# TD-GC-MS Analysis on Antimicrobial Properties of *Vitex negundo* Wood/PF Bio Composites for Drug Store

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The materials for drug store need be antimicrobial. However, Vitex negundo was easy to go mouldy. Therefore, Vitex negundo wood was pretreated by PF and made composite by hot pressing, the antimicrobial properties of wood/PF bio composites was analyzed by thermal desorption-gas chromatography/mass spectrometry (TD-GC/MS). The results showed that *Vitex negundo* wood/PF bio composites contained much phenol and little formaldehyde, suggesting that wood/PF bio composites could inhibit mold growth. And the wood/PF bio composites could safely be used for drug store.

> Key words: Drug store, *Vitex negundo*, Wood/PF bio composites, Antibacterial properties, TD-GC-MS.

Drugs were used to treat manage symptoms of infectious diseases, chronic diseases, and helped relieve suffering and pain. Drugs might be generally safe if they were correctly used and safely stored<sup>1</sup>. However, drugs have both benefits and risks, and there were all risks in taking any drug<sup>2</sup>. Especially, Patients couldn't be helped reduce the risk of harm if drugs were unintentionally polluted and took, resulting that more than 700,000 visits to hospital emergency departments only in the United States each year<sup>3,4</sup>. Though the guidance document had either a long-term or shortterm drug need and put into place effective management systems to support them in the setting, drug poisoning continued to occur on a regular basis<sup>5,6</sup>. In remote rural areas and desolate mountains, drugs were polluted and took very much because storage apparatuses easy went moldy 7-16.

Vitex negundo, which was commonly known as the five-leaved chaste tree, was a large aromatic shrub with quadrangular, densely whitish, tomentose branchlets. It was widely used in folk medicine, particularly in South and Southeast Asia. Vitex negundo was used for treating stored garlic against pests and as a cough remedy in the Philippines. Roots and leaves used in eczema, ringworm and other skin diseases, liver disorders, rheumatic pain, spleen enlargement, gout, abscess, backache; seeds used as vermicide. It was also used to control population of mosquitoes. In the USA, hardiness zone 6-9, its purple flowers bloom most of the summer and it is a popular plant visited by bees and butterflies. However, Vitex negundo contained richly the extractives which had lots of constituents<sup>10-32</sup>, and was easy to go mouldy. To be happy, phenol formaldehyde resin, which contained phenol and formaldehyde, had antibacterial effect. Therefore, Vitex negundo wood was pretreated by PF and made composite by hot pressing, the antimicrobial properties of wood/PF bio composites was analyzed by thermal desorption-gas chromatography/mass spectrometry (TD-GC/MS).

### MATERIALSAND METHODS

*Vitex negundo*, which was grown in Shaoyang Forest, was dried at 105°C for 10 h. Phenol formaldehyde resin had a relative molecular mass of 300 Da, a viscosity of 12–17 MPa·s at

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 $25^{\circ}$ C, a solid content of 40–42%, and a cure time of 80–100 s.

#### Sample preparation

Vitex negundo wood was soaked in phenol formaldehyde resin at room temperature for 30 h, and was air-seasoned for 2 days to about 5%. And the wood was soaked in phenol formaldehyde resin for 90 to 120 s, then dried for 2–3 days. The pieces were formed into wood/PF bio composites by hot pressing with the temperature of 160°C, pressure of 4 MPa, and time of 3 min. then wood/PF bio composites was crushed into 200mu powder<sup>33</sup>.

#### **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 desorped 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  $\times$  0.25 im) 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, quadropole temperature: 150 °C<sup>34</sup>.

#### **RESULTS AND DISCUSSION**

The total ion chromatograms of *Vitex negundo* wood/PF bio composites as measured by TD-GC-MS are shown in Fig.1. The relative content of each component was counted by area normalization. The data was analyzed using the NIST standard MS map software<sup>9-34</sup>. Comparison with reported literature allowed individual

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components to be identified, and the results were listed in Table 1, Table 2 and Table 3.

According to GC/MS result, 20 components were identified from the 21 peaks of Y-1 samples which constituted 97.21% of the total peak area. The result showed that the components were acetic acid, phenol, phenol, benzaldehyde, 2hydroxy-, nonanal, 2-furancarboxaldehyde, 5-(hydroxymethyl)-, salicyl alcohol, phenol, 2,6dimethoxy-, 2,5-dihydroxypropiophenone, vanillin, phenol, 2-methoxy-4-(1-propenyl)-,(e)-, ethanone, 1-(4-hydroxy-3-methoxyphenyl)-, phenol, 2,4bis(1,1-dimethylethyl), benzeneacetic acid, 4hydroxy-3-methoxy-, methyl ester, phenol, 2,6dimethoxy-4-(2-propenyl)-, pentadecane, 2,6,10,14tetramethyl, nonahexacontanoic acid, octacosyl heptafluorobutyrate, eicosane, 1,2benzenedicarboxylic acid, bis 2-methylpropyl) ester, 1,4-dimethyl-8isopropylidenetricyclo[5.3.0.0(4,10)]decane.

According to GC/MS result, 24 components were identified from the 24 peaks of J-1 samples. The result showed that the components were formaldehyde, hydrazine, 1,2-dimethyl-, acetic acid, 2-isopropoxyethylamine, formic acid, acetic acid, phenol, benzaldehyde, 2-hydroxy-, phenol, 4-methyl-, benzoic acid, 2-furancarboxaldehyde, 5-(hydroxymethyl)-, methenamine, salicyl alcohol, phenol, 2,6-dimethoxy-, benzaldehyde, 3-hydroxy-4-methoxy-, phenol, 2-methoxy-4-(1-propenyl)-(e)-, ethanone, 1-(4-hydroxy-3-methoxyphenyl)-, phenol, 2,4-bis(1,1-dimethylethyl), benzeneacetic acid, 4-hydroxy-3-methoxy-, methyl ester, phenol, 2,6-dimethoxy-4-(2-propenyl)-, pentadecane, 2,6,10,14-tetramethyl, ethanol, 2-(dodecyloxy)-, phthalic acid, isobutyl nonyl ester, 1,4-dimethyl-8isopropylidenetricyclo[5.3.0.0(4,10)]decane.

According to GC/MS result, 31 components were identified from the 32 peaks of S-1 samples. The result showed that the components were acetic acid, oxo-, acetic acid, dimethyl ether, acetic acid, phenol, 1h-pyrrole-2-carboxaldehyde, phenol, benzaldehyde, 2-hydroxy-, phenol, 4-methyl-, nonanal, benzoic acid, benzofuran, 2,3-dihydro-, 2-furancarboxaldehyde, 5-(hydroxymethyl)-, salicyl alcohol, 2-furancarboxylic acid, hydrazide, 2-methoxy-4-vinylphenol, phenol, 2,6-dimethoxy-, 2,5-dihydroxypropiophenone, vanillin, phenol, 2-methoxy-4-(1-propenyl)-,(e)-, ethanone, 1-[4-

(methylthio)phenyl], phenol, 2,4-bis(1,1dimethylethyl), benzeneacetic acid, 4-hydroxy-3methoxy-, methyl ester, pentadecane, 3-methyl-, phenol, 2,6-dimethoxy-4-(2-propenyl)-, tridecane, 3-methyl-, n-tetracosanol-1, pentadecane, 2,6,10,14tetramethyl, tetratriacontane, 17-hexadecyl-, 1,2benzenedicarboxylic acid, bis(2-methylpropyl) ester, phenol, 1,4-dimethyl-8isopropylidenetricyclo [5.3.0.0(4,10)]decane.

Comparative results of the GC/MS analysis are shown in Table 4. It can be seen from

the data in the table that the volatiles of the diptreated wood bunches are mainly phenols, alkanes, and aldehydes; the volatiles of the dip-treated board are mainly phenolic acids, and aldehydes; and the volatiles of the non-pretreated plain boards are mainly phenols, aldehydes, and acids. reconstructed timber, the antibacterial properties of the pretreated dipping wood bunches, the hotpressed dipping sheet, and the non-pretreated plain boards were substantial. However, their antibacterial chemical compositions differed.

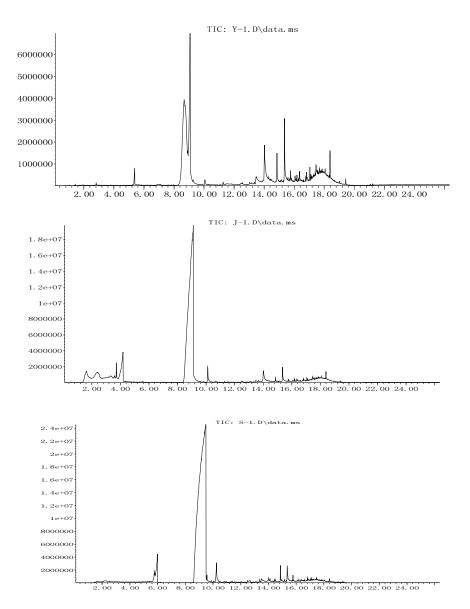


Fig. 1. Total ion of Wood/PF Bio Composites by DT-GC-MS

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No.	RT (min)	Peak area %	Chemical component
1	5.375	1.10	Acetic acid
2	8.690	36.24	Phenol
3	9.057	17.87	Phenol
4	10.064	0.57	Benzaldehyde, 2-hydroxy-
5	11.271	0.18	Nonanal
6	13.484	1.90	2-Furancarboxaldehyde, 5-(hydroxymethyl)-
7	14.030	7.78	Salicyl Alcohol
8	14.869	2.14	Phenol, 2,6-dimethoxy-
9	15.173	0.57	2,5-Dihydroxypropiophenone
10	15.362	5.35	Vanillin
11	15.771	1.65	Phenol, 2-methoxy-4-(1-propenyl)-,(E)-
12	16.065	0.77	Ethanone, 1-(4-hydroxy-3-methoxyphenyl)-
13	16.191	0.44	Phenol, 2,4-bis(1,1-dimethylethyl)
14	16.369	1.89	Benzeneacetic acid, 4-hydroxy-3-methoxy-, methyl ester
15	16.831	1.83	Phenol, 2,6-dimethoxy-4-(2-propenyl)-
16	17.471	7.46	Pentadecane, 2,6,10,14-tetramethyl
17	17.702	2.25	Nonahexacontanoic acid
18	17.869	2.79	Octacosyl heptafluorobutyrate
19	18.058	4.02	Eicosane
20	18.394	3.10	1,2-Benzenedicarboxylic acid, bis 2-methylpropyl) ester
21	19.433	0.10	1,4-Dimethyl-8-isopropylidenetricyclo[5.3.0.0(4,10)]decane

 Table 1. Result of Y-1 samples by DT-GC-MS

Table 2. Result of J-1 samples by DT-GC-MS	Table 2.	Result	of J-1	samples	by DT-GC-MS
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No.	RT	Peak	Chemical component	
	(min)	area %		
1	1.629	4.20	Formaldehyde	
2	2.385	5.32	Hydrazine, 1,2-dimethyl-	
3	3.339	4.06	Acetic acid	
4	3.591	0.96	2-Isopropoxyethylamine	
5	3.728	1.14	Formic acid	
6	4.168	5.28	Acetic acid	
7	9.078	63.83	Phenol	
8	10.127	1.05	Benzaldehyde, 2-hydroxy-	
9	10.683	0.15	Phenol, 4-methyl-	
10	12.551	0.16	Benzoic acid	
11	13.484	0.23	2-Furancarboxaldehyde, 5-(hydroxymethyl)-	
12	13.684	0.18	Methenamine	
13	14.019	2.16	Salicyl Alcohol	
14	14.859	0.47	Phenol, 2,6-dimethoxy-	
15	15.352	1.18	Benzaldehyde, 3-hydroxy-4-methoxy-	
16	15.771	0.47	Phenol, 2-methoxy-4-(1-propenyl)-(E)-	
17	16.065	0.20	Ethanone, 1-(4-hydroxy-3-methoxyphenyl)-	
18	16.191	0.16	Phenol, 2,4-bis(1,1-dimethylethyl)	
19	16.369	0.45	Benzeneacetic acid, 4-hydroxy-3-methoxy-, methyl ester	
20	16.831	0.89	Phenol, 2,6-dimethoxy-4-(2-propenyl)-	
21	17.471	1.84	Pentadecane, 2,6,10,14-tetramethyl	
22	18.058	3.52	Ethanol, 2-(dodecyloxy)-	
23	18.394	1.99	Phthalic acid, isobutyl nonyl este	
24	19.433	0.09	1,4-Dimethyl-8-isopropylidenetricyclo[5.3.0.0(4,10)]decane	

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No. RT		Peak	Chemical component	
	(min)	area %		
1	1.651	0.05	Acetic acid, oxo-	
2	2.217	0.43	Acetic acid	
3	5.795	1.47	Dimethyl ether	
4	5.983	2.81	Acetic acid	
5	9.45	84.52	Phenol	
6	9.865	0.12	1H-Pyrrole-2-carboxaldehyde	
7	10.064	0.09	Phenol	
8	10.253	1.58	Benzaldehyde, 2-hydroxy-	
9	10.820	0.08	Phenol, 4-methyl-	
10	11.376	0.10	Nonanal	
11	12.666	0.32	Benzoic acid	
12	13.369	0.13	Benzofuran, 2,3-dihydro-	
13	13.495	0.63	2-Furancarboxaldehyde, 5-(hydroxymethyl)-	
14	14.020	0.64	Salicyl Alcohol	
15	14.324	0.09	2-Furancarboxylic acid, hydrazide	
16	14.502	0.31	2-Methoxy-4-vinylphenol	
17	14.869	0.73	Phenol, 2,6-dimethoxy-	
18	15.174	0.20	2,5-Dihydroxypropiophenone	
19	15.362	1.25	Vanillin	
20	15.772	0.55	Phenol, 2-methoxy-4-(1-propenyl)-,(E)-	
21	16.076	0.17	Ethanone, 1-[4-(methylthio)phenyl]	
22	16.191	0.13	Phenol, 2,4-bis(1,1-dimethylethyl)	
23	16.370	0.38	Benzeneacetic acid, 4-hydroxy-3-methoxy-, methyl ester	
24	16.621	0.22	Pentadecane, 3-methyl-	
25	16.831	0.65	Phenol, 2,6-dimethoxy-4-(2-propenyl)-	
26	17.114	0.42	Tridecane, 3-methyl-	
27	17.240	0.34	n-Tetracosanol-1	
28	17.461	1.08	Pentadecane, 2,6,10,14-tetramethyl	
29	17.859	0.21	Tetratriacontane, 17-hexadecyl-	
30	18.394	0.23	1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester	
31	19.328	0.04	Phenol	
32	19.433	0.02	1,4-Dimethyl-8-isopropylidenetricyclo[5.3.0.0(4,10)]decane	

Table 3. Result of S-1 samples by DT-GC-MS

Table 4. GC-MS analysis results and comparison

Kind	Treated wood	Wood/PF Bio composites	Untreated wood
Aldehyde	8.00	6.66	3.68
Phenol	60.17	65.97	84.69
Alcohol	7.78	5.68	0.98
Acid	5.24	10.64	3.61
Ester	3.10	2.44	0.61
Alkyl	11.58	1.84	1.95
Ketone	1.34	0.20	0.37
others	2.79	6.55	2.34

The main antibacterial, volatile components of dipping wood bunches included phenol, 2,4-bis(1,1-dimethylethyl), etc., representing 54.55% of the total volatiles. The main antibacterial, volatile components of the hot-pressed dipping sheet included phenol, formaldehyde, 2,4-bis(1,1-dimethylethyl), phenol, and 4-methyl-, accounting for 68.19% of the total volatiles, while the main antibacterial, volatile components of the non-pretreated plain boards included phenol, 2,4-bis(1,1-dimethylethyl), phenol, and 4-methyl-, accounting for 84.86% of the total volatiles.

### CONCLUSIONS

A GC/MS analysis of dip-treated wood bunches, a hot-pressed dipping sheet, and nonpretreated prime boards showed that their antimicrobial properties are substantial. The main antibacterial volatile components of the dip-treated wood bunches accounted for 54.55% of the total volatile matter: the main antibacterial volatile components of the hot-pressed dipping sheet constituted 68.19% of the total volatile matter; and the main antibacterial volatile components of the non-pretreated plain boards accounted for 84.86% of the total volatile matter. The results indicate that a pretreatment consisting of hot dipping with a phenolic resin and using an adhesive phenolic resin can effectively improve the antibacterial properties of V. negundo reconstructed timber.

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