
Comparative Value of a Sorting Procedure and Quantitative Descriptive Analysis to Investigate the Influence of Processing Parameters: Case Study of Hydrolysate Production From Salmon By-Products

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Abstract:

Many papers have recently discussed the value of a free sorting method as a rapid and simple alternative to quantitative descriptive analysis, considered the reference tool for food sensorial characterization. The aim of the present paper is to evaluate whether this method of free sorting can also be used to investigate the influence of processing parameters. An experimental design was applied to production conditions of enzymatic hydrolysates from salmon by-products. The effect of four processing parameters (time and temperature of hydrolysis, sugar and antioxidant addition) on the odor of the hydrolysates was studied using a sorting task with 45 untrained panelists and a quantitative descriptive analysis carried out with 11 trained panelists. This study on 21 enzymatic hydrolysates confirms the similarity of the two sensory maps and shows the value of free sorting in the sensory characteristic description step, especially to avoid missing some descriptors. It also highlights in this example that a holistic approach as sorting can reveal more easily than profiling the significant effects of process parameters on sensory characteristics and the relationships between sensory dimensions and instrumental measurements of volatile compounds.

Practical Applications

Having a rapid and simple method to evaluate the sensory properties of food products and to investigate the effect of processing parameters could be useful during product development steps. Results from the present case study showed that compared with quantitative and descriptive analysis, the holistic approach of sorting task could clearly relate sensory characteristics to processing parameters and seemed efficient for industrial applications and product development.

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1. Introduction

Recent studies have highlighted the value of the sorting technique in the sensory field to assess the similarities of a set of products easily and quickly. This procedure, relatively old and well known in psychology and medical fields (Wild et al. 1965, Morton 1969, Rosch 1973), has seen a renewal of interest for its potential applications in sensory evaluation. The task consists of asking assessors, trained or not, to group products according to their sensory similarities or differences. This technique has been applied to a large range of products and sensory characteristics, from food products such as cheese (Lawless et al. 1995), jellies (Tang and Heymann 2002), yogurts (Saint-Eve et al. 2004), beers (Lelièvre et al. 2008)], and virgin olive oils (Santosa et al. 2010) to non-food products such as fabrics or plastic parts for automobiles (Giboreau et al. 2001, Faye et al. 2004) and also recently to link perceptual experience to textural terms (Varela et al. 2013). The procedure is generally completed by a verbal description of each identified group which leads to a perceptual map based on the dimensions of multidimensional scaling (MDS), the typical analysis performed with sorting data, although alternative approaches have been proposed recently (Abdi et al. 2007, Cadoret et al. 2009). Free sorting has been completed by a hierarchical structure called taxonomic free sorting (Courcoux et al. 2011), which allows a distance to be assigned between groups enabling better discrimination. The value of the sorting technique is also illustrated by a proposed method to test the stability of a sorting map (Blancher et al. 2012). Previous studies suggested that this technique used by naïve consumers could give the same sensory maps as those produced by trained sensory panels (Faye et al. 2006, Cartier et al. 2006, Veramendi et al., 2013). It could therefore offer an alternative to quantitative descriptive analysis, the method widely used in sensory analysis by research and industry to obtain a detailed description of a product in terms of descriptors and intensities (Stone et al 1974) in order to optimise processes or find relationships with consumer preferences. Sorting by untrained panellists appears to be a time-saving alternative to quantitative descriptive analysis for rapid sensory mapping, as it does not require a long stage of panellist selection and training while still producing consistent product maps (Varela and Ares 2012).

The aim of this study is to analyse the efficiency of a sorting task to investigate the influence of processing parameters on the production of enzymatic hydrolysates from salmon by-products. The effect of four processing parameters (time and temperature of hydrolysis, sugar and antioxidant addition) on the odour of the hydrolysates was studied and results from the sorting task were compared with those from a quantitative descriptive analysis. Volatile compounds generated during the processing were also analysed in each hydrolysate and the relationship with both sensory maps was studied to give a complementary point of view on data for the comparison.

2. Materials and methods

2.1. Samples

2.1.1. Preparation

Samples used for this study came from a research project on the production of enzymatic hydrolysates from salmon by-products. This project aimed to investigate the effect of hydrolysis conditions on several quality parameters (Kouakou et al. 2013). Sensory characteristics have been identified as a key factor for further applications in the food industry and were therefore studied more specifically as well as the associated volatile compounds. Samples were prepared from salmon by-products (heads and frames) obtained from a local smoked salmon company (Piriac, France). After this raw material was ground, an enzyme

102 Protamex (Novozymes, Bagsvaerd, Denmark) was added to the mince (0.15% w/w) under
103 different processing conditions. After hydrolysis, all biological reactions were stopped by
104 heating at 95°C for 30 min and samples were centrifuged at 9800 g at 15°C for 30 min. For
105 each processing condition, the aqueous phases collected were separated and sampled into two
106 100-ml plastic flasks for further sensory evaluation, quantitative descriptive analysis and free
107 sorting. All samples were stored at -80°C until evaluation.

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2.1.2. Experimental design

110 An experimental design, based on the Doehlert design completed by some specific
111 experiments, was performed to study the effect of four different independent variables;
112 temperature (from 30°C to 60°C) and time of hydrolysis (from 30 min to 470 min), addition
113 of sugar (xylose, industrial grade, provided by Danisco, Surrey, United Kingdom) or natural
114 antioxidant (a commercial mixture of natural tocopherols and rosemary from the company Jan
115 Dekker International, Wormerveer, the Netherlands) to the mince. This required the
116 preparation of forty samples (Kouakou et al. 2013). A supplementary sample was introduced
117 as a control in sessions of descriptive and quantitative analysis. This control sample was
118 prepared without enzyme at a temperature of 60°C, with a process time of 360 min, without
119 sugar and antioxidant. Thus, 41 samples were finally obtained.

120 It seemed difficult to carry out a sorting task on such a large number so a selection of a
121 sample sub-set, representative of the entire set of hydrolysates, was compiled. A sorting task
122 on odours of around twenty samples was considered achievable. The selection was based on
123 the *D*-optimality criterion, which consists in selecting the 20 products from the 40 candidates
124 such that $\det((X^T X)^{-1})$ is minimal. Linear, quadratic and first order interaction terms for the
125 four processing variables were used to compute the *X* matrix of experiments. This criterion is
126 equivalent to minimising the generalised variance of the estimator (Atkinson and Donev
127 1992). In order to achieve this selection according to the *D*-optimality criterion, an iterative
128 procedure based on the Fedorov exchange algorithm (Fedorov 1972) was used. The control
129 sample was also added so 21 samples were finally presented for the sorting task (Table 1).

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2.2. Sensory evaluation conditions

132 The two sensory methods were performed in the same conditions, in individual partitioned
133 booths controlled for temperature (20°C) and light (day light, T=6500°K). For the descriptive
134 and quantitative method, data were collected with a computerised system (Fizz, Biosystèmes,
135 Dijon, France). The day before the sensory test, samples were thawed overnight at 2°C. Then,
136 the possible difference in colour between samples was masked with a black colouring agent,
137 neutral in smell. About 8 ml of each hydrolysate was poured into a polystyrene crystal flask,
138 assigned a 3 digit-number and kept at 18°C before the test.

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2.3. Sensory methods

141 Two experiments were performed (1) a free sorting with forty-five untrained panellists, (2) a
142 quantitative descriptive analysis with eleven trained panellists.

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2.3.1. Sorting technique

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Untrained panellists

146 The panel was recruited from staff and students of the two research organisations involved in
147 the project, 19 from Ifremer and 26 from Oniris; these 45 people were panellists untrained on
148 hydrolysate products and had no previous experience of this product. However, they could be
149 qualified as initiated in sensory evaluation because they sometimes take part in food tests.
150 Taking into account the results of previous studies, which showed that the stability of a
151 sorting map can be influenced by the complexity of the task and which recommended at least

152 25 people (Faye et al. 2006) or to start a work with 30 evaluations (Cartier et al. 2006), the
153 number of 45 panellists seemed a reasonable figure for this study.

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155 *Free sorting procedure*

156 Panellists received the 21 samples simultaneously in a random order. Previous studies
157 concluded that it was possible to sort until 20 beers (Cholet et al., 2011) and that olfactory
158 fatigue did not affect the results from sorting task with 16 perfumes (Veramendi et al., 2013),
159 it is the reason why we suggested to present 21 samples in order to have a good
160 representativeness of the product space while avoiding a too heavy task for panellists. They
161 were asked to sort the products into groups based on odour similarities. They had to make at
162 least two groups and no more than twenty. Panellists had all the time necessary to perform the
163 task and were required to smell fresh air when necessary. Once performed the sorting,
164 panellists could verify the proximity of samples within each group after a resting time. Then,
165 a description of the odour characteristics was required for each group. Panellists could use
166 their own vocabulary and suggest one or several words to describe each group. No glossary
167 was presented.

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169 2.3.2. Quantitative descriptive analysis

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170 *Trained panellists*

171 Samples were sniffed by a trained panel of 11 people, 8 females and 3 males, selected from
172 members of the internal panel of Ifremer. These panellists have regular training in odour
173 perception and characterisation of seafood products and were selected according to their
174 sensory performances. During the specific training step, individual performances were
175 checked at a multidimensional level with a comparison of samples discrimination results with
176 the discrimination of the group and for each attribute, the consistency in product ranking was
177 evaluated in comparison with the result of the group. Finally 11 out of the 16 panellists who
178 started the study were selected for the final evaluation. The descriptor selection and panellist
179 training are described by Kouakou *et al.* (2013)..

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181

181 *Sensory procedure*

182 A quantitative descriptive analysis (ISO 13299 2003) was performed to quantify nine selected
183 descriptors of odour: global intensity, marine, fat fish, dried fish, roasted, rancid, potato,
184 sulphur and brine fish. In order to evaluate the 41 samples, panellists were required to attend
185 seven sessions of profiling, two per week, in a comparative way. An experimental design was
186 constructed in order to balance for contrast effects. Four parameters were balanced; hydrolysis
187 temperature and time, presence of sugar and antioxidant. Six hydrolysates including the
188 control sample were presented in each session. The intensity of each sensory descriptor was
189 directly scored on an unstructured scale anchored by the terms low intensity (0) and high
190 intensity (10) using data acquisition software. With the aim of comparing the two sensory
191 methods, only the data from the 21 samples used for free sorting were analysed.

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193 2.3.3. Comparison of configurations

194 A global index of proximity of the two factorial configurations, the RV coefficient (Robert
195 and Escoufier 1976), was computed on three dimensions.

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197 **2.4. Volatile compound analysis**

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198 2.4.1. Extraction of the volatile compounds by Headspace Solid Phase

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199 MicroExtraction (HS-SPME)

200 5 ml of hydrolysate was placed in a 20-mL glass vial closed with a screw top equipped with a
201 Teflon septum. The sample was equilibrated for 60 min at 40°C. The extraction of the volatile

202 compounds was performed with a Carboxen/PDMS fibre (85 μm , 1 cm, Carboxen/PDMS
203 StableFlex, Supelco, Sigma-Aldrich Chimie, Lyon, France) for 15 min at 40°C. Analyses
204 were performed on the 41 hydrolysates, the initial experimental design as well as the control
205 sample.

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207 2.4.2 Gas chromatography / Mass spectrometry / FID

208 The apparatus used was a gas chromatograph (Agilent 7890A, Wilmington, DE, USA)
209 equipped with a flame ionisation detector (FID) and coupled to a mass spectrometer
210 (electronic impact source, Agilent 5975CNetwork, Wilmington, DE, USA). The inlet
211 temperature was 260°C, the FID detector temperature 250°C and the MS detector temperature
212 280°C. The carrier gas was helium and the pressure was 62.4 kPa. The splitless mode was
213 used for the injection, and the desorption time was 3 min. The capillary column was a DB-
214 WAX (30 m, 0.25 mm, 0.5 μm , J&W Scientific, Folsom, CA). The program used was 40°C
215 for 10 min, ramp to 120°C at 4°C/min, ramp to 240°C at 20°C/min then equilibrium at 240°C
216 for 5 min. Effluent from the end of the GC was split 1/1 between the MS and the FID. Peaks
217 were integrated with MSD Chemstation software (Agilent Technologies). Mass spectra were
218 recorded in electron impact mode (70 eV) between 33 and 300 m/z mass range at a scan rate
219 of 2.7 scan.s⁻¹.

220 The volatile compounds were identified according to 3 criteria: comparison of their Kovats
221 retention index with the literature, comparison of their mass spectra with those of the Wiley 6
222 library and injection of the corresponding standards. The semi-quantified results were
223 obtained from the FID chromatogram and expressed in peak area. The repeatability of the
224 method was 9%.

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226 2.5. Data Analysis

227 2.5.1. Sensory data analysis

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228 Sorting data

229 From the sorting task, a measure of dissimilarity between two stimuli was considered as the
230 number of subjects who separated these two items into different groups (Faye et al. 2004).
231 This dissimilarity matrix was submitted to a Multidimensional Scaling technique (MDS)
232 which provided a factorial configuration of the stimuli and exhibited the main sensory
233 dimensions of the set of products. A non-metric procedure was used, considering that
234 dissimilarities have only an ordinal interpretation (Borg and Groenen 2005). In order to assess
235 the stability of the resulting configuration and to evaluate whether products were perceived as
236 significantly different from a sensory point of view, confidence ellipses were built using a
237 bootstrapping approach according to the procedure described by Courcoux *et al.* (2011).
238 Cadoret and Husson (2013) showed that ellipses built by a method based on total bootstrap
239 can be interpreted as confidence areas. The volumes of ellipses inform on sensory distances
240 between samples but also on variability between panellist evaluations.

241 To analyse the sensory characteristics of each product, the terms used for one group were
242 associated with each product of the group. A general matrix (products x terms) with the
243 number of occurrences of each term for describing each product was generated from the entire
244 panel. Then, the terms with the same meaning were grouped together by the panel leader and
245 those that appeared less than three times for one product were removed from the final matrix.
246 Correlations between each term and each MDS dimension were computed in order to provide
247 an interpretation of the underlying dimensions.

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249 Profiling data

250 Sensory data were submitted to two-way analysis of variance (ANOVA) with products and
251 panellists as independent factors in order to identify significant product effects and descriptors

252 involved in this discrimination. Significant differences between means were determined using
253 Duncan's multiple range test ($p < 0.05$). A principal component analysis (PCA) without
254 standardisation was performed on the means of the sensory scores of each product and each
255 descriptor using XLSTAT for Windows version 2012 (Addinsoft, Paris, France). As for the
256 sorting data, a procedure of total bootstrapping was applied to set up the confidence ellipses.

257 258 2.5.2. Relation between volatile compounds and sensory data

259 After an identification step, the volatile compounds were grouped according to their probable
260 origin (lipid oxidation, Maillard reactions, fermentation, marine environment, other origin) or
261 according to their main chemical structure (hydrocarbon, alcohols, aldehydes, ketones, acids,
262 furans, sulphurs, pyridine and thiazol). For the two types of classification, the sum of the peak
263 areas for each volatile compound gathered in each group (origin or structure) was calculated.
264 Global matrices (products x volatile compound groups) were obtained. Correlations were
265 calculated between each volatile compound group and each dimension of the product
266 configuration for the sorting task and profiling procedures

267 268 2.5.3. Effect of process parameters on sensory properties of hydrolysates

269 The effect of process parameters on sensory dimensions was assessed by means of ordinary
270 least squares regression (OLSR). For the two sensory procedures (sorting task and
271 quantitative descriptive analysis), the three sensory dimensions were regressed on the factors
272 of the experimental design, using a quadratic model. The resulting regression coefficients are
273 interpreted as the quantification of the main effects of factors, interactions between these
274 factors, and quadratic effects. As the process parameters are not expressed in the same units,
275 the regression coefficients are not directly comparable so the t-values were computed to check
276 the significance of these coefficients. An absolute value of t higher than 2.5 indicates a
277 significant effect of the parameter at the 0.05 level of significance. These t-values were
278 represented in order to compare the contributions of the effects of factors on the different
279 sensory dimensions.

280 281 3. Results

282 3.1. Comparison of the sensory map from free sorting and descriptive and 283 quantitative analysis

284 Free sorting results

285 The first plane of the MDS applied on free sorting data represented 81.2% of the total inertia.
286 The first axis showed a clear discrimination between two main groups of products (Fig. 1a).
287 One of these groups was constituted of twelve products, with the highest coordinates along
288 the first axis. The dimension 2 allowed the separation of three different groups; one group
289 with samples 39, 38 and 20, another including samples 35, 37 and 40 and a specific group
290 with the sample number 3. Fifteen attributes were used to describe these groups. The
291 correlation of these attributes with the MDS dimensions enabled an interpretation of the main
292 sample characteristics (Fig. 1b). On the first dimension, criteria of roasted, brine, cooked and
293 fat fish were associated with the group of twelve products and, at the opposite, seaweed, lean
294 fish, crustacean and sulphur for a second group of products. The attribute "cheese odour" had
295 the highest correlation with dimension 2 and explained some differences between samples.
296 On the first dimension, sample 3, for example, showed a roasted characteristic like the other
297 samples of this group but the position on the second dimension (negative coordinate)
298 indicated at the same time a distinct characteristic, namely a cheese odour in this sample. This
299 specific odour was probably the reason for the separation from the other "roasted" samples.
300 Samples with a negative coordinate on the first dimension presented a larger distribution of
301 location along the second dimension. From the top to the bottom, samples were associated

302 with marine and crustacean characteristics and, to a lesser extent, chemical (samples 38 and
303 39) and to sulphur, seaweed and spoilage odour for samples located on the bottom left side of
304 the figure (35, 37 and 40). The third dimension (not presented) added some information in
305 terms of sample discrimination and allowed a separation of samples 20 and 41 from the
306 others. The terms most frequently associated with these two products were the same as those
307 observed in dimension 2; marine, crustacean and also chemical. However, a detailed study of
308 the description data (Table 2) showed that, compared to samples 38 and 39 on one hand and
309 to samples 35, 37 and 40 on the other hand, the frequencies of attributes quotations used for
310 these two products were different. For example, a marine odour was more frequently
311 described in samples 20 and 41 than in sample 40, while the sulphur odour in contrast was
312 less often noticed compared to samples 35, 37 and 40 but more than in sample 38. The fat fish
313 descriptor was also used with different frequencies, more often than for samples 35 and 37
314 and less than for sample 38.

315 Descriptive and quantitative analysis results

316 Data from profiling were submitted to a two-way analysis of variance with panellists and
317 products as independent factors. This treatment identified significant differences between
318 products and descriptors with the most discriminative power. Comparison of F values for the
319 product effect showed that roasted odour had the highest followed, in decreasing order, by
320 sulphur, global intensity, fat fish, dried fish, potato, marine, rancid and odour of fish in brine.
321 The first plane of the unstandardised principal component analysis (PCA) accounted for
322 85.3% of the total information (Fig. 2a). The first axis (75.1% of total inertia) was mainly
323 created by the criteria roasted, dried fish, global intensity, marine and fat fish while sulphur,
324 fat fish and marine odours were mostly involved in the creation of the second component
325 (10.25% of the inertia) (Fig. 2b). A clear discrimination between samples appeared on this
326 plane. Along axis 1, two groups of products were separated, according to the global intensity
327 of the odour as well as the intensity of roasted and dried notes. As in the sorting procedure, a
328 group of nine samples was found on one hand and a group of twelve samples on the other
329 hand. The second axis presented a more fuzzy separation even though extreme samples were
330 identified. Sample 35 was clearly characterised by a sulphur odour whereas samples 20 and
331 41 had a strong intensity of fat fish odour. The location of the other samples on this axis was
332 mainly modulated by the intensities of these two descriptors. Dimension 3 of the PCA (not
333 shown), created mainly by the descriptors brine note and potato odour added further
334 information to discriminate samples 39, 38 and 14. A specific potato odour, associated with
335 low dried and brine notes allowed these samples to be separated from the others.

336 Comparison of the two sensory maps

337 On the whole, the two procedures led to the same overall conclusion regarding product
338 discrimination. The three-dimensional configurations obtained after running MDS on the
339 sorting data and PCA on the profiling data led to an RV-coefficient equal to 0.81, i.e. good
340 agreement between configurations. Whatever the sensory test used, the first dimension
341 allowed the discrimination of the same two groups of products. One gathered fish
342 hydrolysates with a dominant roasted odour while the second group, which showed a larger
343 within-group variability, was constituted of products with odour characteristics other than the
344 roasted note. The descriptors used in the sorting procedure to qualify this group were
345 chemical, marine, crustacean, seaweed and sulphur odours while for the profiling test, fat fish,
346 marine, rancid, sulphur and potato odours were used. In the two procedures, confidence
347 ellipses for samples 20 and 41 were separated from those of samples 35, 37 and 40. However,
348 only the profiling test highlighted the difference between sample 35 and products 37 and 40
349 and showed the specific potato odour of sample 39. Moreover, with the quantitative and
350 descriptive analysis, it is possible to observe a gradient of intensity among samples with
351 roasted note. Indeed, Fig. 2a shows different locations along the first axis for “roasted”

352 products, in relation to the intensity attributed to this descriptor by the panellists. This
353 information was completely masked in the sorting task; panellists sorted products according
354 to the main odour characteristic, probably without taking into account its intensity.
355 Nevertheless, although profiling could appear a more discriminative procedure, it is important
356 to keep in mind that the step of descriptor selection is essential in the procedure. The example
357 of sample 3 illustrates this point. This product was closer to the “roasted” group in the two
358 configurations (sorting and profiling) but appeared significantly different from this group. If
359 in sorting procedure the term “cheese” odour was the descriptor the most often used to qualify
360 this product, in profiling, no descriptor allowed to identify this characteristic. Indeed, no
361 similar product was present in the range of samples used during the attribute selection step
362 and therefore this special characteristic of cheese odour was not identified. In this case, the
363 sorting task gave more detailed information.

364

365 **3.2. Sensory map and relationship with volatile compounds**

366 The study of the relationships between sensory characteristics and volatile compounds was
367 undertaken in order to find possible explanations for the description of hydrolysate odours..

368 In the case of the sorting procedure, the first dimension of the product configuration showed a
369 high correlation with the group of volatile compounds identified as Maillard reaction products
370 and of marine origin (Table 3) which probably explains the roasted note associated with this
371 dimension. The main group of compounds correlated with dimension 2 was the fermentation
372 origin group since the oxidation group was weakly linked to this dimension. This correlation
373 was mainly due to sample 3, previously described by a cheese odour. The compounds
374 identified in this sample were mainly alcohols (not shown). Regarding compounds from lipid
375 oxidation, the best correlation was observed with the third dimension. For the two groups of
376 hydrolysates identified on the first dimension, with and without sugar, a large distribution of
377 the samples along dimension 3 can be noticed. A general trend of increasing lipid oxidation
378 compounds from the bottom to the top of the dimension 3 was observed (not shown) and
379 seems to explain this correlation.

380 With the profiling procedure, the first dimension of the product configuration showed the
381 same correlation with Maillard and marine origin compounds as the sorting task as well as a
382 correlation between compounds from fermentation and dimension 2. However, compounds
383 from lipid oxidation did not show a clear correlation with any dimension. In this case, the
384 discrimination between samples within the same group, i.e. with and without addition of sugar
385 as described in the next subsection, was less clear and the relationships with compounds from
386 lipid oxidation were weaker. This could be an effect of the profiling procedure; some
387 descriptors, such as the roasted note, would be easier to detect and perhaps contribute to
388 masking or to giving less importance to some attributes such as fat fish or rancid notes. In the
389 profiling test, the distribution of samples according to roasted intensity, along dimension 1,
390 was clear for hydrolysates with sugar, and along dimension 2, according to fat fish intensity
391 and sulphur odour, for hydrolysates without sugar. However, no common dimension enabled
392 a simultaneous distribution of the two groups of products, as in sorting.

393 The study of the chemical structure of compounds did not add any more relevant information.
394 Aldehydes, ketones, furans, acids and sulphurs were associated with compounds from the
395 Maillard reaction and alcohols with a fermentation origin (data not shown) but further
396 differences between the two procedures were not highlighted.

397

398 **3.3 Sensory map and relationship with processing parameters**

399 The effects of processing conditions on sensory characteristics were investigated using a
400 quadratic model of regression on each of the dimensions obtained in MDS or PCA
401 configurations. This model, previously used for the selection of products, involved the main

402 effects, interaction effects and quadratic effects of these factors. This analysis also identified
403 the significant effect by the t-values computed during the regression. These t standardised
404 values were represented on each of the configurations to highlight the main parameters
405 involved in perceived sensory properties. For the sorting task, the first dimension illustrated
406 the high effect of sugar as well as the quadratic effect of sugar (Fig. 3a). This variable
407 explained the separation of samples into two groups along this axis, one with a strong roasted
408 odour and the other without. The quadratic effect of sugar illustrated a non-linear relation in
409 the perception of roasted odour. In fact, as previously described by Kouakou (2013), there
410 was a significant increase in the roasted score between samples treated without sugar and
411 those with 10 g.Kg⁻¹ of sugar but this increase became smaller between 10 and 20 g.Kg⁻¹. The
412 quadratic effect of hydrolysis time was mainly linked to dimensions 1 and 2. A regression
413 analysis of the roasted score only showed a clear optimum between 200 and 300 min of
414 hydrolysis time for samples to which sugar had been added. The time-sugar interaction
415 observed on dimension 2 could illustrate the effect of a long hydrolysis time on the odour of
416 samples with sugar. Sample 3 is an example of these processing conditions where the longest
417 hydrolysis time was applied on a sample with 10 g.Kg⁻¹ of sugar. Dimension 3 of Fig. 3b
418 shows three main effects: hydrolysis time, hydrolysis temperature and the time-temperature
419 interaction. High temperature and long hydrolysis time, without added sugar, led to samples
420 with fat fish, chemical and marine odours (samples 20 and 41) while the interaction indicated
421 that, for the same hydrolysis temperature, the choice of hydrolysis time modulated odours: for
422 example, sample 40 was produced at 60°C for 30 min and presented sulphur, seaweed and
423 spoilage characteristics whereas sample 41 was also hydrolysed at 60°C but for 360 min and
424 its odour was qualified as marine and fat fish.

425 With the sensory profiling data, the regression of each dimension of the PCA on the design
426 factors also showed highly significant linear and quadratic effects of sugar as well as a
427 quadratic effect of hydrolysis time (Fig. 4a). The quadratic effect of time and the time-
428 temperature interaction were also identified as significant effects ($p < 0.10$) (Fig. 4b).
429 Compared to the results obtained from the sorting data, the temperature and the time-sugar
430 interaction were not identified as factors with a significant explanatory power.

431 For the two sensory procedures, sorting task and profiling method, the linear and quadratic
432 effects of sugar were identified as the most significant on hydrolysate sensory properties. It
433 seems that the addition of sugar to hydrolysates led to the same global and dominant
434 characteristic of roasted odour whatever the temperature or the time of hydrolysis. We can
435 suppose that this major effect of sugar limits the analysis of other factor effects. However, it is
436 possible for the two data sets to highlight the quadratic effect of hydrolysis time and the time-
437 temperature interaction.

438 The sorting task data analysis pointed out a significant effect ($p < 0.1$) of hydrolysis time-
439 sugar interaction that was not revealed by profiling data. The assessment of sample 3,
440 prepared with sugar and corresponding to the highest level of time factor, probably explains
441 this result. Moreover, this sample was better discriminated in the sorting task than in the
442 profile test and therefore could contribute to identifying this significant effect. A temperature
443 effect was also observed using the sorting data whereas it was not shown with the profiling
444 data. We can suggest that the scoring of selected descriptors led panellists to recognise and
445 score easily some criteria, such as the roasted note, and perhaps give less importance to the
446 other attributes whereas panellists in the sorting task had more freedom in their assessment
447 and could take into account the global perception of the sample without any influence of one
448 particular characteristic. Dimension 3 (not presented) where the temperature effect was
449 identified, confirmed for the two groups of samples, with or without a roasted note, a
450 distribution of the products according to the temperature, high at the top and low at the
451 bottom. It can also be noticed that sample 41 without enzyme stayed close to samples with

452 enzymes, from a sensory point of view; this could suggest that an increase in the number of
453 small peptides in the soluble phase under enzyme action would not significantly affect the
454 global sensory characteristics of hydrolysates.

455

456 **4. Discussion and Conclusion**

457

458 From a methodological point of view, the results of this study confirm previous conclusions
459 about the value of alternative procedures, such as free sorting, for sensory characterisation.
460 Faye et al. (2004) showed that free sorting applied to the visual description of plastic pieces
461 led to consistent data, a similarity of the sample configuration and the same conclusion about
462 perception-process parameter relationships, compared to descriptive mapping. More recent
463 works and reviews (Cartier et al. 2006, Varela and Ares 2012, Dehlhom et al. 2012, Nestrud
464 and Lawless 2010) have emphasised the benefits of using alternative procedures, such as
465 projective mapping, flash profiling and free sorting, which are generally faster and less time-
466 consuming than the classic descriptive and quantitative analysis while still producing
467 meaningful product configurations, even with untrained panellists. Our study on odour
468 characterisation illustrates this value and also shows that when descriptors are missing in the
469 final list of attributes submitted to panellists, not having been identified during the selection
470 step in profiling, free sorting can be more informative. In fact, the sorting task takes into
471 account the whole space of products to find attributes and can therefore be an interesting step
472 to describe sensory characteristics, even though some authors (Varela and Ares 2012, Chollet
473 et al. 2011) have emphasized that the description by untrained panellists could be less detailed
474 and sometimes more difficult to interpret. Varela and Ares (2012) also highlighted that
475 descriptive analysis was more appropriate to identify small differences between products or to
476 detect differences in intensity and therefore could not be replaced by these new techniques.
477 The results of our case study agree with this fact. For example, panellists who scored the
478 roasted note in salmon hydrolysates during profiling were able to discriminate the intensity of
479 this odour while panellists who did free sorting were not.

480 From a practical point of view, the assessment of sensory characteristics by a free sorting task
481 could be considered a relevant technique to obtain information for companies with no time to
482 train a sensory panel and sufficient to identify the main sensory properties in product
483 development. If more accurate information is needed, such as the intensity of the roasted
484 odour in our study, it could easily be provided by a task like the ranking technique.

485 Regarding the use of a free sorting task for product development and the choice of process
486 parameters, the results of this study on hydrolysates from salmon by-products show that this
487 procedure can highlight process effects more easily than conventional profiling. The
488 temperature effect observed using sorting data but not profiling data, as well as the better
489 correlation between oxidation compounds and dimension 3 in the free sorting configuration,
490 could suggest that a natural task of sorting without any fixed sensory vocabulary can offer
491 more freedom in the panellist assessment and can take into account a global perception which
492 sometimes allows more discrimination. The holistic approach of the sorting procedure shows
493 the power of this tool based on a natural task of difference perception, which does not require
494 any conscious evaluation or analytical quantification as in profiling.

495 However, although free sorting or a more sophisticated approach such as taxonomic free
496 sorting can appear attractive in the industrial context of product development, the method
497 seems less accurate for evaluating the intensity of sensory characteristics. Moreover, the
498 number and characteristics of products to be assessed in the same session could be a
499 restrictive factor. Some adaptations to these tests must be developed to allow a more general
500 use. Nevertheless, this procedure is an attractive test which could be easily used in industrial
501 applications, not only to obtain sensory characteristics but also to optimise a process. In the

502 case of hydrolysate production from salmon by-products, the addition of sugar to modify and
503 mask fish odours has successfully been identified using this method.

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506 **Acknowledgements**

507 This paper is dedicated to Christelle Kouakou, the main contributor to the data collection
508 during her PhD period who suddenly deceased before the publication. The authors want to
509 thank all the members of the two panels from Oniris and Ifremer, for their involvement in
510 sensory sessions.

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589 Table captions

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591 **Table 1** Identification of the 21 experimental points and corresponding process variables,
592 selected from the initial experimental design

593

594 **Table 2** Terms used by panellists to describe samples of salmon hydrolysates associated to a
595 group in the sorting task

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597 **Table 3** Pearson Correlations between volatile compounds classified according to their origin
598 group and each dimension of the product configuration for the sorting task and profiling
599 procedures

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Table 1

Process variables Sample code	Time (min)	Temperature (°C)	Antioxidant (tocopherol and rosemary) (ppm)	Sugar (xylose) (g.kg ⁻¹)
2	30	45	125	10
3	470	45	125	10
6	140	60	125	10
8	140	40	0	10
9	360	50	250	10
11	250	35	250	10
13	250	55	0	10
14	140	40	93.75	0
15	360	50	156.25	20
16	140	50	156.25	20
17	250	35	156.25	20
18	250	45	31.25	20
20	250	55	93.75	0
27	360	50	156.25	20
33	250	45	0	0
35	30	30	0	0
37	30	60	250	0
38	360	30	250	0
39	360	30	0	0
40	30	60	0	0
41	360	60	0	0

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Table 2

Products	roasted	fat fish	marine	lean fish	spoilage	sulphur	brine fish	seaweed	crustacean	paté	tuna	cooked fish	cheese	chemical	rancid
2	13	11	4	1	2	0	5	4	3	3	4	2	0	0	0
3	8	5	3	1	8	4	3	0	1	5	1	3	10	0	1
6	12	12	4	1	2	0	5	2	3	4	4	1	0	1	0
8	13	10	7	1	2	0	5	4	1	4	4	2	0	0	0
9	13	11	6	1	4	0	7	2	2	2	3	2	0	0	0
11	15	7	8	2	3	2	2	3	1	5	2	2	1	1	0
13	11	13	5	5	4	0	4	1	2	2	2	4	0	1	0
14	3	9	7	7	3	10	1	4	2	0	0	1	2	2	1
15	18	9	6	2	5	1	5	0	3	2	1	2	1	0	0
16	14	7	4	2	4	1	3	3	2	5	5	3	0	1	1
17	13	11	4	3	0	1	8	0	2	6	4	3	1	1	0
18	13	7	6	2	2	1	5	2	2	3	3	2	1	0	0
20	3	6	8	7	4	4	2	3	4	0	1	3	1	4	0
27	14	9	8	2	4	2	7	0	3	2	1	4	0	1	1
33	3	12	8	9	4	2	1	5	4	0	1	2	1	1	2
35	0	2	6	8	5	16	0	7	6	0	1	0	3	0	0
37	1	4	6	7	4	11	3	7	4	0	1	1	3	2	0
38	4	11	7	6	6	1	4	4	4	0	1	2	0	3	2
39	4	2	11	4	4	4	2	5	7	1	1	0	1	3	1
40	1	8	3	4	8	13	0	8	4	0	0	1	1	0	0
41	0	6	9	5	2	5	3	7	7	0	1	1	0	3	4
total	176	172	130	80	80	78	75	71	67	44	41	41	26	24	13

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Table 3

Origin	Sorting procedure			Profiling procedure		
	Dim1	Dim2	Dim3	Dim1	Dim2	Dim3
Lipid oxidation	0.15 (0.50)	-0.36 (0.11)	0.53 (0.01)	0.19 (0.41)	0.10 (0.66)	0.18 (0.44)
Maillard reactions	0.69 (0.0006)	-0.14 (0.53)	-0.29 (0.20)	0.77 (0.0000)	-0.009 (0.97)	0.19 (0.40)
Fermentation	0.34 (0.13)	-0.51 (0.02)	0.19 (0.40)	0.30 (0.18)	-0.30 (0.19)	0.05 (0.83)
Marine	0.62 (0.003)	0.19 (0.40)	0.07 (0.75)	0.71 (0.0003)	0.03 (0.90)	-0.0001 (0.99)
Other	-0.19 (0.39)	-0.03 (0.88)	0.03 (0.20)	-0.05 (0.82)	0.10 (0.67)	0.16 (0.50)

614 In brackets, significant level of the correlation
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616 Figure captions

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618 FIG. 1a. SORTING DATA - REPRESENTATION OF FISH HYDROLYSATES WITH 90%
619 CONFIDENCE ELLIPSES ON THE BASIS OF THE FIRST TWO MDS DIMENSIONS

620 Dimension 1 explains 60.9% of the variation and dimension 2 explains 20.3% of the variation

621

622 FIG. 1b. SORTING DATA - CORRELATION OF THE DESCRIPTION TERMS WITH
623 THE FIRST TWO MDS DIMENSIONS

624

625 FIG. 2a. PROFILING DATA - REPRESENTATION OF FISH HYDROLYSATES WITH
626 90% CONFIDENCE ELLIPSES ON THE BASIS OF THE FIRST TWO DIMENSIONS OF

627 PCA. Dimension 1 explains 75.1% of the variation and dimension 2 explains 10.25% of the
628 variation

629

630 FIG. 2b. PROFILING DATA - PROJECTION OF DESCRIPTORS IN THE FIRST PLANE
631 OF PCA

632

633 FIG. 3. SORTING DATA - REPRESENTATION ON THE DIMENSIONS OF MDS OF
634 THE T-VALUES OF THE PROCESSING PARAMETERS FROM A QUADRATIC

635 MODEL REGRESSION

636 (a) first two dimensions, (b) dimensions 1-3

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639 FIG. 4. PROFILING DATA - REPRESENTATION ON THE PRINCIPAL COMPONENTS
640 OF PCA OF THE T-VALUES OF THE PROCESSING PARAMETERS FROM A

641 QUADRATIC MODEL REGRESSION

642 (a) first two dimensions, (b) dimensions 1-3

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 652 FIG. 1a. SORTING DATA - REPRESENTATION OF FISH HYDROLYSATES WITH 90%
 653 CONFIDENCE ELLIPSES ON THE BASIS OF THE FIRST TWO MDS DIMENSIONS
 654 Dimension 1 explains 60.9% of the variation and dimension 2 explains 20.3% of the variation
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 657 FIG. 1b. SORTING DATA - CORRELATION OF THE DESCRIPTION TERMS WITH
 658 THE FIRST TWO MDS DIMENSIONS
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 661 **Fig. 1a**

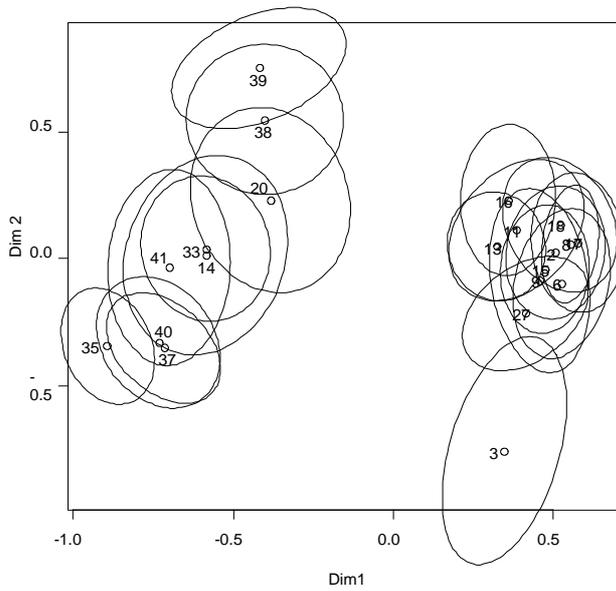
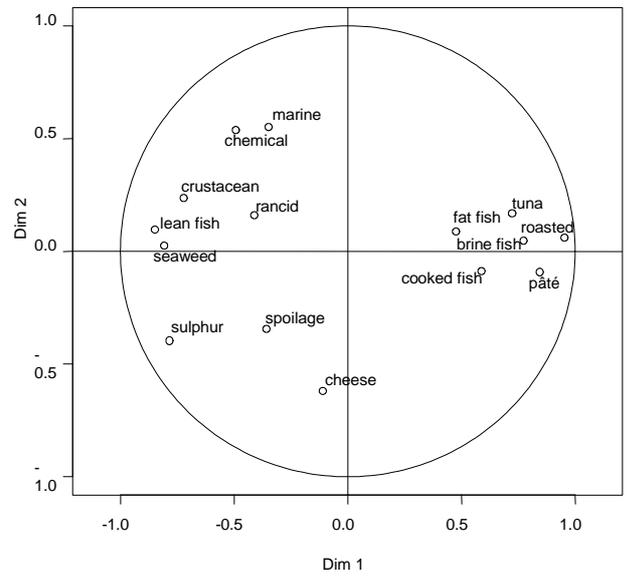


Fig. 1b



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FIG. 2a. PROFILING DATA - REPRESENTATION OF FISH HYDROLYSATES WITH 90% CONFIDENCE ELLIPSES ON THE BASIS OF THE FIRST TWO DIMENSIONS OF PCA. Dimension 1 explains 75.1% of the variation and dimension 2 explains 10.25% of the variation

FIG. 2b. PROFILING DATA - PROJECTION OF DESCRIPTORS IN THE FIRST PLANE OF PCA

Fig. 2a

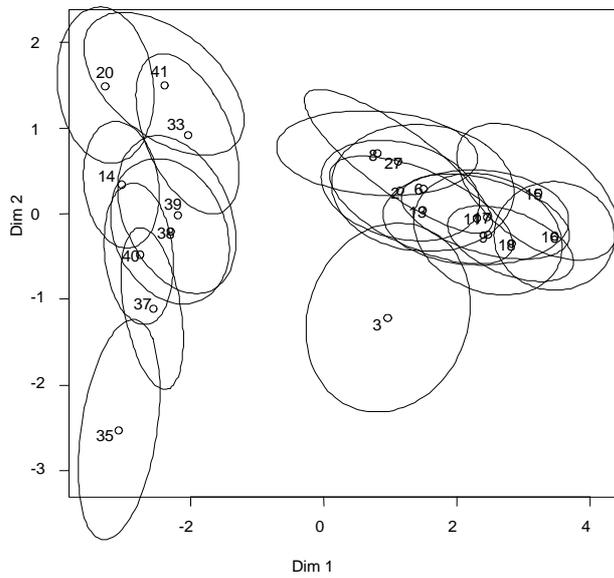
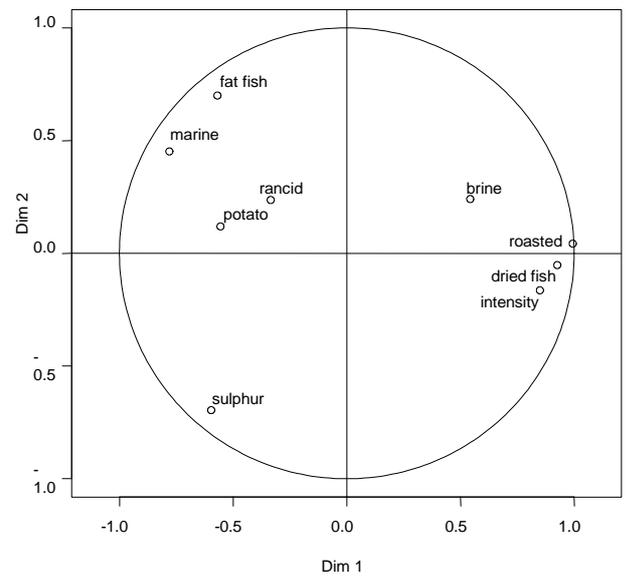


Fig. 2b



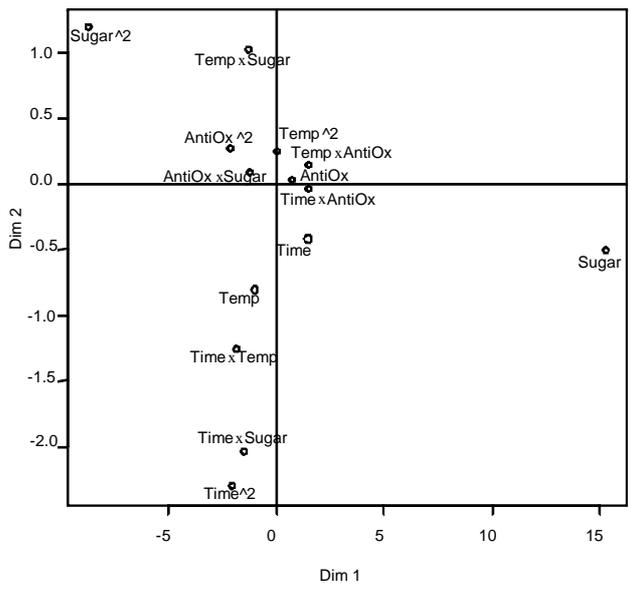
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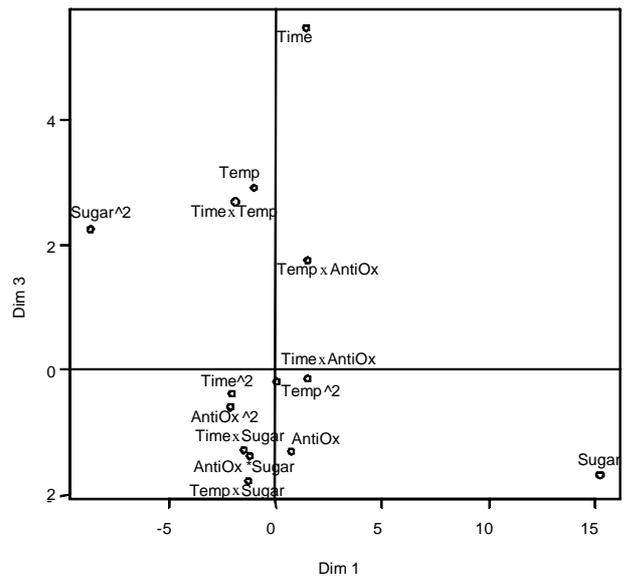
FIG. 3. SORTING DATA - REPRESENTATION ON THE DIMENSIONS OF MDS OF THE T-VALUES OF THE PROCESSING PARAMETERS FROM A QUADRATIC MODEL REGRESSION

(a) first two dimensions, (b) dimensions 1-3

(a)



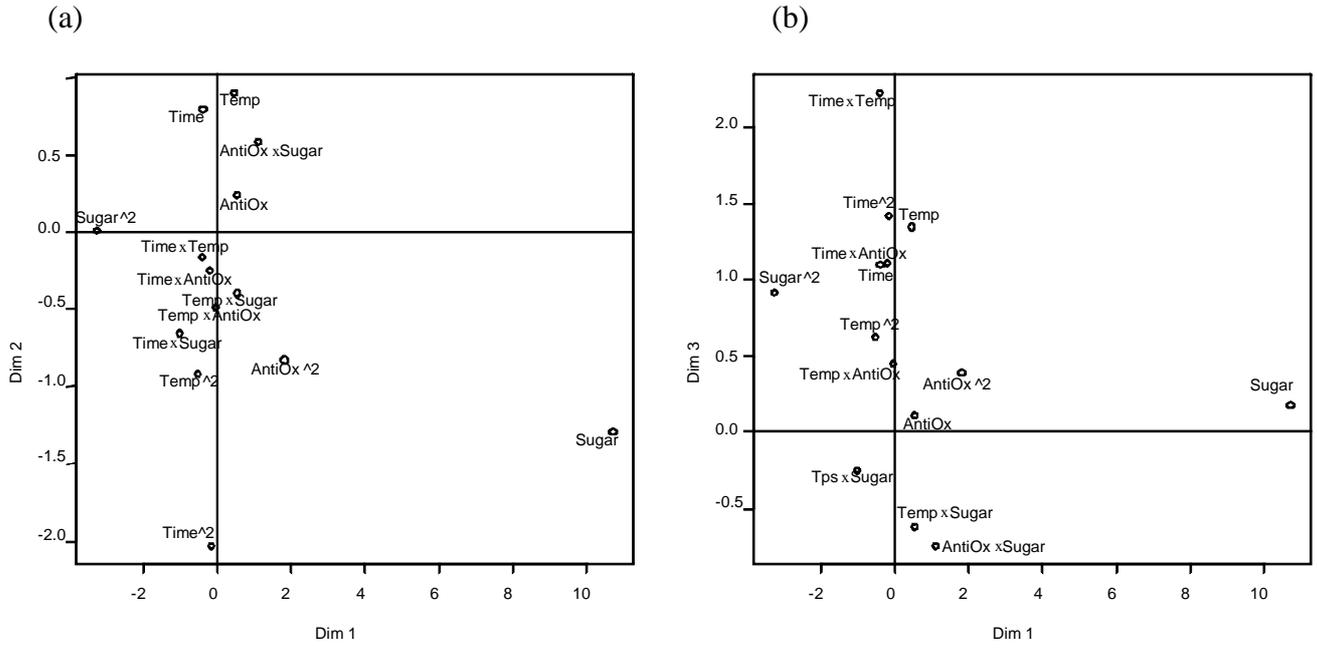
(b)



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FIG. 4. PROFILING DATA - REPRESENTATION ON THE PRINCIPAL COMPONENTS OF PCA OF THE T-VALUES OF THE PROCESSING PARAMETERS FROM A QUADRATIC MODEL REGRESSION
(a) first two dimensions, (b) dimensions 1-3



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