

TD-GC-MS Analysis on Antibacterial and Safety Characteristics of Volatiles from Bamboo during Cavitation, Carbonization and Activation for Public Health

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In this study, bamboo was subjected to cavitation, carbonization and activation, to reveal the antibacterial activity and improve its environmental friendliness. The resulting products were characterized by TD-GC-MS. The results indicated that volatiles from cavitated rod and activated carbon comprised mainly acidic compounds, while those from charcoal were mainly phenolic. The volatiles had antibacterial activity. However, this suggests that bamboo activated carbon may be a safer alternative to charcoal for indoor use.

Key words: Bamboo activated carbon; Bamboo charcoal; cavitated rod; Environmental friendliness; antibacterial activity; volatiles.

Bamboo is a widely used and sustainable bioresource. It is a grass, rather than a tree. Bamboo is fully mature after 6–7 years, and can regrow from its existing root structure when its stalks are cut. This makes bamboo a sustainable and environmentally friendly resource¹. It is commonly used as a food source, and to make household goods including furniture, sporting goods, dinnerware, jewelry and handbags^{2,3}. Bamboo could potentially replace less sustainable crops in many areas, and create a source of income⁴. It is an important economic source for low income people in China and India [5,6]. In practice, only 40% of bamboo biomass is efficiently used, with the remainder becoming waste. Bamboo charcoal is prepared by burning

fragments of plants 5 years or older in an oven at 500–1200 °C. Bamboo charcoal can consume a significant proportion of bamboo's biomass and waste, and benefits the environment by reducing pollutant residue. The use of bamboo charcoal in China has been documented since 1486 AD, during the Ming Dynasty in Chuzhou Fu Zhi. In modern Japan and China, bamboo charcoal is largely used as a fuel for drying tea and cooking.

Bamboo biomass has an extraordinary microstructure. Bamboo charcoal is highly absorbent, and can be used to purify water by removing organic impurities^{7,8}. Bamboo charcoal can absorb atmospheric odors from residential housing environments. It can prevent the growth of harmful bacteria, because its high porosity and surface area help to regulate humidity, and absorb sweat and other chemicals⁹. The combustion of bamboo charcoal emits negative ions, which helps to counteract positive ions emitted from televisions, computers, and modern electronics⁹.

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Adding bamboo charcoal to the diets of fish and poultry may also increase their growth rates⁷. However, the adsorption of bamboo charcoal is limited, and bamboo charcoal often requires further activation.

In recent years, research on activation and application of bamboo charcoal has increased. The activated carbon from waste Nigerian bamboo has been used to remediate industrial pollutants¹⁰. Furthermore, waste bamboo activated with HNO₃ has been shown to more effectively remove metal ions from waste streams, via different metal recovery processes, than activated carbon from coconut and palm kernel shells^{11,12}. Bamboo activated carbon was prepared, carbonized, and impregnated with different concentrations of four acids at 800 °C in a muffle furnace for 2 h, to determine an optimal activation agent for adsorbing BTX vapor as 0.143 M trioxonitrate (v) acid^{11,12}. Activated carbon fibers prepared from bamboo at 850 °C have exhibited high adsorption capacities for iodine and methylene blue, with higher activation temperatures yielding a more crystalline microstructure¹³. After KOH activation, bamboo activated carbon exhibits a narrow micropore range, having narrow micropores with diameters ~0.55 nm, which contributes to a high CO₂ adsorption¹⁴. Bamboo activated carbon treated with NaCl at elevated temperatures for up to 180 min, exhibited a color removal efficiency of ~77%, and its adsorption of acidic dye obeyed Langmuir behavior¹⁵. In summary, bamboo activated carbon can be used to adsorb and remove organic species, lead, chlorine, unpleasant tastes and odors in effluent, and colors from gas/liquid streams. Bamboo charcoal and activated carbon have received much research attention, particularly their processing parameters and product advantages. However, their environmental characteristics received little attention¹⁶⁻¹⁸.

In the current study, bamboo is subjected to cavitation, carbonization and activation, to reveal the antibacterial activity and improve its environmental friendliness. The resulting materials are analyzed by thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS).

MATERIALS AND METHODS

Preparation

Bamboo (*Phyllostachys heterocycla*) was collected from the Jinggangshan Forest Region, P. R. China. The bamboo was broken into pieces, and extruded into a cavitated rod (zhu-1) at 180 200 °C by a cavitation extrusion machine. The cavitated rod was carbonized in oxygen free air at 600 °C for 6 h, to yield bamboo charcoal (zhu-2). The bamboo charcoal was activated at 800 900 °C in a steam of activated carbon (zhu-3), using an apparatus manufactured by Japanese Mountain King Power Engineering Co., Ltd. Japan.

TD-GC-MS analysis

Samples (5 g) were placed in the sample tubes of a Master TD thermal desorber (DANI Co., Ltd., Italy), and the sample tubes were purged with 120 °C He for 30 min. The trap adsorption temperature was set to 120 °C, trap resolution temperature to 130 °C, valve temperature to 130 °C, and transmission line temperature to 130 °C. The volatiles were desorbed for 15 min and analyzed by an online linked gas chromatograph/mass spectrometer (GC/MS), an Agilent 6890N+5795C GCMSTM (Agilent Co., Ltd., USA), which was linked to a mass selective detector. An elastic quartz capillary column (DB-5MS; 30 m × 0.25 mm × 0.25 μm) coated with a neutral phase (Hewlett-Packard-5 cross-linked 5% phenyl methyl silicone) was used. The carrier gas was helium and the injection port temperature was 280 °C. The GC temperature program began at 45 °C for 3 min, increased at 8 °C/min to 120 °C, then increased at 20 °C/min to 300 °C, was held for 5 min, followed by a split injection at a ratio of 30:1. The MS program scanned over a range of 29–500 AMU (m/z), at an ionizing voltage of 70 eV. The flow velocity of the He carrier gas was 1.2 mL/min. Ion source temperature: 230 °C, quadrupole temperature: 150 °C¹⁹.

RESULTS AND DISCUSSION

The total ion chromatograms of the cavitated rod, charcoal and activated carbon as measured by TD-GC-MS are shown in Figure 1. The relative content of each component was

counted by area normalization. The data was analyzed using the NIST standard MS map software. Comparison with reported literature allowed individual components to be identified (listed in Table 1, Table 2 and Table 3).

Analysis on molecules of volatiles from bamboo during activated carbon preparation

According to GC/MS result, 17 components were identified from the 21 peaks of volatiles from cavitated rod. The result showed that the components were acetic acid (55.31%, 2.689/3.801 min), formic acid (21.97%, 1.871/3.004 min), furfural (19.14%, 5.595/6.004 min), phenol (0.55%, 8.900 min), 1-hydroxy-2-butanone (0.40%, 4.924 min), butyrolactone (0.39%, 7.463 min), 5-methyl-2-

furancarboxaldehyde (0.34%, 8.271/8.449 min), 2-furanmethanol (0.28%, 6.403 min), 1H-pyrrole-2-carboxaldehyde (0.28%, 9.603 min), 1,2-dimethoxy-ethene (0.28%, 4.169 min), 2-methoxy-phenol (0.20%, 10.967 min), 4-ethyl-phenol (0.19%, 12.624 min), 2,3-dihydro-benzofuran (0.19%, 13.527 min), [2R-(2 α , 4 α , 8 α)]-1,2,3,4,4a,5,6,8a-octahydro- α , α , 4a, 8-tetramethyl-2-naphthalene-methanol (0.17%, 17.335 min), 1,2-ethanediol, diacetate (0.14%, 6.571 min), [1aR-(1 α , 3 α , 7 β)]-1a,2,3,3a,4,5,6,7b-octahydro-1,1,3a,7-tetramethyl-1H-cyclopropa[a]naphthalene (0.13%, 17.167 min), [1S-(1 α , 4 α , 8 α)]-1,2,4a,5,8,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-naphthalene (0.04%, 16.905 min).

Table 1. The analytical results of cavitated rod by TD-GC-MS

No.	R.T. (min)	P. A. (%)	Name
1	1.871	0.65	formic acid
2	2.689	15.65	acetic acid
3	3.004	21.32	formic acid
4	3.801	39.66	acetic acid
5	4.169	0.28	1,2-dimethoxy-ethene
6	4.924	0.40	1-hydroxy-2-butanone
7	5.595	15.48	furfural
8	6.004	3.66	furfural
9	6.403	0.28	2-furanmethanol
10	6.571	0.14	1,2-ethanediol, diacetate
11	7.463	0.39	butyrolactone
12	8.271	0.21	5-methyl-2-furancarboxaldehyde
13	8.449	0.13	5-methyl-2-furancarboxaldehyde
14	8.900	0.55	phenol
15	9.603	0.28	1H-pyrrole-2-carboxaldehyde
16	10.967	0.20	2-methoxy-phenol
17	12.624	0.19	4-ethyl-phenol
18	13.527	0.19	2,3-dihydro-benzofuran
19	16.905	0.04	[1S-(1 α , 4 α , 8 α)]-1,2,4a,5,8,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-naphthalene
20	17.167	0.13	[1aR-(1 α , 3 α , 7 β)]-1a,2,3,3a,4,5,6,7b-octahydro-1,1,3a,7-tetramethyl-1H-cyclopropa[a]naphthalene
21	17.335	0.17	[2R-(2 α , 4 α , 8 α)]-1,2,3,4,4a,5,6,8a-octahydro- α , α , 4a, 8-tetramethyl-2-naphthalenemethanol

Table 2. The analytical results of bamboo charcoal by TD-GC-MS

No.	R.T. (min)	P.A. (%)	Name
1	1.965	-25.63	formic acid
2	3.182	-46.89	acetic acid, hydroxy-, methyl este
3	4.095	0.92	propanoic acid
4	4.210	1.81	1-hydroxy-2-butanone
5	5.459	39.50	furfural
6	5.983	4.46	2-furanmethanol
7	6.162	2.28	1-(acetyloxy)-2-propanone
8	7.305	18.38	butyrolactone
9	7.515	2.23	2,5-hexanedione
10	8.249	4.58	5-methyl-2-furancarboxaldehyde
11	8.522	1.01	methyl 2-furoate
12	8.784	16.48	phenol
13	9.267	5.28	1-(2-furanyl)-1-propanone
14	9.760	11.52	3-methyl-1,2-cyclopentanedione
15	10.295	3.47	2-methyl-phenol
16	10.925	15.23	2-methoxy-phenol
17	11.187	4.44	3-methyl-2-butanamine
18	11.470	6.10	maltol
19	12.540	11.62	4-ethyl-phenol
20	12.907	0.76	2-propyl-phenol
21	13.432	3.42	2,3-dihydro-benzofuran
22	14.082	4.10	4-ethyl-2-methoxy-phenol
23	14.439	1.12	5-acetoxymethyl-2-furaldehyde
24	14.880	10.84	2,6-dimethoxy-phenol
25	15.184	1.01	3,5-dihydroxy-benzoic acid
26	16.821	0.30	hexadecane
27	17.062	0.66	cedrol
28	17.324	0.97	[2R-(2 α , 4 α , 8 α)]-decahydro- α , α , 4a-trimethyl-8-methylene-2-naphthalenemethanol

From the GC/MS results, 26 components were identified from 28 peaks of volatiles from bamboo charcoal. The result showed the components as furfural (39.50%, 5.459 min), butyrolactone (18.38%, 7.305 min), phenol (16.48%, 8.784 min), 2-methoxy-phenol (15.23%, 10.925 min), 4-ethyl-phenol (11.62%, 12.540 min), 3-methyl-1,2-cyclopentanedione (11.52%, 9.760 min), 2,6-dimethoxy-phenol (10.84%, 14.880 min), maltol (6.1%, 11.470

min), 1-(2-furanyl)-1-propanone (5.28%, 9.267 min), 5-methyl-2-furancarboxaldehyde (4.58%, 8.249 min), 2-furanmethanol (4.46%, 5.983 min), 3-methyl-2-butanamine (4.44%, 11.187 min), 4-ethyl-2-methoxy-phenol (4.10%, 14.082 min), 2-methyl-phenol (3.47%, 10.295 min), 2,3-dihydro-benzofuran (3.42%, 13.432 min), 1-(acetyloxy)-2-propanone (2.28%, 6.162 min), 2,5-hexanedione (2.23%, 7.515 min), 1-hydroxy-2-butanone (1.81%, 4.210 min), 5-acetoxymethyl-2-furaldehyde (1.12%, 14.439 min), methyl 2-furoate (1.01%, 8.522 min), 3,5-dihydroxybenzoic acid (1.01%, 15.184 min), [2R-(2 α ,4 α ,8 α)]-decahydro- α , α ,4a-trimethyl-8-methylene-2-naphthalenemethanol (0.97%, 17.324 min), propanoic acid (0.92%, 4.095 min), 2-propyl-phenol (0.76%, 12.907 min), cedrol (0.66%, 17.062 min), hexadecane (0.30%, 16.821 min).

According to GC/MS result, 30 components were identified from 34 peaks of volatiles from bamboo activated carbon. The result showed that the components were acetic acid (51.85%, 2.511/2.972/3.843 min), furfural (13.31%, 5.448/6.015 min), 2,6-dimethoxy-phenol (5.30%, 14.88 min), phenol (4.29%, 8.805/8.900 min), formic acid (4.28%, 3.14 min), 4-ethyl-phenol (2.56%, 12.54 min), 2-methoxy-phenol (2.41%, 10.904 min), ethyl ether (2.06%, 4.168 min), butyrolactone (1.33%, 7.536 min), 4-ethyl-2-methoxy-phenol (1.29%, 14.072 min), 1-hydroxy-2-butanone (1.13%, 4.997 min), 3-methoxy-pyridine (1.03%, 9.298 min), vanillin (1.00%, 15.404 min), 2,5-dihydroxypropiophenone (0.85%, 15.184 min), 2,3-dihydro-benzofuran (0.81%, 13.421 min), maltol (0.72%, 11.481 min), *N*-methyl-1,3-propanediamine (0.70%, 11.208 min), 3-methyl-1,2-cyclopentanedione (0.64%, 9.791 min), 5-methyl-2-furancarboxaldehyde (0.57%, 8.281 min), (*E*)-2-methoxy-4-(1-propenyl)-phenol (0.57%, 15.782 min), 2-furanmethanol (0.55%, 6.445 min), 2-hydroxy-3,4-dimethyl-2-cyclopenten-1-one (0.47%, 10.327 min), 1H-pyrrole-2-carboxaldehyde (0.45%, 9.634 min), 1-(2-furanyl)-ethanone (0.40%, 7.074 min), 2-furancarboxylic acid, hydrazide (0.38%, 8.512 min), 5-acetoxymethyl-2-furaldehyde (0.34%, 14.439 min), 1,2-ethanediol, diacetate (0.29%, 6.613 min), 2,6,10,14-tetramethyl-pentadecane (0.27%, 17.460 min), 2,6,10,14-tetramethyl-

Table 3. The analytical results of bamboo activated carbon by TD-GC-MS

No.	R.T. (min)	P.A. (%)	Name
1	2.511	18.57	acetic acid
2	2.972	2.70	acetic acid
3	3.140	4.28	formic acid
4	3.843	30.58	acetic acid
5	4.168	2.06	ethyl ether
6	4.997	1.13	1-hydroxy-2-butanone
7	5.448	13.07	furfural
8	6.015	0.24	furfural
9	6.445	0.55	2-furanmethanol
10	6.613	0.29	1,2-ethanediol, diacetate
11	7.074	0.40	1-(2-furanyl)-ethanone
12	7.536	1.33	butyrolactone
13	8.281	0.57	5-methyl-2-furancarboxaldehyde
14	8.512	0.38	2-furancarboxylic acid, hydrazide
15	8.805	1.39	phenol
16	8.900	2.90	phenol
17	9.298	1.03	3-methoxy-pyridine
18	9.634	0.45	1H-pyrrole-2-carboxaldehyde
19	9.791	0.64	3-methyl-1,2-cyclopentanedione
20	10.327	0.47	2-hydroxy-3,4-dimethyl-2-cyclopenten-1-one
21	10.904	2.41	2-methoxy-phenol
22	11.208	0.70	<i>N</i> -methyl-1,3-propanediamine
23	11.481	0.72	maltol
24	12.540	2.56	4-ethyl-phenol
25	13.421	0.81	2,3-dihydro-benzofuran
26	14.072	1.29	4-ethyl-2-methoxy-phenol
27	14.439	0.34	5-acetoxymethyl-2-furaldehyde
28	14.880	5.30	2,6-dimethoxy-phenol
29	15.184	0.85	2,5-dihydroxypropiophenone
30	15.404	1.00	vanillin
31	15.782	0.57	(<i>E</i>)-2-methoxy-4-(1-propenyl)-phenol
32	17.051	0.08	cedrol
33	17.460	0.27	2,6,10,14-tetramethyl-pentadecane
34	18.058	0.09	2,6,10,14-tetramethyl-hexadecane

hexadecane (0.09%, 18.058 min), cedrol (0.08%, 17.051 min).

Analysis on Antibacterial and Safety Characteristics from bamboo during activated carbon preparation

The TD-GC-MS results indicated the molecular distributions of the cavitated rod, charcoal and activated carbon. The retention times of the components of these samples showed a particular trend. Species with retention times of ≤ 10 , ≤ 15 and > 15 min constituted 99.08, 0.58 and 0.34% of the cavitated rod, respectively. Species with retention times of ≤ 10 , ≤ 15 and > 15 min constituted 35.93, 61.10 and 2.94% of the charcoal, respectively. Species with retention times of ≤ 10 , ≤ 15 and > 15 min constituted 82.56, 14.60 and 2.86% of the activated carbon, respectively. This suggested that the 120 °C volatiles of the cavitated rod and activated carbon predominantly had retention times of ≤ 10 min, and that the 120 °C volatiles of the charcoal had retention times of 10–15 min. Acids, phenol and its derivatives, and other species constituted 77.28, 0.94 and 21.78% of the

cavitated rod, respectively. Acids, phenol and its derivatives, and other species constituted 1.93, 62.50, and 35.54% of the charcoal, respectively. Acid, phenol and its derivatives, and other species constituted 56.13, 17.27, and 26.62% of the activated carbon, respectively. This suggested that the 120 °C volatiles of the cavitated rod and activated carbon were mainly acidic compounds, and that the 120 °C volatiles of the charcoal were mainly phenol and its derivatives. Phenol and its derivatives were largely produced during carbonization. Activation volatilized and decreased the content of phenol and its derivatives. This means that bamboo activated carbon is a safer alternative to bamboo charcoal for indoor use. The cavitated rod contained 2-furanmethanol. The bamboo charcoal contained maltol (6.10%), 2-furanmethanol, 1-(acetyloxy)-2-propanone, 2,5-hexanedione, 1-hydroxy-2-butanone and cedrol. The bamboo activated carbon contained vanillin, maltol (0.08%), 2-furanmethanol and cedrol. The bamboo charcoal and activated carbon exhibited a fragrance, with the charcoal smelling more ‘burnt’ than the activated carbon.

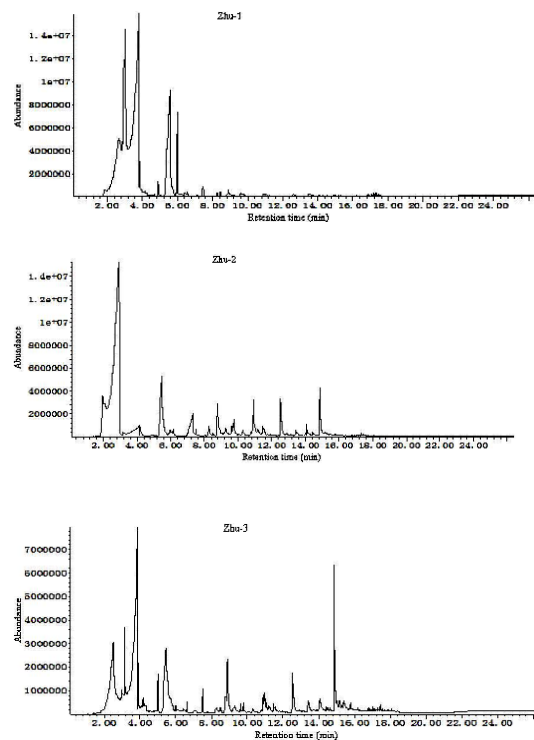


Fig. 1. Total ion chromatogram of bamboo during activated carbon preparation

CONCLUSIONS

The chemical structure of bamboo during cavitation, carbonization and activation were characterized by TD-GC-MS. The 17, 26 and 30 components were identified in the cavitated rod, charcoal, activated carbon by TD-GC-MS. Volatile species of the cavitated rod and activated carbon largely had retention times of ≤ 10 min, while those of the charcoal were 10–15 min. Volatiles from the cavitated rod and activated carbon were mainly acidic compounds, while those from bamboo charcoal were mainly phenolic. This result suggests that bamboo activated carbon is a safer alternative to bamboo charcoal for indoor use. Furthermore, the charcoal and activated carbon exhibited a pleasant fragrance, with the charcoal smelling more ‘burnt’ than the activated carbon.

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