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1 **Quality-driven design of sponge cake: insights into reactivity, furan**  
2 **mitigation and consumer liking**

3

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13

14 **Suggested abbreviated title**

15 Quality-driven design of sponge cake: insights into liking and furan

16 **ABSTRACT**

17 This work highlights the importance of considering reactivity into the quality-driven design of  
18 heat-treated foods, which should cover the mitigation of process-induced contaminants and  
19 the improvement of the sensory properties of the foodstuff. The joint effects of formulation  
20 and baking conditions on reactivity and several quality aspects (i.e. volatile generation,  
21 physical properties, sensory and consumer tests), followed by product optimization (i.e.  
22 consumer liking and furan mitigation) were studied. While key markers are affected by all  
23 factors and their interactions, the effect of sugar and whole egg are the clearest. Furan  
24 would be predominantly generated from glucose via caramelization and/or Maillard reaction,  
25 whereas the formation of Strecker aldehydes and lipid oxidation products would be  
26 favoured by precursors in whole egg. Formulations with a low glucose content, baked at low  
27 temperatures/short times lead to optimal products. Egg-based ingredient content may be  
28 set according to preference or by applying different optimization approaches.

29

30

31 **KEYWORDS**

32 formulation, process conditions, heat-treated food, furfural, browning reactions, sensory  
33 evaluation, multivariate analysis, optimization

## 34 **1. Introduction**

35 Among common foodstuffs, thermally-treated products, such as baked goods, are widely  
36 consumed by population and are particularly important both from food safety and quality  
37 standpoints. During heat processing, food components and minor constituents may undergo  
38 different chemical pathways such as non-enzymatic browning and lipid oxidation, leading to  
39 a wide number of compounds. Some may impact aroma or colour, while others may be of  
40 health concern. Furan, in particular, has been widely addressed among process-induced  
41 toxicants in recent years, and is a current public health concern due to its possible  
42 carcinogenicity (European Food Safety Authority (EFSA), 2017; International Agency for  
43 Research on Cancer, 1995; Joint FAO/WHO Expert Committee on Food Additives, 2011).  
44 Its occurrence has been documented in a large number of foods including bakery products  
45 (Crews & Castle, 2007; Wegener & López-Sánchez, 2010; Zoller, Sager, & Reinhard, 2007).  
46 Even though strategies have been reported for its mitigation (Anese, Manzocco, Calligaris,  
47 & Nicoli, 2013; Rannou, Laroque, Renault, Prost, & Sérot, 2016), it remains a challenge  
48 since this compound can be produced from different precursors via several reaction  
49 pathways (i.e. caramelization, Maillard reaction, lipid oxidation, thermal amino acid  
50 degradation) (Crews & Castle, 2007; Perez Locas & Yaylayan, 2004; Srivastava et al.,  
51 2018; Stadler, 2012).

52 Sensory properties of baked goods (e.g. biscuits, cookies, bread) have been studied  
53 together with one or more additional aspects of quality (e.g. physical, chemical, nutritional)  
54 (Adebiyi, Obadina, Adebo, & Kayitesi, 2017; Gupta, Bawa, & Abu-Ghannam, 2011; Martins  
55 et al., 2015; Mudgil, Barak, & Khatkar, 2016; Poinot et al., 2008), but few research works  
56 consider them along with food safety. Indeed, only a handful of studies have been  
57 performed on furan mitigation along with sensory evaluation, namely in pasta (Manzocco et  
58 al., 2014), meat sauces and biscuits (Anese, Bot, & Suman, 2014). Moreover, to the best of

59 our knowledge, only one piece of work has been done on furan mitigation and consumer  
60 testing, specifically in tomato juice (Bahçeci, Akıllıoğlu, & Gökmen, 2015). Needless to say,  
61 although quality driven design of bakery products has been carried out in several works  
62 (Demirekler, Sumnu, & Sahin, 2004; Hadiyanto, Esveld, Boom, van Straten, & van Boxtel,  
63 2008; Purlis, 2014), these have mainly focused on colour and texture-related  
64 measurements as quality criteria, overlooking other important aspects, such as the  
65 occurrence of safety-related compounds, sensory properties, consumer acceptability, and  
66 the role that reactivity plays in these.

67

68 Clearly, there is a current need for developing mitigation strategies for furan in baked goods  
69 and heat-treated foods in general, while considering their sensory quality. Sponge cake is a  
70 suitable foodstuff for studying the relationship between reactivity and quality and further  
71 develop optimization strategies. Indeed, its complex chemical composition and structure  
72 resulting from diverse ingredients (i.e. sugar, egg, fat, flour, salt), which may undergo  
73 several reaction pathways during thermal treatment, make it an interesting product to study.  
74 In fact, it contains furan at the ppb level (Cepeda-Vázquez, Blumenthal, Camel, & Rega,  
75 2017) and a number of aroma compounds typically found in bakery products (Cepeda-  
76 Vázquez, Rega, Descharles, & Camel, 2018; Maire, Rega, Cuvelier, Soto, & Giampaoli,  
77 2013; Pozo-Bayón, Ruíz-Rodríguez, Pernin, & Cayot, 2007; Rega, Guerard, Delarue, Maire,  
78 & Giampaoli, 2009).

79

80 This paper deals with the effects of both formulation and baking conditions on sponge cake  
81 reactivity and quality and its further optimization. A holistic approach covering several  
82 aspects, including volatile generation, physical and physicochemical properties, sensory  
83 characteristics and consumer liking, is used for understanding the underlying phenomena,

84 while a quality optimization strategy based on furan mitigation and liking is presented. This  
85 is achieved through experimental design for effectively studying the relationship between  
86 each factor and each of the variables (linear, quadratic) and interactions among factors  
87 themselves. The paper finally discusses whether optimization criteria are conflicting or not  
88 and hence, whether a trade-off must be considered.

89

90

## 91 **2. Materials and methods**

### 92 *2.1. General*

93 An experimental design considering both sponge cake formulation (sucrose, glucose, whole  
94 egg and egg white content) and baking conditions (time and temperature) was first carried  
95 out. Several responses were then acquired through four blocks of variables, namely: volatile  
96 compound analysis, physical and physicochemical measurements, sensory evaluation and  
97 consumer tests. All responses were used for studying the effect of formulation and baking  
98 conditions on sponge cake quality and gaining insight into its relationship with reactivity.  
99 Furan content and consumer liking were in particular used for optimizing sponge cake, both  
100 from a food safety and sensory quality point of view.

101

102 Sponge cakes studied here are slim in order to induce crust formation and enhance  
103 reaction pathways, making it possible to obtain a large variety of products having different  
104 properties (e.g. chemical, sensory).

105

### 106 *2.2. Chemicals and standards*

107 Furan (purity: 99%), furfural (purity: 99%), 2-methylbutanal (purity: 95%), 2,5-  
108 dimethylpyrazine (purity: 99.9%), benzaldehyde (purity: 99%), hexanal (purity: 98%), 2-

109 pentylfuran (purity: 97%), 1-hydroxy-2-propanone (purity: 90%) and d4-furan (purity: 98%)  
110 were supplied by Sigma-Aldrich. 3-Methylbutanal (purity: 98%) was obtained from Merck,  
111 2,3,5-trimethylpyrazine was supplied by Acros (purity: 99%) and d4-furfural (purity: 99.7%)  
112 was obtained from CIL Cluzeau, CDN Isotopes (Pointe-Claire, Canada). Methanol (purity:  
113 99.9%) was purchased from Carlo Erba (Val-de-Reuil, France). Ultrapure water (> 5  
114 MΩ.cm) was produced using a Millipore Elix 3 System from Millipore SAS (Molsheim,  
115 France).

116

### 117 *2.3. Ingredients*

118 Flour was kindly provided by Grands Moulins de Paris (Ivry-sur-Seine, France). Caster  
119 sucrose was obtained from Tereos (Lille, France) and glucose (dextrose monohydrate) was  
120 kindly provided by Roquette Frères (Lestrem, France). Pasteurized whole egg and egg  
121 white were supplied by Agrodoubs (Flagey, France) and Ovoteam (Massy, France). Non-  
122 hydrogenated palm oil was obtained from Palva Celys (Rezé, France). NaCl was supplied  
123 by Salinor (Clichy, France). Sucrose, glucose and salt were stored at room temperature;  
124 flour, whole egg, egg white and palm oil were stored at -20°C. Flour, whole egg and egg  
125 white were thawed at 4°C and palm oil in a water bath at 50°C prior to use.

126

### 127 *2.4. Sponge cake preparation*

128 Sponge cakes were formulated in line with Cepeda-Vázquez et al., (2018) and according to  
129 the experimental design, using the following recipe: 125 g of flour, 125 g of sugar, 225 g of  
130 egg-based ingredient, 20 g of non-hydrogenated palm oil and 5 g of NaCl, where sugar  
131 refers to sucrose and/or glucose and egg-based ingredient to whole egg and/or egg white.

132

133 *2.4.1. Batter preparation*

134 Sponge cake batter (500 g) was prepared according to Cepeda-Vázquez et al., (2017) by  
135 mixing and beating the egg-based ingredient (whole egg and/or egg white), the sugar  
136 (sucrose and/or glucose) and salt using a kitchen appliance (Kenwood Chef, KM300, UK)  
137 for 10 min at maximum speed. Flour was gently added during 1.5 min at minimum speed  
138 and the whole was further beaten for 0.5 min. Palm oil was gently added during 15 s at  
139 minimum speed and the batter beaten for 1 min at the same speed. Formulations  
140 containing egg white-only were further battered for 120 s for proper incorporation.

141

142 *2.4.2. Baking trials*

143 Aliquots of 20 g of batter were poured into 21 aluminium molds (8.0 cm x 4.5 cm x 3.5 cm)  
144 and baked in an instrumented oven (Bongard, Wolfisheim, France), specifically designed to  
145 ensure thermal homogeneity (Fehaili, Courel, Rega, & Giampaoli, 2010). Baking  
146 temperature and time were set according to the design of experiments (DOE): 155-185°C  
147 for 15-25 min.

148 Resulting sponge cakes, of about 1.8 cm high, were sampled and stored according to the  
149 analysis for which they were bound. One sponge cake was sampled for colour  
150 measurements, while four were used for other instrumental analyses. These were put into  
151 hermetically sealed glass jars immediately after baking and then frozen at -20°C for further  
152 composite sampling (see 2.5). The remaining cakes, bound for sensory and consumer tests,  
153 were cut into bite-sized rectangular pieces of approximately 4.0 cm x 2.25 cm containing  
154 both crust and crumb, put into re-sealable zipper storage plastic bags, cooled at room  
155 temperature, sealed and then frozen at -20°C.

156

157 *2.5. Composite sampling*

158 For each row of the experimental design, four frozen sponge cakes were ground by means  
159 of a Grindomix GM200 knife mill, equipped with a stainless steel bowl and titan knives  
160 (Retsch GmbH, Haan, Germany). Each batch was ground using the following conditions:  
161 3,000 rpm for 10 s, then 6,000 rpm for up to 20 s. Composite samples were immediately put  
162 into hermetically sealed glass jars and stored at -20°C until analysis (adapted from Cepeda-  
163 Vázquez et al., (2017)).

164

#### 165 *2.6. Physical and physicochemical properties*

166 Dry matter and pH determination were carried out as in previous studies (Cepeda-Vázquez  
167 et al., 2018). Colour measurement was performed on the upper face of a whole cake at 3  
168 points by using a 6834 spectro-guide sphere gloss colorimeter (BYK-Gardner, Germany)  
169 and expressed in the CIELab colour space (adapted from Cepeda-Vázquez et al., (2018);  
170 Poinot et al., (2008)).

171

#### 172 *2.7. HS trap / GC-MS procedure*

173 Several volatile compounds were studied according to Cepeda-Vázquez et al., (2018).  
174 While furan and furfural were defined as relevant furanic quality markers for safety and  
175 aroma, respectively, additional aroma compounds commonly found in baked goods were  
176 also considered. These compounds are representative of different classes (furans,  
177 aldehydes, pyrazines, etc.) and reaction pathways (mainly caramelization, Maillard reaction,  
178 lipid oxidation and Strecker degradation).

179 Quantitative analysis for furan and furfural and semi-quantitative analysis for additional key  
180 aroma markers (n=3) were performed simultaneously by means of a TurboMatrix  
181 Headspace Sampler HS 40 Trap (Perkin Elmer, Llantrisant, UK) equipped with an air  
182 monitoring trap containing a two-sorbent bed (Carbotrap and Carbosieve SIII, Perkin Elmer),

183 and a Trace GC Ultra gas chromatography system coupled to an ISQ single quadrupole  
184 mass spectrometer (Thermo Scientific, Rodano, Italy) (Cepeda-Vázquez et al., 2017, 2018).

185

#### 186 *2.7.1. Standard solutions for furan and furfural quantification*

187 Stock and standard solutions were prepared in a room at 18°C, while HS samples were  
188 prepared in a separate room to avoid any cross-contamination from the surrounding air.

189 Stock solutions of d4-furan, d4-furfural, furan and furfural in methanol were prepared  
190 separately at a concentration of 2.5 g l<sup>-1</sup>, put into hermetic amber glass bottles and stored at  
191 -20°C for up to one month. A standard solution of d4-furan at a concentration of 25 ng µl<sup>-1</sup>  
192 was prepared daily by diluting the stock solution in ultrapure water. In order to adapt isotope  
193 concentration to different samples, six mixed standard solutions were prepared daily by  
194 diluting the standard solution of d4-furan and the stock solution of d4-furfural in ultrapure  
195 water (concentrations ranged from 0.03 to 4.1 ng µl<sup>-1</sup> for d4-furan and from 1.2 to 735 ng µl<sup>-1</sup>  
196 for d4-furfural). They were then kept at 4°C until use. All glassware was previously baked  
197 out at 55°C for at least 24 h to avoid any contamination prior to analysis (Cepeda-Vázquez  
198 et al., 2017).

199

#### 200 *2.7.2. HS sample preparation*

201 During vial preparation, sponge cake composite samples were kept in an ice bath to avoid  
202 volatile loss. Aliquots of ground sponge cake (0.588 g, dry basis) and ultrapure water (9.412  
203 g) were weighed into 20 ml vials for a 16 water / sample amount ratio (dry basis) and 10 g  
204 of total amount (water + sample amount, dry basis) (Cepeda-Vázquez et al., 2017). All  
205 amounts were exactly measured in mass. A volume of 40 µl of the corresponding d4-furan  
206 + d4-furfural standard solution was added according to the estimated furan and furfural  
207 concentration of each sample. Vials were capped with aluminium polytetrafluoroethylene

208 coated silicone septa at each step of preparation and sealed immediately after labeled  
209 standards' addition to avoid loss of volatiles. Vials were then vortexed for 5 s at maximum  
210 speed. All HS vials were previously baked out in a muffle furnace at 350°C for at least 1 h  
211 to avoid any HS glassware contamination before analysis (Cepeda-Vázquez et al., 2017).

212

### 213 *2.7.3. HS trap conditions*

214 Conditions were set as previously described for the optimization of HS trap joint extraction  
215 of furan and furfural (Cepeda-Vázquez et al., 2017).

216

### 217 *2.7.4. GC-MS analysis*

218 GC-MS was adapted from Cepeda-Vázquez et al., (2018). A 100% polyethylene glycol  
219 column (ZB-WAX) was used for separation (60 m x 0.25 mm x 0.5 µm; Zebron, USA).  
220 Helium was used as carrier gas at a constant flow rate of 1.2 ml min<sup>-1</sup>. Injection was done in  
221 split mode (split ratio of 1:8). The GC oven was programmed as follows: initial temperature  
222 40°C (held for 4 min), then raised at 10°C min<sup>-1</sup> until 150°C and then raised at 20°C min<sup>-1</sup>  
223 until reaching a final temperature of 240°C (held for 8 min). MS transfer line and ion source  
224 temperatures were set to 230°C and 200°C, respectively. The ionization mode was electron  
225 impact (EI), 70 eV. Data acquisition was done in nine segments, including a full scan mode  
226 from 25 to 150 m/z at 0.2 s per scan and the corresponding selected ion monitoring (SIM)  
227 mode with a dwell time of 0.1 s each, except for furfural and d4-furfural 0.05 s each (furan,  
228 m/z 39, 68; d4-furan, m/z 42, 72; 2-methylbutanal, m/z 41, 44, 57; 3-methylbutanal 41, 44,  
229 57; hexanal, m/z 44, 56; 2-penylfuran, m/z 81, 138; 1-hydroxy-2-propanone, m/z 43, 74;  
230 2,5-dimethylpyrazine, m/z 42, 108; 2,6-dimethylpyrazine, m/z 42, 108; 2,3,5-  
231 trimethylpyrazine, m/z 42, 122; furfural, m/z 39, 95, 96; d4-furfural, m/z 42, 98, 100;  
232 benzaldehyde, m/z 77, 106 [quantifier ions in italics]). Chromatographic peak areas for each

233 compound were calculated by extracting the quantifier ions from the SIM mode acquisition  
234 data using Quan Browser, Xcalibur 2.1.0 SP1, build 1160 (Thermo Fisher Scientific Inc.,  
235 USA) and used both for quantitative and semi-quantitative analyses. Native molecules were  
236 confirmed by means of the Wiley 8 and NIST 08 mass spectra libraries, calculation of  
237 normal alkane retention index (RI) and comparison to NIST Chemistry WebBook SRD 69  
238 (2017) indices, and analysis of pure standards (Cepeda-Vázquez et al., 2018).

239 The reported method performances for furan and furfural quantification are the following:  
240 repeatability (RSD:  $\leq 3.3\%$  for furan and  $\leq 2.6\%$  for furfural), intermediate precision (RSD:  
241  $4.0\%$  for furan and  $4.3\%$  for furfural), linearity ( $R^2$ : 0.9957 for furan and 0.9996 for furfural),  
242 limit of detection (LOD; furan:  $0.50 \text{ ng g}^{-1}$  sample, dry basis; furfural:  $10.2 \text{ ng g}^{-1}$  sample, dry  
243 basis), limit of quantification (LOQ; furan:  $0.99 \text{ ng g}^{-1}$  sample, dry basis; furfural:  $41.1 \text{ ng g}^{-1}$   
244 sample, dry basis) (Cepeda-Vázquez et al., 2017). Experimental reproducibility, from batter  
245 preparation to HS trap analysis, has been reported as 6.4 and 6.8% for furan and furfural  
246 quantitation, respectively, and ranged from 6 to 15% for the semi-quantitative analysis of  
247 selected markers (Cepeda-Vázquez et al., 2018).

248

## 249 *2.8. Sensory description*

250 Bite-sized frozen samples were thawed at room temperature 30 min prior to descriptive  
251 analysis by 5 panelists well aware of sensory descriptive methodologies (3 females, 2  
252 males) for sensory description. Term generation was first carried out individually in 2  
253 sessions of about 30 min each, as follows: 9 products were randomly presented per session  
254 and panelists were free to generate descriptors into 7 categories, namely appearance,  
255 texture, odour, mouthfeel, taste, aroma and persistence; a blank category was also  
256 provided for additional terms. A total of 179 terms were collected. Semantic consensus was  
257 then achieved in a discussion session where panelists were presented with a full set of

258 samples. Synonyms were identified and a total of 18 terms were selected as appropriate for  
259 evaluation; protocols were defined accordingly. Evaluation of 18 samples was then carried  
260 out in 2 sessions in individual cabins, where water and napkins were available for panelists.  
261 Samples were put into plastic cups labeled with a 3-digit code randomly assigned and  
262 presented monodically (one by one), according to a XLSTAT-generated serving order. Nine  
263 products were assessed per session using the 18 previously selected descriptors and a  
264 rating-based variant of the check-all-that-apply and flash profile techniques. Evaluating  
265 sessions took about 30 min each.

266

### 267 *2.9. Consumer tests*

268 Consumers who volunteered to take the tests were recruited at AgroParisTech (Massy  
269 Center) in France (n=50). Tests were carried out at the Sensory Perception and  
270 Sensometrics Laboratory in individual cabins. Verbal and written information about the  
271 study was provided to participants and consent was given orally.

272 Samples were thawed, prepared and labeled as for the sensory description; a full set of 18  
273 samples was presented to consumers in one session, monodically following a XLSTAT-  
274 generated serving order. Consumers were asked to select the corresponding 3-digit code  
275 and indicate overall liking using the ISO 9-point hedonic scale (where 1 = "extremely  
276 dislike", 5 = "neither like nor dislike" and 9 = "extremely like") (ISO, 2003). Water and  
277 napkins were available at each cabin. Average time for hedonic tests was around 15 min  
278 per participant.

279 Approval from an ethical board was not considered necessary for this study since sponge  
280 cake is a common foodstuff. Additionally, the furan content of our samples remained at a  
281 low level overall when compared to other widely consumed food products, such as baby-  
282 food, bread, cereal snacks or coffee (Zoller et al., 2007).

283

## 284 *2.10. Optimization procedure*

285 The step-by-step procedure, from selection of factors to optimization, is briefly described  
286 below.

287

### 288 *2.10.1. Selection of factors*

289 Formulation and baking conditions were considered at once for studying reactivity and  
290 optimizing sponge cake quality. Previous studies showed that sugar and egg are the two  
291 most impacting categories of ingredients on key quality marker generation in sponge cake,  
292 including furan, furfural and other selected aroma markers (Cepeda-Vázquez et al., 2018).  
293 Hence, two variables related to recipe were selected: sucrose content ( $S$ , %, within sugar  
294 category) and whole egg content ( $E$ , %, within egg-based ingredient category). In other  
295 words, a mixed approach was considered within each of these variables, making it possible  
296 to study both sucrose and glucose, and also whole egg and egg white. Among process  
297 conditions, only baking variables were taken into account since these are likely the most  
298 important factors influencing the extent of reaction pathways leading to safety and quality-  
299 related compounds in baked goods (mainly through caramelization, Maillard reaction and  
300 lipid oxidation). Baking time ( $t$ ) and temperature ( $T$ ) were thus selected.

301

### 302 *2.10.2. Optimal design of experiments*

303 An optimal, tailor-made experimental plan consisting of 18 rows was designed to study and  
304 optimize sponge cake quality (Table 1). Response surface models were postulated  
305 including all quadratic and linear terms, as well as second order interactions.

306 The experimental plan was designed considering orthogonality among all factors, meaning  
307 that the effects of the formulation and baking variables could be differentiated from one  
308 another.

309 The following low (-1), middle (0) and high (+1) levels were selected for each factor: *S*,  
310 0/50/100% (within sugar category); *E*, 0/50/100% (within egg-based ingredient category); *t*,  
311 15/20/25 min; *T*, 155/170/185°C. Sucrose and whole egg levels were set according to  
312 previous formulation studies (Cepeda-Vázquez et al., 2018), while screening trials were  
313 carried out for setting time and temperature levels. In these, the extreme formulations were  
314 studied (i.e. glucose-based and also egg white-containing sponge cakes, which were  
315 reported to have the highest and lowest furanic compound content and darkest and lightest  
316 colour, respectively) (Cepeda-Vázquez et al., 2018). During such trials, batters were baked  
317 at 140, 170 and 200°C for 10, 15, 20, 25 and 30 min, yielding a wide range of samples.  
318 Sensory acceptability and analytical sensitivity were the criteria to narrow the baking  
319 boundaries for the experimental design. Unbaked and burnt products were left out since  
320 they were out of the acceptability range. Such unbaked cakes were also likely to be below  
321 the furan detection limit, based on the results by Cepeda-Vázquez et al., (2018).

322

### 323 *2.10.3. Model fitting and multivariate optimization*

324 Multiple linear regression (MLR) was performed by standard least squares (SLS) for each  
325 response (5% for type I error). For volatile compounds, responses were defined as  
326 logarithmic functions (ln) of furan content, furfural content and chromatographic peak areas  
327 for the additional aroma compounds to respect homoscedasticity hypothesis of MLR. Thirty-  
328 five models were obtained accordingly (11 on volatiles, 5 on physicochemical properties, 18  
329 on sensory descriptors and 1 on consumer preference) in order to study the main effects  
330 and interactions.

331 A sponge cake optimization strategy was then generated by means of a prediction profiler  
332 based on the resulting models for mean consumer preference and furan content, using  
333 desirability functions. The model for furfural content was also added for illustrative purposes.  
334 Terms in the furan reduced model were considered into the joint profiler. Desirabilities were  
335 defined as the maximum value for liking, the minimum value for furan and none for furfural.  
336 As a result, single values for each factor were obtained by maximizing overall desirability.

337

### 338 *2.11. Additional data treatment*

339 To visualize and interpret our results, we performed a Principal Component Analysis (PCA)  
340 on mean values for each set of experiments (individually): for volatile compounds, physical  
341 and physicochemical properties, sensory data (correlation method), as well as for consumer  
342 data (covariance method) (Greenhoff & MacFie, 1999). Factors of the DOE were added as  
343 supplementary variables in all PCA to identify their potential linear links to volatile  
344 compound generation, sponge cake properties and consumer liking.

345 To identify the discriminating variables (i.e. the ones that enable us to differentiate the set of  
346 experiments), two-way ANOVA (Product, Panelist) was carried out on sensory data (one  
347 per descriptor). To identify the differences in terms of liking for the experiments, two-way  
348 ANOVA (Product, Consumer) was carried out on hedonic scores. Agglomerative  
349 Hierarchical Clustering (AHC) was also performed on consumer data (standardized by  
350 consumer - using Euclidean distance for dissimilarities and Ward's method for  
351 agglomeration) to identify potential clusters of consumers in terms of liking.

352 Formulation and baking experimental design, MLR and optimization by means of desirability  
353 functions were done in JMP 10.0.0 (SAS Institute Inc.). Sensory and consumer  
354 experimental set-up, ANOVA, AHC and PCA were carried out in XLSTAT 2014.5.04  
355 (Addinsoft SARL).

356

357

### 358 **3. Results and discussion**

#### 359 *3.1. Furan and furfural content*

360 Furan content was low in most samples and remained at a  $\text{ng g}^{-1}$  level, while that of furfural  
361 ranged from  $\text{ng g}^{-1}$  to  $\mu\text{g g}^{-1}$ . Interestingly, furan and furfural reduced models show that their  
362 contents are affected by sponge cake formulation, baking and their interactions in very  
363 similar ways (all linear terms, most quadratic effects and interactions,  $R^2$ : 0.965 for furan  
364 and 0.991 for furfural,  $P > F: < 0.0001$  for both variables) (Table 2). This suggests that  
365 predominant pathways in furan generation are those common to furfural (i.e. caramelization  
366 and Maillard reaction) when coupling sponge cake formulation to baking conditions. This  
367 result confirms previous findings showing that changes in fat leading to mild lipid oxidation  
368 did not exert a significant effect on furan content (Cepeda-Vázquez et al., 2018).  
369 Differences in content levels do suggest however, that furfural's generation is favoured over  
370 that of furan regardless of the formulation or baking conditions. Similar results were  
371 observed when studying sponge cake formulation alone (Cepeda-Vázquez et al., 2018) and  
372 in a kinetic study in cake models specifically designed to follow furan and furfural's  
373 formation via caramelization and Maillard reaction (Srivastava et al., 2018). Overall, this  
374 suggests that furfural (less volatile and easier to analyze than furan), could indeed be a  
375 proper marker of furan generation in this type of product or reaction system.

376

#### 377 *3.2. Reactivity in sponge cake*

378 Furan, furfural and selected key markers, as well as physical and physicochemical  
379 properties were considered for studying reactivity. According to the PCA on responses of all  
380 markers, principal components 1, 2 and 3 account for 95.8% of variability (Figure 1a and b).

381 Even though the DOE was built considering orthogonality among the effects under study,  
382 both loading plots depicting them as supplementary variables show that these are no longer  
383 orthogonal, meaning that interactions among some DOE factors do take place (Figure 1a  
384 and b). This was also confirmed by the corresponding response surface models, where not  
385 only the main effects but also several interactions were observed for all compounds ( $R^2 \geq$   
386 0.911 and  $P > F: < 0.0001$  for all models). This clearly suggests that the generation of  
387 volatile compounds in sponge cake is due to formulation and baking conditions, as well as  
388 their interactions.

389 Moreover, in the PC1 vs PC2 plot, DOE factors follow two axes: sugar type is highly related  
390 to PC2 (25.2%), i.e. sucrose (+), glucose (-), while whole egg (+), time (+) and temperature  
391 (+) relate to PC1 (51.4%) to a minor extent. However, in the PC2 vs PC3 plot, whole egg  
392 appears to be anticorrelated to time and temperature, forming a new axis strongly related to  
393 PC3 (19.2%) (Figure 1a and b).

394 Furan, furfural and 1-hydroxy-2-propanone, all found in one cluster, contribute positively to  
395 PC1 and PC3 and negatively to PC2. This means that sugar, egg-based ingredients as well  
396 as baking time and temperature, all contribute to their formation, either directly or through  
397 their interactions. Pyrazines, all grouped into another cluster, would be predominantly  
398 affected by sugar (related to PC2) and by whole egg to a much lower extent, baking time or  
399 temperature (related to PC1 and PC3). More importantly, these clusters exhibit different  
400 behaviours when considering PC2 (highly related to sugar category): furan, furfural and 1-  
401 hydroxy-2-propanone correlate to glucose (-), while pyrazines correlate to sucrose (+)  
402 (Figure 1a and b). These important findings suggest that, when considering the nature of  
403 sugar, furan, furfural and 1-hydroxy-2-propanone are predominantly formed from glucose, a  
404 reducing sugar, either via caramelization or Maillard reaction, while pyrazines are majorly

405 formed from sucrose, a non-reducing sugar, via hydrolysis and further Strecker degradation  
406 (often considered into the Maillard reaction pathway).

407 Other aroma compounds are found in a third cluster: hexanal, 2-pentylfuran and Strecker  
408 aldehydes (benzaldehyde, 3-methylbutanal, 2-methylbutanal) (Figure 1a and b). While 2-  
409 pentylfuran can be formed through lipid oxidation from polyunsaturated fatty acids (PUFAs)  
410 and also via the interaction of lipid oxidation products and amino groups (Adams, Bouckaert,  
411 Van Lancker, De Meulenaer, & De Kimpe, 2011; Belitz, Grosch, & Schieberle, 2009; Grein,  
412 Huffer, Scheller, & Schreier, 1993; Hidalgo, Gallardo, & Zamora, 2005; Perez Locas &  
413 Yaylayan, 2004; Whitfield, 1992), hexanal can only be formed via lipid oxidation. Hence, a  
414 high correlation between hexanal and Strecker aldehydes was initially unexpected, but their  
415 high correlation to whole egg (mostly related to PC3) might explain it. Indeed, whole egg is  
416 an important source of both PUFAs and free amino acids (U.S. Department of Agriculture  
417 (USDA), 2016), which are precursors of hexanal and Strecker aldehydes, respectively. As  
418 of 2-pentylfuran, previous studies on sponge cake formulation showed that it was somewhat  
419 correlated to hexanal and also to Strecker aldehydes (Cepeda-Vázquez et al., 2018).  
420 Hence, its correlation to them in this work, that is, when considering baking time and  
421 temperature in addition to formulation, is not surprising.

422 Another interesting result is the finding that Strecker aldehydes and pyrazines are not  
423 grouped into one cluster, although they both depend on free amino acids and sugar  
424 degradation products (dicarbonyls for Strecker aldehydes, and aminoketones or 1-hydroxy-  
425 2-propanone along with  $\text{NH}_3$  and hexanal for pyrazines).

426

427 Physical and physicochemical properties are adequately represented in the PC1 and PC2  
428 plot (PC1: 77.9%, PC2: 13.4%) (Figure 1c). Although DOE factors are less well represented  
429 in the same plot, again two axes are visible: the first relating to sucrose content and the

430 second one partially to temperature and whole egg content to a much lower extent. Baking  
431 time appears to be correlated to both axes. Clearly, all properties are dependent on all  
432 formulation and baking variables, since they are not aligned to the axes. Overall, long  
433 baking times, high temperatures, and formulations with whole egg and glucose, that is more  
434 advanced thermal reactions, all relate to low lightness ( $L^*$ ) and pH values, as expected.  
435 These two properties are indeed the best indicators of browning progression according to  
436 modeling ( $R^2$ : 0.976 and 0.928, respectively, and  $P > F$ :  $<0.0001$  for both variables).

437

### 438 3.3. *Sponge cake sensory quality*

439 Regarding sensory data, most descriptors were found useful for discriminating sponge cake  
440 samples, except for caramel odour ( $P > F$  for product: 0.151) and nut aroma, which was  
441 close to the set significance level ( $P > F$  for product: 0.055). All the discriminating sensory  
442 terms, along with nut aroma, were further analyzed by PCA.

443 Most descriptors are well represented in the PC1 vs PC2 plot, accounting for 73.0% of  
444 variability of sensory data (Figure 2). Interestingly, while some descriptors are scattered,  
445 two clusters are clearly visible (lower left and lower right) and differentiated by PC1. The  
446 first one groups variables related to “typical” descriptors for sponge cake, such as sponge  
447 cake and egg aroma and odour, as well as sweet taste. The second one includes  
448 descriptors related to “severe” thermal reactions, like caramel and burnt notes, colour, bitter  
449 taste and is opposite to spongy mouthfeel and sticky texture. The “typical” cluster is highly  
450 related to sucrose content, and to short time and low temperatures (or their interaction) to a  
451 minor extent, while the “severe” group is related to long baking time and high temperature  
452 (or their interaction), and to glucose to some extent. A very important finding is that whole  
453 egg is highly correlated to both “typical” and “severe” descriptors, since it is negatively  
454 correlated to PC2.

455 Firmness and crusty mouthfeel are both related to time and temperature, which is not  
456 surprising, but they also are to whole egg; chewy mouthfeel interestingly relates to glucose  
457 and egg white (Figure 2).

458 Concerning hedonic scores, AHC resulted in two clusters of 21 and 29 participants,  
459 respectively (supplementary figure A). According to ANOVA results, consumers in cluster 1  
460 rank products higher ( $P > F: 0.001$ ), yet clusters seem to have similar class profiles  
461 (supplementary figure B). PCA on liking also shows that consumers are mostly grouped into  
462 one cluster, both in the PC1 vs PC2 (59.2%) and PC1 vs PC3 plots (54.9%) (Figure 3a and  
463 b). Liking appears to be highly related to sucrose, opposite to baking time and temperature,  
464 and influenced by whole egg to a minor extent. A response surface model considering  
465 preference scores for all participants ( $n=50$ ) confirms that all formulation and baking factors,  
466 as well as some interactions, impact liking ( $R^2: 0.987$ ,  $P > F: < 0.0001$ ). Sponge cakes with  
467 a high sucrose and whole egg content, baked during a short time at low temperature are  
468 preferred overall.

469

#### 470 *3.4. Linking reactivity and sponge cake quality*

471 Results discussed in previous sections prove that formulation (sucrose, glucose, whole egg  
472 and egg white content) and baking conditions (time and temperature) do have an effect on  
473 the reaction pathways occurring in sponge cake and its quality. Furthermore, interactions  
474 among formulation and baking factors were identified and quantified. More importantly,  
475 these factors impact volatile generation, physical, physicochemical and sensory properties  
476 as well as liking differently, that is to say they interact in different ways. These findings  
477 underline the need of using a holistic approach by studying several factors and also several  
478 aspects of the product at once. In this regard, gaining further insight might be achieved by  
479 applying sensometric or chemometric tools to study the relationship between different

480 blocks of variables or variables from different blocks, together or aside from factors. For  
481 instance, it would be interesting to determine how the selected aroma markers correlate to  
482 preference, how the latter is impacted by texture or to define how "typical" sponge cake or  
483 "severe" heat treatment descriptors, L\* and pH relate to preference and furan content.

484

### 485 3.5. *Optimizing sponge cake: liking and furan mitigation*

486 Furan mitigation and consumer preference, here defined as food safety and sensory quality  
487 criteria, are both affected by formulation and baking factors as explained before. More  
488 importantly, they exhibit opposite behaviours in relation to most factors and furan content  
489 level remains low overall despite changes in these. Consequently, there is enough room for  
490 finding optimal sponge cakes with low furan content that fall within consumers' preference.

491 A set of optimal sponge cake formulation and baking conditions was found, considering  
492 both quality criteria simultaneously, based on desirability function maximization (Figure 4a).  
493 High sucrose content (with respect to glucose), low temperature and short baking time, all  
494 contribute to maximizing liking and minimizing furan, within the studied ranges. These  
495 quality criteria are however conflicting with respect to whole egg content, since they are  
496 both positively influenced by such factor. A trade-off would naturally be considered, as  
497 depicted in Figure 4a, yet the predicted furan content remains below LOD (0.50 ng g<sup>-1</sup>  
498 sample, dry basis) along the boundaries of these factor, when keeping the rest of them at  
499 their optimal values. This means that amino acids and lipids in whole egg would not favour  
500 furan generation as much as sugar type, and that whole egg would do more for liking than  
501 for furan generation, when keeping the remaining variables at optimal values. As a result,  
502 increasing whole egg content for maximizing liking alone might as well be considered  
503 (Figure 4b).

504 Also, the fact that the optimal values are reached at the low boundaries for time and  
505 temperature suggests that there might be greater room for optimization if setting even lower  
506 values for these two variables. However, it must be noted that lower values could also lead  
507 to unbaked products reducing hedonic score and hence, a quadratic form might therefore  
508 be observed for these factors. Finally, prediction profiler depicts similar curves for furan and  
509 furfural for both optimization scenarios (Figure 4a and b), confirming that these compounds  
510 behave in similar ways.

511

512

#### 513 **4. Conclusions**

514 Furan content and consumer liking, selected as food safety and sensory quality criteria for  
515 sponge cake optimization respectively, are influenced in opposite ways by most formulation  
516 and baking factors. Overall, high sucrose content (in relation to glucose), low temperature  
517 and short baking times yield sponge cakes with a minimal furan content and a maximal  
518 consumer liking score, while whole egg content may be set according to preference only,  
519 since furan level would remain around LOD levels. It must also be noted that other  
520 optimization strategies could be used instead, such as defining a threshold for furan content  
521 and/or preference, once regulations on furan content are defined, and depending on the  
522 criteria set for meeting consumer needs.

523 Regarding reactivity, several reaction pathways seem to occur when considering both  
524 formulation and baking conditions in sponge cake, according to the study of key markers.  
525 Furan, furfural and 1-hydroxy-2-propanone would be predominantly produced from glucose  
526 via caramelization and/or Maillard reaction, while pyrazines would be mainly generated from  
527 sucrose via sugar hydrolysis and further Strecker degradation. The latter, as well as lipid  
528 oxidation would also occur and be highly related to egg yolk (in whole egg). Moreover,

529 furfural, whose analysis might be easier than that of furan, could indeed be a proper marker  
530 of furan generation in this type of products given their close relationship.

531 This work contributes to the development of quality-driven design strategies adapted for  
532 heat-treated food, based on safety and sensory criteria. It also advances the understanding  
533 of the link between reactivity and quality through a holistic approach. This strategy could  
534 also be broadened to other quality aspects (e.g. loss of nutritional value, antioxidant content,  
535 etc.) or easily applied to thermally-treated products with different properties (e.g. sensory,  
536 chemical composition, structure, dimensions, etc.). It could be further explored by applying  
537 sensometric or chemometric tools as well, for gaining further insight into how different  
538 quality aspects relate to each other (e.g. sensory description and consumer liking), or how  
539 variables from different aspects also relate (e.g. aroma markers from GC-MS analysis and  
540 aroma descriptors from sensory tests; furfural content and both colour measurements and  
541 as perceived by panelists, etc).

542

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547

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551

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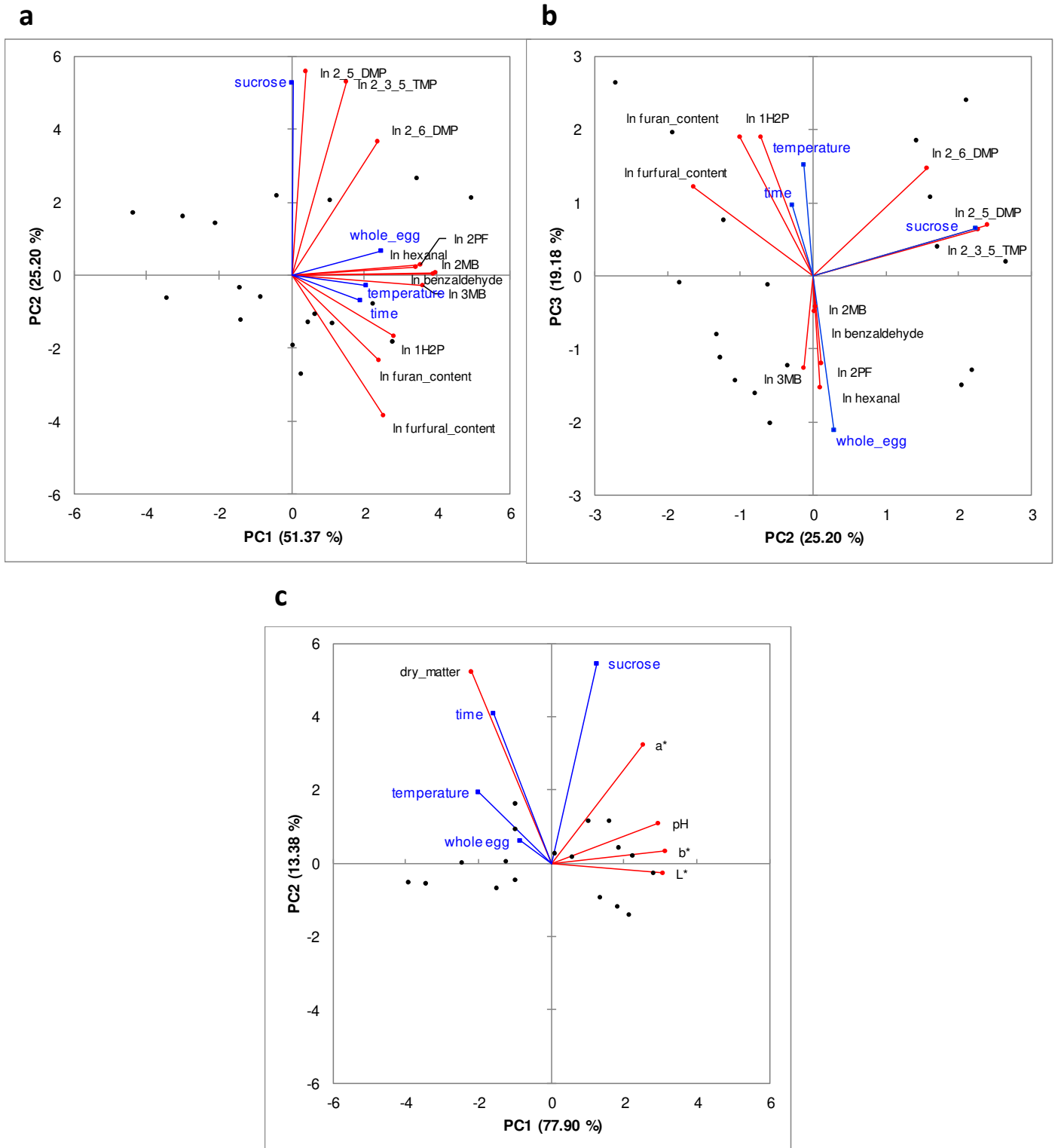
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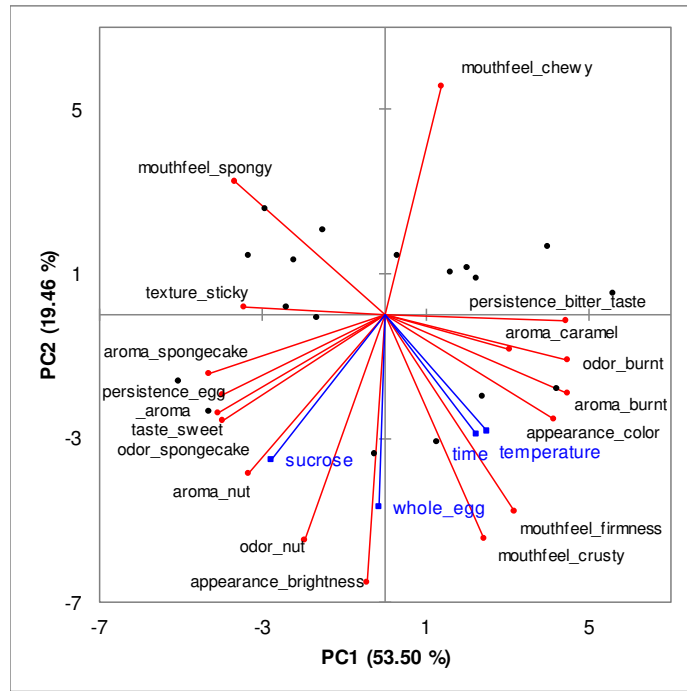
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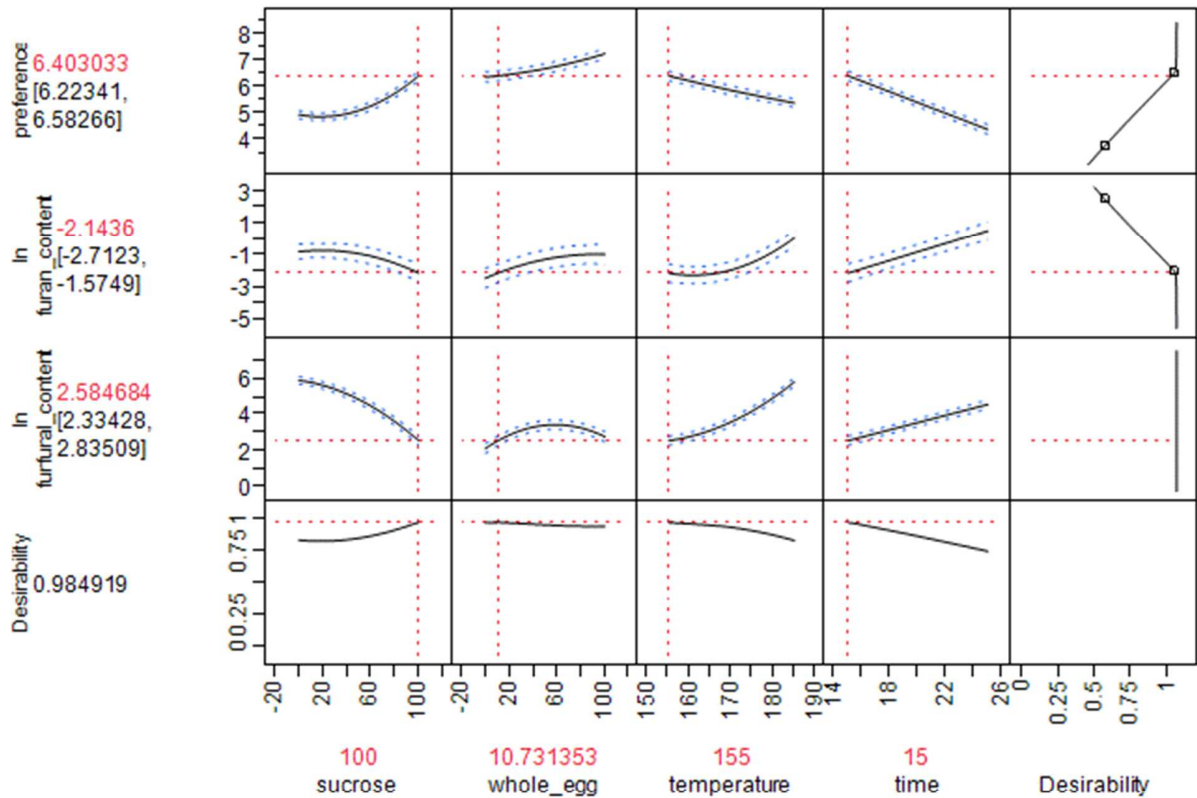
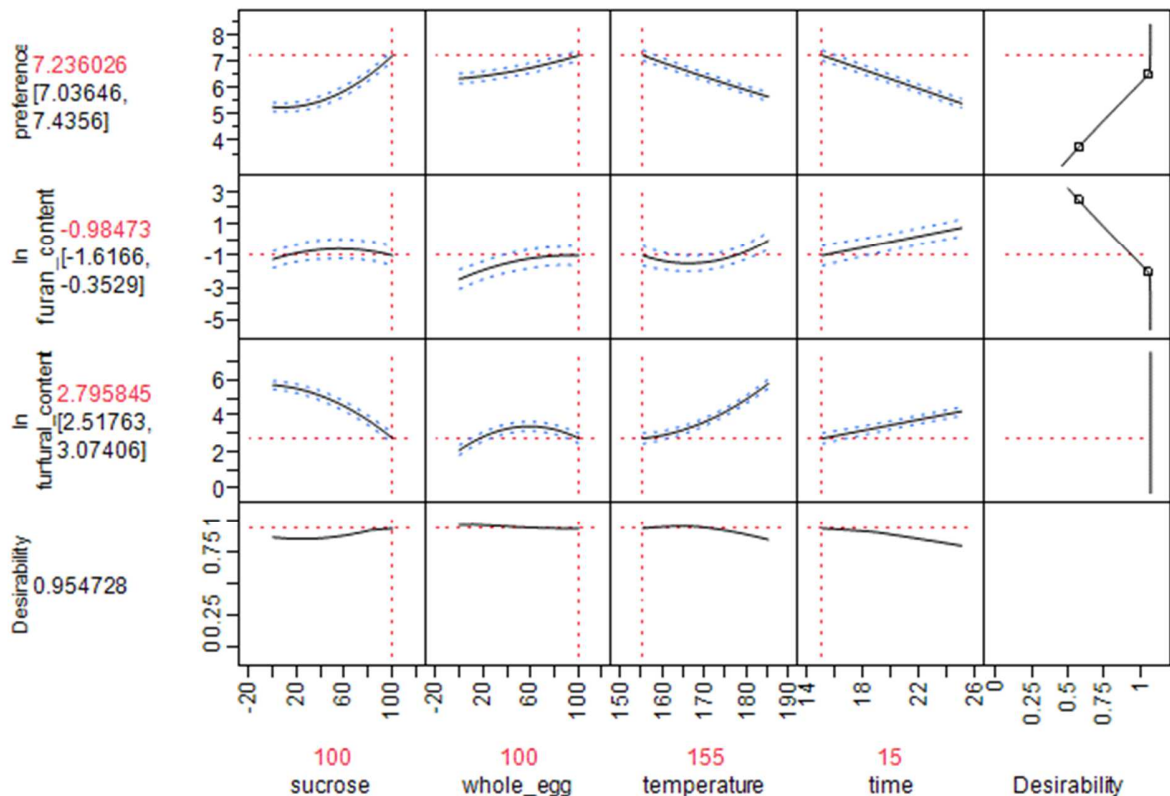


**Figure 1 PCA biplots on selected volatile markers and physical-physicochemical properties.**  
 ln: logarithmic functions; 2MB: 2-methylbutanal; 3MB: 3-methylbutanal; 2PF: 2-pentylfuran; 1H2P: 1-hydroxy-2-propanone; 2,5 DMP: 2,5-dimethylpyrazine; 2,6 DMP: 2,6-dimethylpyrazine; 2,3,5 TMP: 2,3,5-trimethylpyrazine; L\*: lightness, a\*: green (-) to red (+); b\*: blue (-) to yellow (+); variables for PCA (•); supplementary variables (■)



**Figure 2 PCA biplot on sensory descriptors.**  
 Variables for PCA (•); supplementary variables (▪)



**a****b**

**Figure 4 Prediction profilers with desirability functions.**



















Response surface models for preference, logarithmic functions (ln) of furan and furfural content, and desirability function according to factors; desirabilities are defined as the maximum value for preference, minimum value for furan content and none for furfural content; (a) set of optimal formulation and baking conditions; (b) set of optimal values for sucrose, temperature and time and maximal value for whole egg.

**Table 1 Optimal response surface design on sponge cake formulation and process conditions**

<b>Run</b>	<b>Sucrose</b>	<b>Whole egg</b>	<b>Baking temperature</b>	<b>Baking time</b>
	<i>S (%)</i> <sup>a</sup>	<i>E (%)</i> <sup>b</sup>	<i>T (°C)</i>	<i>t (min)</i>
1	1	0	1	1
2	-1	1	1	-1
3	-1	1	-1	1
4	1	1	1	0
5	1	1	0	-1
6	1	-1	1	-1
7	-1	-1	-1	1
8	0	0	-1	-1
9	1	-1	0	1
10	1	1	-1	1
11	-1	-1	-1	-1
12	-1	0	0	0
13	1	-1	-1	0
14	-1	-1	1	1
15	-1	1	-1	-1
16	-1	1	1	1
17	0	1	0	1
18	0	-1	1	0

<sup>a</sup> within sugar category; <sup>b</sup> within egg-based ingredient category

**Table 2 Furan and furfural content according to experimental design along with response surface models**

	<i>Run</i>	<i>Furan</i>	<i>Furfural</i>		<i>Parameter estimate</i>	<i>Furan</i> <sup>a</sup>	<i>Furfural</i> <sup>a</sup>	
<b>content (ng g<sup>-1</sup> sample, dry basis)</b>	1		179.5 ± 31.1	12297.5 ± 669.3	<b>modeling</b>	<i>Intercept</i>	1.E+02	7.E+01
	2		1.7 ± 0.2	3107.4 ± 53.9		<i>sucrose</i>	6.E-02	-5.E-02
	3		1.7 ± 0.3	1634.1 ± 12.1		<i>whole_egg</i>	1.E-01	5.E-02
	4		4.0 ± 0.2	877.2 ± 26.1		<i>temperature</i>	-1.E+00	-8.E-01
	5		0.5 ± 0.0	53.3 ± 58.4		<i>time</i>	-9.E-01	-2.E-01
	6		0.6 ± 0.1	168.2 ± 31.5		<i>sucrose*sucrose</i>	-2.E-04	-3.E-04
	7		7.1 ± 1.6	1687.3 ± 72.1		<i>sucrose*whole_egg</i>	2.E-04	4.E-05
	8		0.4 ± 0.0	218.8 ± 96.3		<i>whole_egg*whole_egg</i>	-2.E-04	-4.E-04
	9		4.2 ± 0.5	272.9 ± 14.0		<i>sucrose*temperature</i>	-3.E-04	3.E-04
	10		2.4 ± 0.2	70.8 ± 6.5		<i>whole_egg*temperature</i>	-5.E-04	-
	11		0.4 ± 0.1	296.2 ± 16.0		<i>temperature*temperature</i>	4.E-03	3.E-03
	12		4.7 ± 0.4	3751.4 ± 185.8		<i>sucrose*time</i>	-	-
	13		0.4 ± 0.1	26.3 ± 3.3		<i>whole_egg*time</i>	-1.E-03	-6.E-04
	14		721.3 ± 43.0	43861.8 ± 1749.3		<i>temperature*time</i>	7.E-03	3.E-03
	15		0.3 ± 0.0	279.3 ± 19.8		<i>time*time</i>	-	-
	16		155.6 ± 4.3	25781.7 ± 958.4				
	17		4.8 ± 0.5	1606.8 ± 20.4				
	18		176.8 ± 16.7	8710.8 ± 215.6				

means and 95% confidence intervals (n=3)

<sup>a</sup> ln of content