



Arab Univ.  
J. Agric. Sci.,  
Ain Shams Univ.,  
Cairo, 15(1), 71-87, 2007

## PREPARATION AND FLAVOUR EVALUATION OF HIGH QUALITY FREEZE DRIED SEASONING BLEND

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**Keywords:** Spices, Curry, Seasoning, Volatile oil, Sensory evaluation

### ABSTRACT

Combinations of different spices were used at variable concentrations in the preparation of various curry blends. The sample showed the highest sensory attributes in comparison with control curry sample was compounded with different spices, onion and garlic at variable concentrations in corresponding seasoning blend. The oleoresin of the seasoning blend that possessed the highest quality scores was prepared and subjected to freeze drying process after dispersion on suitable carrier. The flavour contribution percentages of the characteristic notes; light sweet top, medium aromatic, full boiled spicy and pungent spicy were calculated for each curry and seasoning blends. The hydro distilled (HD) oils of the selected curry and its constituents were subjected to GC-MS analysis. The results revealed the high contribution of the components possess spicy note in the raw curry sample. The components possess pungent notes showed less representation.  $\beta$ -Caryophellene was the major compounds in the HD oil of raw seasoning blends followed by  $\delta$ -carene and ar-turmenone. The qualitative and quantitative variation in the volatile compounds separated from the HD oil of the freeze dried sample may be attributed to the interaction between such components and the used carrier (10% maltodextrin in water).

### INTRODUCTION

Herbs and spices are rarely used singly and often complement each other when used in well balanced mixture or seasoning. Skill in blending seasoning is acquired by practical experience of the raw materials available, their relative flavouring

strength and qualities as well as their interrelationship with the profile of the main dietary items in the end product (Pszczola, 2001).

Seasonings are compounds containing one or more spices, or spice extractives, which when added to a food, either during its manufacture or in its preparation, before it is served, enhance the natural flavour of the food and thereby increase its acceptance by the consumer (Underriner, 1994).

The stages of building up a seasoning depend upon whether one is attempting to match an existing seasoning or formulating one for a designated use (Heath and Reineccius, 1986a). The matching of an existing seasoning involves first an agreement on the target sample and its profile. It is necessary to carry out some preliminary analysis to assess the organoleptic characteristics. Thereafter, the building up of the matching seasoning follows the usual lines of blending appropriate ingredients and repeated testing until matching product is achieved.

Spice oleoresins are being used in increasing amounts in industrial scale food preparation since they are easier to handle than spice powders and are free of microorganisms unlike essential oils; oleoresins contain nonvolatile fractions such as the compounds responsible for pungency or hot, fixed oils, antioxidants and pigments (Giese, 1994). Oleoresins are consistent in flavour and aromas sterile and stable during storage. A weakness of the oleoresins is that they are very viscous and thick, making them difficult to weight, handle and mix in processing operation.

New seasoning technology can be used to solve this problem. Oleoresins can be dispersed over a suitable carrier and then subjected to drying. Using a new drying technology as an alternative to the conventional method create seasonings

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(Received January 10, 2007)

(Accepted January 17, 2007)

homogenous in composition and unique in function and application (Sinton, 2004).

Curry powder is one of the classic standardized seasoning blends. It is a special blend of selected spices, skillfully put together for a single dish. Usually different curries are prepared for use at the same meal.

Traditionally turmeric as the main ingredient in combination with other spices such as paprika, chili, ginger, clove and cinnamon are used in various combinations of curries powder.

Turmeric (*Curcuma longa* L.) has been used in traditional medicine for treatment of several kinds of diseases (Srinivasan, 2005). Curcumin is a yellow coloured phenolic pigment obtained from powdered rhizome of turmeric. Extensive investigations on curcumin have demonstrated its varied therapeutic effects as anti-inflammatory, antibacterial, antiviral, antifungal, antitumor, antispasmodic and hepatoprotective (Aggarwal *et al* 2004). Ginger (*Zingiber officinale* Roscoe) and pepper (*Piper nigrum* L.) are the classic pungent spices used for food flavouring. Ginger is also used medicinally (Srinivasan, 2005). Concentrated extracts of the aroma, flavour, and colouring compounds (oleoresins) of these spices are important commercial products that have generally been prepared with liquid solvents such as ethanol, acetone, and dichloroethane (Purselove *et al* 1981a,b and c). A strongly antibacterial and antioxidant activity has been revealed for clove essential oils (Blank *et al* 1991).

Extensive studies have been carried out to determine the chemical composition of the volatile oils of different spices (Siatis *et al* 2005; Cheng *et al* 2006 and Kimbaris *et al* 2006); however, no data could be found concerning the volatiles analysis of their seasoning blends.

The main objective of the present study was to use new technology in preparation of high quality seasoning. To achieve this aim different curries were prepared and the best one was compounded with other aromatic plants in corresponding seasoning blends. A correlation between the flavour contribution percentages and the volatile compounds in the selected seasoning blend was investigated.

## MATERIALS AND METHODS

### Plant materials and chemicals

Dry clean spices, a curry sampled (control); dry garlic and onion were purchased from the same local market, authentic compounds and

standard n-paraffin (C<sub>8</sub>-C<sub>22</sub>) were purchased from Sigma – Aldrich Co.s (St.Louis, MN, USA), and Merck (Darmstadt, Germany). All other chemicals were of analytical grade.

### 1- Preparation of different curry and seasoning blends

- 1.1- Spices used in preparation of curry blends were separately grounded and blended at variable concentrations as shown in **Table (1)**.
- 1.2- Formulations of five seasoning blends are shown in **Table (2)**. Each blend contained the curry sample that possessed the best sensory characteristics in combinations with other the aromatic plants to achieve the desired flavour quality.

### 2- Sensory evaluation

- 2.1- *Similarity of attributes of curry sample to the control curry sample*: The panelists (10 assessors) rated the similarity of different attributes (odour, colour, taste and over all acceptability) of the prepared curries to the control curry sample on a category scale 1.0(no similarity) to 10.0 (identical) the results obtained by the panelists were averaged and the standard deviation was calculated **Table (3)** (Mayer and Grosch, 2001).
- 2.2- *Sensory quality assessment of seasoning blends*: The different sensory attributes (odour, colour, taste and appearance) of the prepared five blends under investigation **Table (4)** were estimated and scored by 10 assessors (Chemistry of flavour and Aroma Dept., NRC). The grading system was based on a total score of 10 of which 3.5 was awarded for odour, 3.5 for taste, 1.5 for colour and 1.5 for appearance **Table (4)**.
- 2.3- *Sensory profile of the prepared curry and seasoning blends*: Each of the curry and seasoning formulation was converted from a weight relationship to a flavouring profile. The weight % of each constituent was multiplied by the respective flavour index (Heath and Reineccius, 1986b) to give the flavour contribution, which in turn was converted into percentage figure.

Table 1. Composition of different curry blends.

Constituents	Curry1	Curry2	Curry3
Turmeric ( <i>Curcuma longa</i> )	28.8	44.8	44.8
Coriander ( <i>Coriandrum sativum</i> )	20.0	6.4	---
Black Pepper ( <i>Piper nigrum</i> )	10.0	16.0	16.0
Cumin ( <i>Cuminum cyminum</i> )	10.0	3.2	---
Ginger ( <i>Zingiber officinale</i> )	9.0	12.8	12.8
Fenugreek ( <i>Trigonella foenum-graecum</i> )	9.0	16.0	16.0
Clove ( <i>Syzygium aromaticum</i> )	4.5	---	3.2
Celery ( <i>Apium graveolens</i> )	4.5	---	---
Caraway ( <i>Carum carvi</i> )	2.2	---	---
Garlic Powder ( <i>Allium sativum</i> )	1.0	---	---
Mace ( <i>Myristica fragrans</i> )	1.0	---	---
Cinnamon ( <i>Cinnamomum verum</i> )	---	0.6	0.6
Cardamom ( <i>Elettaria cardamomum</i> )	---	0.2	---
Paprika ( <i>Capsicum annum</i> )	---	---	6.4
Bay Leaf ( <i>Laurus nobilis</i> )	---	---	0.2
Total	100.0	100.0	100.0

Table 2. Composition of different seasoning blends

Constituents	Seasoning 1	Seasoning 2	Seasoning 3	Seasoning 4	Seasoning 5
Curry 3	6.9	12.5	15.8	15.8	15.4
Onion powder ( <i>Allium cepa</i> )	20.7	18.8	15.8	15.8	15.4
Garlic Powder ( <i>Allium sativum</i> )	13.8	12.5	10.5	15.8	15.4
Paprika ( <i>Capsicum annum</i> )	27.6	25.0	31.6	32.8	32.1
Black Pepper ( <i>Piper nigrum</i> )	6.9	6.3	5.3	5.3	5.2
oregano ( <i>Origanum vulgare</i> )	6.9	6.3	5.3	2.5	2.5
Chili ( <i>Capsicum nightshade</i> )	3.4	3.0	2.6	0.1	---
Bay Leaf ( <i>Laurus nobilis</i> )	---	3.1	2.6	1.4	1.3
Salt	13.8	12.5	10.5	10.5	12.7
Total	100.0	100.0	100.0	100.0	100.0

### 3- Preparation of the oleoresins and freeze dried seasoning blends

The oleoresin of the seasoning blend that showed the best sensory result was extracted with ethanol under continuous stirring at room temperature for 3 hours. The suspension was filtered and the ethanol was removed by vacuum distillation. The oleoresin was dispersed on maltodextrin solution (10% in distilled water) and subjected to freeze drying.

### 4- Isolation of volatile compounds

Each spice onion, garlic, curry blend and seasoning blend were subjected to hydro distillation for 3 hours using Clevenger type apparatus. The obtained oil was dried over anhydrous sodium sulfate. The collected essential oils were immediately analyzed using GC and GC-MS.

### 5- Gas chromatographic (GC) analysis

GC analysis was performed by using Hewlett-Packard model 5890 equipped with a flame ionization detector (FID). A fused silica capillary column DB5 (60m x 0.32 mm id) was used. The oven temperature was maintained initially at 50°C for 5 min, then programmed from 50 to 250°C at a rate of 4°C/min. Helium was used as the carrier gas, at flow rate 1.1ml/min. The injector and detector temperatures were 220 and 250°C, respectively. The retention indices (Kovats index) of the separated volatile components were calculated using hydrocarbons (C<sub>8</sub>-C<sub>22</sub>, Aldrich CO.) as references.

### 6- Gas chromatographic-mass spectrometric (GC-MS) analysis

The analysis was carried out by using a coupled gas chromatography Hewlett-Packard model (5890)/mass spectrometry Hewlett-Packard-MS (5970). The ionization voltage was 70 eV, mass range m/z 39-400amu. The GC condition was carried out as mentioned above. The isolated peaks were identified by matching with data from the library of mass spectra (National Institute of Standard and Technology) and compared with those of authentic compounds and published data (Adams, 1995). The quantitative determination was carried out based on peak area integration.

## RESULTS AND DISCUSSION

### Sensory evaluation

#### 1- Curry blends

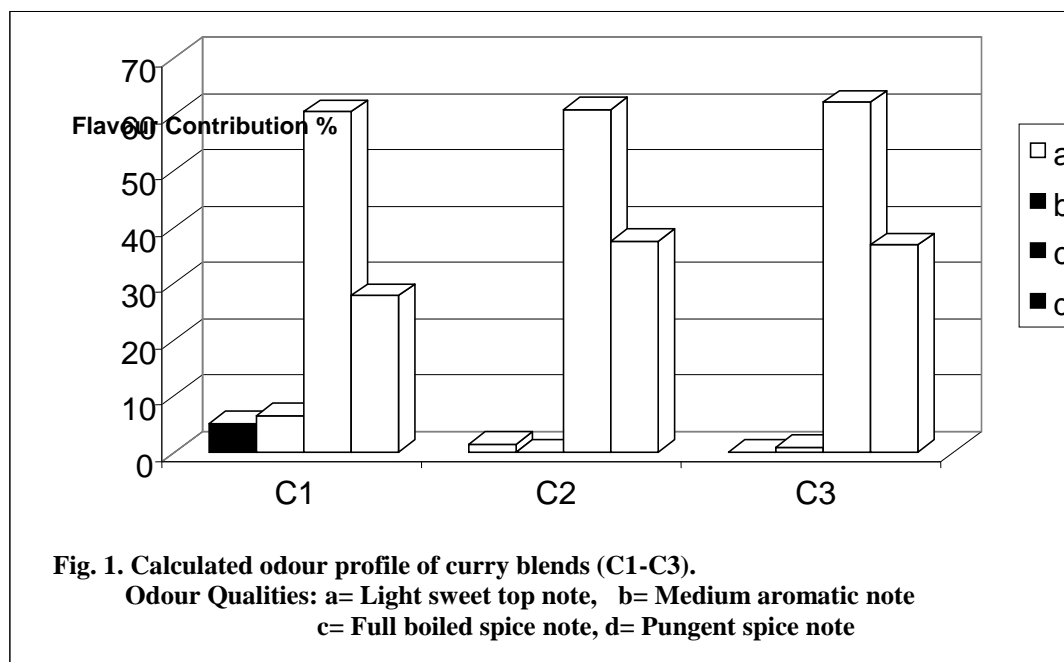
The sensory evaluation of the three prepared curry blends was carried out by rating the similarity of the different quality characteristics (odour, taste, colour, overall acceptability) in comparison with those of the control curry sample. As shown in **Table (3)** curry blend (C3) possessed the highest quality. The flavour contribution percentages of each curry blend was calculated and cited in **Fig. (1)** (Heath and Reineccius, 1986b). It is obvious that spicy note exhibited the highest contribution in all curry blends with slight increase in C3, whereas the pungent note showed the lowest value in C1. These results are mainly correlated to the change in composition of the different curry blends **Table (3)**.

Table 3. Similarity of the sensory attributes of prepared curry blends to control curry

Curry	Similarity Score <sup>a</sup>			
	Colour	Flavour	Taste	Over all acceptability
Blend	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD
C1	7.65 ± 2.03 <sup>b</sup>	6.40± 1.43	6.55 ±1.61	6.83±1.19
C2	8.60±1.17	8.00±1.49	8.05±0.89	8.22±0.72
C3	9.5±1.25	9.65±1.20	9.50±0.85	9.55±0.89

a- The similarity was scored by ten panelists on a category scale from 1.0 (not perceptible) to 10.0 (Strongly perceptible).

b- The values are the mean of 10 panelists ± S.D.



## 2- Seasoning blends

The curry blend C3 which exhibited the highest sensory quality was used in combination with other constituents **Table (4)** in preparation of five seasoning blends. A detailed sensory analysis concerning aroma, taste, colour and appearance of each seasoning blend was carried out. The total quality scores (TQS) were also calculated. It is obvious that blend S5 possessed the highest TQS. The flavour contribution percentage of each seasoning blend was calculated and cited in **Fig. (2)**. As shown in the **Fig. (2)** seasoning S1 possessed the highest pungent spice note whereas S5 showed the least one. This may be attributed to the high concentration of black pepper and chili in S1 compared with the other seasoning blends. Chili, ginger and black pepper are the classic pungent spices used for food flavour (**Catchpole et al 2003**). The variations in the seasoning blend constituents gave rise to remarkable variations in their flavour contribution percentage for each odour quality.

### Volatile components

#### 1- Hydrodistilled (HD) oil of curry blend C3 and its constituents

To prove the results of the odour sensory profile it was of interest to subject the hydro distilled oil of selected curry blend (C3) to high resolution

gas chromatography– mass spectrometric (HRGC-MS) analysis. Also, the volatile components in HD oil of the individual spices constituting this curry blend were present in **Table (3)** to illustrate their role on the overall odour profile of C2. Taking into consideration the concentration of each constituent in the blend (**Table 1**).

More than 90 volatile components were separated and identified. However only the most potent components for each spice in the curry blend were listed in **Table (5)** with their area percentages. Identification of the volatile components was carried out by comparison of their mass spectra and retention time with those of reference standards (Aldrich Co.,) and published data (**Adams, 1995**). As shown in **Table (5)** the total yield of ar-turmenone, turmenone, and turmenol comprised about 77% of the HD oil of dry turmeric under investigation. This result is in accordance with **Zwaving and Bos (1992)** who identified these compounds in high concentration about 75%, in *Curcuma domestica* Val (*C. longa*. L.)

As shown in **Table (5)** sotolone (3-hydroxy-4,5-dimethyl,2(5H) fuenone) is the dominant compound in the HD oil of fenugreek, it comprised 34.51 % of the total identified compounds. Sotolone has been identified as the key characteristic compound responsible for the fenugreek odour of dried *Lactarius helvus* (**Rapior and Fons, 2000**). **Girardon et al (1986)** Proposed sotolone as the character impact compounds of

Table 4. Sensory evaluation of five seasoning blends

Seasoning Blends	Quality	MS*	Scores**
S1	Aroma	3.5	2.52 ± 0.12
	Taste	3.5	2.66 ± 0.11
	Colour	1.5	1.01 ± 0.08
	Appearance	1.5	1.07 ± 0.08
	TQS	10.0	7.26 ± 0.1
S2	Aroma	3.5	2.55 ± 0.13
	Taste	3.5	2.38 ± 0.13
	Colour	1.5	1.13 ± 0.1
	Appearance	1.5	1.07 ± 0.11
	TQS	10.0	7.13 ± 0.12
S3	Aroma	3.5	2.80 ± 0.11
	Taste	3.5	2.63 ± 0.12
	Colour	1.5	1.31 ± 0.12
	Appearance	1.5	1.21 ± 0.1
	TQS	10.0	7.77 ± 0.11
S4	Aroma	3.5	2.73 ± 0.13
	Taste	3.5	2.59 ± 0.13
	Colour	1.5	1.23 ± 0.11
	Appearance	1.5	1.17 ± 0.10
	TQS	10.0	7.72 ± 0.12
S5	Aroma	3.5	2.83 ± 0.11
	Taste	3.5	2.85 ± 0.11
	Colour	1.5	1.22 ± 0.11
	Appearance	1.5	1.22 ± 0.10
	TQS	10.0	8.12 ± 0.11

MS\* : Maximum score

Scores\*\* : Average of 10 panelists ± S.D

TQS : Total quality score

fenugreek, it contributes to its spicy / curry note. The author have pointed out the structural similarity between sotolone and 4-hydroxy-L-isoleucine (HIL) which is the most abundant free amino acid in fenugreek seed (**Sauvaire et al 1984**). The aroma characteristic of sotolone changes from seasoning- like and curry like at high concentration to caramel-like at low concentration (**Blank and Schieberle, 1993**). The other identified compounds were previously reported in the volatiles of fenugreek oils (**Mazza et al 2002 and Ahmadiani et al 2004**).

$\beta$ -ionone was the predominate component (29, 33%) among the volatile compounds identified in paprika HD oil **Table (5)**.  $\beta$ -ionone and cyclocitral

(6.00%) are carotene derivatives. **Guadayol et al (1997)** suggested that the polyenic chain in  $\beta$ -carotene undergoes oxidation easily, yielding cyclic and noncyclic products containing often an oxygenated functional group in trimethyl cyclohexane ring or oxygenated functional group in allylic chain.

The identified compounds in HD black pepper oil  $\beta$ - Caryophyllene (27.33%) was the major component followed by  $\delta$ -Carene (24.81%), limonene (15.51%),  $\beta$ -Pinene (9.52%) and  $\alpha$ -Pinene (5.76%). These results are in quite agreement with those reported by **Pino et al 1990; Plessi et al 2002 and Singh et al 2004**. In comparison with the other spices under investigation, the clove

buds HD oil showed very low number of separated volatile components (Table 5). In accordance with the previously published data (Porta *et al* 1998 and Wenqiang *et al* 2007) eugenol was the most abundant compound (82.82%) followed by  $\beta$ -Caryophyllene (14.46%). The HD oil of cinnamon bark was found to contain mainly cinnamaldehyde (90.20%) in addition to eugenol (2.20%) and  $\beta$ -Caryophyllene (2.18%). These major constituents have been reported in some previous studies related to the chemical analysis of cinnamon spices (Yang *et al* 2005 and Cheng *et al* 2006).

It is obvious from Table (5) that 1,8- cineole is the major identified compound (35.37%) in the HD oil of bay leaves under investigation followed by  $\alpha$ -terpinyl-acetate (14.59%), eugenol (11.72%),  $\beta$ - Caryophyllene (7.86%), sabinene (2.79%). These results are in accordance with those of Kilic *et al* 2004. More early Buttery *et al* 1974 stated that 1,8- cineole is the major component for Mediterranean bay oil. The HD oil of ginger was dominated by  $\alpha$ -zingiberene,  $\alpha$ -muurolene  $\beta$ -sesquiphellandene and  $\alpha$ - bisabolene. These compounds were reported previously by Chen and Ho, 1988 in ginger oil.

As shown in Table (5) eugenol is the predominant compound in HD oil of raw curry (C3) blend (20.6%) it possesses spicy clove note (Pino *et al* 1990). ar-Turmenone which is the most potent odorants in turmeric comprised (18.18%) of the total identified components in HD oil followed by  $\beta$ -caryophellene (17.82%). The later mentioned compound was described as woody spicy (Pino *et al* 1990). On the otherhand, the components responsible for pungent note such as  $\alpha$ -muurolene,  $\alpha$ -zingiberene,  $\alpha$ -bisabolene,  $\alpha$ -farnesene and sesquiphellanderren (Shao *et al* 2003) and those responsible for worm note such as  $\delta$ -carene (Pino *et al* 1990) were identified in less concentration (Table 5). These results confirm the results of the calculated odour profile cited in Fig. (1).

## 2- Hydrodistilled oil of seasoning blend (S5) and its constituents

The oleoresin of the seasoning blend (S5) that showed the best sensory results was prepared and subjected to freeze drying after dispersion on maltodextren. The HD oil of S5 constituents of other than C3 (oregano, onion and garlic) were investigated. Table (6) shows the percentage composition of the most potent odorants in the HD oil of dry onion and garlic. All the identified compounds were previously identified in onion (Gya-

wali *et al* 2006) and garlic (Edris and Fadel, 2002 and Kimbaris *et al* 2006). The composition of the HD oil of oregano is shown in Table (7). Thymol was the predominant compounds (26.77%) followed by  $\gamma$ -terpinene (17.10%), p-cymene (9.25%), linalyl acetate (7.54%) and carvacrol (5.68%). Russo *et al* 1998 studied the relationship between chemical composition and biotypes in oregano spice (*Origanum vulgare* ssp. hritum). The authors stated that the significant quantitative differences among different essential oils were apparent only between the two isomeric phenols, carvacrol or noncrystallizable phenol, and thymole or crystallizable phenol and their biosynthetic precursors  $\gamma$ -terpinene and p-cymene. The concentration of these compounds varied greatly among the samples, in particular those of carvacrol and thymole. In all cases, however, the sum of the two phenols and their precursors constituted the bulk of each essential oil (72.08-82.86%).

The HD oils of S5 (raw, oleoresin and freeze dried) were subjected to GC-MS analysis. The most potent odorants were identified and cited in Table (7) with their relative area percentages as well as the method of identification.  $\beta$ -Caryophyllene was the major identified components (18.77%) in raw S5 followed by  $\delta$ - carene (10.48%), ar-turmenone (6.73%) and  $\beta$ - sesquiphellandrene (4.50%). These results are in quite agreement with the calculated odour quality profile of S5 Fig. (2). The high volatile components were absent in the oleoresin sample whereas most of the sesquiterpenes showed noteciable increase. ar-Turmenone was the major identified components (27.62%) followed by  $\beta$ -caryophyllene (21.89%).

The qualitative and quantitative variations in the volatile compounds identified in HD oil of the freeze dried seasoning blend (S5) may be attributed to the interaction between the maltodextren used as carrier and the individual volatile compounds in the hydro distilled oil.

From the aforementioned results it can be concluded that advanced drying technology can be used for preparation of convenient seasoning blends suit with the consumer preference. The calculated flavour contribution to the main odour seasoning attributes can be used for predication of the appropriate seasoning formulation. In future study the odour seasoning profile of freeze dried S5 will be evaluated according to the international methods to clarify the correlation between the changes in volatile compounds and odour sensory qualities during storage.









Table 6. The most potent odorants identified in dry garlic and onion volatile oils

Onion		Garlic	
Compounds	Area %	Compounds	Area %
Dimethyl disulfide	1.19	Methyl allyl sulfide	2.89
2,5- Dimethyl thiophene	2.72	Dimethyl disulfide	0.49
2,4- Dimethyl thiophene	2.40	Dimethyl trisulfide	0.53
2-Methyl propyl disulfide	2.76	Diallyl disulfide	19.20
Z-Propenyl methyl disulfide	5.62	Methyl butyl trisulfide	0.72
E-Propenyl methyl disulfide	1.51	Methyl allyl trisulfide	11.84
Dimethyl trisulfide	1.17	2-Vinyl-[4H]-1,2-dithiin	0.47
Z-Propenyl propyl disulfide	7.77	1,4-Dimethyl tetrasulfide	1.80
E-Propenyl propyl disulfide	4.85	Diallyl trisulfide	46.02
Methyl propyl trisulfide	2.50	3,5-Diethyl 1,2,4-trithiolne	0.58
Methyl propenyl trisulfide	2.32	Diallyl tetrasulfide	7.46
Dipropyl trisulfide	33.71		
3,4-Diethyl-1,2,4-trithiolane	1.87		
Dimethyl tetrasulfide	1.10		
Z-propenyl propyl trisulfide	10.86		
E-propenyl propyl trisulfide	7.64		

Compounds listed according to their elution on DB5 Column.

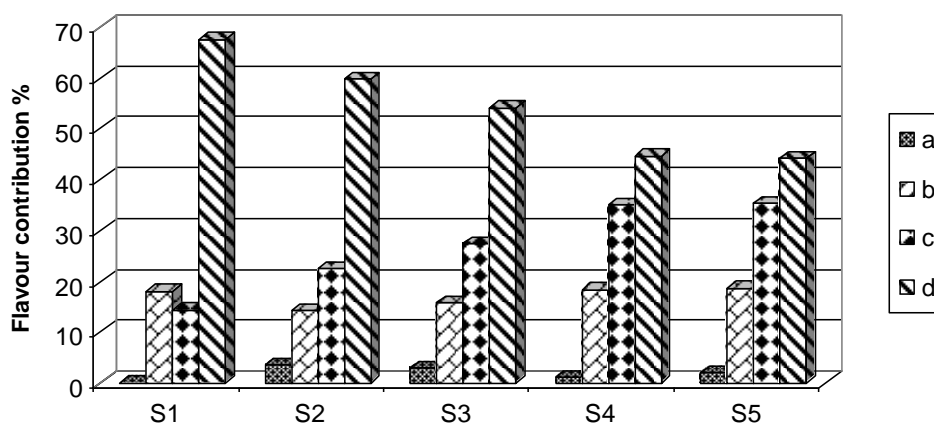


Fig. 2. Calculated odour profile of seasoning blends (S1-S5).

Odour Qualities: a= Light sweet top note, b= Medium aromatic note  
c= Full boiled spice note, d= Pungent spice note







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