# STUDY ON THE COMPOSITION OF ESSENTIAL OIL FROM FLOWER BUD AND BUD OF SOPHORA JAPONICA L. IN HOABINH AND HUNGYEN PROVINCES

NGHIÊN CÚU THÀNH PHẦN TINH DẦU TÙ HOA VÀ NU SOPHORA JAPONICA L. Ở TỈNH HÒA BÌNH VÀ HƯNG YÊN

# Nguyen Thi Thu Huyen, Pham Van Thiem, Phan Dinh Chau

Hanoi University of Technology

# ABSTRACT

In this study we reported identified compounds of the essential oil from the fresh bud and the flower of Sophora Japonica L. cultivated in Vietnam analyzing by GC-MS. The major components were caryophyllene, α-humulene, (-)-caryophyllene oxide, 8-heptadecene, phytol and tricosane which were detected in both flower oil and bud oil. The results also showed that these oils were poor in monoterpenes. The major groups of the essential oil of bud and flower were found to be sesquiterpenes (11.45%, 56.69%), oxygenated sesquiterpenes (8.47%, 21.05%) and oxygenated diterpenes (15.62%, 5.72%), respectively. Moreover, the changing of the composition of flower oil and bud oil both from Hoabinh and Hungyen provinces was shown. Key word index: Sophora Japonica L., essential oil, flower, bud.

# TÓM TẮT

Trong nghiên cứu này chúng tôi công bố các hợp chất nhân biết bằng phân tích GC-MS của tinh dầu nu và hoa của Sophora Japonica L. trồng ở Việt nam. Các cấu tử chính được phát hiện trong cả tinh dầu hoa và tinh dầu nụ là caryophyllene, α-humulene, (-)-caryophyllene oxide, 8-heptadecene, phytol và tricosane. Các kết quả cũng chỉ ra rằng các dầu này ít monoterpene. Các nhóm chính trong . tinh dầu nụ, tinh dầu hoa đã được tìm thấy là sesquiterpene (11.45%, 56.69%), các dẫn xuất sesquiterpene có chứa oxy (8.47%, 21.05%) và diterpene có chứa oxy (15.62%, 5.72%). Sự thay đổi thành phần hóa học của dầu nụ và dầu hoa, từ Hòa Bình và Hưng Yên cũng được chỉ ra trong nghiên Cứu.

#### **I. INTRODUCTION**

Flower buds of Sophora Japonica L. are a medicinal herb for many decades in Vietnam. To unveil discover their pharmacology, chemical studies were undertaken to reveal the rutin content which has been reported as 35.1% in the material [1]. Some other ingredients also have been identified including quercetin, isorhamnetin-3-rutinoside, luteolin 7-O-β-Dglucoside, kaempferol, azukisaponin, soyasaponin, kaikasaponin,  $\beta$ -sitosterol, isorhamnetin, kaempferol-3-rutinoside, betulin, sophoradiol, sophorin and acids [2, 3]. Furthermore, some volatile constituents in the dried flower bud have been shown in our recent study [4]. Although the chemical composition of the flower buds was investigated, the composition of their oil has not been reported before. Thus, the present paper presented on the constituents of essential oil of fresh flower and bud of S. Japonica L. using GC-MS technique.

#### **II. EXPERIMENTAL**

*Plant material:* The fresh flower and bud of S. Japonica L. were collected random in December 2008 from Hoabinh province and in January 2009 from Hungyen province, Vietnam.

*Oil isolation:* The fresh flower (2.0 kg) and fresh bud (2.0 kg) of S. Japonica L. were cleaned from impurities. Each of them was ground and distilled by Clevenger type apparatus for 8h. After decanting and drving sodium over anhydrous sulfate. the corresponding white colored oils were recovered in yield of 0.01% and 0.02 % (v/w fresh), respectively. The content of essential oil obtained from Hoabinh sample was similar to Hungyen sample. The oils were stored in sealed brown vials kept in refrigerator for analysis afterward.

Analysis: GC/MS analyses were carried out on a Shimadzu 2010 GC/MS system equipped with a DB-5 fused silica column (30m x 0.25mm, film thickness 0.25um). The column temperature was kept at 70°C for 2 min and increased to 230°C at a rate of 7°C/min and kept constant at 230°C for 15 min. The flow rate of helium as carrier gas was 1 mL/min. The mass spectrometer was taken at 70eV with 0.5sec interval, scan speed (1666) scanning from 40 to 800 m/z. The ion source and interface temperature were set at 200°C and 250°C, respectively. Approximate 1mg portion of each sample was dissolved in 1.5ml n-hexane solvent and 0.5µl volume was injected into the column using a split ratio of 10:1 with injection temperature set to 250°C.

*Identification of components:* Identification of the constituents of oil was made by comparison of their mass spectra and retention indices (RI) with those given in the literature and MS data. Percentage of the identified compounds was computed from peak areas. Total oil was set at 100%.

# **III. RESULTS AND DISCUSSION**

The chemical composition of flower and bud oils is given in Table I and a chromatogram of essential oil of fresh bud also was shown in Figure I. A total of 15 (20) compounds were identified, constituting over 63.94% (84.53%) of composition of bud oil from Hungyen and Hoabinh sample. For the flower oil, a total of 22 compounds were identified, representing 81.11% of Hungyen sample and 97.75% of Hoabinh sample.

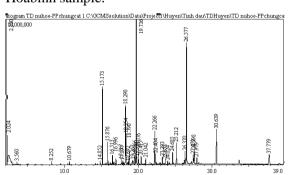


Fig.1 Chromatogram of essential oil from fresh bud of S. Japonica L. cultivated in Hoabinh province

Table I showed that these oils were poor in monoterpenes but rich in sesquiterpenes, oxygenated sesquiterpenes and diterpenes. The major groups in the oil were found to be sesquiterpenes (11.45%, 56.69%), oxygenated 21.05%) sesquiterpenes (8.47%)and oxygenated diterpenes (15.62%, 5.72%) in samples 2 and 4, respectively. Caryophyllene, *α*-humulene and hexacosane were predominating constituents in sesquiterpene hydrocarbon and *n*-alkane groups. The main components of the flower oil, bud oil in four samples were caryophyllene (26.84%, 9.08%, 25.17%, 44.61%), α-humulene (5.77%, 2.37%, 6.05%, 10.96%), (-)-caryophyllene oxide (1.47%, 5.89%, 9.96%, 17.45%), 8-heptadecene (10.62%, 6.39%, 4.81%, 7.05%), phytol (5.99%, 15.37%. 10.81%, 5.72%) and hexacosane (19.57%, 6.20%), respectively.

The differences of composition between flower oil and bud oil were exhibited in Table I. Some constituents were found in the flower oil such as methyl butyrate,  $\alpha$ -copaene,  $\beta$ -elemene,  $\delta$ -cadinene. pentadecane. phenanthrene. farnesyl acetate, eicosane while they were unshown in the bud oil. 3-Hexaneone, 2hexaneone, 3-hexaneol, 2-hexaneol, nerolidol, isophytol components were absent from the flower oil whereas they were found in the bud oil. Moreover, terpenoids were present such as hexahydrofarnesyl acetone and phytol which is connected biogenesis to the and biotransformation of chlorophyll in plant. The differences in their contents will be characterized for the particular ontogenetic stage. Although phytol was the main component after flowering and hexahydrofarnesyl acetone were present only at flowering stage, their content in flower and bud was approximately equal in the experiment. Thus, the differences of composition between flower oil and bud oil shows qualitative rather than quantitative variation.

Table I also shows the change of the content of major groups in essential oil from Hungyen and Hoabinh samples. The sesquiterpene content of bud oil in Hungyen (32.61%) was higher than in Hoabinh (11.45%) but the monoterpene, oxygenated sesquiterpene, oxygenated diterpene and nonterpene content was lower than in Hoabinh sample. For flower oil in Hungyen, the nonterpene and oxygenated diterpene content was higher than in Hoabinh sample but the monoterpene, sesquiterpene and

oxygenated sesquiterpene content was lower than in Hoabinh sample. These changes of chemical composition depend on geographic, climatic and harvesting condition. However, to prove this hypothesis, fresh flower and bud from different regions should be collected and carefully analyzed.

Table I: Constituents of essential oil from flowers and buds of S. Japonica L. cultivated in Hoabinh and Hungyen provinces

$\mathbf{N}^{0}$	Similarity	RI <sup>a</sup>	Component <sup>b</sup>	Content <sup>c</sup> (%)			
				Bud		Flower	
				Sample 1	Sample 2	Sample 3	Sample 4
				(Hungyen)	(Hoabinh)	(Hungyen)	(Hoabinh
					(Houthin)		)
1.	96	688	2-pentanone	1.40	-	2.08	1.39
2.	97	-	methyl butyrate	-	-	0.17	0.30
3.	96	-	3-hexanone	0.66	0.27	-	-
4.	97	-	2-hexanone	0.98	0.36	-	-
5.	96	797	3-hexanol	0.39	0.14	-	-
6.	97	873	2-hexanol	0.46	0.23	-	-
7.	96	1030	l-limonene	-	0.08	1.00	1.09
8.	97	1104	nonanal	-	0.50	0.19	0.35
9.	95	1379	α-copaene	-	-	0.25	0.45
10.	96	1393	β-elemene	-	-	0.23	0.41
11.	98	1438	caryophyllene	26.84	9.08	25.17	44.61
12.	98	1455	α - humulene	5.77	2.37	6.05	10.96
13.	96	1500	pentadecane	-	-	0.23	0.36
14.	94	1520	δ-cadinene	-	-	0.13	0.26
15.	93	1539	nerolidol	-	0.33	-	-
16.	96	1573	(-)-caryophyllene oxide	1.47	5.89	9.96	17.45
17.	95	1602	humulene epoxide II	-	1.28	1.59	2.90
18.	93	1615	tetradecanal	-	0.45	0.59	0.44
19.	96	1672	8-heptadecene	10.62	6.39	4.81	7.05
20.	93	1752	phenanthrene	-	-	0.23	0.38
21.	98	1843	farnesyl acetate	-	-	0.20	0.32
22.	97	1845	hexahydrofarnesyl acetone	0.33	0.97	0.32	0.38
23.	95	1901	palmitic acid, methyl ester	1.16	0.46	-	0.50
24.	94	1944	isophytol	0.3	0.25	-	-
25.	96	2000	eicosane	-	-	0.19	0.13
26.	97	2128	phytol	5.99	15.37	10.81	5.72
27.	96	2200	docosane	1.32	2.71	0.70	1.67
28.	97	2301	tricosane	6.25	17.83	10.01	0.63
29.	96	2598	hexacosane	-	19.57	6.20	-
Monoterpene hydrocarbons				0	0.08	1.00	1.09
Sesquiterpene hydrocarbons				32.61	11.45	31.83	56.69
Oxygenated sesquiterpenes				1.80	8.47	12.07	21.05
Oxygenated diterpenes				6.29	15.62	10.81	5.72
Nonterpenes				23.24	48.91	25.40	13.20
Unknown				36.06	15.47	18.89	2.25
Total identified				63.94	84.53	81.11	97.75

<sup>a</sup>RI retention index: measured relative to *n*-alkane on DB-5 column and referenced in [5] references

<sup>b</sup> The compounds listed in order of elution from DB-5 column.

<sup>c</sup>: The content based on the percentage of peak area

- = Undetected compound

In the experience, we smelt a similar scent found from cooking glutinous rice and distilling leaves of Pandanus amaryllifolius Roxb. during distillation. The special flavor differs from scent found in the fresh or dried material. Table I shows that these oils have components which some are chemical composition of *pandanus* leaf and glutinous rice such as 3-hexaneone, 2-hexaneone, 3-hexaneol, β-caryophyllene, 3-hexaneol. Although 2acetyl-1-pyrroline, a constituent has been reported to be an important flavor component in the materials, was undetected in flower and bud oils so the result explained the similar scent between these materials [6,7].

# **IV. CONCLUSIONS**

In the paper, quantification of essential oil exists in fresh flower and bud of *S. Japonica* L. has been performed using GC-MS. The major components were caryophyllene,  $\alpha$ -humulene, (-)-caryophyllene oxide, 8-heptadecene, phytol and hexacosane detected in both flower oil and bud oil. On the other hand, the changing composition of oil samples in flower and bud, in Hoabinh and Hungyen were shown in the study.

# REFERENCES

- 1. Le Thi Anh Dao; Scientific information Vietnam National University, Hanoi pedagogic University, (1), 47 49 (1997).
- Reneta Gevrenova, Gerassim Kitanov, Dessislava Ilieva; Pharmaceutical Biology, Vol.45 (2),149 – 155 (2007).
- 3. *Kimura Masayuki, Yamada Hiromi*; Journal of the Pharmaceutical Society of Japan, Vol. 104 (4), 340-346 (1984).
- 4. Nguyen Thi Thu Huyen, Pham Xuan Thang, Phan Dinh Chau, Pham Van Thiem; Journal of science and technology in Vietnam, Vol. 45 (1B), 497-502 (2007).
- 5. http://www.kovats.org/
- 6. http://www.uni-graz.at/~katzer/engl/Pand\_ama.html
- 7. E. Lupotto, G. Mellerio, F. Corana, S. Cavigiolo, D. Greppi; Proceedings of the XLIX Italian society of Agricultural genetics annual congress potenta, Italy (2005).
- *Contact*: Nguyen Thi Thu Huyen Tel: (+84)912.411.769; E-mail: thuhuyen\_hut06@yahoo.com Center for Education and Development of Chromatography, Hanoi University of Technology No. 1, Dai Co Viet Str., Hanoi, Vietnam