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# 龙桑一号桑叶精油的挥发性组分

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**摘 要:**桑品种龙桑一号(*Morus alba*)栽培于中国东北黑龙江省齐齐哈尔市甘南林场桑产业科技示范园。2009年7月、8月和9月的桑品种龙桑一号干桑叶精油中的挥发性组分经水蒸馏提取后进行了气相色谱-质谱(GC-MS)分析。结果表明桑叶精油的共有组分是植醇(醇类化合物)、六氢金合欢丙酮(酮类化合物)、二十七烷和二十五烷(烃类化合物)。棕榈酸为7月和8月采集桑品种龙桑一号干桑叶精油的共有第一主成分。顺式-$\beta$-金合欢烯、反式-$\beta$-金合欢烯、$\beta$-甜没药烯、反式-$\alpha$-佛手柑油烯和$\alpha$-姜黄烯是9月采集桑品种龙桑一号干桑叶精油的主要芳香组分。萜类化合物相对含量较高的9月份采集的干桑叶精油具有药用价值。

**关键词:**桑;精油;棕榈酸;萜类化合物;气相色谱-质谱法

**中图分类号:**Q946.85　　　　**文献标识码:**A

# Volatile Components of Essential Oil from Mulberry Variety "Longsang 1" Leaves

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**Abstract:** The mulberry variety "Longsang 1" (*Morus alba*) was cultivated in Gannan Forest Mulberry Industry Science and Technology Demonstration Field of the Qiqihar City in Heilongjiang Province in Northeastern China. In July,August and September of 2009,the leaves of "Longsang 1" were collected,then the volatile components of the essential oils in the leaves were hydrodistillated and analyzed by gas chromatography-mass spectrometry. The results showed that phytol (alcohols),hexahydrofamesyl acetone (ketones),heptacosane,and pentacosane (hydrocarbons) were the common volatile components of the essential oil. Hexadecanoic acid was dominant in the essential oil from leaves collected in July and August. (E)-$\beta$-farnesene,(Z)-$\beta$-farnesene,$\beta$-bisabolene,trans-$\alpha$-bergamotene and $\alpha$-curcumene were the main volatile flavor compounds in the essential oil from leaves collected in September. Air-dried mulberry leaves collected in September has relatively high medicinal value due to their higher content of terpenoids in the essential oil.

**Key words:** *Morus alba*; essential oil; hexadecanoic acid; terpenoids; gas chromatography-mass spectrometry

## Introduction

Leaves of *Morus alba* L. (Moraceae) are traditional medicine and used for silkworm production in China [1-4]. They also can be developed as beverages (tea etc.),foods (noodles,pastries etc.),and cosmetics (perfume,cream,bath,shampoo etc.) [5-7]. The volatile aromatic constituents of essential oil are the most important for the mulberry leaves product development. For the volatile components of essential oil from *M. alba* leaves,the different researchers had different major compounds in *M. alba* leaves because of their different growing sites,sampling and analyzing methods etc. Li's research showed that 3,7,11,15-tetramethyl 2-hexadecimal alcohol was the principal volatile component of leaves [8],while (*E*)-$\beta$-ocimene (53.62%) and $\alpha$-pinene (11.81%) were the main volatile constituents in Wu *et al*. study [9]. The hexadecyl alcohol or anethol was the first principal volatile component of the essential oil in Li's or Zhou's researches [10,11],respective-

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ly; while Sun's result was that *n*-octadecane (9.11%), di-2-methylpropyl phthalate (8.92%), 3, 7, 11, 15-tetramethyl 2-hexadecyl alcohol (7.19%) were the main constituents[12]. Additionally, Li *et al.*[13] studied the volatile components of the *M. alba* (2-year age, three mulberry cultivars) fresh leaves, diethyl phthalate (24.44%) and 1-penten-3-ol (12.02%) were the main constituents of the seedling mulberry, isopentyl alcohol (15.65%), 1-hexanol (10.99%), γ-terpinen (10.79%) were the main constituents of the Lu 7946, linalyl propionate (58.65%) and diethyl phthalate (6.89%) were the main constituents of Nongsang 14; For *M. alba* dried leaves of 10 different areas, Sun *et al.*[14] study showed that hexadecanoic acid, diisobuthyl phthalate, methyl hecadecanoate, hexahydrofamesyl acetone, pristane, *n*-nonadecome, megastigmatrienone, α-zoneone, β-zoneone, 1-(2,6,6-trimethyl-1, 3-cyclohexadien-1-yl) 2-buten-1-one, β-cydocitral, propiophenone, phenethyl alcohol, phenylethanal, 2,4-heptenal, cyclohexyl cyclooctane, heptanal were their common volatile components with hydrodistillated method

Mulberry variety "longsang 1" cultivated in Gannan Forest Mulberry Industry Science and Technology Demonstration Field, Qiqihar, Heilongjiang Province, P. R. China, is a fruit and leaves dual-purpose new cultivar with high-yield, high-quality and cold resistance. The purpose of this study is to analyse the volatile components of essential oils from leaves collected in different months by gas chromatography-mass spectrometry, and to provide a reference for the quality control and product development of mulberry variety "longsang 1" leaves.

## Materials and Methods

### Plant materials

The leaves of *M. alba* (3-year age, grafted seedlings) were collected from Gannan Forest Mulberry Industry Science and Technology Demonstration Field, Qiqihar, Heilongjiang Province, P. R. China in July, August, and September of 2009, respectively. Voucher specimens were identified by professor Mu Li-Qiang (Northeast Forestry University) and deposited in Sericultural Research Institute of Heilongjiang Province of China.

### Isolation of Essential Oils

Air-dried whole leaves samples (240 g) were hydrodistilled in 5 L glass apparatus for 3 h, then extracted with 20 mL ether. The ether phase was dried with anhydrous sodium sulfate for 15 min, then the anhydrous sodium sulfate was removed and the dried ether phase was placed in 40 ℃ water bath to evaporate ether solvent, then the pale-yellow color essential oil was obtained.

### GC-MS Analysis

0.04 g/mL *M. alba* leaves essential oils *n*-hexane (HPLC pure) samples were analyzed by using GC-MS 6890N-5973 insert (Agilent, USA).

Analysis conditions of essential oils in July or August are as follows: DB-17MS capillary column, column length 30 m, internal diameter 0.25 mm, film thickness 0.25 μm, FID detector; The temperature programs were 60 ℃ (4 min) to 150 ℃ at a rate of 20 ℃/min, then to 240 ℃ (5 min) at a rate of 5 ℃/min. The injector temperature was 260 ℃, the injection volume was 1.0 μL, split ratio 25∶1, carrier gas, He at a rate of 1 mL/min; temperature of MS transfer line was 280 ℃, EI with 70 eV. Ion source temperature was 230 ℃, scan range was 15 ~ 260 u. The temperature programs of essential oil in September were 60 ℃ (4 min) to 240 ℃ (10 min) at a rate of 5 ℃/min. The injector temperature was 260 ℃, the injection volume was 1.0 μL, splitless, carrier gas, He at a rate of 1 mL/min; temperature of MS transfer line was 280 ℃, EI with 70 eV. Ion source temperature was 230 ℃, scan range was 15 ~ 260 u. The temperature programs of September had slight difference with July and August due to different sampling date and following other sample batches' programs.

## Results and Discussion

Total ions chromatograms of essential oils from leaves collected in July, August, and September of 2009, respectively, were reported in Figure 1.

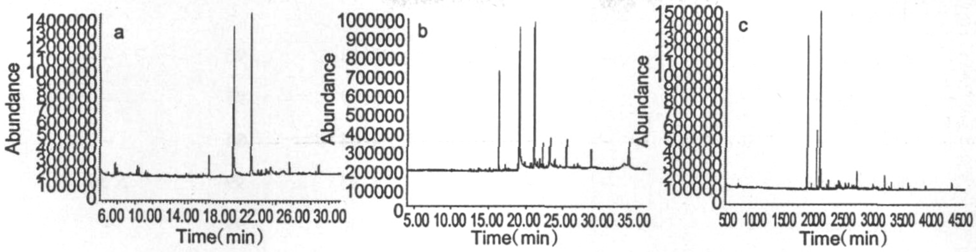


**Fig. 1 Total ions chromatograms of essential oil from air-dried *Morus alba* leaves collected in July (a), August (b), and September (c), respectively**

The identification of the volatile constituents of essential oils from air-dried *M. alba* leaves collected in July, August, and September of 2009, respectively, were based on comparison of their retention time, and mass spectra with those obtained from the Wiley libraries and the literature and the mass fraction of all compounds were calculated by the area normalization method. The main components of the essential oil from leaves collected in each month were reported in Table 1.

The essential oil of leaves collected in July was analyzed to include 7 kinds of compounds classification: three hydrocarbons (4.94%), one aldehyde (0.61%), three alcohols (32.00%), one ketone (2.99%), three acids (52.11%), one ester (0.72%), and unknown (6.66%). For the compounds classification in August, 6 kinds of compounds classification were tested except for aldehyde. Hexadecanoic acid was the principal volatile component in July and August. Moreover, hexadecanoic acid, phytol, and hexahydrofamesyl acetone were the main constituents of the essential oil of air-dried *M. alba* leaves in July and August.

The essential oil of leaves collected in September had 4 kinds of compounds classification: nine hydrocarbons (82.61%), one alcohol (3.20%), one ketone (3.53%), and unknown (10.67%). α-Curcumene (32.77%) was the principal volatile component. In addition, α-curcumene, trans-α-bergamotene (29.00%), and (*Z*)-β-farnesene (12.15%) were the main constituents of the essential oil of air-dried *M. alba* leaves collected in September. The comparison results with volatile components of essential oils from "longsang 1" leaves collected in different months showed that phytol, hexahydrofamesyl acetone, heptacosane, and pentacosane were their common volatile components. Hexadecanoic acid was the common principal volatile component between the essential oil from leaves collected in July and August. (*E*)-β-farnesene, (*Z*)-β-farnesene, β-bisabolene, trans-α-bergamotene, and α-curcumene were the main volatile compounds with aroma in the essential oil from leaves collected in September. Hexadecanoic acid (44.00%), phytol (29.81%), linolenic acid (5.40%), and hexahydrofamesyl acetone (2.99%) were the main constituents of the essential oil of leaves collected in July. Linolenic acid is an essential fatty acid needed by the body to maintain good skin condition.[15,16] Due to the medicinal value of terpenoids[17], the essential oil of air-dried mulberry leaves collected in September with high relative content of terpenoids has utilization value.

**Table 1 Main volatile composition of essential oil from air-dried *Morus alba* leaves collected in July, August, and September respectively**

| Classification | Compound name | Essential oil of leaves collected in July | | Essential oil ofleaves collected in August | | Essential oil of leaves collected in September | |
|---|---|---|---|---|---|---|---|
| | | Mass fraction(%) | Match(%) | Mass fraction(%) | Match(%) | Mass fraction(%) | Match(%) |
| Hydrocarbons | tetracosane | 0.90 | 91 | 0.87 | 98 | – | – |
| | pentacosane | 1.96 | 98 | 3.36 | 99 | 1.20 | 93 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | heptadecane | 2.08 | 86 | – | – | – | – |
| | docosane | – | – | 0.53 | 96 | – | – |
| | octacosane | – | – | 2.69 | 95 | – | – |
| | 1-eicosene | – | – | 4.64 | 84 | – | – |
| | heptacosane | – | – | 2.83 | 99 | 2.36 | 91 |
| | nonacosane | – | – | 6.46 | 99 | – | – |
| | *trans*-α-bergamotene | – | – | – | – | 29.00 | 90 |
| | (*E*)-β-farnesene | – | – | – | – | 0.99 | 95 |
| | (*Z*)-β-farnesene | – | – | – | – | 12.15 | 90 |
| | β-bisabolene | – | – | – | – | 2.09 | 98 |
| | α-curcumene | – | – | – | – | 32.77 | 98 |
| | heneicosane | – | – | – | – | 1.25 | 91 |
| | hexacosane | – | – | – | – | 0.80 | 91 |
| Alcohols | phytol | 29.81 | 95 | 24.09 | 95 | 3.20 | 91 |
| | *trans*-*p*-mentha-2,8-dienol | 1.32 | 87 | – | – | – | – |
| | *cis*-*p*-mentha-2,8-dien-1-ol | 0.87 | 83 | – | – | – | – |
| Ketones | hexahydr ofamesyl acetone | 2.99 | 91 | 10.94 | 96 | 3.53 | 91 |
| Acids | hexadecanoic acid | 44.00 | 98 | 38.11 | 97 | – | – |
| | linoleic acid | 2.71 | 96 | – | – | – | – |
| | linolenic acid | 5.40 | 91 | – | – | – | – |
| Esters | dibutyl phthalate (DBP) | 0.72 | 83 | 1.05 | 94 | – | – |
| | diisobutyl phthalate (DIBP) | – | – | 0.55 | 83 | – | – |
| Aldehydes | (*E*)-2-hexenal | 0.61 | 95 | – | – | – | – |

## Conclusion

The essential oils of air-dried mulberry variety "longsang 1" (*Morus alba*) leaves collected in July, August, and September of 2009, respectively, were found to commonly contain hydrocarbons, alcohols, and ketones.

The comparison results with volatile components of essential oils from mulberry variety "longsang 1" (*Morus alba*) leaves collected in different months showed that phytol, hexahydrofamesyl acetone, heptacosane, pentacosane were their common volatile components. Hexadecanoic acid was the common first principal volatile component between the essential oil from mulberry variety "longsang 1" leaves collected in July and August. (*E*)-β-farnesene, (*Z*)-β-farnesene, β-bisabolene, *trans*-α-bergamotene and α-curcumene were the main volatile compounds with aroma in the essential oil from mulberry variety "longsang 1" leaves collected in September. The essential oil of air-dried mulberry leaves collected in September with high relative content of terpenoids has medicinal value.

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