42.99
C = N	286.75	9.69	286.739	13.16
C-O-C	287.58	4.88	287.61	8.01
pyrrolic N	399.48	18.58	399.45	61.08
pyridinic N	-	-	398.709	31.03
graphitic N	-	-	400.15	5.14
C = O	531.76	3.15	530.44	25.49
C-O	531.059	74.30	531.539	63.20
C-O-C	533.26	3.70	533.04	11.32
- Indicates that the sample is not tested				

Textural characterization

Surface area

The pore size of BN-AC is 1.688 nm, belonging to micropore (< 2 nm). The specific area of blood powder was 0.826 cm³/g, and the specific surface area of BN-AC and micropores were 283.719 m²/g and 135.036 m²/g, respectively. The precursor has almost no pore size and specific surface area, and the specific surface area increases by 343.5 times after activation with magnesium chloride solution to prepare BN-Ac. Table 4 shows that the waste blood containing protein can successfully prepare bio-nitrogen doped activated carbon.

Table 4. Specific surface area and pore structure parameters of precursor and BN-AC

Sample	S _{BET} /m ² g ⁻¹	S _{micro} /m ² g ⁻¹	V _{total} /cm ³ g ⁻¹	V _{micro} /cm ³ g ⁻¹	D/nm
precursor BN-AC	0.826	-	-	-	-
	283.720	135.036	0.289	0.128	1.688
S _{BET} , total specific surface area; S _{micro} , specific surface area of micropores; V _{total} , total pore volume; V _{micro} , micropore volume; -, is not tested.					

By SEM analysis

The morphology including the porosity of the prepared BN-AC could be clearly observed from their SEM micrographs, as shown in Fig 1. Without any activation, BN-AC from blood(a) shows compact and smooth surface with little pores. On the surface of BN-AC (b) activated by magnesium chloride, there are small particles and unevenly distributed, and a small amount of floc structure with loose structure. The loose structure will make the microporous structure become developed. Results of SEM analysis indicate that BN-AC with different porosity have been successfully prepared.

Determination of adsorption performance

The methylene blue adsorption value represents the decolorization ability of the adsorbent, and the methylene blue adsorption value is also an important index for the characterization of the liquid adsorption performance of activated carbon. Iodine adsorption value indicates the developed degree of micropores greater than 1.0 nm of activated carbon, which is the performance of the adsorption capacity of activated carbon to small molecule impurities.

Table 5. Determination results of BN-AC adsorption values

Sample	Methylene blue adsorption value (mg/g)	Iodine adsorption value(mg/g)
precursor	0	0
BN-AC	600.00	734.95
Ordinary AC	450.00	680.24

Table 5 is the adsorption value measurement results of the precursor, BN-AC and ordinary activated carbon. The precursor has no adsorption property due to its small specific surface area. The methylene blue adsorption capacity of BN-AC is 600 mg/g, and the iodine adsorption capacity is 734.95 mg/g. The adsorption capacity of methylene blue on normal activated carbon is 450 mg/g, and the adsorption capacity of iodine is 680.24 mg/g. The methylene blue and iodine adsorption values of BN-AC were higher than those of ordinary AC, which indicated that the prepared bio-nitrogen doped activated carbon had potential adsorption properties.

Conclusions

Compared with ordinary activated carbon, the specific surface area of bio-nitrogen doped activated carbon is lower, but the methylene blue and iodine adsorption values of BN-AC were higher than those of ordinary AC. It may be that the nitrogen containing functional groups in BN-AC provide a favorable effect on the adsorption performance. The total alkalinity and total acidity of functional groups on the surface of BN-AC were 0.720 mmol/g and 0.313 mmol/g respectively. In the infrared spectrum, N-H, NH₂, C = N

functional groups exist at 3415 cm^{-1} and 1624 cm^{-1} . XPS analysis showed that the surface of the precursor has only one Pyrrolic N, and the surface of BN-AC contains Pyridinic N, Pyrrolic N and Graphitic N, the N content of the precursor was successfully preserved.

Declarations

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Author contributions Xingping Zhang performed literature study, data analysis, making figures and tables and technical writing. Haichao Li supervised the the results and data analysis and performed the technical revisions of the manuscript. Guangle Wang, Qingsong Ji and Tian Liang were performed revisions of figures and tables. All authors read and approved the final manuscript.

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Consent to participate Not applicable

Consent to publish Not applicable

Competing interests Not applicable

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Data availability and materials Not applicable

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Figures

Figure 1

SEM images of precursor (a) and BN-AC (b)

Figure 2

FTIR of precursor and BN-AC

Figure 3

X-ray photoelectron spectrograms of precursor and BN-AC (a) and fine spectrograms of BN-AC (b-e)