J Food Sci Nutr
Vol 13, p 18～22 (2008)
DOI: 10.3746/jfn.2008.13.1.018

J Food Science and Nutrition

Characterization of Volatile Components in Field Bean (*Dolichos lablab*) Obtained by Simultaneous Steam Distillation and Solvent Extraction

Joo-Shin Kim[1] and Hau Yin Chung[2†]

[1]*Kwangil Synthesis Plant Co. Ltd., Seoul 155-055, Korea*
[2]*Department of Biology, Food and Nutritional Sciences Programme, and Food Science Laboratory, The Chinese University of Hong Kong, N.T., Hong Kong*

Abstract

Volatile components in field bean (*Dolichos lablab*) were collected by simultaneous steam distillation and solvent extraction and analyzed by gas chromatography-mass spectrometry. One hundred and five components were identified including alcohols (32), ketones (18), aldehydes (9), acid (1), alkanes (5), aromatics compounds (4), esters (2), furans (2), naphthalene (1), pyrazines (4), pyridine (3), sulfur-containing compounds (4) and terpenes (7) and miscellaneous compounds (13). Relatively high concentration of *n*-hexanal found in the field bean might be undesirable to some consumers.

Key words: field bean, volatile components, SDE, GC-MS

INTRODUCTION

Legumes contain significant amount of protein, vitamins, minerals and dietary fiber and are good alternatives to red meat and fatty foods. This contributes to the increased consumption of legumes in recent years (1). Cooked legumes are good source of nutrients, but undercooked legumes have poor nutritive value since they may contain antinutrient factors such as protease inhibitors, phytate, tannins and lectins which reduce the availability of nutrients (1-5). Legumes can be processed and transformed into different ingredients such as flour, protein concentrate or protein isolate, etc. for the production of finished products (6).

Field bean, *Dolichos lablab* is often used as an ingredient in preparing deserts and soups in Asia, but as ornamental in the USA (7). The plant is a vine and it can grow to a length of 9 m, and has a larger seed than does soybean. The plant takes about 90～150 days to reach maturity and survives in poor soil conditions (7). Some functional properties of the bean were reported by Chau et al. (8) and more recently, bioactive peptides derived from the bean were found to stimulate cholecystokinin from the enteroendocrine cells and suppress food intake (9).

Many researchers have focused on the nutritional values and health benefits of various beans (10-16), limited data is available on the volatile components of cooked field beans (17), which may affect its acceptance. Therefore, the objective of this study was to determine the profile of volatile components in the field bean and to deduce its acceptance by consumers.

MATERIALS AND METHODS

Sample preparation

Field beans (*Dolichos lablab*) were purchased from a supermarket in Hong Kong. Their moisture content was determined according to the AOAC method (18). They were lightly blended for 40 s using a blender (model no. MX-T2GN, National brand, Masushita Electric Co. Ltd., Taipei, Taiwan) to obtain particles sizes between 250～355 μm using standard testing sieves (ASTM E-11 Specification no. 45 and 60, VWR Scientific, West Chester, USA) for simultaneous steam distillation and solvent extraction.

Simultaneous steam distillation-solvent extraction (SDE)

Ground samples (35 g) were transferred to a stainless steel pocket (15 cm×5 cm) which was loaded on a platform formed by three 20-cm spatulas in a 5-L round bottom flask (19). One mL of 2,4,6-trimethylpyridine (internal standard, IS) at a concentration of 10 μg/mL was added to each sample. Five hundred mL of double distilled water and 50 mL of redistilled dichlormethane were used in the sample flask and solvent flask, respectively. Each sample was extracted for 2 hr in a Likens and Nickerson (20) type SDE apparatus. Triplicate extractions were carried out. Extracts were concentrated

†Corresponding author. E-mail: anthonychung@cuhk.edu.hk
Phone: +852-2609-6149, Fax: +852-2603-5745

with a stream of prepurified nitrogen gas to a 0.5 mL volume, dried by passing through the 2.5-g anhydrous sodium sulfate into 1.5-mL amber vials and stored at -80°C until further analyses.

Gas chromatography-mass spectrometry (GC-MS)

A Hewlett-Packard 6890 GC coupled with an MS was used. A 60-m fused silica open-tubular column (Supelcowax 10, 0.25 mm i.d., 0.25 μm film thickness, Bellefonte, PA) was installed in the GC. The GC was operated under the following conditions: initial and final temperatures and holding times were 35°C for 5 min and 195°C for 110 min, respectively; the ramp rate was 2°C/min. Linear flow rate was 30cm/s and split rate was 10:1. The MS conditions were as follows: electron ionization voltage, 70 eV; mass range, 33- 450 a.m.u.; electron multiplier voltage, 1,118 V; scan rate, 3.49 scans/min. Five μL of the extract was injected into the GC-MS.

Qualification and quantification of field bean

Positive identification of each component was based on matches of both retention time and mass spectrum with those of its authentic standard. Quantification was carried out by the generation of calibration curves for each positively identified component.

RESULTS AND DISCUSSION

The moisture content of field bean was 9.12% which was lower than that found by Chau et al. (8) at 11.4%. One hundred and five compounds were distributed into fourteen categories, as shown in Table 1. Fig. 1 shows a typical chromatogram of the field beans. Among the chemical classes, alcohols and ketones were the two largest classes in number with 32 and 18 compounds, respectively. Miscellaneous compounds and aldehydes were comprised of 13 and 9 compounds, respectively. The rest of the chemical classes contained 7 or less compounds. Each chemical class has at least one volatile component with very high mean concentration of more than 25 μg/kg dry sample (Table 1). The gas chromatography-mass spectrometry analysis also showed that the volatile compounds in field beans contained high percentages of ketones (40.4%), alcohols (30.8%), aldehydes (9.2%). Compound classes with concentration below 9% but higher than 1% include miscellaneous compounds (4.7%), acids (3.5%) and furans (1.8%). The remainder of the classes had concentration below 1%. Nineteen compounds had micromoles per kilogram dry weight sample higher than 1. Five compounds were found to have relatively higher concentrations than 5 μmole/kg which include 2-propanone (35.15 μmole/kg), 1-hydroxy-2-propanone (30.11 μmole/kg), 2,3-butanedone (19.82 μmole/kg), 1-hexanol (16.10 μmole/kg) and hexanal (9.08 μmole/kg). Fernandes and Nagendrappa (17) identified 42 compounds in the *D. lablab* pod exudate, which mainly consisted of fatty acids and their methyl esters. In this study, many more components were identified in the pod.

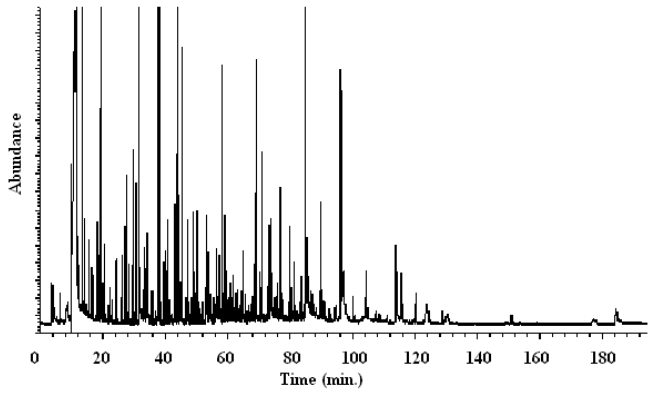

Fig. 1. A typical total ion chromatogram for volatile components of field bean (*Dolichos lablab*)

In the headspace of ground raw and cooked soybeans, Del Rosario et al. (21) reported that 62 and 71 compounds were identified, respectively and 47 compounds were common to both raw and cooked soybeans. Some compounds identified in the soybeans were also found in the field bean (Table 1).

Samoto et al. (22) reported that 17 components were identified in the off-flavor soy protein isolates. By removing the oil-body associated proteins, the researchers observed a decrease in the intensities of the off-flavor. Thirteen out of 17 compounds found in the soy protein isolate were also identified in the field bean including pentanal, hexanal, (*E*)-2-pentenal, heptanal, octanal, nonanal, benzaldehyde, 2-heptanone, 2-pentylfuran, 1-penten-3-ol, 1-pentanol, 1-hexanol and 1-octen-3-ol.

Fourteen compounds contributing to the beany odor in the soy protein isolate were also reported by Boatright and Lei (23) using the gas chromatography/olfactometry (GC/O) technique. The most potent components in the headspace of the isolate as evaluated by a group of sniffers was *n*-Hexanal. Boatright and Lei (23) described it as having an oxidized/nutty odor. Overall, nine components including pentanal, *n*-hexanal, benzaldehyde, 2-pentylfuran, 2,3-butanedione, 2,3-pentanedione, 2-heptanone, dimethyldisulfide and dimethyl trisulfide were found. Compared to the red bean, both beans have six common components. Further evaluation of the soy protein isolate using the aroma extract dilution analysis (AEDA) by the same group revealed that dimethyl trisulfide had the highest dilution factor and was followed

Table 1. Volatile components in *Dolichos lablab*

Compound name[1)]	RI[2)]	MW[3)]	*m/z*[4)]	Conc.[5)] (μg/kg)
Acid (1)				
hexadecanoic acid	2800<	256.24	256	542.0±316.6
Alkanes (5)				
undecane	1096	156.19	57	2.6±0.5
7-oxabicyclo[4.1.0]heptane	1155	98.07	83	77.5±6.6
tridecane	1298	184.22	85	4.0±0.1
tetradecane	1398	198.24	85	5.9±0.2
pentadecane	1498	212.25	85	7.2±0.5
Aromatics (4)				
methylbenzene[a,b]	1037	106.08	91	26.0±4.3
1,4-dimethylbenzene[a,b]	1133	106.08	106	2.5±0.2
1,3-dimethylbenzene[a,b]	1180	106.08	106	6.9±0.5
ethenylbenzene	1260	104.06	104	1.8±0.1
Ester (2)				
ethyl hexadecanote	2249	284.27	88	7.8±4.0
ethyl linoleate	2521	308.27	308	13.2±0.4
Furans (2)				
2-ethylfuran	1000>	96.06	81	124.0±9.1
2-pentylfuran[a,b,c,e]	1234	138.10	81	156.9±6.8
Miscellaneous compounds (13)				
1-methyl-1*H*-pyrrole	1141	81.06	81	5.72±0.8
furfural	1469	96.02	96	449±47.0
2-acetylfuran	1509	110.04	95	10.5±1.6
1*H*-pyrrole	1521	67.04	67	35.5±3.2
5-methylfurfural	1577	110.04	110	21.1±4.8
2-formyl-1-methylpyrrole	1624	109.05	109	7.5±1.2
2-hydroxymethylfuran	1663	98.04	98	79.5±18.3
(*E*)-anethole	1832	148.09	148	1.4±0.2
benzeneacetonitrile	1932	117.06	117	2.8±0.1
2-acetylpyrrole	1973	109.05	94	61.4±20.1
2-formylpyrrole	2027	95.04	66	4.7±0.8
1*H*-indole	2449	117.06	117	48.5±9.3
diisobutyl phthalate	2533	278.34	149	2.8±1.0
Pyrazines (4)				
pyrazine[b]	1210	80.04	80	20.2±0.9
2,6-dimethylpyrazine[a,b]	1326	108.07	108	32.5±0.9
ethylpyrazine[b]	1333	108.07	107	5.6±0.7
2,3-dimethylpyrazine	1344	108.07	108	3.9±0.4
Pyridine (3)				
pyridine	1176	80.04	79	26.9±5.4
2,4,6-trimethylpyridine	1362	121.09	121	nd
2-acetylpyridine	1605	110.07	79	108.3±10.6
Terpenes (7)				
l-linalool	1548	154.14	93	75.5±7.3
junipene	1569	204.19	161	3.1±0.3
α-terpineol	1697	154.14	136	4.6±0.8
(E)-geraniol	1846	154.14	69	9.5±0.3
geranyl acetone	1856	194.17	151	15.5±1.5
β-ionone	1940	192.15	177	2.2±0.3
(*Z*)-jasmone	1944	164.12	164	5.0±0.8
Aldehydes (9)				
pentanal[a,b,c,d]	1000>	86.07	58	194.5±13.5
hexanal[a,b,c,d,e]	1082	100.09	56	909.3±89.2
(*E*)-2-pentenal[c]	1129	84.06	55	68.4±6.4
heptanal[b,c]	1186	114.10	70	28.4±2.8
octanal[b,c]	1291	128.12	41	13.5±1.0
nonanal[a,b,c]	1396	142.14	98	17.6±0.4
(*E*,*E*)-2,4-heptadienal	1496	110.07	81	10.7±0.3
benzaldehyde[b,c,d]	1527	106.04	106	116.1±24.5
phenylethanol	1648	120.06	91	70.8±16.9
Alcohols (32)				
2-butanol[a,b]	1021	74.07	45	291.4±23.9
1-propanol[a,b]	1034	60.06	59	40.1±4.8
2-methyl-3-buten-2-ol	1037	86.07	71	55.4±3.3
2-methyl-1-propanol[a,b]	1088	74.07	43	103.3±6.4
3-pentanol[a]	1105	88.09	59	9.1±0.5
2-pentanol[a]	1117	88.09	45	20.7±1.2
1-penten-3-ol[a,b,c]	1159	86.07	57	41.7±5.8
3-hexanol	1195	102.10	59	3.7±0.2
3-methyl-1-butanol[a,b]	1205	88.09	55	164.7±6.8
1-pentanol[a,b,c]	1248	88.09	42	362.8±13.9
cyclopentanol	1299	86.13	57	4.7±0.2
2-heptanol[a,b]	1318	116.12	45	28.0±0.1
1-hexanol[a,b,c]	1352	102.10	56	1643.9±36.6
(*Z*)-3-hexenol	1384	100.09	67	78.3±0.4
3-octanol[a,b]	1393	130.14	59	48.8±0.5
2-butoxyethanol	1400	118.10	45	56.9±7.6
(*E*)-2-hexen-1-ol	1405	100.09	41	34.4±1.3
2-octanol[a,b]	1418	130.14	45	4.5±0.0
1-octen-3-ol[a,b,c]	1451	128.12	57	508.5±6.1
1-heptanol	1454	116.12	70	52.9±2.7
2-ethyl-1-hexanol	1489	130.14	43	2.9±0.3
1-octanol	1557	130.14	56	13.9±0.8
2-chlorocyclohexanol	1655	134.61	57	21.5±0.3
α-methyl-benzenemethanol	1814	122.07	107	10.3±0.7
2-nitrophenol	1831	139.03	139	7.2±0.6
2-methoxyphenol	1863	124.05	109	83.3±13.2
benzenemethanol	1878	108.06	108	228.0±24.8
benzeneethanol	1912	122.07	91	121.56±15.8
2,6-bis(1,1-dimethylethyl)-4-methylphenol	1916	220.18	205	7.7±1.2
phenol	2010	94.04	94	71.6±10.4
3-methylphenol	2094	108.06	108	426.1±16.2
2,4-bis(1,1-dimethylethyl)-phenol	2312	206.17	191	18.9±6.3
Ketones (18)				
2-propanone[a,b]	1000>	58.04	58	2040.1±666.0
2-pentanone	1000>	86.07	86	29.1±4.5
2,3-butanedione[b,d]	1000>	86.04	86	1705.2±223.4
1-penten-3-one	1019	84.06	55	26.9±2.3
2,3-pentanedione[b,d]	1061	100.05	100	357.4±71.8
2-hexanone[b]	1079	100.09	58	9.9±1.3
(*E*)-3-penten-2-one	1125	84.06	69	59.7±1.8
4-methyl-3-penten-2-one	1131	98.07	83	4.4±0.1
2-heptanone[b,c,d]	1182	114.10	58	111.0±11.4
3-hydroxy-2-butanone	1285	88.05	45	303.0±32.2
cyclohexanone	1289	98.07	55	7.9±1.0
1-hydroxy-2-propanone	1301	74.04	43	2229.0±529.9
6-methyl-5-hepten-2-one	1340	126.10	108	12.3±0.2
2-methyl-2-cyclopenten-1-one	1369	96.06	67	10.4±0.2
2-undecanone	1600	170.17	58	10.1±2.3
γ-butyrolactone	1632	86.04	42	154.6±36.2
2-tridecanone	1810	198.20	58	23.4±2.9
γ-nonalactone	2032	156.12	85	149.1±23.1
Sulfur-containing compounds (4)				
dimethyldisulfide[b,d]	1073	93.99	94	9.1±29.3
dimethyl trisulfide[d,e]	1383	125.96	126	5.0±2.8
2-(methylthio)ethanol	1531	92.03	61	43.4±6.9
2-formylthiophene	1698	112.00	112	4.0±0.3
Naphthalene (1)				
naphthalene	1745	128.06	128	42.8±2.9

[1)]Compound reported in other references; References: [a]del Rosario et al. (21)－raw; [b]del Rosario et al. (21)－heated; [c]Boatright and Lei (23)－by gas chromatography/olfactometry; [d]Boatright and Lei (23)－by aroma extract dilution analysis; [e]Samoto et al. (22).
[2)]Retention index. [3)]Molecular weight. [4)]Mass/charge: specific fragment used for the determination of area ratio.
[5)]Concentration and standard deviation (μg/kg) on a dry weight basis.

by (*E*,*E*)-2,4-decadienal, 2-pentylpyridine, (*E*,*E*)-2,4-nonadienal, *n*-hexanal, 1-phenylethanone and 1-octen-3-one. However, in field bean, only dimethyl trisulfide and *n*-hexanal were found whereas in red bean, only 1-phenylethanone and *n*-hexanal were found. Although dimethyl trisulfide was cited as a potent component in the soy protein isolate, its concentration was quite low in the field bean, at 5.0 μg/kg, making it less likely to contribute significantly to the malodor than *n*-hexanal (909.3 μg/kg).

As *n*-hexanal was present in soy-based products possessing off-flavor, it is one of the important common off-flavor components found in all samples. Apparently, it could be a major contributor to off-flavor if it is found in other types of beans (21-23). *n*-Hexanal has a fatty, green, grassy, powerful and penetrating odor (24). Different methods have been proposed to control its presence in some specific products (25-27).

In this study, field bean contained 909 μg/kg of *n*-hexanal, which is more than three times the value found in the red beans (263.8 μg/kg) (28). Since *n*-hexanal has a low threshold value of 5.75×10^{-8} g/L (29), its calculated odor activity value (OAV) is much higher than the rest of the compounds in the same class (30), and it is higher than that the OAV found for the red bean. Apparently, the high OAV in the field bean would suggest a more distinctive green odor contributed by *n*-hexanal. For consumers who are not familiar with this odor, they will find it rather undesirable.

In conclusion, field beans had more than one hundred components distributed among 14 chemical classes. Nineteen compounds dominated the composition of the bean based on their micromolar value/kg samples. *n*-Hexanal was among the dominant compounds identified based on OAV calculation. It contributed strongly to the odor of the field beans, which may not be desirable to some consumers.

REFERENCES

1. Morrow B. 1991. The rebirth of legumes. *Food Technol* 45: 96-121.
2. Nielsen SS. 1991. Digestibility of legumes proteins. *Food Technol* 45: 112-114.
3. Silverstein LJ, Swanson BG, Moffett DF. 1996. Procyanidin from black beans (*Phaseolus vulgaris*) inhibits nutrient and electrolyte absorption in isolated rat ileum and induces secretion of chloride ion. *J Nutr* 126: 1688-1695.
4. Kamlesh S, Neelam K, Saroj B. 2002. Variability in physico-chemical properties and nutrient composition of newly released ricebean and fababean cultivars. *J Food Compos Anal* 15: 159-167.
5. Boniglia C, Fedele E, Sanzini E. 2003. Measurement by ELISA of active lectin in dietary supplements containing kidney bean protein. *J Food Sci* 68: 1283-1286.
6. Uebersax MA, Ruengsakulrach S, Occena LG. 1991. Strategies and procedures for processing dry beans. *Food Technol* 45: 104-108, 110-111.
7. Christman S. 2004. *Dolichos lablab*. No. 612. Floridata. http://www.floridata.com/ref/D/doli_lab.cfm. Accessed Jan 12, 2008.
8. Chau CF, Cheung PCK, Wong YS. 1997. Functional properties of protein concentrates from three Chinese indigenous legume seeds. *J Agric Food Chem* 45: 2500-2503.
9. Sufian MK, Hira T, Asano K, Hara H. 2007. Peptides derived from dolicholin, a phaseolin-like protein in country beans (*Dolichos lablab*), potently stimulate cholecystokinin secretion from enteroendocrine STC-1 cells. *J Agric Food Chem* 55: 8980-8986.
10. Hughes JS. 1991. Potential contribution of dry bean dietary fiber to health. *Food Technol* 9: 122-126.
11. Grant G, Edwards J, Pusztai A. 1995. Alpha-Amylase inhibitor levels in seeds generally available in Europe. *J Sci Food Agric* 67: 235-238.
12. Chau CF, Cheung PCK, Wong YS. 1997. Effects of cooking on content of amino acids and antinutrients in three Chinese indigenous legume seeds. *J Sci Food Agric* 75: 447-452.
13. Chau CF, Cheung PCK, Wong YS. 1998. Hypocholesterolemic effects of protein concentrates from three Chinese indigenous legume seeds. *J Agric Food Chem* 46: 3698-3701.
14. Itoh T, Itoh Y, Mizutani M, Fujishiro K, Furuichi Y, Komiya T, Hibasami H. 2002. A hot water extract of adzuki (*Vigna angularis*) induces apoptosis in cultured human stomach cancer cells. *J Jpn Soc Food* 49: 339-344.
15. Han KH, Fukushima M, Kato T, Kojuma M, Ohba K, Shimada K, Sekikawa M, Nakano M. 2003. Enzyme-resistant fractions of beans lowered serum cholesterol and increased sterol excretions and hepatic mRNA levels in rats. *Lipids* 38: 919-924.
16. Han KH, Fukushima M, Shimizu K, Kojima M, Ohba K, Tanaka A, Shimada K, Sekikawa M, Nakano M. 2003. Resistant starches of beans reduce the serum cholesterol concentration in rats. *J Nutr Sci Vitaminol* 49: 281-286.
17. Fernandes AA, Nagendrappa G. 1979. Chemical constituents of *Dolichos lablab* (Field Bean) pod exudate. *J Agric Food Chem* 27: 795-798.
18. AOAC. 1980. *Official Methods of Analysis*. 14th ed. Association of official analytical chemists, Washington, DC. p 31.
19. Chung HY, Shing Yung IK, Kim J-S. 2001. Comparison of volatile components in dried scallops (*Chlamys farreri* and *Patinopecten yessoensis*) prepared by boiling and steaming methods. *J Agric Food Chem* 49: 192-202.
20. Likens ST, Nickerson GB. 1964. Detection of certain hop oil constituents in brewing products. *Am Soc Brew Chem Proc* 2: 5-13.
21. Del Rosario R, de Lumen BO, Habu T, Flath RA, Mon TR. 1984. Comparison of headspace volatiles from winged beans and soybeans. *J Agric Food Chem* 32: 1011-1015.
22. Samoto M, Miyazaki C, Kanamori TA, Akasaka T, Kawamura Y. 1998. Improvement of the off-flavor of soy protein isolate by removing oil-body associated proteins and polar lipids. *Biosci Biotechnol Biochem* 62: 935-940.
23. Boatright WL, Lei Q. 1999. Compounds contributing to the beany odor of aqueous solutions of soy protein isolates.

J Food Sci 64: 667-670.
24. Sigma-Aldrich. 2003. *Flavors and Fragrances*. International ed, Sigma-Aldrich, Milwaukee, WI, USA.
25. Maheshwari P, Ooi ET, Nikolov ZL. 1995. Off-flavor removal from soy-protein isolate by using liquid and supercritical carbon dioxide. *J Am Oil Chem* 72: 1107- 1115.
26. Peeyush M, Murphy PA, Nikolov ZL. 1997. Characterization and application of porcine liver aldehyde oxidase in the off-flavor reduction of soy proteins. *J Agric Food Chem* 45: 2488-2494.
27. Wang ZH, Dou J, Macura D, Durance TD, Nakai S. 1998. Solid phase extraction for GC analysis of beany flavors in soymilk. *Food Res Int* 30: 503-511.
28. Kim J-S, Chung HY. 1997. Analysis of the volatile components in red bean (*Vigna angularis*). *J Appl Biol Chem* 50: 120-126.
29. Devos M, Patte F, Rouault J, Laffort P, Van Gemert LJ. 1990. *Standardized Human Olfactory Thresholds*. Oxford University Press, New York, NY, USA.
30. Guadagni DG, Buttery RG, Harris J. 1966. Odour intensities of hop oil component. *J Sci Food Agric* 17: 142-144.

(Received January 14, 2008; Accepted February 21, 2008)