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Investigating sensory characteristics and volatile components in boiled scallop aroma using chemometric techniques

Kae Morita[a], Kikue Kubota[a], Tetsuo Aishima[b,*]

[a]*Department of Nutrition and Food Science, Ochanomizu University, 2-1-1 Otsuka, Bunkyo-ku, Tokyo 112-8610, Japan*
[b]*Chemometrics & Sensometrics Laboratory Ltd, 1–197 Sengen-cho, Saitama, Saitama 330-0842, Japan*

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Abstract

A three-level full-factorial design for pH (2, 6.7 and 11.4) and parts (S: mantle muscle, M: adductor muscle and W: mantle and adductor muscle) was applied to investigating the influence of these factors on generating the boiled scallop aroma. Quantitative descriptive analysis, using nine attributes, was used to describe the aroma property of boiled scallop. M and W samples, at pH 6.7, showed higher scores for the characteristics "boiled scallop", "*kamaboko* (steamed *surimi*)" and "sweet". "Sour" and "irritate" were characteristic of M and W samples at pH 2 and pH 11.4, respectively. Response surfaces clearly showed how pH and parts influenced the generation of each attribute. Partial least squares regression (PLSR) models, calculated using influential GC–MS peaks, were highly predictable. Considering aroma properties of influential volatile components, selection by PLSR is easily interpretable in relation to each attribute. © 2001 Published by Elsevier Science Ltd.

Keywords: Scallop; Boiled flavor; Chemometrics; Experimental design; Quantitative descriptive analysis

1. Introduction

Scallop is a popular seafood, globally, because of its characteristic flavour. It is cooked in many different ways, such as boiling, frying, deep-frying and steaming. There are many recipes and people use parts of scallop, depending on dishes. In Japanese dishes, e.g., *sushi* and *sashimi* (sliced raw fish or seafood), both mantle muscle of scallop, called *kai-himo* (shell-string), and adductor muscle are cooked and eaten similarly.

Kounosu, Watanabe, Koriyama, and Shirai (1988) determined the taste-active components in a soup of cooked scallop by an omission test. Suzuki, Ichimura, and Etoh (1990) compared the amounts of 84 compounds in raw and boiled scallop and found that dimethyl sulfide and breakdown compounds of polyunsaturated fatty acids were important for raw scallop aroma but Maillard reactants were essential for cooked aroma. Boiled flavours in scallop were described by sensory evaluation, using 32 terms, including nine aroma descriptors (Phleger, Holtz, Grimes, Leighton, & Jacobsen, 1978).

Heating conditions influence sensory properties of cooked aromas. Experimental designs (Morgan, 1991) are efficient to screen out important factors from many candidates and to optimize cooking conditions. In our previous research, experimental designs were successfully applied to selecting influential factors and to investigating effects of the factors on cooked prawn aroma (Morita, Kubota, & Aishima, 2001). In flavour research, sensory evaluation is essential because of its high sensitivity and capability for quantitatively describing sensory properties of foods. Correlating two data sets supplied by sensory and instrumental analyses, using chemometric techniques, we can obtain useful information to understand the flavour problem (Aishima & Nakai, 1991).

In the current work, quantitative descriptive analysis and gas chromatography–mass spectrometry (GC–MS) analysis were applied to boiled scallop broths prepared by a three-level full-factorial design (3^2 FFD). Relationships between aroma properties in boiled scallop and volatile components were investigated by partial least squares regression (PLSR) analysis.

* Corresponding author. Tel.: +81-48-649-6114; fax: +81-84-643-5666.
E-mail address: aishima@abox9.so-net.ne.jp (T. Aishima).

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2. Materials and methods

2.1. Sample preparation

Live scallops (*Patinopecten yessoensis*) were purchased from a local market in Tokyo. After removing shells and internal organs, mantle muscle and adductor muscle were obtained. The mantle muscle (140 g) and/or adductor muscle (240 g) was cut into 5 mm squares and placed in a three-necked 1000-ml flask. After adding 608 ml of deionized distilled water, the mixture was refluxed for 30 min. A broth was obtained by filtering the boiled mixture through three layers of cotton gauze (Hakujuji Co., Tokyo, Japan). The broth was divided into 32-ml portions and preserved in 50-ml glass vials at −50 °C until sensory evaluation and preparations of aroma concentrates were performed. The levels of sample pH were adjusted with HCl or NaOH for sensory evaluation, and with citric acid or NaOH for preparation of aroma concentrates, respectively.

Table 1
3^2 Fll-factorial design matrix

Expt. No.	Sample name	Factor	
		pH ($x1$)	Part ($x2$)
1	S-2	−1	−1
2	S-6.7	0	−1
3	S-11.4	1	−1
4	W-2	−1	0
5	W-6.7	0	0
6	W-6.7	0	0
7	W-6.7	0	0
8	W-11.4	1	0
9	M-2	−1	1
10	M-6.7	0	1
11	M-11.4	1	1
	−1[a]	2	Mantle muscle
	0[a]	6.7	Mantle & adductor muscle
	1[a]	12	Adductor muscle

[a] Factor level.

Table 2
Sensory attributes selected for quantitive descriptive analysis from preliminary sessions

Attribute	Characteristic
Sweet	Sweet aroma in cooked food
Boiled corn	Sweet aroma in boiled corn
Sea breeze	Smell of sea breeze
Sour	Tomato sauce-like
Irritate	Ammonia-like
Dried fish	Aroma of dried sea food
Boiled egg	Smell of boiled egg
Kamaboko	Aroma of *Kamaboko* (steamed *surimi*)
Boiled scallop	Typical aroma in boiled scallop

2.2. Experimental design

A two-factor three-level full-factorial design (3^2 FFD; Morgan, 1991) for parts and pH was applied to scallop samples (Table 1).

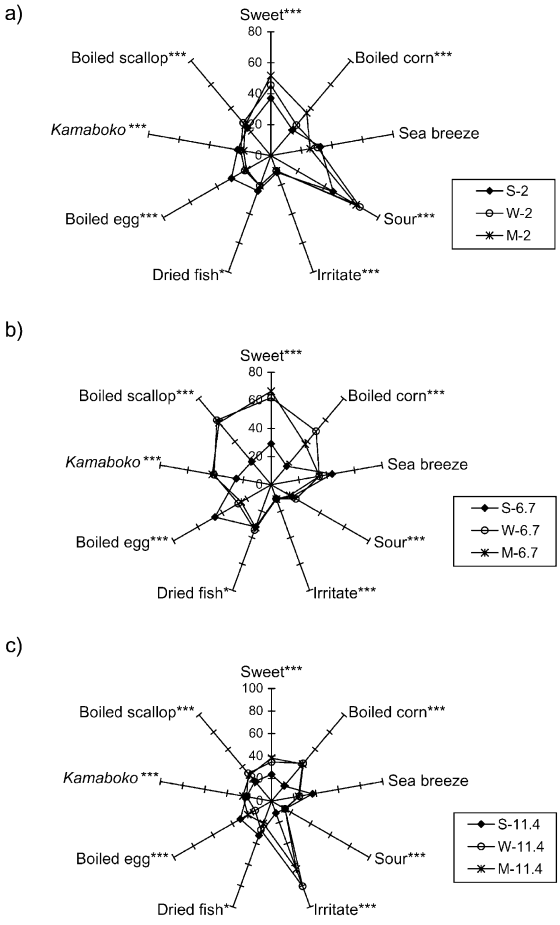

Fig. 1. Comparison of sensory profiles for boiled scallop samples at three different pH levels, a: low, b: medium and c: high. Statistical test was performed based on all 9 samples. $*P<0.05$. $***P<0.001$.

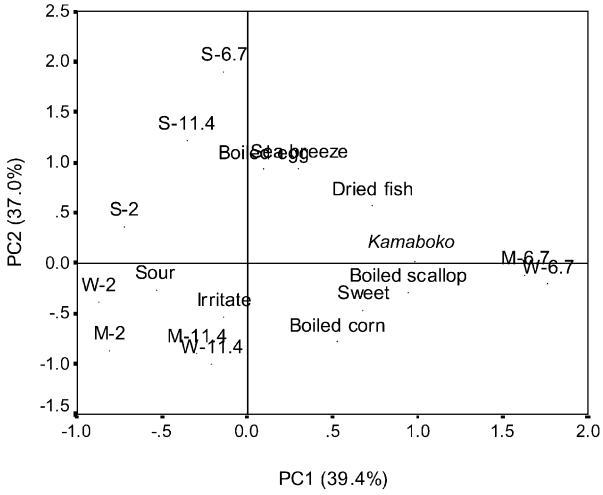

Fig. 2. Biplot of PC scores and factor loadings in PCA applied to boiled scallop samples.

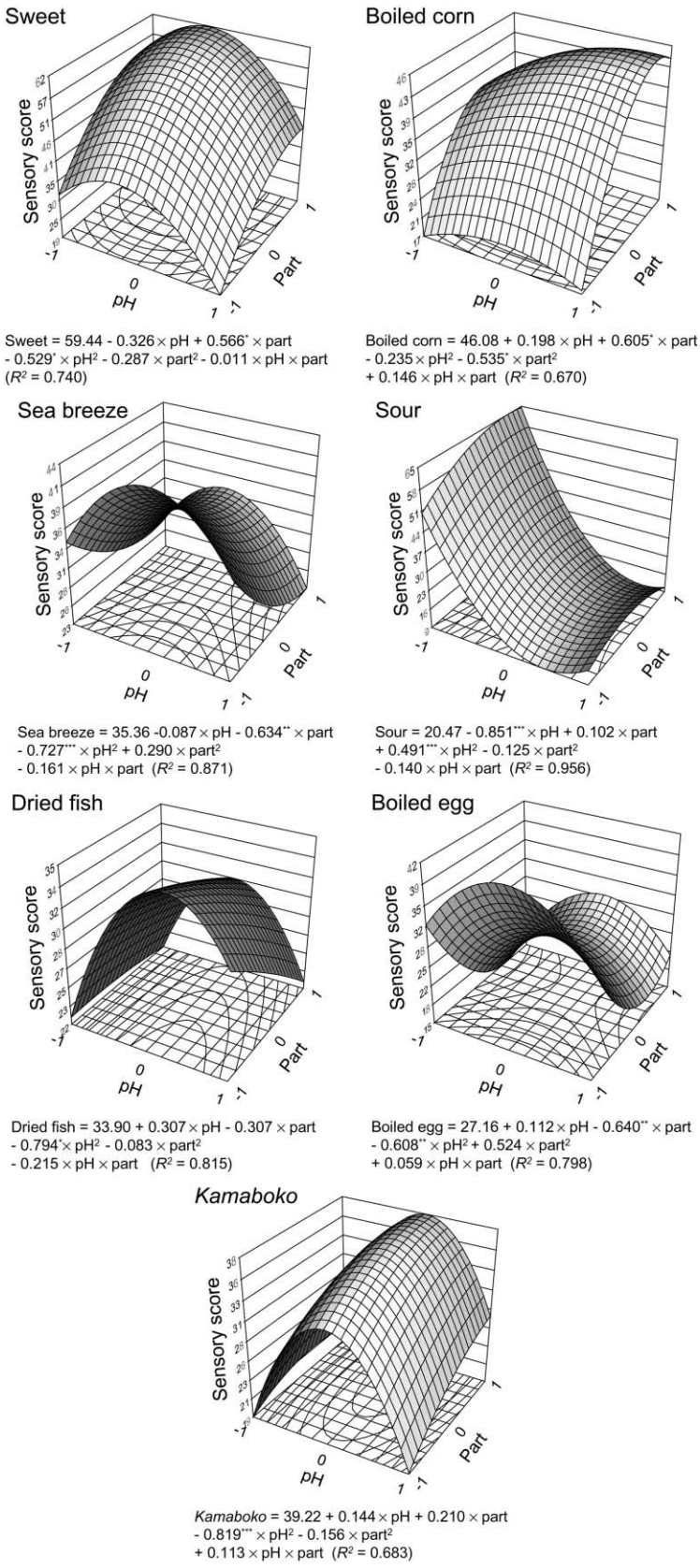

Fig. 3. Response surfaces and MLR models for 7 attributes. On the axis for pH, −1, 0 and 1 stand for pH 2, 6.7 and 11.4, respectively, and for parts,—1, 0 and 1 indicate mantle muscle, mantle and adductor muscle, and adductor muscle, respectively. In equations, *, ** and *** indicate $P<0.05$, $P<0.01$and $P<0.001$, respectively.

2.3. Sensory evaluation

For evaluating scallop samples, a quantitative descriptive analysis was performed for nine sensory attributes selected from preliminary sessions (Table 2), using a 15-cm line scale, by a well-trained panel, consisting of 14 females aged 21–29. Each sample, prepared from 32 ml of sample solution at 40 °C, was placed in a 260 ml disposable plastic cup, covered with a plastic Petri dish and served to each panellist. For sensory evaluation, a panellist opened the Petri dish and sniffed. Every sample was coded with a 3-digit random number. Ten samples were randomly separated into three sets, each composed of 3, 4 and 4 samples, and evaluated in the following three sessions. In every session, the randomly arranged three or four samples were served to each panellist, who was asked to evaluate them according to the order. All samples were tested once.

2.4. Preparation of aroma concentrates from 3^2 FFD samples

Each sample, prepared from 65 g of scallop, was extracted by simultaneous distillation-extraction (SDE) with 80 ml of dichloromethane for 2 h using a modified Likens–Nickerson apparatus (Nickerson & Likens, 1966). One ml of dichrolomethane, containing 2.4 ppm methyl decanoate, was added to the extract as the internal standard (IS) for GC–MS analysis. After concentrating the dichloromethane extract in a 100-ml round-bottom flask at 45 °C, the extract was transferred into a 0.8-ml test tube for further concentration it to about 10 mg with dried N_2 gas.

2.5. GC–MS analysis

Aroma concentrate (1.5 μl) was injected into an HP5790 gas chromatograph (Hewlett-Packard, Palo Alto, CA), coupled with an HP5972 mass spectrometer. GC–MS was operated with an ionization voltage at 70 eV and ion source temp of 150 °C. The GC conditions were as follows; column: DB-WAX (60 m×0.25 mm i.d., film thickness: 0.25 μm: J & W Scientific Inc., Folsom, CA), carrier gas: He with a flow rate of 1.0 ml/min, oven temp: 60 °C, held for 4 min and then elevated to 180 °C at 2 °C/min, injection temp.: 200 °C, and detector temp.: 220 °C. Peak components were identified by matching their mass spectra with those in the Wiley Library of MS spectra (Hewlett-Packard) and their retention indices. Each peak area was converted into a ratio to that of IS (internal standard) for the statistical analysis.

2.6. Statistical analysis

For experimental designs, TrialRun (SPSS Inc., Chicago, IL) was used. Analysis of variance (ANOVA), principal component analysis (PCA) and multiple linear

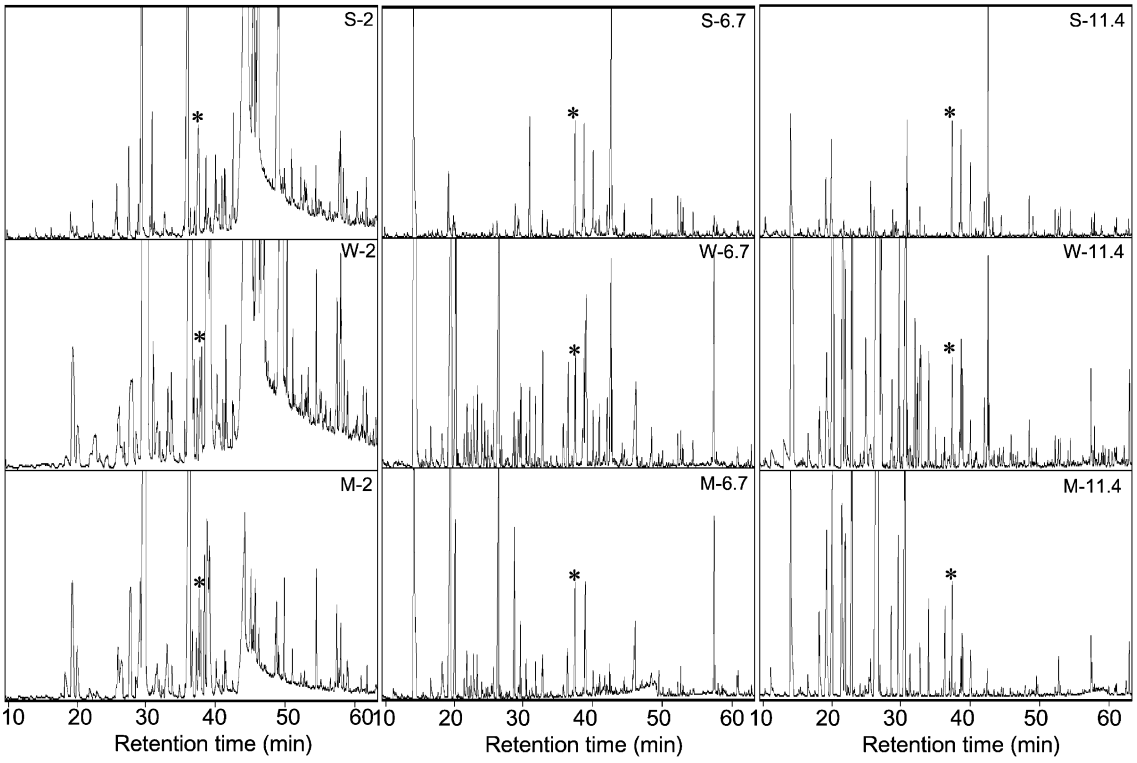

Fig. 4. Total ion chromatograms of aroma concentrates of scallop samples.

regression analysis (MLR) were performed by SPSS ver. 9.0. The coefficients of determination (R^2) in MLR were adjusted, based on their degrees of freedom. PLSR analysis was performed by Unscrambler ver.7.01 (CAMO ASA, Trondheim, Norway).

3. Results and discussion

3.1. Sensory profiles in 3^2FFD samples

Sensory profiles for boiled scallop samples, prepared according to a 3^2 FFD for pH (2, 6.7 and 11.4) and parts (S: mantle muscle, W: mantle and adductor muscle and M: adductor muscle) are shown in Fig. 1. Regarding W and M samples, high scores were observed for "sweet", "*kamaboko* (steamed *surimi*)" and "boiled scallop" at pH 6.7, "sour" at pH 2 and "irritate" at pH 11.4. However, the score for "boiled corn" in W-6.7 was higher than that in M-6.7. Scores for "sea breeze" and "boiled egg" in S samples were higher than those in other samples.

Two principal components (PC), accumulating 76.4% (PC1: 39.4% and PC2: 37.0%) of the total variance were extracted by PCA. A biplot, shown in Fig. 2, indicates closeness of W and M samples, regardless of pH levels. "*Kamaboko*", "sweet" and "boiled scallop" formed a group near to W-6.7 and M-6.7; on the other hand, both "boiled egg" and "sea breeze" were located close to S-6.7 and S-11.4. "Sour" was close to all samples at pH 2 but "irritate" and all W-11.4 and M-11.4 samples grouped together.

3.2. Effects of pH and part on generating each attribute

Main, second-order and interactive effects of each attribute were calculated, based on responses from 3^2 FFD experiments by MLR analysis. As Fig. 3 shows, MLR models for 7 attributes, except for "irritate" and "boiled scallop", were statistically significant ($R^2 \geqslant 0.670$). In MLR models for six attributes, except for "boiled corn", all second-order effects of pH were highly significant. Among them, response surfaces for "dried fish" and "*kamaboko*" were ridge-like because second-order effects of pH on these two attributes were negatively large but all other effects were rather small. The response surface for "sweet" was parabolic with the maximum point at the intermediate level of pH and adductor muscle. This parabolic response surface was derived from the significant large main effect of part and negatively large second-order effects of pH and part. Response surfaces for "sea breeze" and "boiled egg" were saddle-like because of the large negative second-order effects of pH and main effects of parts but the positive second-order effects of part. As indicated by MLR models, "boiled corn" and "sour" were mainly influenced by part and pH, respectively. To investigate the generation of each attribute in the boiled scallop aroma, applying the experimental design combined with the response surface methodology succeeded in revealing the influences of each factor quantitatively and visually.

3.3. Relationships between attributes and volatile components in boiled scallop

GC profiles in 3^2 FFD samples differed from each other, and samples at pH 6.7 are comparatively shown in Fig. 4. One hundred and twelve peaks commonly present in more than two profiles were used as predictors in PLSR analysis. First, PLSR analyses were performed using all 112 peaks, and then 20 influential peaks were selected according to the PLS loading

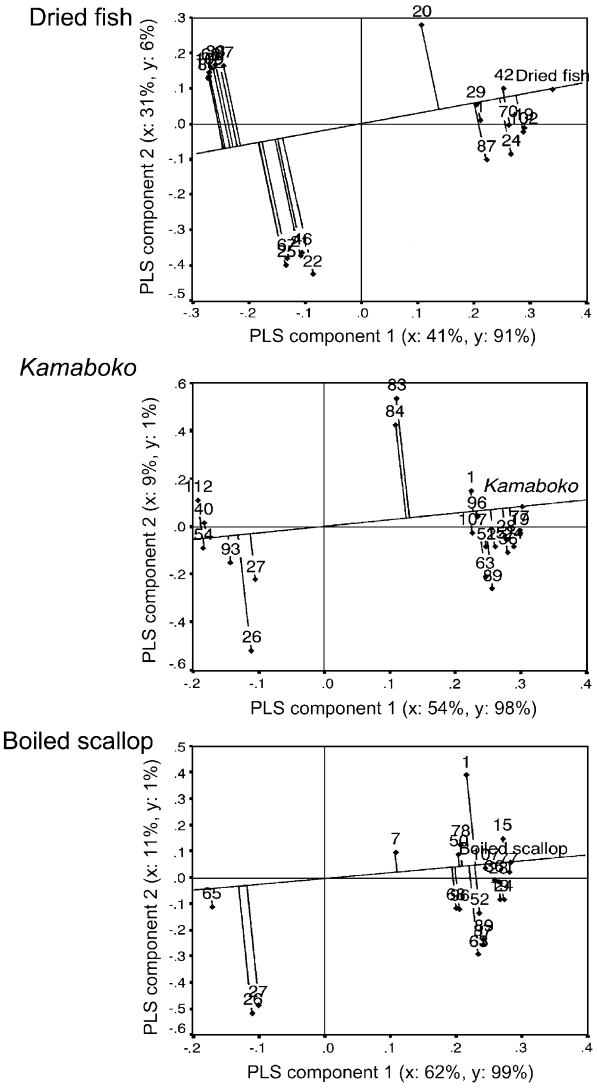

Fig. 5. Plots of volatile components heavily loading on PLS components 1 and 2 for "dried fish", *kamaboko* and boiled scallop.

weights on peaks. All PLSR models calculated, based on the influential peaks for each attribute, were highly predictable ($R^2 \geqslant 0.944$) as shown in Fig. 5.

As shown in Fig. 6b, c, scores of W-6.7 and M-6.7 were higher in both "*kamaboko*" and "boiled scallop", which were close to each other in the biplot shown in Fig. 2. Fourteen common peaks were selected as contributing to "*kamaboko*" and "boiled scallop" (Fig. 5; Table 3). Dimethyl disulfide (leek, onion and vegetable; Girard & Durance, 2000), 3-hydroxy-2-butanone and 2-acetylpyrrole (cookie-like; Fors, 1983), showing positive contribution to both "*kamaboko*" and "boiled scallop", increased after boiling (Suzuki et al., 1990). Furthermore, several Maillard reactants such as 2-acetylfuran (pleasant and sweet; Fors, 1983) and 2-thiophenecarbaldehyde (coconut-like; Fors, 1983) were selected as important to "*kamaboko*" and/or "boiled scallop. On the other hand, (*E, Z*)-2,6-nonadienal, green and vegetable notes (Tanchotikul & Hsieh, 1989), contributed negatively to "boiled scallop".

Scores for "dried fish" were higher in S-6.7, M-6.7, W-6.7 and S-11.4 (Fig. 6a). Several peaks, including dimethyl disulfide and alcohols, showed similar contributions to "dried fish" as to "*kamaboko*" and "boiled scallop" (Fig. 5; Table 3). However, negative correlations of thermally generated compounds with "nutty"

Table 3
Peaks and compounds selected by PLSR as closely relating to "dried fish", "*kamaboko*" and "boiled scallop".

No.	Compund	Positively relating to	Negatively relating to
1	Dimethl disulfide	Dried[a], *Kamaboko*, Scallop[b]	
7	3-Penten-2-ol	Scallop	
15	3-Hydroxy-2-butanone	*Kamaboko*, Scallop	
19	3-Methyl-2-pentanol	Dried, *Kamaboko*, Scallop	
20	(*E*)-2-Pentenol	Dried	
21	2,5-Dimethylpyrazine		Dried
22	*N*,*N*-Dimethylformamide		Dried
24	3-Pentanol	Dried, *Kamaboko*, Scallop	
25	2,3-Dimethylpyrazine		Dried
26	*N*,*N*-Dimethyleyanamide		*Kamaboko*, Scallop
27	2-Hydroxy-3-propanone		*Kamaboko*, Scallop
28	Hexanol	*Kamaboko*, Scallop	
29	2-Methyl-2-cyclopeten-1-one	Dried	
36	*N*,*N*-Dimethylacetamide	*Kamaboko*, Scallop	
38	5-Methyl-1(3*H*)-furanone		Dried
40	Acetic acid		*Kamaboko*
42	1-Octen-3-ol	Dried	
46	Tetramethylpyrazine		Dried
50	2-Acetylfuran	Scallop	
52	unknown	*Kamaboko*, Scallop	
54	2,3,5-Trimethyl-6-ethylpyrazine		*Kamaboko*
63	Dimethylsulfoxide	*Kamaboko*, Scallop	
65	(*E*,*Z*)-2,6-Noadienal		Scallop
66	2-Methylbenzofuran		Dried
67	4,5-Dimethylfurfural		Dried
68	unknown	Scallop	
70	2-Octen-1-ol	Dried	
77	3-Acetyl-1-methylpyrrole	*Kamaboko*, Scallop	
78	Thiomethyl disulfide	Scallop	
80	Nonanol		Dried
83	2-Thiophenecarbaldehyde	*Kamaboko*	
84	1-(5-Methyl-2-pyrazinyl-)-1-ethonone	*Kamaboko*	
87	unknown	Dried, Scallop	
89	3-Methyl-2-thiophenecarboxyaldehyde	*Kamaboko*, Scallop	
93	2(5*H*)-Furanone		*Kamaboko*
96	Dimethylthioformamide	*Kamaboko*, Scallop	
97	Hexanoic acid		Dried
102	2-Penethylethanol	Dried	
107	2-Acetylpyrrole	*Kamaboko*, Scallop	
109	2-Methyoxy-6-vinylphenol		Dried
112	Octanoic acid		Dried, *Kamaboko*

[a] Dried fish.
[b] Boiled scallop.

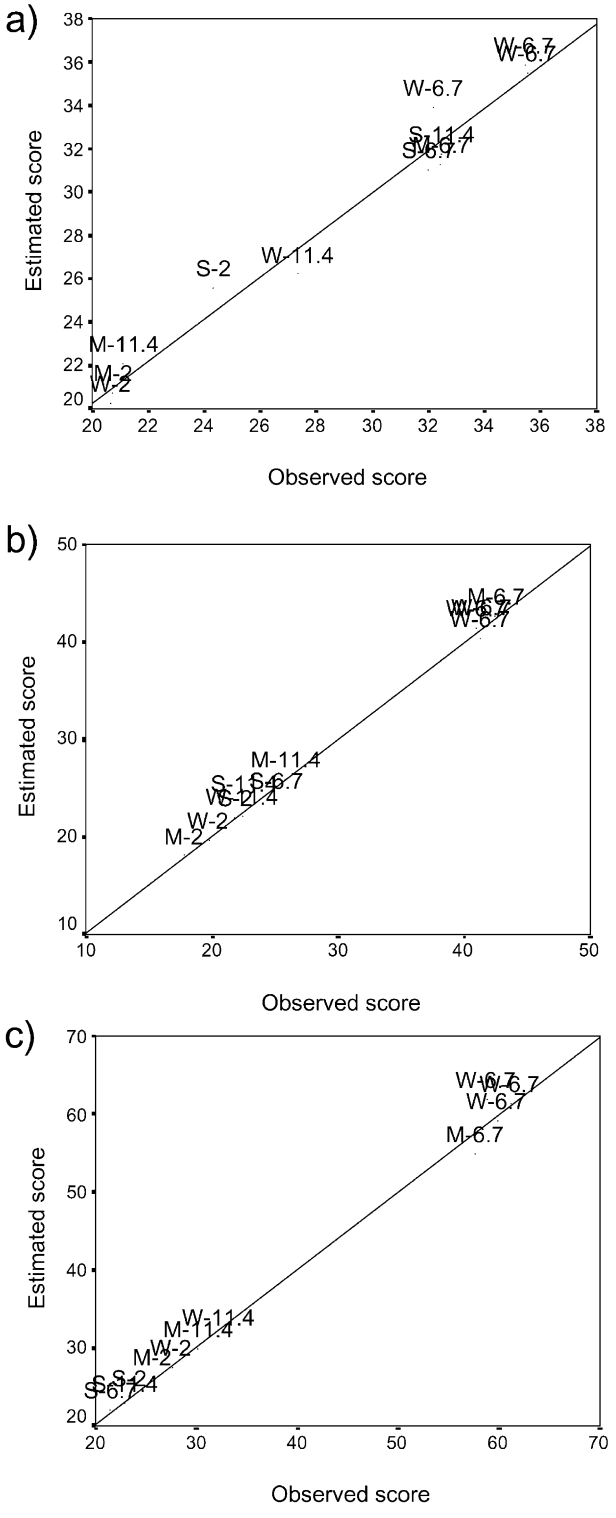

Fig. 6. Relationships between observed and estimated scores for a: dried fish, b: *kamaboko* and c: boiled scallop, based on PLSR models.

and/or "roasted" aroma to "dried fish" suggested that this attribute had existed in raw scallop.

4. Conclusions

Aroma characteristics in boiled scallop were quantitatively described using nine attributes. "Sweet", "*kamaboko*" and "boiled scallop", and "sea breeze" well-characterized the aromas in boiled adductor muscle and mantle muscle, respectively. A 3^2 FFD for parts and pH, combined with the response surface methodology, indicated how these factors influenced the generation of the boiled scallop aroma. PLSR models, using selected influential peaks for each attribute, were highly predictive. Considering attributes and aromas in individual peak components, selection of volatile components by PLSR, for each attribute, seems meaningful.

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