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# Preliminary investigation on concentrating of acetol from wood vinegar

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ABSTRACT

Acetol, as one of the components in biomass pyrolysis liquid, is a high value added compound for medicine synthesis. Benefit may be obtained if acetol can be extracted from the pyrolysis liquid, while the instability of acetol makes the concentrating difficult. In this paper, the concentrating of acetol from wood vinegar is preliminarily investigated, and the conditions of distillation, solvent extraction, and $Na_2CO_3$ effect on the concentrating result are discussed. Herein the content of acetol can be concentrated from below 4% in the raw wood vinegar to above 60%, and the number of main components reduce from over 20 to 5, while the yield of acetol is still rather low. It was found that in the organic solution distillation process, acetol can be easily concentrated from 1% to above 40%, while a further distillation of the concentrated acetol system was rather hard. The conversion of acetol in the distillation process was probably an important cause to the low yield of acetol, and a lower distillation temperature was advantageous for the concentrating of acetol.

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## 1. Introduction

With declining petroleum resources, and more environmental concerns on fossil fuels, it is imperative to develop sustainable fuels and chemicals from renewable resources [1–4]. As one of the important renewable resources, biomass can be converted to liquid products by thermochemical approach for pyrolysis-oil and chemicals [2–7]. Pyrolysis-oil may be used as a transport fuel after upgrading treatment [8–12] or by emulsion with diesel [13]. Chemicals like methanol, acetic acid can be obtained from the pyrolysis liquid a long time ago [3]. Recently, hydrothermal approach was applied in treating the biomass to produce chemicals like liquid alkanes [2,6,14,15] or the unique compound like 2-(4-hydroxy-benzyl)-4-methyl-phenol [16]. The bio-refinery is getting prosperous these years while the high value added chemicals that can be obtained are still rather few, compared with the number of components found in the pyrolysis-oil.

Acetol (1-hydroxy-2-propanone), as one of the major component in the liquid product by pyrolysis of biomass, is a valuable compound for medicine synthesis [17]. At present, there are two main approaches in producing acetol [18]. The first, acetol can be synthesized by the reaction between bromoacetone and sodium or potassium formate or acetate, followed by hydrolysis of the ester with methyl alcohol. The second, acetol can be prepared by the treatment of glycerol or propylene glycol at 200–300 °C with a dehydrogenating catalyst. While the two methods are both in high cost due to the expensive bromoacetone or catalyst. So it is worthwhile to take efforts to extract acetol from the pyrolysis liquid. While the instability of acetol makes the concentrating difficult. Till now there is no report on the method of extraction of acetol from pyrolysis liquid.

In this paper, the concentrating of acetol from wood vinegar was preliminarily probed. The conditions of distillation, solvent extraction, and $Na_2CO_3$ effect on the concentrating result were tested and phenomena were discussed.

## 2. Analytical method

The wood vinegar and the concentrated products were analyzed by GC–MS apparatus (Agilent-6890-5972). The column was AB-FFAP (30 m × 0.25 mm × 0.25 μm). Analytical conditions: inlet heater, 275 °C; MSD temperature, 150–155 °C; temperature program, initial 50 °C for 5 min, increase to 110 °C by 20 °C/min for 5 min, and finally to 230 °C by 20 °C/min for 6.5 min. The contents of the components in different systems were represented by peak area percentages for relative comparisons under different conditions, since the aim of the work was for qualitative probe.

## 3. Results and discussion

The raw wood vinegar was analyzed by GC–MS, the total ion chromatogram (TIC) was shown in Fig. 1, and the composition was listed in Table 1. It can be seen from Fig. 1 that water (80.97%) was the predominant component in wood vinegar, and the other main components were acetic acid (7.28%), methyl

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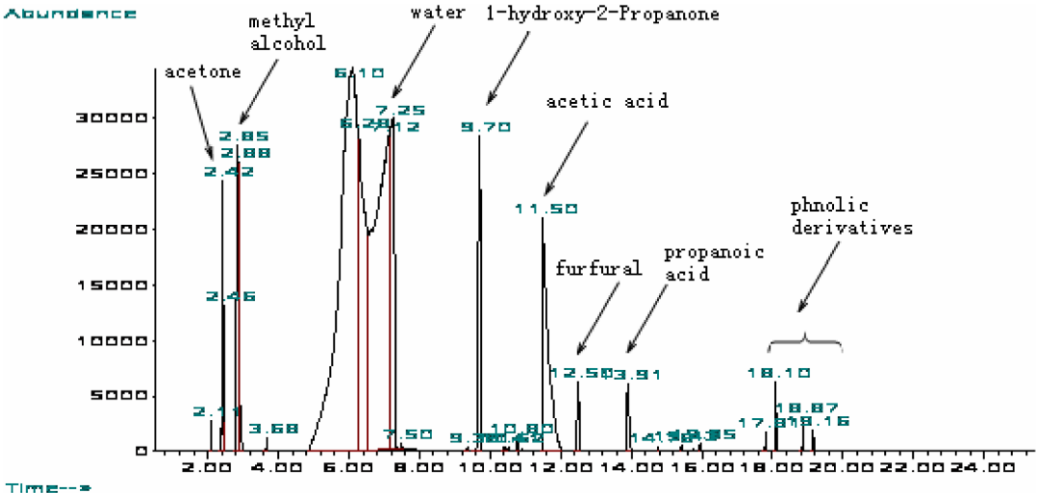


**Fig. 1.** Total ion chromatogram of the raw wood vinegar.

**Table 1**
Composition of raw wood vinegar and latter 50% evaporated fraction.

| No. | RT/min | ID | Area%$^{Raw}$ | Area%$^{L50\%}$ |
|---|---|---|---|---|
| 1 | 2.11 | Acetaldehyde | 0.12 | |
| 2 | 2.42 | Acetone | 0.99 | |
| 3 | 2.46 | Acetic acid, methyl ester | 0.44 | |
| 4 | 2.84–2.88 | Methyl alcohol | 4.55 | 0.038 |
| 5 | 3.69 | 2,3-Butanedione | 0.06 | 0.182 |
| 6 | 6.1–7.24 | Water | 80.97 | 79.715 |
| 7 | 7.5 | Propanenitrile | 0.03 | |
| 8 | 9.37 | Formamide | 0.02 | |
| 9 | 9.7 | 2-Propanone, 1-hydroxy- | 3.41 | 4.182 |
| 10 | 10.41 | 2-Cyclopenten-1-one | 0.04 | |
| 11 | 10.52 | 2-Cyclopenten-1-one, 2-methyl- | 0.01 | |
| 12 | 10.8 | Butanoic acid, 2-oxo- | 0.1 | |
| 13 | 11.49 | Acetic acid | 7.28 | 14.241 |
| 14 | 12.51 | Furfural | 0.61 | 0.078 |
| 15 | 13.91 | Propanoic acid | 0.7 | 0.972 |
| 16 | 14.78 | 2-Furancarboxaldehyde, 5-methyl- | 0.02 | |
| 17 | 15.43 | Butanoic acid | 0.03 | 0.034 |
| 18 | 15.95 | 1-Penten-3-one, 2-methyl- | 0.03 | |
| 19 | 17.81 | Cyclooctane | 0.06 | |
| 20 | 18.1 | Phenol, 2-methoxy- | 0.33 | 0.128 |
| 21 | 18.87 | Phenol, 2-methoxy-4-methyl- | 0.11 | 0.025 |
| 22 | 19.16 | Phenol | 0.11 | 0.099 |

Raw – raw wood vinegar.
L50% – later 50% evaporated fraction.

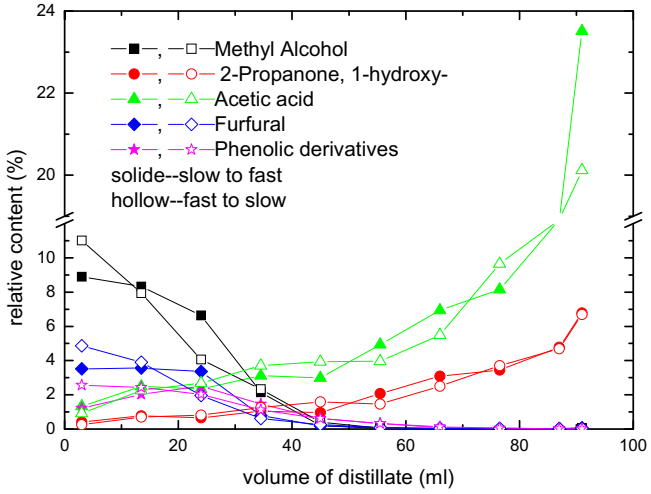


**Fig. 2.** Components in different distillation fractions under two distilling conditions.

alcohol (4.55%), acetol (3.4%), acetone (0.99%), propanoic acid (0.7%), furfural (0.61%), and phenol derivatives (0.55%). Among the main components, acetol was most distinct in the ratio of content to market price, indicating that it was worthwhile to take efforts to extract acetol from the wood vinegar.

The raw wood vinegar (100 ml) was firstly distilled at atmospheric pressure. Two heating rates were tested, one case was slow heating in the beginning and fast heating later, and the other case was from fast to slow. With distillation going, the evaporated fractions were collected in the order of 3 ml, 10.5 ml × 8, and 4 ml. The composition of each evaporated fraction was analyzed, and the change of the composition with distillation process was shown in Fig. 2. The composition of the latter 50% evaporated fraction was listed in Table 1.

It can be seen from Fig. 2 that the heating rate had very little influence on the content of acetol during the distillation process, and therefore a constant heating rate for distillation was adopted. The contents of methanol, furfural, phenol derivatives were higher in the early 45% evaporated fraction, and then the contents of theirs decreased with distillation going. The contents of acetol and acetic acid were lower in the early distilled fractions, and then increased distinctly after 45% volumetric fraction. it can be seen from Table 1 that the content of acetol in the latter 50% fraction was higher, and the contents of volatile components and phenol derivatives were lower than those in the original raw wood vinegar. So the latter 50% evaporated fraction can be used as the source material for further treatment.

Acetol (145 °C) has similar boiling point with acetic acid (117.9 °C) and has high solubility in water, so the separation of acetol by direct distillation of the water solution with much acetic acid inside is hard. So solvent extraction to transfer acetol from water phase to organic phase was considered. According to the like dissolves the like principle, the solvent like acetone must have high solubility to acetol, but these solvents dissolve with water very well too, so the screen for appropriate solvent is needed. Herein four solvents of toluene, chloroform, ethyl acetate, hypnone were tested. Raw wood vinegar (5 ml) was blended with solvent (5 ml) in a test tube, mixing by fast shaking up and down for 5 min, laying aside for 10 min, and then the organic layer was analyzed by GC–MS. The peak areas of the main components in different solvents were shown in Fig. 3. It can be seen from Fig. 3 that the ratio of ace-

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