Kasetsart J. (Nat. Sci.) 43 : 189 - 196 (2009)

Comparative Study of Scented Compound Extraction from *Plumeria obtusa* L.

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ABSTRACT

Various scented compound extraction methods were studied using *Plumeria obtusa* L. flowers: water distillation, steam distillation, water-steam distillation, hexane extraction, petroleum ether extraction, and cold and hot enfleurage. Chemical compounds of the extracts from each method were analyzed by gas chromatography-mass spectrometry (GC-MS). The results showed the percentage yield of the extracts was 0.0167, 0.0045, 0.0342, 0.4170, 0.3510, 0.3969 and 12.2400%, respectively. The major chemical component in the essential oils from all distillation methods and both solvents was benzyl salicylate, but the extracts from cold and hot enfleurage were linalool and n-undecanoic acid, respectively.

Key words: *Plumeria obtusa* L., distillation, solvent extraction, enfleurage, essential oil, absolute, GC-MS, major compound

INTRODUCTION

Frangipani essential oil is used as an ingredient in cosmetics and is excellent for aromatherapy uses, including, to scent candles, freshen potpourri, add fragrance to massage oils and of course as a perfume which has a very pleasing smell (Woodspirits Natures Essentials, 2003; Aromatic Ltd. 2001-2008).

Plumeria obtusa L., a member of Apocynaceae family, is commonly known as frangipani or the temple tree. In Thailand, it is most famously called lanthom.

The fragrance of *Plumeria* flowers has previously been studied and the yield and chemical compounds from water distillation reported (Kamariah *et al*., 1999; Norsita *et al*., 2006). However, there are no reports on other extraction methods. Thus, the objective of this research was to undertake a comparative study of some extraction methods and chemical analysis of the floral scent obtained.

MATERIALS AND METHODS

Extraction procedures

Distillation

Essential oil was extracted from fresh frangipani flowers by three distillation methods: water, steam and water-steam (Ernest *et al*., 1947;

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Oyen and Dung, 1999). In each method, 300 g of flowers were distilled for 4 h. With each sample, the light yellow oil was collected on the water surface, dehydrated with anhydrous Na_2SO_4 and kept in an opaque bottle.

Solvent extraction

Fresh flowers (70 g) were soaked in 1 L of hexane or petroleum ether for 1 hr, then, solvent evaporation under reduced pressure at low temperature was used to produce the concrete yield. The final absolute yield was extracted from the concrete yield using absolute ethanol (Ernest *et al*., 1947).

Enfleurage

The cold enfleurage procedure involved coating 200 ml of palm stearin on a glass frame. This was allowed to cool and harden. Fresh flowers (2 kg) were separated and placed on the glass allowing the scent components to diffuse into the fatty layer until the frangipani scent was discernable. For the hot enfleurage procedure, 100 g of fresh flowers were soaked in 400 ml of hot palm oil, heated at 60-70°C for 30 mins and then allowed to cool down before being placed in a refrigerator overnight. The floral scent in the pomade from each method were extracted with cold absolute alcohol. The extracts were obtained after evaporation to dryness (Ernest *et al*., 1947).

Chemical analysis

Identification of the chemical compounds in the extracts was performed using gas chromatography-mass spectrometry (GC-MS), with a QP 5050A Shimadzu and capillary column DB-5 (5%-phenyl)-methylpolysiloxane 0.25 μm, 60 m, 0.25 mm (i.d.). The type of detector was FID. The temperature programs used were: 80°C for the oven, 200°C for the injector and 230°C for the detector. The injector volume was 1 μl and the carrier gas was helium 99.999%. Retention time (Rt) was 40 mins, solvent cutime was 5 mins and mass range 40 was to 400 m/z.

RESULTS AND DISCUSSION

Distillation

The essential oil yields from water, steam and water-steam distillation were 0.0167, 0.0045 and 0.0342%, respectively (Figure 1) and were light yellow with a concentrated floral scent. The yield from water-steam distillation was much higher than for the other methods.

Chemical analysis by GC-MS of the water distillation found 19 compounds and the major compound was benzyl salicylate (31.32%), that showed peak no 15 at Rt 37.659 mins (Figure 2 and Table 1). The steam and water-steam distillations found 13 and 21 compounds, respectively. The major compound from the steam method was 27.58 % benzyl salicylate (Figure 3 and Table 2). The water-steam method showed a peak at Rt 37.658 mins and the major compound was 31.90% benzyl salicylate (Figure 4 and Table 3). Kamariah *et al*. (1999) also reported detecting benzyl salicylate, with 39% as the main component in frangipani oil from water distillation. Benzyl salicylate has a mild sweet odor, whereas benzyl benzoate is almost odorless in *P. obtusa* (Norsita *et al*., 2006).

Solvent extraction

The yields of concrete and absolute from hexane extraction were 0.5377 and 0.4170%, respectively and from petroleum ether extraction

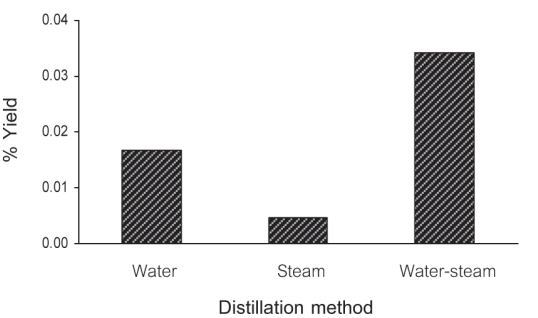

Figure 1 Comparison of the extraction yields of frangipani oil by distillation method.

were 0.4351 and 0.3510%, respectively. Characteristics of the concretes from both solvents were light yellow wax and strong odor. The hexane absolute was a light yellow liquid with a mild odor of frangipani. The petroleum ether absolute was deep yellow, had a strong odor, which was more similar to fresh flowers than that from hexane.

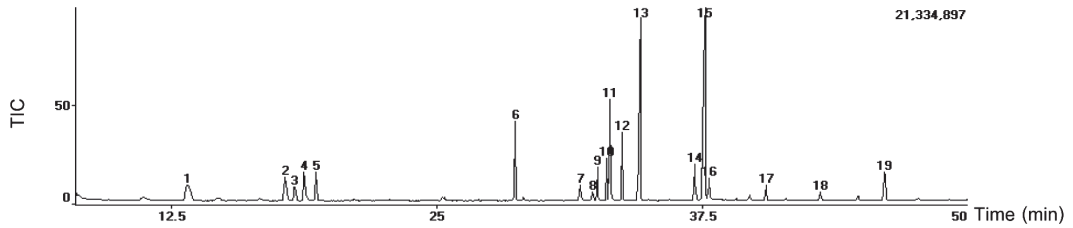

Figure 2 Chromatogram of essential oil from water distillation.

Table 1 Chemical compounds of essential oil from water distillation.

Peak No.	Retention time (min)	%Relative peak area	Possible compounds
1	13.280	4.72	linalool
2	17.877	4.36	(Z)-geraniol
3	18.314	2.02	(Z)-citral
4	18.762	3.79	(E)-geraniol
5	19.318	3.66	(E)-citral
6	28.702	4.85	(Z)-beta-farnesene
7	31.773	1.22	1-hexadecene
8	32.346	0.33	2-methylpentadecane
9	32.577	1.97	alpha-farnesene
10	33.022	2.61	(Z)-farnesol
11	33.192	6.70	(E)-farnesol
12	33.746	4.37	(E)-farnesal
13	34.601	18.90	benzyl benzoate
14	37.155	2.71	1-octadecanol
15	37.659	31.32	benzyl salicylate
16	37.865	1.65	eicosane
17	40.531	1.33	(E)-farnesyl acetate
18	43.072	0.61	(E)-farnesyl acetate
19	46.100	2.89	heneicosane

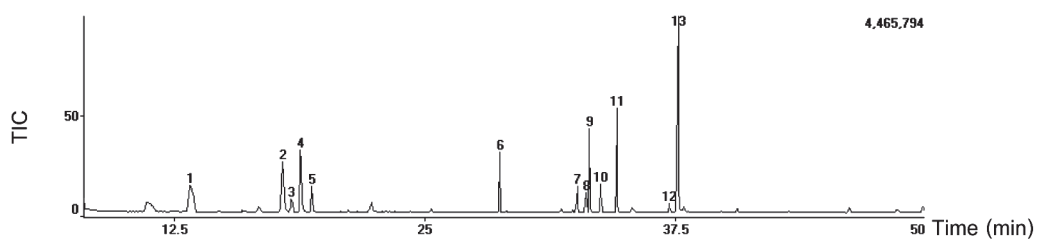

Figure 3 Chromatogram of essential oil from steam distillation.

The chemical identification by GC-MS of the hexane absolute found 17 compounds (Figure 5 and Table 4) and from the petroleum ether absolute found 13 compounds (Figure 6 and Table 5), with the major compound of both absolutes being benzyl salicylate (44.69 and 42.63%, respectively).

Enfleurage

The absolute yields of the cold and hot enfleurage were 0.3842 % (soft fat) and 12.24 % (light yellow), respectively.

Using GC-MS analysis, 13 compounds were detected in the cold enfleurage and the major component was 23.13 % linalool (Figure 7 and Table 6). In the hot enfleurage there were 12 compounds and n-undecanoic acid (31.75%) was the major component (Figure 8 and Table 7). Only three compounds: linalool, benzyl benzoate and benzyl salicylate, were found in both absolutes.

CONCLUSIONS

The characteristics and chemical compounds of frangipani oil and absolute differed depending on the extraction method. The percentage yield of frangipani oil from the three distillation methods of water, steam and water-steam was 0.0167, 0.0045, and 0.0342%, respectively, while the yield of absolute from hexane and petroleum ether was 0.4170 and 0.3510%, respectively. The absolute extraction yield from the cold and hot enfleurage was 0.3842 and 12.24%, respectively. The major compound of the essential oil and absolute from solvent extraction was benzyl salicylate, from cold enfleurage was linalool and from hot enfleurage was n-undecanoic acid. The isolation of frangipani absolute by hexane extraction was considered the most appropriate method at a pilot scale for extract

Table 2 Chemical compounds of essential oil from steam distillation.

Peak No.	Retention time (min)	%Relative peak area	Possible compounds
1	13.327	11.18	linalool
2	17.923	13.70	(Z)-geraniol
3	18.363	2.54	(Z)-citral
4	18.813	12.15	(E)-geraniol
5	19.363	4.60	(E)-citral
6	28.753	4.68	(Z)-beta-farnesene
7	32.627	2.27	alpha-farnesene
8	33.072	1.41	(Z)-farnesol
9	33.235	6.95	(E)-farnesol
10	33.789	2.36	(E)-farnesal
11	34.609	9.89	benzyl benzoate
12	37.206	0.69	1-octadecanol
13	37.669	27.58	benzyl salicylate

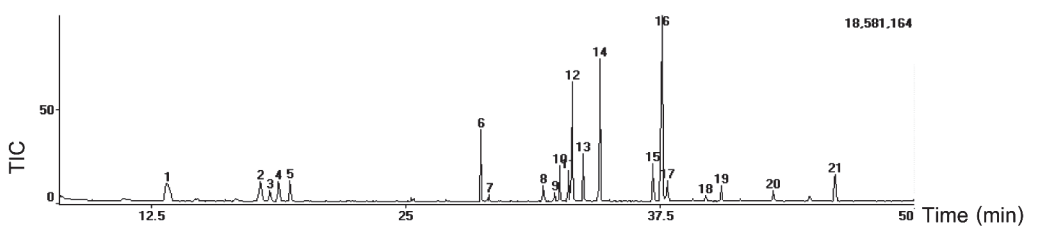

Figure 4 Chromatogram of essential oil from water-steam distillation.

use in perfume or cosmetic materials because this method is cheaper and more convenient than the enfleurage methods and it yielded a higher percentage than the distillation method. The pure essential oil from water-steam distillation was suitable for aromatherapy in uses such as massage oil.

ACKNOWLEDGEMENTS

The authors acknowledge financial support by the Kasetsart University Research and Development Institute (KURDI) and the Kasetsart Agricultural and Agroindustrial Product Improvement Institute (KAPI).

Table 3 Chemical compounds of essential oil from water-steam distillation.

Peak No.	Retention time (min)	%Relative peak area	Possible compounds
1	13.316	6.49	linalool
2	17.886	4.33	(Z)-geraniol
3	18.327	1.66	(Z)-citral
4	18.777	3.12	(E)-geraniol
5	19.324	3.09	(E)-citral
6	28.718	5.03	(Z)-beta-farnesene
7	29.104	0.39	(E)-beta-farnesene
8	31.790	1.40	1-hexadecene
9	32.360	0.45	2-methylpentadecane
10	32.592	2.62	alpha-farnesene
11	33.035	2.14	(Z)-farnesol
12	33.210	9.29	(E)-farnesol
13	33.754	3.42	(E)-farnesal
14	34.592	13.28	benzyl benzoate
15	37.166	3.04	1-octadecanol
16	37.658	31.90	benzyl salicylate
17	37.880	1.95	eicosane
18	39.763	0.46	unknown[1]
19	40.549	1.69	unknown[2]
20	43.096	1.05	(E)-farnesyl acetate
21	46.121	3.20	heneicosane

unknown[1] = m/z 41(100) 51(36.9) 68 (33.2) 77(40.6) 93(71.8) 105(35.1);
unknown[2] = m/z 41(100) 51(28.7) 69(24.6) 77(29.9) 93(51.3) 105(34.3).

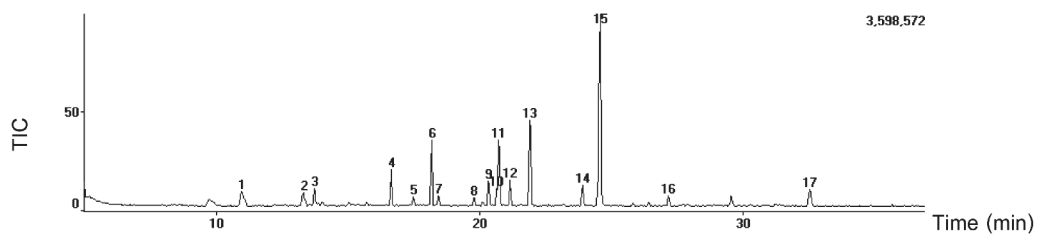

Figure 5 Chromatogram of hexane absolute.

Table 4 Chemical compounds of hexane absolute.

Peak No.	Retention time (min)	%Relative peak area	Possible compounds
1	10.981	3.66	linalool
2	13.345	3.93	(Z)-geraniol
3	13.751	3.10	(E)-geraniol
4	16.661	2.72	(E)-geranylacetnoe
5	17.510	0.69	2,4-di-t-butylphenol
6	18.199	5.53	(Z)-nerolidol
7	18.462	0.84	unknown[1]
8	19.807	0.88	1-hexadecene
9	20.363	2.56	alpha-farnesene
10	20.682	1.74	(Z)-farnesol
11	20.754	7.22	(E)-farnesol
12	21.177	2.84	(E)-farnesal
13	21.936	11.67	benzyl benzoate
14	23.927	2.80	1-octadecanol
15	24.610	44.69	benzyl salicylate
16	27.181	1.43	unknown[2]
17	32.554	3.69	isoeicosane

unknown[1] = m/z 41(100) 43(87) 55 (48) 56(25) 57(35) 68(24) 83(14) 97(15);
unknown[2] = m/z 41(100) 50(15.8) 51(23) 53(17) 68(21) 69(31) 77(33) 93(35) 105(32).

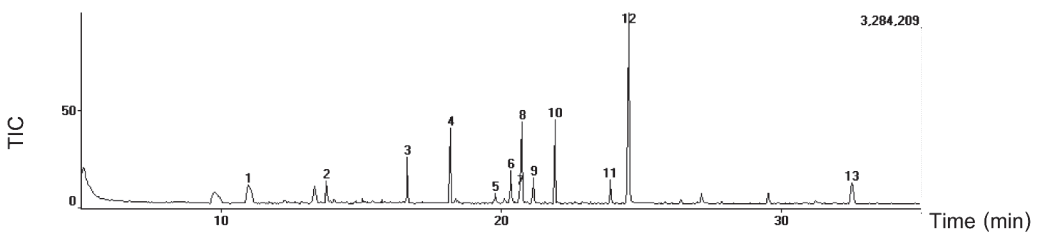

Figure 6 Chromatogram of petroleum absolute.

Table 5 Chemical compounds of petroleum absolute.

Peak No.	Retention time (min)	%Relative peak area	Possible compounds
1	10.993	6.04	linalool
2	13.753	4.44	(E)-geraniol
3	16.658	3.96	(E)-geranylacetnoe
4	18.196	6.53	(Z)-nerolidol
5	19.801	1.16	1-hexadecene
6	20.361	3.39	alpha-farnesene
7	20.678	1.54	(Z)-farnesol
8	20.751	9.49	(E)-farnesol
9	21.171	2.72	(E)-farnesal
10	21.929	10.64	benzyl benzoate
11	23.920	3.33	1-octadecanol
12	24.587	42.63	benzyl salicylate
13	32.534	4.12	Isoeicosane

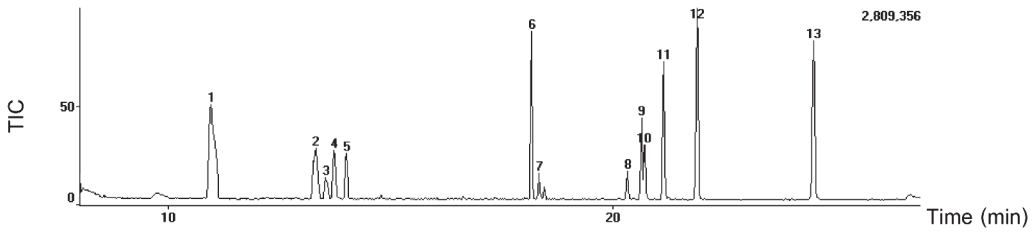

Figure 7 Chromatogram of cold enfleurage absolute.

Table 6 Chemical compounds of cold enfleurage absolute.

Peak No.	Retention time (min)	%Relative peak area	Possible compounds
1	10.962	23.13	linalool
2	13.323	8.31	(Z)-geraniol
3	13.542	1.87	(Z)-citral
4	13.732	4.69	(E)-geraniol
5	14.006	4.14	(E)-citral
6	18.182	8.16	(Z)-beta-farnesene
7	18.354	0.81	(E)-beta-farnesene
8	20.344	1.42	(E)-nerolidol
9	20.665	4.67	(Z)-farnesol
10	20.733	3.40	(E)-farnesol
11	21.165	9.04	(E)-farnesal
12	21.925	14.73	benzyl benzoate
13	24.551	15.62	benzyl-salicylate

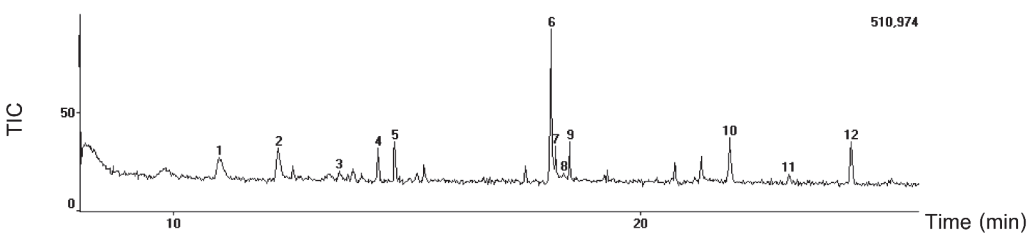

Figure 8 Chromatogram of hot enfleurage absolute.

Table 7 Chemical compounds of hot enfleurage absolute.

Peak No.	Retention time (min)	%Relative peak area	Possible compounds
1	10.989	3.80	linalool
2	12.254	3.94	octanoic acid
3	13.550	4.01	unknown[1]
4	14.381	6.69	(E,E)-2,4-decadienal
5	14.746	8.49	(E,E)-2,4-dodecadienal
6	18.094	31.75	n-undecanoic acid
7	18.195	7.91	farnesol
8	18.358	4.84	geranyl valerate
9	18.492	5.94	ethyl pentadecanoate
10	21.919	9.72	benzyl benzoate
11	23.193	3.22	unknown[2]
12	24.514	9.70	benzyl salicylate

unknown[1] = m/z 40(33) 41(37) 43.00 (70)44(56) 51(36) 55(100) 70(29) 71 (23) 79(23) 83 (37) 98 (42) 26(23);
unknown[2] = m/z 41(33) 43(100) 45 (20) 57 (26) 58 (53) 59(11) 60(15)69(17) 71(13) 149 (11.20).

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