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Scented Extracts and Essential Oil Extraction from *Michelia alba* D.C.

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ABSTRACT

The study assessed extractions of scents and essential oils from *Michelia alba* D.C., with water, steam, water-steam distillation, cold enfleurage using palm stearin and hot enfleurage using palm oil, hexane and petroleum ether extraction. The yields of *M. alba* essential oils from the three distillations were 0.225, 0.120 and 0.199%, respectively. The percentage yields of *M. alba* absolutes obtained from cold and hot enfleurage, hexane and petroleum ether extractions at saturation point (1,500 g of flower/ 200 ml of fat, 400 g/ 400 ml of oil, 200 and 150 g/L of solvent) were 0.3511, 2.7506, 0.0457 and 0.0497%, respectively. The chemical composition of the essential oils and absolutes was analyzed by GC-MS. The results showed that linalool was the major component of the oils and indole, linalool and phenylethyl alcohol were the major components of the absolutes.

Key words: *Michelia alba* D.C., distillation, enfleurage, solvent extraction, GC-MS

INTRODUCTION

Michelia alba D.C. ("White Champaka") is a fragrant plant in the Magnoliaceae family. Originating from southern Asia, it has acclimatized to many regions of the world. As a tree, *M. alba* is small to medium-sized (10-20 feet high) (Smitinand, 2001) and can be cultivated as a garden plant for ornamental purposes in most tropical and lower subtropical countries. The white elongated bell-shaped flowers have a strong sweet fragrance, bloom year round and emit a powerful scent, which can be detected several meters away. They are used as religious offerings or in garlands and their sweet, pungent, alluring fragrance makes them an ideal ingredient for aromatherapy products (Baussan, 2006).

Floral scents are secondary metabolites produced in small amounts but having a strong scent (David, 1998). They are composed of either essential oil or volatile oil and nonvolatile substances. The scents are synthesized in various plant organs and have complex constituents that are too difficult to imitate (Dudareva and Negre, 2005). Nowadays, the demand for volatile oil is expanding and there are different methods to extract these scents. Various scented extracts such as 'essential oil' are produced using a distillation method. 'Pomades' are the fats, which have absorbed essential oils by the cold or hot

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enfleurage method, 'Concretes' is a solid, wax-like substance obtained from solvent extraction and 'Absolute' is composed of aromatic molecules extracted from "concretes" by alcohol (Douglas, 2006).

This study aimed to: 1) find suitable methods to extract floral scents from *M. alba*, 2) study the chemical composition of the extracts obtained and 3) find the optimum ratio between the flowers and the extracting materials for use in enfleurage and solvent extraction.

MATERIALS AND METHODS

Extraction by distillation

An amount of 500 g of fresh *M. alba* flowers was extracted with 1,000 ml of distilled water for 4 h using each of three methods; water, steam and water and steam distillation. The final essential oils were separated using anhydrous sodium sulphate (applied from Parliament, 1998) and kept for further analysis.

Extraction by enfleurage

Cold enfleurage

A total of 100 ml of palm stearin was melted at 80°C and spread over two glass frame sheets. *M. alba* flowers were placed on one layer of the sheet coated with the fat and then covered with the other sheet. After approximately one day, the flowers were removed and replaced by fresh flowers. The process was repeated until the fat was saturated with floral scent. Six ratios between flowers and the palm stearins: 1,000, 1,500, 2,000, 2,500, 3,000 and 3,500 g/200 ml, were studied for the optimum absorption of the flowering scents. The scented fat or pomade was then mixed with alcohol to separate any aromatic substances that were removed from the solvent by a vacuum evaporator to leave the absolute content.

Hot enfleurage

A total of 400 ml of palm oil was heated up to 60°C for 30 min. Six treatments: 300, 350, 400, 450, 500 and 550 g flowers/ 400 ml of palm oils, were investigated. The mixture was then cooled down and kept in a refrigerator overnight. The oil was then agitated for several days. The resulting oil was filtered and then extracted by alcohol to obtain the absolute content (applied from Gupta, 1952).

Extraction by solvent

Hexane and petroleum ether were used to extract the scents from *M. alba* flowers. Six ratios: 100, 150, 200, 250, 300 and 350 g of flower were soaked with 1 L of each solvent for 1 h. After removing the debris from the flowers, the solvent was then removed from the extracts by rotary evaporation under vacuum to obtain the concrete contents. *M. alba* absolutes were extracted from the concrete by alcohol (applied from Parliament, 1998).

Physical appearances, such as color, odor and other characteristics of all extracts were observed. The yield of extract from each method was assessed, compared and used to determine the saturation point of the fat, oil and solvent to absorb the scents from *M. alba*.

GC-MS analysis

GC-MS analysis of all extracts was performed on 60 m × 0.25 mm DB–5 capillary columns of GC-MS QP 5050A Shimadzu using a temperature program rate of 60-250°C. Other identical operating conditions were 57 min total time and 5 min of solvent cut time and 40 to 400 m/z of mass range using 1 μl of injector volume. Helium was used as a carrier gas. The components of the extracts were identified by comparison with mass spectral data from standard library (TUTOR.LIB, NIST12.LIB and NIST62.LIB) and literature data (Adam, 2007).

RESULTS AND DISCUSSION

Extraction by distillation

The highest yield of Champaka oil was obtained from water distillation (0.225%) followed by water-steam distillation (0.119%) and steam distillation (0.120%) (Figure 1). The physical appearance of the oils was a deep yellow liquid with a boiled-flower odor (Table 1). GC-MS analysis of the oils showed that there were 7, 9 and 9 volatile compounds, respectively. Their major component was linalool (85.78, 91.74 and 83.38%, respectively) (Figure 2, Table 2).

Extraction by enfleurage

The saturation point of palm stearin (cold enfleurage) was 1500 g flowers/200 ml and its yield was 0.3511% (Figure 3a). Palm oil could absorb proximal yields of 2.7506 and 2.7962% at 400 and 450 g/400 ml, respectively. These results implied that the saturation point of palm oil was near 400g/400 ml. However, the saturation point of fat depends on the essential oil content in the flowers and properties of the fat. Pensuk *et al*. (2007) reported that buffalo fat could extract the scent from *M. alba* with a ratio of 1,400 g flower/ 100 g of fat.

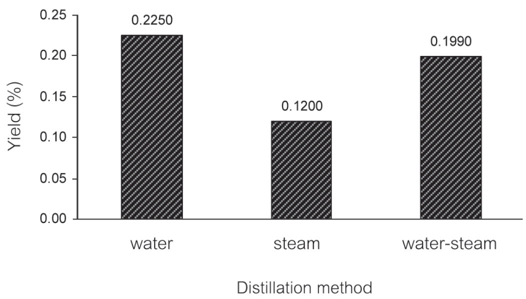

Figure 1 Yields of *M. alba* essential oils from three distillation methods.

The results indicated that hot enfleurage gave a higher yield of *M. alba* scent than cold enfleurage. This was probably due to the heat used in the extraction process. Moreover, absolutes from both methods contained palm wax and palm oil, which was extracted by ethanol used in the process. The wax could be discarded by filtrating or centrifuging (Pensuk *et al*., 2007), but the oil was hard to remove. Thus, yields were quite different. The chemical composition of absolute from *M. alba* by cold enfleurage consisted of three main compounds (Figure 2d). Indole (67.89%) was the major component of this absolute (Table 2), which was similar to the results from Pensuk *et al*. (2007). Absolute from hot enfleurage consisted of only two substances (Figure 2e, Table 2) with linalool (91.00%) as a major component. The characteristics that differed from those in distillation extracts are shown in Table 1.

Extraction by solvent

The results of hexane and petroleum ether extraction are show in Figure 4. The optimum ratio of hexane extraction was 200 g flower/l and the percentage yield of hexane concrete and absolute was 0.0840 and 0.0457%, respectively (Figure 4a). Petroleum ether could absorb the scents from *M. alba* with minor constant yields of 0.0784 and 0.0497% and 0.0582 and 0.0505% of concrete and absolute from 150 and 200 g/l, respectively (Figure 4b). These results implied that the saturation point of petroleum ether was 150 g/l.

Table 1 Physical appearances of *M. alba* essential oil and absolute from different extraction methods.

Extraction methods	Physical appearances
Distillation	Deep yellow oil with boiled flower odor
Enfleurage	Reddish-brown and wax-liked liquid. Odor of the liquid from cold enfleurage was more similar to fresh flowers than that from hot enfleurage.
Solvent	Brown and wax-like semi solid with odor similar to fresh flowers but more pungent. Petroleum ether extracts showed stronger scent than hexane extracts.

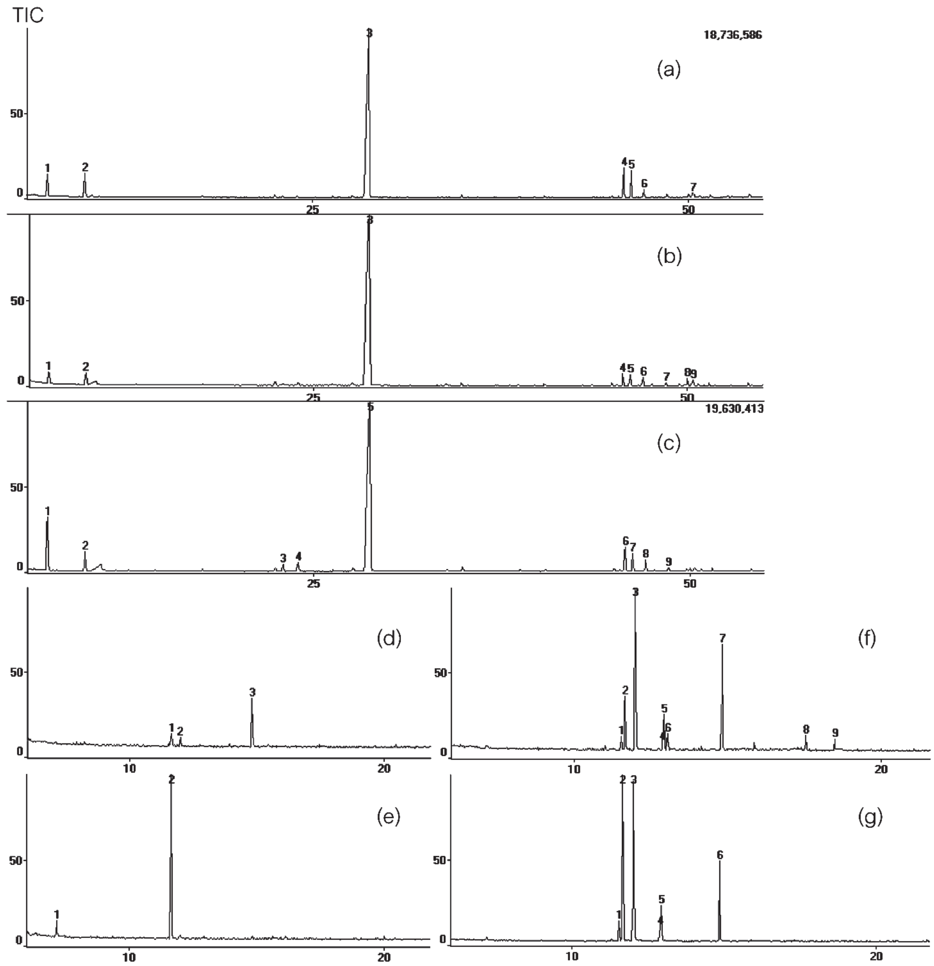

Figure 2 Chromatogram of *M. alba* essential oils from distillation with: (a) water; (b) steam; and (c) water-steam and absolutes from: (d) cold enfleurage; (e) hot enfleurage; (f) hexane extraction; and (g) petroleum ether extraction.

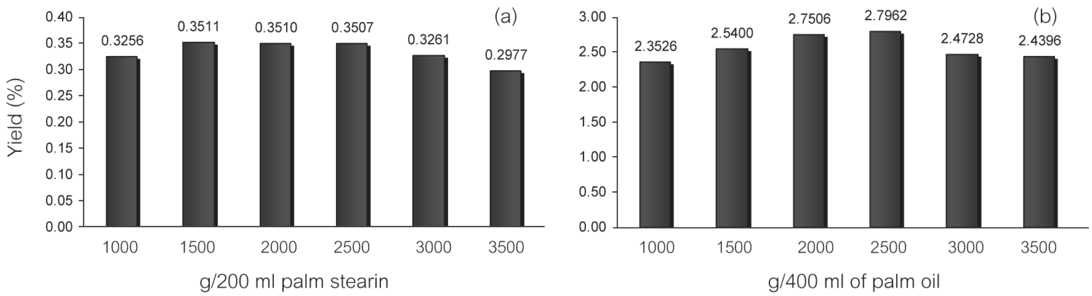

Figure 3 Yield of *M. alba* absolutes from: (a) cold enfleurage; and (b) hot enfleurage.

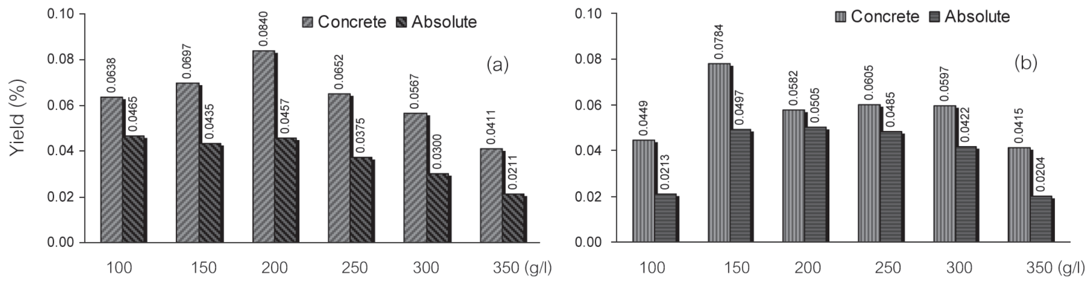

Figure 4 Yield of *M. alba* concretes and essential oils from extraction using: (a) hexane; and (b) petroleum ether.

Absolute from hexane extraction contained nine compounds (Figure 2f) and its major components were phenylethyl alcohol (39.10%) and indole (25.98%) (Table 2). Petroleum ether extract consisted of six compounds (Figure 2g) which had phenylethyl alcohol (34.93%) and linalool (34.86%) as major components (Table 2).

Solvent extraction was considered the most suitable method for extracting essential oil from aromatic plants in order to avoid the decomposition of aromatic compounds by heat (Parliament, 1998). However, this can be done only under safe conditions. Steam distillation could be used with Champaka oil extraction, though some chemicals could be lost by heat and the boiling-flower odor could contaminate the oil. Another interesting method was enfleurage, especially cold enfleurage, which produced high yields and had an odor most similar to that of fresh flowers (Gupta, 1952).

The cold methods, such as cold enfleurage, hexane and petroleum ether extraction produced high quality extracts from *M. alba* because of their components, linalool and indole, which are the major components of *M. alba* and are used in many perfumes. In particular, indole, (a natural compound that increases the perceived odor strength and improves the stability of other aromatic compounds in volatile oils), is used as a fixation compound in various perfume formulas (Basketter *et al*., 2002). Thus, the content of indole in scented extracts might be related to the different odor produced from each extraction method.

CONCLUSIONS

According to the results of extraction of *M. alba* essential oils and scents, the yields of the oils from the three distillations were 0.2250, 0.1200 and 0.1990, respectively. Their chemical composition showed that linalool was the major component in all extracts. Percentage yields of absolutes from cold and hot enfleurage, hexane and petroleum ether extractions at their saturated point were 0.3511, 2.7506, 0.0457 and 0.0497, respectively. Major components of these absolutes were indole, linalool and phenylethyl alcohol. The odor of the Champaka getting from entleurage extraction was more similar to fresh flowers than others.

ACKNOWLEDGEMENT

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Table 2 Chemical compositions of M. alba essential oils and scented extracts from different extraction methods.

Extraxction method	Peak No.	KI*	%Relative peak area	Compounds
Water distillation	1	-	2.43	methyl 2-methylbutyrate
	2	-	3.14	ethyl 2-methylbutyrate
	3	1095	85.78	linalool
	4	1389	3.82	beta-elemene
	5	1403	3.59	methyl eugenol
	6	1417	0.92	beta-caryophyllene
	7	1491	0.32	methyl isoeugenol
Steam distillation	1	-	1.31	methyl 2-methylbutyrate
	2	-	1.47	ethyl 2-methylbutyrate
	3	1095	91.74	linalool
	4	1389	1.42	beta-elemene
	5	1403	1.36	methyl eugenol
	6	1417	1.00	beta-caryophyllene
	7	1452	0.30	alpha-humulene
	8	1484	0.82	germacrene D
	9	1489	0.59	beta-selinene
Water-steam distillation	1	-	5.58	methyl 2-methylbutyrate
	2	-	2.28	ethyl 2-methylbutyrate
	3	1032	0.98	cis-beta-ocimene
	4	1044	1.28	trans-bata-ocimene
	5	1095	83.38	linalool
	6	1389	2.77	beta-elemene
	7	1403	2.04	methyl eugenol
	8	1417	1.33	beta-caryophyllene
	9	1452	0.35	alpha-humulene
Cold enfleurage	1	1095	20.43	linalool
	2	1106	11.67	Phenyl ethyl alcohol
	3	1290	67.89	indole
Hot enfleurage	1	-	9.00	ethyl 2-methylbutyrate†
	2	1095	91.00	linalool
Hexane	1	1067	3.32	cis-linalool oxide (furanoid)
	2	1095	13.30	linalool
	3	1106	39.10	phenylethyl alcohol
	4	1170	1.87	cis-linalool oxide (pyranoid)
	5	1173	8.02	trans-linalool oxide (pyranoid)
	6	1174	2.93	terpinen-4-ol
	7	1290	25.98	indole
	8	1497	3.09	Methyl-p-tert-butylphenyl acetate
	9	1588	2.40	1-Hexadecene
Petroleum ether	1	1067	4.00	cis-linaloolxide (furanoid)
	2	1095	34.86	linalool
	3	1106	34.93	phenylethyl alcohol
	4	1169	2.49	Ethyl benzoate
	5	1173	6.33	trans-linaloolxide (pyranoid)
	6	1290	17.40	Indole

*KI: kovatís index on DB-5 column (Adams, 2007)

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