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Effects of Drying and Roasting to Effectively use a Discarded Part of Asparagus (Asparagus officinalis L.)

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Abstract

Asparagus (*Asparagus officinalis* L.) has a characteristic flavor and useful components, although the lower stem is not suitable for eating because it has a fibrous skin like wood, being normally discarded. Therefore, to propose drying and roasting as methods for using the hard lower stem of asparagus, usually discarded, useful components in the asparagus stem after drying and roasting were analyzed. The rutin content was decreased significantly by drying and roasting. The ascorbic and folic acids contents were almost unchanged by drying but decreased by roasting. The fructose content was increased by drying, although glucose was almost unchanged. Both were decreased significantly by roasting. The sucrose content was increased by drying but unchanged by roasting. The increase or decrease in some free amino acids were caused after drying, and most of free amino acids disappeared after roasting. Furthermore, characteristic volatile compounds derived from the Maillard reaction were caused by drying and roasting. Most of the useful components in the hard lower stem of asparagus can be concentrated by drying, and characteristic volatile compounds be added by drying and roasting. Therefore, it is promising that the asparagus stem, an unused resource, is able to be utilized as a useful food material by drying and roasting

Introduction

Asparagus (*Asparagus officinalis* L.) is a perennial herbaceous plant of the family Liliaceae, and a vegetable having a characteristic flavour and useful components such as asparagine, rutin, ascorbic and folic acids (Chitrakar et al., 2019). There is relatively high production of asparagus around the world: yields in 2020 were 5,671,000 and 26,700 t worldwide and in Japan, respectively (MAFF, 2021). Generally, asparagus is delivered after the spears are adjusted to a standard length by cutting the lower stem away. The part cut away is not suitable for eating because it has a fibrous skin like wood, being normally discarded (Zhang et al., 2014). However, this hard stem has useful components unique to asparagus (Chitrakar et al., 2019). Therefore, if a method of utilizing this by-product without loss of components can be proposed, it would represent an effective practical use of unused resources. To date, some drying methods (Nindo et al., 2003), concentration by spray drying (Siccama et al., 2021), treatment with hot water (Jaramillo-Carmona et al., 2019), and applications in cookies (Liu et al., 2016) and juice (Zhang et al., 2014) have been reported as methods for processing this unused part of asparagus. However, these processes are sophisticated to maintain its quality. Therefore, it is important to propose a simpler processing method that can be easily used by asparagus producers.

Drying is a normal food processing method. In Japan, dried shiitake mushrooms called ‘Hoshishiitake’ and dried radish strips called ‘Kiriboshidaikon’ have been used as dried foods (Kitaoka-Saito et al., 2020). Drying can not only improve the preservability of food due to the decrease in water content but also concentrate the components. In particular, ‘Hoshishiitake’ has been used as a material of the umami component in soup stoch (Kasuga & Aoyagi, 2005). Therefore, it may be able to be used as a material in soup stock by dried asparagus stem.

Also, tea is a beverage drunk across the whole world. There are many types of tea such as green, roasted green, oolong, black and Pu'er tea, obtained by different processing methods despite being from the same plant (*Camellia sinensis*). Among them, roasted green tea is often drunk in Japan, and has the characteristic fragrance obtained by roasting. The tea leaf is produced by roasting the steamed and dried green tea leaf (Mizukami, 2012). Therefore, roasted asparagus stem is able to be expected to add characteristic fragrance.

As asparagus stem has been successfully applied to materials of soup stock and tea, it is expected that drying would better preserve the abundant useful components. In addition, roasting is a promising process because not only umami taste but also flavour is important for soup stock (Tokitomo, 2017). In this study, the useful components in dried and roasted asparagus stem were therefore analysed to propose a simple method for utilizing this unused part of asparagus.

Materials And Methods

Preparation of Asparagus Samples

Asparagus was harvested at Shiraishi farm (Tokyo, Japan) in August 2021, and the stems closer to the ground 27 cm from the top were used as samples. Drying of the asparagus samples was performed with a multipurpose electric drying machine (DSJ-3-1A, Shizuoka Seiki Co. Ltd., Fukuroi, Japan) at 40℃ for 48 h. Roasting of the dried samples was performed with a home-made roasting machine at approximately 150℃ for 20 min while measuring the temperature. Asparagus samples before drying were refrigerated immediately after harvest and sent to the Laboratory of Food Technology in Nippon Veterinary and Life Science University (Musashino, Japan) where they were kept cold. The dried and roasted samples were put in a plastic bag immediately after these treatments and sent to the same laboratory at room temperature. Component analysis of these samples was performed immediately after arrival.

Component Analysis

Water Contents

The water content of asparagus samples was calculated using the weight after drying at 105℃ for 4 h. The water content of asparagus before and after drying and after roasting was 93.5, 8.9 and 2.6%, respectively.

Rutin

Rutin was measured according to the report by Kato et al. (2019). One gram of asparagus sample before drying or 0.1 g of dried or roasted sample was mixed with 20 mL of 80% ethanol solution, respectively, and homogenized with a homogenizer (ULTRA-TURRAX®, model: T10B, IKA®-Werke GmbH & Co., KG, Staufen, Germany). The supernatant obtained by centrifuging the homogenate (5℃, 10,000 × *g*, 10 min) was filtered through a 0.45 μm filter and subjected to high performance liquid chromatography (HPLC;

Chromaster , Hitachi High-Tech Science Co., Tokyo, Japan). The HPLC operating conditions were as follows: column, Inertsil™ ODS-3 (5 μm, 4.6 × 250 mm I.D., GL Science Inc., Tokyo, Japan); mobile phase, 10 mmol/L sodium dihydrogen phosphate:CH_3CN = 90:10 - 30 min - 50:50; flow rate, 1.0 mL/min; detector, UV detector (5410, 280 nm); column oven temperature, 40°C; and injection volume, 20 μL. Rutin content was obtained using a standard curve.

Ascorbic Acid

Ascorbic acid was measured according to the liquid chromatograph's technical notes (LC Technical Note LC009; GL Science Inc.). One gram of asparagus sample before drying or 0.1 g of dried or roasted sample was mixed with 15 mL of 5% metaphosphoric acid, respectively, and homogenized with a homogenizer (ULTRA-TURRAX®, model: T10B, IKA®-Werke GmbH & Co.). The supernatant obtained by centrifuging the homogenate (5°C, 10,000 × *g*, 10 min) was filtered through a 0.45 μm filter and subjected to HPLC (Chromaster , Hitachi High-Tech Science Co.). The HPLC operating conditions were as follows: column, Inertsil™ ODS-3 (5 μm, 4.6 × 250 mm I.D.; GL Science Inc.); mobile phase, 0.2% phosphoric acid containing 5 mmol/L sodium 1-hexanesulfonate:CH_3CN = 95:5; flow rate, 1.0 mL/min; detector, UV detector (5410, 210 nm); column oven temperature, 40°C; and injection volume, 20 μL. Ascorbic acid content was obtained using a standard curve.

Folic Acid

Folic acid was measured according to the reports by Aiso et al. (1998). One gram of asparagus sample before drying or 0.1 g of dried or roasted sample was mixed with 10 mL of 0.1 mol/L phosphate buffer (pH 7.0) containing 1 (w/v)% ascorbic acid and 0.1 (v/v)% 2-mercaptoethanol, respectively, homogenized with a homogenizer (ULTRA-TURRAX®, model: T10B, IKA®-Werke GmbH & Co.) and heated at 121°C for 15 min in an autoclave (KTS-2322, ALP Co. Ltd., Hamura, Japan). After cooling to room temperature, the homogenate was centrifuged (5°C, 10,000 × *g*, 10 min) and the obtained supernatant was measured with folic acid ELISA kit (Cell Biolabs, Inc., CA, USA). Folic acid content was obtained using a standard curve.

Sugars

Sugars were measured according to the report by Baert (1997). One gram of asparagus sample before drying and 0.1 g of dried or roasted sample was mixed with 9.0 and 9.9 mL of deionized water, respectively, and homogenized with a homogenizer (ULTRA-TURRAX®, model: T10B, IKA®-Werke GmbH & Co.). The homogenate was centrifuged (5°C, 10,000 × *g*, 10 min) and the obtained supernatant was filtered through a 0.45 μm filter and subjected to HPLC (Hitachi High-Tech Science Co.). The operating conditions of HPLC were as follows: column, Inertsil™ NH2 (5 μm, 4.6 mm i.d. × 250 mm, GL Science Inc.); mobile phase, DW:CH_3CN = 85:15; flow rate, 1.0 mL/min; detector, RI detector (L-7490); column oven temperature, 40°C; and injection volume, 20 μL. The contents of fructose, glucose and sucrose were obtained using standard curves.

Free Amino Acids

Free amino acids were measured according to a previous report (Kobayashi et al., 2021). The above supernatant was mixed with 3% sulfosalicylic acid at 1:1 (v/v) and left to stand under refrigeration overnight. After that, the supernatant obtained by centrifuging the mixture (5°C, 10,000 × *g*, 10 min) was filtered through a 0.45 µm filter and subjected to an automated amino acid analyzer (JLC500-500/V2, JEOL Ltd., Akishima, Japan).

Volatile Compounds

Volatile compounds were extracted according to a previous report (Ikeura et al., 2010). Thirty grams of asparagus sample before drying and 3 g of dried or roasted sample was mixed with 270 and 27 mL of deionized water, respectively, and homogenized with a homogenizer (ULTRA-TURRAX®, model: T10B, IKA®-Werke GmbH & Co.). The homogenate was centrifuged (5°C, 10,000 × *g*, 10 min) and the obtained supernatant was suction-filtered with a filter made of glass fiber. The obtained filtrate was passed through a glass column (2 cm i.d. × 40 cm length) with an infill of 10 mL of porapak Q resin (polydivinylbenzene, 50-80 mesh, Waters Co. Ltd., Milford, MA, USA). After the column was washed with 100 mL of deionized water, volatile components adsorbed to the column were eluted with 100 mL of diethyl ether. The eluate after adding 50 µL of 500 mg/L *n*-hexadecane-d_{34} solution (in dichloromethane) as an internal standard was dehydrated with anhydrous sodium sulphate overnight, concentrated to approximately 50 µL with nitrogen gas flow and analysed by gas chromatography-mass spectrometry (GC-MS).

The GC-MS analysis was performed according to a previous report (Kobayashi & Odake, 2019) using an Agilent 7890 GC system coupled to a 5975C inert MSD with triple-axis detector (Agilent Technologies, CA, USA) and a CP-WAX capillary column (0.25 mm i.d. × 30 m, thickness 0.25 µm; Agilent Technologies). The oven temperature was held at 40°C for 4 min, raised to 240°C at 8 °C/min and held for 2 min. Helium gas was used as a carrier gas at a linear velocity of 30 cm/s. The injection, interface and ion source temperatures were set at 240, 230 and 200°C, respectively. One microlitre of sample was injected using an autosampler in split ratio of 20:1. Volatile compounds were determined with the NIST mass spectrum database and the concentrations were relative values calculated with the internal standard.

Statistical Analysis

All experiments were performed in triplicate. The data presented are the means and standard deviations. The results for dried and roasted samples were not only actual values but also as values when the water content was converted into that of the sample before drying. Significant differences were evaluated by the Tukey-Kramer test ($p < 0.05$).

Results And Discussion

Rutin and Ascorbic and Folic Acids

The contents of rutin and ascorbic and folic acids in the hard lower stem of asparagus before and after drying and after roasting are shown in Table 1. The rutin content was decreased significantly by drying and roasting, although ascorbic and folic acids were concentrated. However, by converting the water content of dried and roasted asparagus into that before drying, it was understood that the contents of ascorbic and folic acids in dried asparagus were maintained but decreased by roasting. It has been reported that rutin in asparagus hardly decreases on cooking method other than boiling (Drinkwater et al., 2015), although it was decreased significantly by drying in this study. There are no reports about the behavior of rutin in dried asparagus. Rutin is considered to be degraded enzymatically in drying, because the reported optimal temperature of the rutin-degrading enzyme in buckwheat is 50℃ (Yasuda et al., 1992). In addition, ascorbic acid disappeared after roasting at 150℃ but was hardly affected by drying at 40℃ in this study. This result agreed with a report that ascorbic acid in asparagus decreased to approximately half by drying with steam at 95-97℃ (Nindo et al., 2003). Similarly, folic acid in asparagus was hardly decreased by drying but reduced by 58% by roasting. This result is the same as that reported by Tamene et al. (2019) who found that folic acid was decreased to half by the baking process in making injera from tef flour.

Sugars

The sugar content of the hard lower stem of asparagus before and after drying and after roasting is shown in Table 2. The contents of fructose and glucose were increased by drying but almost unchanged by roasting. However, by converting the water contents of dried and roasted asparagus into that before drying, it was confirmed that the fructose content was increased by drying and glucose was almost unchanged although both were decreased significantly by roasting. The sucrose content was concentrated by drying and roasting. However, it was revealed to be increased by drying but almost unchanged by roasting when the water content of dried and roasted asparagus was converted into that before drying. It has been reported that these sugars are almost unchanged by steam at 99±1℃ (Sergio et al., 2021), that fructose and glucose are destroyed by heat (Maeda et al., 1962) and that sucrose has better heat stability than fructose and glucose (Yoshimatsu & Furukawa, 1974). Therefore, the present results that fructose and glucose were decreased by roasting although sucrose was almost unchanged were supported. Yamaguchi et al. (2017) also discussed that the increase of sucrose content caused by heating tomato is induced via a plural metabolic system such as the decomposition of starch and oligosaccharides. The increase of fructose and sucrose in asparagus after drying might be caused by the same phenomena as above because inulin is a component of asparagus (Chitrakar et al., 2019).

Free Amino Acids

The free amino acid contents of the hard lower stem of asparagus before and after drying and after roasting are shown in Table 3. The free amino acid included most in the asparagus stem was Asn. Free amino acids other than Glu, Phe, His, Lys and Trp in asparagus stem were concentrated by drying. However, it was confirmed that free amino acids other than P-ser, a-ABA, Met and MEA were decreased or disappeared when the water content of dried and roasted asparagus was converted into that before

drying. Furthermore, free amino acids other than P-ser in asparagus stem decreased or disappeared after roasting. Maeda (1990) reported that most of the free amino acids in *Italian ryegrass* (*Lolium multiflorum*, LAM.) were decreased by drying at 120°C. Maeda et al. (1969) also noted that free amino acids were reduced by inducing the Maillard reaction on heating along with seasoning with reducing sugars. Therefore, it was recognized that the decrease in these free amino acids was due to the Maillard reaction, because fructose and glucose, reducing sugars, in asparagus stem were decreased by roasting in this study.

Volatile Compounds

The volatile compounds in the hard lower stem of asparagus before and after drying and after roasting are shown in Table 4, 5 and 6, respectively. Seventeen alkanes, seven alcohols, six aldehydes, six acids, three esters, a ketone and three other volatile compounds were identified by GC-MS in asparagus stem before drying. In addition, eight alkanes, four alcohols, seven aldehydes, four acids, six esters, two ketones and two other volatile compounds were detected in dried asparagus stem. Furthermore, five alkanes, an alcohol, three aldehydes, two acids, four esters, six ketones, nineteen nitrogen-containing compounds and three other volatile compounds were detected in roasted asparagus stem. 1-Octen-3-ol, reported to be a main volatile compound of asparagus (Siccama et al., 2021), was identified in asparagus before and after drying. 3-Methyl-butanal detected in dried asparagus stem is a volatile compound formed by Strecker degradation of Leu and Ile (Wu et al., 2014) and has been detected in spray-dried tomato (Jeyaprakash et al., 2020). Furans such as furfural and 5-hydroxymethylfurfural identified are volatile compounds derived from the Maillard reaction (Zeppa et al., 2002) and have been reported to be generated by heat decomposition of glucose (Duangkaew et al., 2017). In addition, 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one, a volatile compound generated by the Maillard reaction between glucose and His, has an antioxidant effect (Yu et al., 2013). 5-Hydroxymethylfurfural and 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one, having scorched and sweet flavours, respectively, are also induced by roasting, and are formed by the Maillard reaction between fructose and some types of free amino acids (Nishihori & Kawakishi, 1995). Furthermore, furaneol, formed by drying, is a volatile compound formed by the Maillard reaction (Yokoyama et al., 2020) with a caramel-like flavour and contributes most to the flavour of roasted green tea (Mizukami, 2012). Therefore, it is recognized that dried asparagus stem has many characteristic volatile compounds generated by the Maillard reaction. The phenomena can be confirmed by the decrease in free amino acids caused by drying. Heterocyclic compounds generated from roasted asparagus stem have been also reported as volatile compounds caused by roasted (Wu et al., 2014). It has been recognized that volatile compounds of tea leaf including mainly furans, pyrroles and pyrazines are increased by roasting (Zhu et al., 2021). In particular, it has been noted that pyrazines are necessary for forming desired roasting flavour (Mizukami et al., 2008). Ulrich et al. (2001) reported that pyrazines derived from the Maillard reaction by heat are present in the volatile compounds obtained from asparagus by simultaneous distillation extraction. 2-Ethyl-5-methyl-pyrazine and 2-ethyl-6-methyl-pyrazine are formed by the reaction between L-ascorbic acid and Thr or Ser (Yu & Zhang, 2010). In addition, methyl-pyrazine, 2,3-dimethyl-pyrazine, 2-hydroxy-3-methyl-2-cyclopenten-1-one, furaneol and 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one are formed by the Maillard reaction between glucose

and three types of free amino acids of Gly, Arg or Lys (Yokoyama et al., 2020). Most of the volatile compounds identified in dried and roasted asparagus stem corresponded with those obtained by the Maillard reaction of brown sugar (Liu et al., 2021). Among them, furfural, benzeneacetaldehyde, dihydro-2-methyl-3(2H)-furanone, 2-furanmethanol and butyrolactone are reported to enhance sweetness (Liu et al., 2021). Furthermore, it has been noted that syringaldehyde, detected in roasted asparagus stem, contributes to the sweet flavour in beer (Sterckx et al., 2011). Therefore, a roasting process at a higher temperature than drying forms more volatile compounds derived from the Maillard reaction with nitrogen-containing compounds and could add desired flavour to asparagus as with roasted green tea. This reaction can be recognized by a decrease in fructose, glucose, ascorbic acid and most free amino acids.

Conclusion

Decreasing the water content of food by drying can improve its preservability and concentrate the components. In addition, roasting can add pleasant volatile compounds as roasted green tea. In this study, it was revealed that ascorbic and folic acids, sugars and most free amino acids could be concentrated by drying the hard lower stem of asparagus which is usually discarded. On the other hand, non-volatile components other than sucrose and P-ser were decreased by roasting the dried asparagus, although volatile compounds peculiar to roasted asparagus could be added. Therefore, it is promising that the hard lower stem of asparagus, an unused resource, is able to be utilized as a useful food material by drying and roasting. However, a more proper method such as temperature and time for drying and roasting, and optimal temperature and time for eluting components are needed to be investigated, since some components were decreased by drying and roasting in this study.

Declaration

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Conflict of Interest The authors declare no competing interests.

Data Availability Statement The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

Authorship Contribution Fumiyuki Kobayashi: Methodology, Validation, Formal analysis, Investigation, Data Curation, Writing - Original Draft, Visualization. Ryusuke Kimura: Conceptualization, Data Curation, Supervision. Jutaro Mochizuki: Conceptualization, Resources. Naoko Tateishi: Conceptualization, Resources. Sachiko Odake: Investigation, Writing - Review & Editing.

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Tables

Table 1 The contents of rutin, ascorbic and folic acids in asparagus stem before and after drying and after roasting

	Before drying		After drying		After roasting	
Rutin (mg/100g)	11.82±0.449	c	0.28±0.049	a	1.47±0.189	b
		b	0.03±0.005	a	0.04±0.005	a
Ascorbic acid (mg/100g)	6.17±0.563	a	57.45±8.444	b	15.43±3.854	a
		b	5.48±0.806	b	0.43±0.106	a
Folic acid (μg/100g)	6.31±0.185	a	70.22±17.641	b	127.83±3.843	c
		b	6.70±1.684	b	3.52±0.106	a

The values in upper- and under-side's columns of 'After drying' and 'After roasting' are the component contents of actual measurement and when water content is converted into the water content of 'Before drying', respectively.

Different superscript lowercase letters indicate significant differences in components by the Tukey-Kramer method ($p < 0.05$).

Table 2 The contents of fructose, glucose and sucrose in asparagus stem before and after drying and after roasting

	Before drying		After drying		After roasting	
Fructose (g/100g)	0.99±0.024	a	22.39±3.682	b	1.19±0.274	a
		b	2.14±0.351	c	0.03±0.008	a
Glucose (g/100g)	0.64±0.019	a	10.59±2.815	b	0.04±0.024	a
		b	1.01±0.269	b	0.00±0.001	a
Sucrose (g/100g)	0.15±0.009	a	2.51±0.361	b	3.57±1.328	b
		a	0.24±0.035	b	0.10±0.037	a

The values in upper- and under-side's columns of 'After drying' and 'After roasting' are the component contents of actual measurement and when water content is converted into the water content of 'Before drying', respectively.

Different superscript lowercase letters indicate significant differences in components by the Tukey-Kramer method ($p < 0.05$).

Table 3 Free amino acids in asparagus stem before and after drying and after roasting

	Before drying		After drying		After roasting	
P-Ser	8.5±0.04	a	532.5±52.57	c	179.2±4.30	b
		a	50.8±5.02	b	4.9±0.12	a
Asp	85.4±0.86	a	348.0±41.49	b	101.9±33.63	a
		c	33.2±3.96	b	2.8±0.93	a
Thr	57.4±0.72	b	144.9±22.73	c	17.6±7.31	a
		c	13.84±2.17	b	0.49±0.20	a
Ser	199.1±2.11	b	427.0±58.05	c	60.0±27.17	a
		c	40.8±5.54	b	1.7±0.75	a
Asn	1696.2±17.16	b	4225.8±976.96	c	386.7±138.94	a
		c	403.4±93.27	b	10.7±3.83	a
Glu	152.9±1.33	b	140.0±23.41	b	0.0±0.00	a
		b	13.4±2.24	a	0.0±0.00	a
Gln	272.7±2.79	b	973.2±138.41	c	0.0±0.00	a
		c	92.9±13.21	b	0.0±0.00	a
Gly	72.8±0.76	b	192.8±58.67	c	41.4±7.38	a
		c	18.4±5.60	b	1.1±0.20	a
Ala	155.3±0.77	b	828.3±175.53	c	75.6±26.93	a
		c	79.1±16.76	b	2.1±0.74	a
a-ABA	14.0±0.12	a	1103.6±128.17	b	0.0±0.00	a
		b	105.4±12.24	c	0.0±0.00	a
Val	97.3±0.72	b	741.2±71.17	c	34.8±11.61	a
		c	70.8±6.80	b	1.0±0.32	a
Met	8.8±0.05	a	325.5±65.25	b	0.0±0.00	a
		a	31.1±6.23	b	0.0±0.00	a
Ile	46.7±0.13	a	201.7±9.92	b	53.0±5.62	a
		c	19.3±0.95	b	1.5±0.15	a
Leu	66.0±0.42	b	194.1±21.12	c	20.9±12.33	a
		c	18.5±2.02	b	0.6±0.34	a

Tyr	19.2±0.05	a	64.0±14.0	b	0.0±0.00	a
		b	6.1±1.34	a	0.0±0.00	a
Phe	19.7±0.11	b	0.0±0.00	a	0.0±0.00	a
		b	0.0±0.00	a	0.0±0.00	a
GABA	95.2±0.76	a	434.0±109.22	b	49.7±11.46	a
		c	41.4±10.43	b	1.4±0.32	a
MEA	27.8±0.42	a	1004.7±150.05	b	0.0±0.00	a
		b	95.9±14.32	c	0.0±0.00	a
His	19.4±0.34	b	0.0±0.00	a	0.0±0.00	a
		b	0.0±0.00	a	0.0±0.00	a
Lys	77.7±1.17	b	0.0±0.00	a	0.0±0.00	a
		b	0.0±0.00	a	0.0±0.00	a
Trp	19.0±7.96	b	0.0±0.00	a	0.0±0.00	a
		b	0.0±0.00	a	0.0±0.00	a
Arg	29.2±0.69	b	88.9±15.49	c	0.0±0.00	a
		b	1.48±0.69	a	0.0±0.00	a

The values in upper- and under-side's columns of 'After drying' and 'After roasting' are the component contents of actual measurement and when water content is converted into the water content of 'Before drying', respectively.

Different superscript lowercase letters indicate significant differences in components by the Tukey-Kramer method ($p < 0.05$).

Table 4 Volatile compounds in the hard lower stem of asparagus

KI [z]	Compounds [y]	Conc. (ng/g) [x]
Hydrocarbons		
1000	Decane	319.8±83.04
1051	5-Methyl-decane	20.4±4.47
1057	2-Methyl-decane	47.6±8.54
1067	3-Methyl-decane	26.8±5.34
1100	Undecane	347.0±52.01
1121	Ethylbenzene	18.8±7.04
1130	*p*-Xylene	10.2±3.55
1136	*m*-Xylene	13.1±5.27
1167	(1-Methylethyl)-benzene	20.3±5.52
1178	*o*-Xylene	9.8±3.93
1219	1-Ethyl-2-methyl-benzene	7.4±1.73
1279	Mesitylene	11.4±3.82
1300	Tridecane	7.4±1.64
1400	Tetradecane	8.8±3.26
1500	Pentadecane	10.6±2.70
1600	Hexadecane	15.9±0.25
1800	Octadecane	10.1±0.89
Alcohols		
975	Ethanol	48.3±22.28
1242	1-Pentanol	19.7±4.35
1348	1-Hexanol	45.5±23.58
1444	1-Octen-3-ol	9.5±1.37
1529	2,5-Dimethyl-3-hexanol	35.6±20.15
1612	2-(2-Ethoxyethoxy)-ethanol	7.1±2.38
1789	1-(2-Butoxyethoxy)-ethanol	37.0±6.90
Aldehydes		
1079	Hexanal	113.8±39.24

1214	(*E*)-2-Hexenal	22.0±8.28
1324	(*Z*)-2-Heptenal	8.6±2.39
1431	(*E*)-2-Octenal	11.1±0.68
1467	(*E*,*E*)-2,4-Heptadienal	13.6±4.80
2571	Vanillin	827.9±859.35
Acids		
1457	Acetic acid	25.9±7.53
2057	Octanoic acid	58.1±26.62
2266	*n*-Decanoic acid	88.6±20.78
2477	Dodecanoic acid	155.6±70.95
2684	Tetradecanoic acid	275.9±285.07
2889	*n*-Hexadecanoic acid	751.9±167.63
Esters		
961	Ethyl acetate	205.4±72.78
994	Methyl butanoate	31.4±18.89
1864	Butyl butanoate	27.8±3.37
Ketones		
1653	Acetophenone	22.9±1.54
Others		
1227	2-Pentyl-furan	9.3±3.01
1962	Benzothiazole	63.3±48.98
2193	2-Methoxy-4-vinylphenol	86.1±14.28

[z] KI on CP-WAX column

[y] compounds identified by GC-MS

[x] 1.0 ng/g of detectable limit

Table 5 Volatile compounds in dried asparagus stem

KI [z]	Compounds [y]	Conc. (ng/g) [x]
Hydrocarbons		
1000	Decane	1,4745±1,071.6 (140.8±102.30) [w]
1051	5-Methyl-decane	98±59.4 (9.4±5.67)
1057	2-Methyl-decane	147±30.8 (14.0±2.94)
1067	3-Methyl-decane	117±73.5 (11.2±7.01)
1100	Undecane	1,675±1,036.5 (159.9±98.95)
1121	Ethylbenzene	118±81.3 (11.3±7.77)
1130	*p*-Xylene	66±42.0 (6.3±4.01)
1136	*m*-Xylene	84±59.6 (8.1±5.69)
1167	(1-Methylethyl)-benzene	106±68.8 (10.1±6.57)
1177	*o*-Xylene	66±43.3 (6.3±4.14)
1191	*d*-Limonene	24±10.0 (2.2±0.96)
1279	Mesitylene	72±45.7 (6.9±4.36)
1304	1,2-Diethyl-benzene	26±19.4 (2.5±1.85)
1400	Tetradecane	30±10.6 (2.9±1.01)
1500	Pentadecane	79±27.7 (7.6±2.65)
1700	Heptadecane	83±4.5 (7.9±0.43)
1800	Octadecane	73±8.7 (6.9±0.83)
1900	Nonadecane	52±19.0 (5.0±1.82)
2065	Cubenene	112±57.5 (10.7±5.49)
2166	2-Isopropyl-5-methyl-9-methylene-bicyclo[4.4.0]dec-1-ene	162±99.5 (15.5±9.50)
2193	α-Copaene	178±112.6 (17.0±10.75)
2369	Cyclotetradecane	959±539.1 (61.1±64.19)
Alcohols		
975	Ethanol	677±736.3 (64.7±70.29)
1242	1-Pentanol	65±44.5 (6.2±4.25)
1444	1-Octen-3-ol	253±115.2 (24.2±10.99)

1612	2-(2-Ethoxyethoxy)-ethanol	78±36.5 (7.4±3.49)
1715	5-Methyl-2-furanmethanol	57±22.2 (5.5±2.12)
1789	1-(2-Butoxyethoxy)-ethanol	498±197.7 (47.5±18.88)
1825	1,1'-Oxybis-2-propanol	530±152.1 (50.6±14.52)
1875	2,2'-Oxybis-1-propanol	467±128.3 (44.6±12.25)
2074	Elemol	275±223.0 (26.3±21.29)
Aldehydes		
969	3-Methyl-butanal	70±68.4 (6.7±6.52)
1079	Hexanal	262±134.9 (25.0±12.87)
1324	(*E*)-2-Heptenal	150±84.6 (14.4±8.07)
1393	Nonanal	35±13.3 (3.3±1.27)
1431	(*E*)-2-Octenal	48±20.2 (4.5±1.93)
1462	Furfural	92±71.4 (8.7±6.82)
1526	Benzaldehyde	108±46.3 (10.3±4.42)
1644	Benzeneacetaldehyde	127±60.0 (12.1±5.72)
2494	5-Hydroxymethylfurfural	703±162.3 (67.1±15.50)
2566	Vanillin	1,284±335.5 (122.5±32.03)
2572	*E*-15-Heptadecenal	1,454±462.8 (138.8±44.18)
Acids		
1457	Acetic acid	421±273.6 (40.2±26.12)
1689	2-Methyl-2-propenoic acid	300±166.3 (28.7±15.88)
1941	2-Ethyl-hexanoic acid	225±170.5 (21.5±16.28)
1945	Heptanoic acid	127±86.2 (12.2±8.23)
2057	Octanoic acid	1,008±387.1 (96.2±36.96)
2477	Dodecanoic acid	911±200.0 (87.0±19.09)
2684	Tetradecanoic acid	1,724±1013.2 (164.6±96.72)
2848	(*E*)-Cinnamic acid	17,933±8,408.6 (1,712.0±802.75)
2889	*n*-Hexadecanoic acid	2,660±1,031.8 (253.9±98.51)
Esters		

949	Ethyl formate	282±224.8 (26.9±21.47)
961	Ethyl acetate	2,273±971.6 (217.0±92.76)
994	Methyl butanoate	174±166.3 (16.6±15.88)
1842	Butoxyethoxyethyl acetate	12,381±6,986.1 (1,182.0±666.95)
1864	Butyl butanoate	361±139.7 (34.5±13.33)
2122	Diisobutyl adipate	4,359±2,453.3 (416.1±234.21)
2211	Methyl hexadecanoate	112±87.1 (10.7±8.31)
2691	Dibutyl phthalate	1,199±287.2 (114.5±27.42)
2903	Butyl citrate	14,448±6,002.7 (1,379.3±573.07)
Ketones		
1408	3-Octen-2-one	92±42.1 (8.7±4.02)
2026	Furaneol	1,005±510.3 (96.0±48.72)
Nitrogen-containing compounds		
1968	2-Acetyl-1H-pyrrole	1,794±1,006.9 (171.3±96.13)
2109	Methimazole	1,431±612.7 (136.7±58.49)
2262	2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one	20,837±11,308.7 (1,989.2±1,079.61)
Others		
1227	2-Pentyl-furan	57±24.8 (5.4±2.37)
1729	2-Butoxyethyl ether	59±32.1 (5.6±3.07)
2294	2,4-Bis(1,1-dimethylethyl)-phenol	1,712±394.8 (163.5±37.69)

[z] KI on CP-WAX column

[y] compounds identified by GC-MS

[x] 10.0 ng/g of detectable limit

[w] concentration when water content was converted to value before drying

The values in parentheses are the component contents when water content is converted into the water content of ‘Before drying’.

Table 6 Volatile compounds of roasted asparagus stem

KI [z]	Compounds [y]	Conc. (ng/g) [x]
Hydrocarbons		
1000	Decane	1,078±384.7 (29.7±10.60) [w]
1051	5-Methyl-decane	86±31.6 (2.4±0.87)
1057	2-Methyl-decane	177±59.0 (4.9±1.63)
1067	3-Methyl-decane	119±29.3 (3.3±0.81)
1100	Undecane	1,780±543.1 (49.1±14.97)
1121	Ethylbenzene	124±40.4 (3.4±1.11)
1130	*p*-Xylene	78±29.7 (2.2±0.82)
1136	*m*-Xylene	65±25.2 (1.8±0.69)
1167	(1-Methylethyl)-benzene	99±33.4 (2.7±0.92)
1191	*d*-Limonene	53±21.3 (1.5±0.59)
1200	Dodecane	108±35.1 (3.0±0.97)
1219	1-Ethyl-2-methyl-benzene	73±24.4 (2.0±0.67)
1279	Mesitylene	98±38.4 (2.7±1.06)
1300	Tridecane	195±50.9 (5.4±1.40)
1400	Tetradecane	306±83.5 (8.4±2.30)
1500	Pentadecane	683±209.9 (18.8±5.79)
1600	Hexadecane	746±141.8 (20.6±3.91)
1666	Phytane	284±89.3 (7.8±2.46)
1700	Heptadecane	673±195.8 (18.6±5.40)
1766	3-Methyl-heptadecane	254±54.8 (7.0±1.51)
1800	Octadecane	718±76.3 (19.8±2.10)
1900	Nonadecane	525±117.7 (14.5±3.24)
2000	Eicosane	1,112±442.2 (30.7±12.19)
2369	Cyclotetradecane	1,148±281.1 (31.7±7.75)
Alcohols		
975	Ethanol	926±310.2 (25.5±8.55)
1652	2-Furanmethanol	3,921±1,938.1 (108.1±53.42)

Aldehydes		
1079	Hexanal	94±70.1 (2.6±1.93)
1462	Furfural	3,277±1,564.7 (90.3±43.12)
2023	1H-Pyrrole-2-carboxaldehyde	3,253±954.2 (90.0±26.30)
2190	5-Acetoxymethylfurfural	2,431±1,151.1 (67.0±31.73)
2494	5-Hydroxymethylfurfural	11,489±5,360.1 (316.7±147.74)
2566	Vanillin	4,103±2,424.7 (113.1±66.83)
2936	Syringaldehyde	946±494.8 (26.1±13.64)
Acids		
1457	Acetic acid	417±211.4 (11.5±5.83)
1683	Crotonic acid	1,058±594.0 (29.2±16.37)
2184	3-Methyl-2-furoic acid	844±389.2 (23.3±10.73)
2477	Dodecanoic acid	988±225.6 (27.2±6.22)
2684	Tetradecanoic acid	2,284±880.2 (63.0±24.26)
2848	(*E*)-Cinnamic acid	11,292±5,563.0 (311.2±153.34)
2889	*n*-Hexadecanoic acid	2,820±1,401.0 (77.7±38.62)
Esters		
961	Ethyl acetate	3,101±1,184.3 (85.5±32.64)
994	Methyl butanoate	177±77.2 (4.9±2.13)
1313	Methyl lactate	106±57.6 (2.9±1.59)
1531	Furfuryl acetate	875±410.9 (24.1±11.33)
1589	Dimethyl butanedioate	184±48.4 (5.1±1.33)
1842	Butoxyethoxyethyl acetate	6,473±1,930.3 (178.4±53.21)
2077	Methyl cinnamate	578±145.8 (15.9±4.02)
2122	Diisobutyl adipate	2,584±721.8 (71.2±18.89)
Ketones		
991	2-Pentanone	241±111.4 (6.6±3.07)
1261	Dihydro-2-methyl-3(2H)-furanone	587±296.4 (16.2±8.17)
1504	1-(2-Furanyl)-ethanone	884±420.6 (24.4±11.59)

1788	2-Hydroxy-3,4-dimethyl-2-cyclopenten-1-one	543±152.6 (15.0±4.21)
1825	2-Hydroxy-3-methyl-2-cyclopenten-1-one	1,103±446.8 (30.4±12.31)
1890	3-Ethyl-2-hydroxy-2-cyclopenten-1-one	336±102.4 (9.3±2.82)
2026	Furaneol	2,788±1,213.7 (51.2±50.28)
Nitrogen-containing compounds		
1006	Tetrahydro-thiazole	417±155.2 (11.5±4.28)
1180	Pyridine	150±65.6 (4.1±1.81)
1265	Methyl-pyrazine	293±149.90 (8.1±4.13)
1322	2,5-Dimethyl-pyrazine	361±162.4 (10.0±4.48)
1327	2,6-Dimethyl-pyrazine	376±179.7 (10.4±4.95)
1332	Ethyl-pyrazine	155±78.3 (4.3±2.16)
1347	2,3-Dimethyl-pyrazine	187±83.3 (5.1±2.30)
1383	2-Ethyl-6-methyl-pyrazine	280±142.9 (7.7±3.94)
1390	2-Ethyl-5-methyl-pyrazine	262±123.1 (7.2±3.39)
1404	2,6-Dimethyl-4-pyridinamine	563±271.2 (15.5±7.48)
1432	2,6-Diethyl-pyrazine	136±72.3 (3.7±1.99)
1444	3-Ethyl-2,5-dimethyl-pyrazine	477±194.8 (13.2±5.37)
1509	Pyrrole	109±62.3 (3.0±1.72)
1583	3-Methoxy-pyridine	220±80.0 (6.0±2.20)
1779	Methyl nicotinate	527±206.2 (14.5±5.68)
1818	1-Acetyl-1,2,3,4-tetrahydro-pyridine	219±79.8 (6.0±2.20)
1968	2-Acetyl-1H-pyrrole	7,982±3,810.5 (220.0±105.03)
2101	1-Methyl-1H-pyrrole-2-carboxaldehyde	2,274±1,445.4 (62.7±39.84)
2242	5-Hydroxymaltol	6,516±3,062.0 (179.6±84.40)
2262	2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one	7,074±3,721.4 (195.0±102.58)
2413	3-Pyridinol	6,466±2,598.7 (178.2±71.63)
Others		
1634	Butyrolactone	1,670±857.3 (46.0±23.63)
2306	2,3,5-Trimethyl-1,4-benzenediol	466±190.3 (12.8±5.24)

2509	4-Methoxythiophenol	1,168±684.1 (32.2±18.86)

[z] KI on CP-WAX column

[y] compounds identified by GC-MS

[x] 10 ng/g of detectable limit

[w] concentration when water content was converted to value before drying

The values in parentheses are the component contents when water content is converted into the water content of ‘Before drying’.