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**Yogurt Enrichment with Grape Pomace: Effect of Grape Cultivar on Physicochemical, Microbiological and Sensory Properties**

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1 **Yogurt enrichment with grape pomace. Effect of grape cultivar on physico-chemical,**  
2 **microbiological and sensory properties**

3

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16

17 **Running head : Grape skin flour and yogurt quality**

18

19 **Keywords :** Yogurt, Grape skin, Grape pomace, Phenolic compounds, Volatile compounds

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27 **Summary**

28 Grape skin flours obtained from grape pomace of Chardonnay, Moscato and Pinot noir  
29 varieties were used as sources of polyphenolic compounds in yogurt formulation during  
30 three weeks of storage. Yogurt containing grape skin flour presented significantly higher  
31 total phenolic content (+55%), antioxidant activity (+80%) and acidity (+25%) whereas  
32 lower pH, syneresis (-10%) and fat (-20%) than control. Procyanidin B1 and vanillic acids  
33 were detected only in the yogurt added of Pinot noir flour while gallic acid, catechin and  
34 quercitrin were the major phenolic compounds found in the yogurts with Moscato or  
35 Chardonnay grape skins. Significant differences were highlighted for acidity and lactose  
36 content while total phenolic content, antioxidant activity and lactic acid bacteria trend were  
37 stable after production and storage. The liking test performed with consumers showed a  
38 loss of textural quality for yogurts fortified with grape skin flours.

39

40

41 **Practical applications**

42 Grape skin is a nutritious, but underused, by-product of winemaking containing fibre and  
43 antioxidants. Using a suitable production design, a new fortified yogurt formulation with  
44 grape by-product could be optimized for enhance antioxidant consumers' daily intake.

45 The use of grape skin flour in the development of value-added food products will be a step  
46 toward making new functional foods, and partially solving waste management problem  
47 from wine production. The results of this study would provide an opportunity of dairy-  
48 producer to develop a novel product in agreement with consumers' preferences. This  
49 research represents a new approach in the development of novel dairy foods with high  
50 nutritional quality and with great potential applications on food industry.

51

52

## 53 **Introduction**

54 Grape (*Vitis vinifera L.*) is one of the world's largest fruit crops. Winemaking process uses  
55 a considerable amount of fresh grape generating a huge mass of solid by-products that  
56 correspond to approximately 13% of the total grape weight. This by-product, usually  
57 referred to as grape pomace (GP), is generated after destemming and pressing grapes and is  
58 composed of grape seeds and skins. The disposal of GP is costly and complicated due to  
59 characteristics of its composition, such as its high sugar content and low pH. If not  
60 properly treated, these characteristics pose a crucial environmental problem (Cheng et al.  
61 2010).

62 Currently GP has different non-food applications: cattle feed (Özvural and Vural 2011),  
63 solid fuel for gas production, compost-fertiliser, effective adsorbent of pollutant heavy  
64 metals and even for the production of high-added value materials (e.g., pullulan and  
65 laccase) (Arvanitoyannis et al. 2006). Because it is well known that GP is an interesting  
66 source of fibre and antioxidants with significant nutritional activities, some research has  
67 been performed towards using GP for food applications. For example, grape skin flour  
68 obtained from GP has been used in baked goods (Walker et al. 2014), corn breakfast cereal  
69 (Camire et al. 2007), and tomato puree (Lavelli et al. 2014) whereas grape seed flour has  
70 been added to bread (Hoye and Ross 2011), meat (Özvural and Vural 2011), cereal bars,  
71 pancakes and noodles (Rosales Soto et al. 2012), and minced fish muscle (Sánchez-Alonso  
72 et al. 2007).

73 GP antioxidants can be considered completely safe in comparison with synthetic  
74 antioxidants and include polyphenol components such as anthocyanins, flavanols,  
75 catechins and proanthocyanidins (Rosales Soto et al. 2012). These compounds have a high  
76 antioxidant activity, which gives them potential health-promoting and disease-protective  
77 effects (Choi et al. 2010; Hogan et al. 2010). For this reason, these compounds have  
78 recently been considered as food additives or novel ingredients that can introduce extra

79 health benefits to various food products (Peng et al. 2010) and, at the same time, could be a  
80 solution for the waste disposal problem.

81 Yogurt is already considered to be a healthy food because it contains viable probiotic  
82 bacteria, however, it does not contain fibre and phenolic antioxidant compounds  
83 (Karaaslan et al. 2011). Available data on the GP addition into yogurt (Tseng and Zhao  
84 2013) are encouraging regarding the feasibility of using GP as novel ingredient. The  
85 objective of this study was to investigate the influence, over three weeks of storage at 4 °C,  
86 of GP addition from different unfermented grape varieties (Chardonnay, Moscato and  
87 Pinot noir) on gross composition, phenolic and volatile compounds, antioxidant activity,  
88 lactic acid bacteria and consumer preferences of yogurt.

89

## 90 **Materials and Methods**

### 91 **Chemicals**

92 n-Hexane, sulphuric acid, sodium hydroxide, ethanol, methanol, trifluoroacetic acid, 2-  
93 octanol, 2,2-diphenyl-1-picrylhydrazyl (DPPH), Folin-Ciocalteu's phenol reagent, sodium  
94 carbonate, pyruvic acid, lactic acid, citric acid, acetic acid, propionic acid, butyric acid,  
95 tartaric acid, malic acid, glucose, lactose, fructose, gallic acid, protocatechuic acid,  
96 procyanidin B1, 2,3,4-trihydroxybenzoic acid, catechin, vanillic acid, epicatechin, rutin  
97 and quercitrin were purchased from Sigma-Aldrich (Milan, Italy). All chemicals were of  
98 reagent or HPLC grade level. Ultra-pure water was produced with a Milli-Q System  
99 (Millipore, Milan, Italy).

100

### 101 **Grape skin flour preparation**

102 Non-fermented GP of three *Vitis vinifera* varieties Chardonnay, Moscato and Pinot noir  
103 were provided from a winemaking factory (Fontanafredda, Alba, Italy). Skins were  
104 mechanically separated, stored at -20 °C until drying, dried in an oven (Memmert, UFE

105 550, Germany) at 54 °C for 48 h and then ground with a Retsch ZM200 grinder (Retsch  
106 Gmbh, Germany) to obtain grape skin flour (GSF) with a particle size of less than 250 µm.  
107 GPF was sterilized in an autoclave at 121 °C for 15 minutes before use in yogurt  
108 production.

109

### 110 **Yogurt production**

111 Yogurt was prepared using UHT whole milk (fat 36.0 g/kg, proteins 31.0 g/kg and  
112 carbohydrates 48.0 g/kg) purchased at the local market. Milk was put in a vat and milk  
113 powder 3% (w/w) was added. When the temperature reached 42 °C, milk was inoculated  
114 with starter culture YO-MIX 401 (Santamaria, Burago di Molgora, Italy), containing a  
115 mixture of *Streptococcus thermophilus* and *Lactobacillus delbrueckii* subsp. *bulgaricus*  
116 (2:1).

117 The inoculated milk was fermented at 42 °C until a final pH of 4.8 was obtained  
118 (approximately 6.5 h). At this point the sterile GPF was mixed with yogurt to reach a  
119 concentration of 60 g/kg and separated into pots. Samples were stored at 4 °C and analyses  
120 were performed immediately after production and at 1, 7, 14, and 21 days of storage. Two  
121 different yogurt productions were realised. Within each production yogurt was divided in  
122 four batches in which one without GSF (Control) and three fortified yogurts (FY) named  
123 Chardonnay, Moscato and Pinot noir.

124

### 125 **Physicochemical characteristics of GPF**

126 The moisture content of the GSF was determined using a Eurotherm EUR thermo-balance  
127 (Gibertini, Milano, Italy) at 105 °C. Protein, fat and ash contents were determined  
128 according to AOAC official methods of analysis (Tseng and Zhao, 2013). The  
129 carbohydrate content was estimated by difference. Dietary fibre (TDT, SDF and IDF) was

130 measured using the Megazyme Total Dietary analysis kit (Lee et al. 1992). All analyses  
131 were performed in triplicate.

132

### 133 **Physicochemical characteristics of yogurt**

134 pH was measured with a Crison microph 2002 pH-metre (Crison Strumenti SpA, Carpi,  
135 Italy). Titratable acidity was determined via a potentiometric method (IDF 1991) and  
136 expressed as lactic acid per 100 g of yogurt. Yogurt syneresis was determined according to  
137 Celik et al. (2006), with some modifications. Yogurt (20 g) was centrifuged at 16,800 ×g  
138 for 20 min at 4 °C using a Megafuge 11 R centrifuge (Thermo Fischer Scientific, Waltham,  
139 MA, USA). Syneresis was expressed as the volume of separated whey per 100 mL of  
140 yogurt (Wacher-Rodarte et al. 1993). Samples were analysed in triplicate.

141

### 142 **Extraction of bioactive compounds**

143 The extraction was carried out according to McCue and Shetty (2005), with slight  
144 modifications. Briefly, each yogurt sample (10 g) was diluted with distilled water (2.5 mL)  
145 and centrifuged (16.800 ×g, 40 minutes, 4 °C). The supernatant was harvested and filtered  
146 through a 0.45-µm polypropylene membrane filter (VWR, Milan, Italy). Extraction was  
147 carried out in triplicate on different pots and extracts were stored at 4 °C until analysis.

148

### 149 **TPC and RSA of yogurt**

150 The total phenolic content (TPC) was determined in triplicate using an assay modified  
151 from Apostolidis et al. (2007). Briefly, 1 mL of extract was transferred into a test tube and  
152 mixed with 1 mL of 95% ethanol and 5 mL of distilled water. To each sample, 500 µL of  
153 50% (v/v) Folin-Ciocalteu reagent were added and the resulting sample was mixed. After 5  
154 min, 1 mL of 5% Na<sub>2</sub>CO<sub>3</sub> was added and the reaction mixture was allowed to stand in the  
155 dark at room temperature for 60 min. Just before the end of the incubation time, samples



156 were centrifuged (16.800 ×g, 10 minutes, 20 °C) and the supernatant absorbance was read  
157 at 725 nm with a UV-visible spectrophotometer (UV-1700 PharmaSpec, Shimadzu, Milan,  
158 Italy). The absorbance values were converted to the total phenolics and were expressed as  
159 micrograms of gallic acid equivalents per gram of sample (µg GAE/g). Standard curves  
160 were established using various concentrations of gallic acid in water ( $R^2= 0.997$ ).

161 The radical scavenging activity (RSA) was determined using the 2,2-diphenyl-1-  
162 picrylhydrazyl radical (DPPH<sup>\*</sup>) assay modified by Gadow et al. (1997). A sample extract  
163 (75 µL or distilled water for the blank) was placed in a test tube, and 3 mL of a  $6 \times 10^{-5}$  M  
164 methanolic solution of DPPH<sup>\*</sup> were added. The decrease in absorbance at 515 nm was  
165 determined at the steady state (60 min of incubation at room temperature in the dark) after  
166 a previous centrifugation step. All determinations were performed in triplicate on different  
167 pots. The inhibition percentage (IP) of the DPPH<sup>\*</sup> by yogurt extracts was calculated  
168 according to the formula

$$169 \text{ IP} = [(A_{0\text{min}} - A_{60\text{min}})/A_{0\text{min}}] \times 100$$

170 where  $A_{0\text{min}}$  is the absorbance of the blank at  $t = 0$  min, and  $A_{60\text{min}}$  is the absorbance of  
171 samples at 60 min.

172

### 173 **HPLC-DAD analysis**

#### 174 **Phenolic compound profiles**

175 HPLC-DAD analysis of yogurt extract was performed using a Thermo-Finnigan Spectra-  
176 System HPLC system (Thermo-Finnigan, Waltham, USA) equipped with a P2000 binary  
177 gradient pump system, a SCM 1000 degasser, an AS 100 automatic injector, a UV6000LP  
178 DAD and the ChromQuest software for data processing. Separation was achieved on a  $C_{18}$   
179 RP Lichrosphere 250 × 4.6 mm, 5 µm (Merck, Milan, Italy) column, equipped with a  $C_{18}$   
180 RP Lichrosphere guard column 5 µm (Merck, Milan, Italy). The mobile phase was  
181 composed of trifluoroacetic acid/ultra-pure water (0.1:99.9, v/v) (A) and methanol (B). The

182 flow rate was 1 mL/min and the injection volume was 20  $\mu$ L. The elution program was as  
183 follows: initial conditions of 95% A, held for 2 minutes, 80% A over 8 min, 25% A over  
184 57 min, 0% A over 13 min, 95% A over 5 min. DAD spectra were recorded in full scan  
185 modality over the wavelength range of 200 to 600 nm and at a discrete wavelength of 525  
186 nm. Identification was achieved by comparing the retention times and spectra with those of  
187 authentic standards. Phenolic compounds were quantified using the following external  
188 standards: gallic acid ( $\lambda_{\max}$ =270,  $R^2$ =0.9998, LOD=0.01 mg/L), procyanidin B1 ( $\lambda_{\max}$ =277,  
189  $R^2$ =0.9997, LOD=0.50 mg/L), (+)-catechin ( $\lambda_{\max}$ =280,  $R^2$ =0.9995, LOD=1.00 mg/L), (-)-  
190 epicatechin ( $\lambda_{\max}$ =280,  $R^2$ =0.9998, LOD=0.50 mg/L), rutin ( $\lambda_{\max}$ =356,  $R^2$ =0.9998,  
191 LOD=0.06 mg/L) and quercitrin ( $\lambda_{\max}$ =350,  $R^2$ =0.9999, LOD=0.09 mg/L). Protocatechuic  
192 acid, 2,3,4-trihydroxybenzoic acid, and vanillic acid were quantified using the gallic acid  
193 calibration curve. The precision, evaluated by calculating the RSD% of the retention time  
194 and the peak area for each analyte collected over a period of 3 weeks, was 1.90-7.89% for  
195 gallic acid, 1.82-10.54% for protocatechuic acid, 1.18-6.04% for procyanidin B1, 1.59-  
196 9.57% for 2,3,4-trihydroxybenzoic acid, 1.32-15.74% for (+)-catechin, 0.29-10.65% for  
197 vanillic acid, 2.13-9.17% for (-)-epicatechin, 1.22-11.36% for rutin, and 1.39-9.34% for  
198 quercitrin.

199

## 200 **Sugar and acid determination**

201 Ion-exchange high-performance liquid chromatography was used to determine the organic  
202 acid and sugar contents. The method of Adhikari et al. (2002) was used with slight  
203 modification.

204 Yogurt samples (5 g) were added to 20 mL of 0.013 N H<sub>2</sub>SO<sub>4</sub> (mobile phase) and mixed  
205 for 30 min with a horizontal shaker (PBI, Milano, Italy) at 100 oscillation/min. The slurry  
206 was subsequently centrifuged for 30 min at 5000  $\times$ g and 10  $^{\circ}$ C and the supernatant was  
207 filtered through a 0.45  $\mu$ m polypropylene membrane filter (VWR, Milan, Italy).

208 The HPLC system (Thermo Quest, San Jose, CA) was equipped with an isocratic pump  
209 (P1000), a multiple autosampler (AS3000) fitted with a 20  $\mu$ L loop, a UV detector  
210 (UV100) set to 210 and 290 nm, and a refractive index detector (Spectra System RI-150,  
211 Thermo Electro Corporation). The detectors were connected in series. Data were collected  
212 using ChromQuest ver. 3.0 (Thermo Finningan).

213 The analyses were performed isocratically at 0.8 mL/min and 65 °C with a 300  $\times$  7.8 mm  
214 i.d. cation exchange column (Aminex HPX-87H) equipped with a cation H<sup>+</sup> microguard  
215 cartridge (Bio-Rad Laboratories, Hercules, CA). The mobile phase was 0.013 N H<sub>2</sub>SO<sub>4</sub>,  
216 which was prepared by diluting reagent grade sulphuric acid with ultrapure water and  
217 degassing under vacuum. Identification was achieved by comparison with retention times  
218 of authentic standards. A total of eight organic acids and three sugars were investigated,  
219 including pyruvic acid, lactic acid, citric acid, acetic acid, propionic acid, butyric acid,  
220 tartaric acid, malic acid, glucose, lactose and fructose.

221

## 222 **Analysis of volatile compounds**

223 The volatile compounds in the yogurt samples were extracted using headspace solid phase  
224 micro-extraction (HS-SPME) and analysed by gas chromatography/mass spectrometry  
225 (GC/MS). The analysis was carried out as described by Coda et al. (2011) with slightly  
226 modifications. All samples were analysed in triplicate. The analysis was conducted using a  
227 20 mL vial filled with 1.5 g of sample to which was added 5  $\mu$ L of 2-octanol in ultra-pure  
228 water (92.8 mg/L) as an internal standard. After an equilibration time of 30 min at 37 °C,  
229 the extraction was performed using the same temperature for 40 min with a 50/30  $\mu$ m  
230 DVB/CAR/PDMS fibre (Supelco, Milan, Italy) with stirring (250 rpm) before injection.  
231 The fibre was desorbed at 260°C for 4 min in splitless mode. GC/MS analysis was  
232 performed with a Shimadzu GC-2010 gas chromatograph equipped with a Shimadzu QP-  
233 2010 Plus quadrupole mass spectrometer (Shimadzu Corporation, Kyoto, Japan) and a DB-

234 WAXETR capillary column (30 m × 0.25 mm, 0.25 μm film thickness, J&W Scientific  
235 Inc., Folsom, CA, USA).

236 The carrier gas (He) flow-rate was 1 mL/min. The temperature program began at 40 °C for  
237 5 min, and then the temperature was increased at a rate of 10 °C/min<sup>1</sup> to 80 °C and 5  
238 °C/min to 240 °C for 5 min. The injection port temperature was 250 °C, the ion source  
239 temperature was 240 °C and the interface temperature was 230 °C. The detection was  
240 carried out by electron impact mass spectrometry in total ion current mode (TIC), using an  
241 ionization energy of 70 eV. The acquisition range was *m/z* 30–330. The identification of  
242 volatile compounds was confirmed by injection of pure standards and the comparison of  
243 their retention indices (a mixture of a homologous series of C<sub>5</sub>-C<sub>28</sub> was used), MS data  
244 reported in the literature and in the database (<http://webbook.nist.gov/chemistry/>).  
245 Compounds for which pure standards were not available were identified on the basis of  
246 mass spectra and retention indices available in the literature. Semiquantitative data (μg/kg)  
247 were obtained by measuring the relative peak area of each identified compound in relation  
248 to that of the added internal standard.

249

### 250 **Microbiological analysis**

251 For each yogurt type, sampling points were analysed using traditional microbiological  
252 methods (CFU). Streptococci were counted on M-17 agar (Oxoid, Milan, Italy) and  
253 lactobacilli were counted on Man Rogosa Shape agar (Oxoid, Milan, Italy). Both medium  
254 were incubated under microaerophilic conditions at 37 °C for 48 h.

255

### 256 **Liking tests**

257 Because a previous acceptance test that was done on a small scale with a restricted panel  
258 (data not shown) indicated that better liking was found for the Moscato and Chardonnay  
259 yogurts, we chose to use only the white varieties for liking test.

260 To assess the sensory acceptability of yogurt samples, a Central Location Test was  
261 conducted in Turin (Italy). The consumer test was performed at a stand for the University  
262 of Gastronomic Sciences during a public event named “European Researchers’ Night”. 256  
263 regular consumers of yogurt (48% males, 52% females, 18-86 years, mean age 24)  
264 voluntarily participated in the sensory evaluation. Written informed consent was obtained  
265 from each subject after the experiment was described to them.

266 The test consisted of a sensory evaluation of the fortified yogurts (Moscato and  
267 Chardonnay) and of the control sample. Yogurt samples (10 g) were served under blind  
268 conditions in opaque white plastic cups (38 mL) sealed with a clear plastic lid and coded  
269 with a random three-digit number. Samples were served in completely randomized order,  
270 with the control served as the last sample for all subjects to limit the contrast effect  
271 (Meilgaard et al. 2006). Consumers were asked to stir each sample with a plastic teaspoon,  
272 observe its appearance, smell and taste it, and rate the yogurts for appearance, odour, taste,  
273 flavour, texture and overall acceptance. Liking was expressed on a 9-point hedonic scale  
274 ranging from ‘dislike extremely’ (1) to ‘like extremely’ (9) (Peryam and Pilgrim 1957).  
275 Purchase interest (*Would you buy this yogurt?*) was also rated on a 7-point scale (1=  
276 absolutely no, 7= absolutely yes). Participants were required to rinse their mouth with still  
277 water for about one minute between samples. Consumers took between 15 and 20 minutes  
278 to complete the evaluation. Liking data (appearance, odour, taste, flavour, texture and  
279 overall acceptance) and declared purchase interest from consumers were independently  
280 submitted to a two-way ANOVA model, assuming sample and subject as main effects, by  
281 performing LSD ( $p < 0.05$ ).

282

### 283 **Data analysis**

284 A one-way analysis of variance (ANOVA) with Duncan’s test for mean comparison was  
285 used to highlight significant differences among samples. All calculations were performed

286 using the STATISTICA for Windows statistical software (Release 7.0; StatSoft Inc., Tulsa,  
287 OK, USA).  
288

## 289 **Results and Discussion**

### 290 **Chemical composition of GSF and yogurts**

291 Fat values were significantly different among varieties, with the lowest value for Pinot  
292 noir, probably due to more loss of grape seeds during preparation of the GSF (Table 1).  
293 Pinot noir showed also the lowest protein value (88.3 g/kg), whereas the highest was for  
294 Chardonnay, at 97.0 g/kg. The highest values of soluble, insoluble and total dietary fibre  
295 were found in Moscato (90.2, 390.9 and 481.0 g/kg respectively) followed by Chardonnay  
296 and Pinot noir.

297 Concerning fortified yogurt (FY), the lowest protein contents (Table 2) were observed in  
298 Pinot noir (208.4 g/kg) and Chardonnay (216.5 g/kg) yogurts, while the highest was found  
299 in Moscato yogurt (246.5 g/kg). Fat evaluation revealed that FY containing Pinot noir had  
300 a lower value than yogurt containing Moscato, with fat contents of 214.4 and 242.9 g/kg  
301 ( $p<0.05$ ), respectively.

302 Carbohydrates concentration were significantly different between FY samples; they were  
303 higher in Pinot noir yogurt, followed by Chardonnay and Moscato yogurts. Moisture was  
304 significantly different between yogurts and the Moscato FY had the highest value,  
305 followed by yogurt containing Chardonnay and Pinot noir.

306

### 307 **pH, acidity and syneresis of yogurt**

308 High significant differences ( $p<0.001$ ) were found for pH with respect to storage time and  
309 yogurt type, except on the 14<sup>th</sup> day (Table 3). The addition of GSF to yogurt instantly  
310 reduced the pH from 4.59 to 4.22-4.26, as previously reported by Tseng and Zhao (2013).  
311 The reduction in pH during storage corresponded to an increase in acidity (Tseng and  
312 Zhao, 2013). The highest increase was found in Moscato yogurt (+17.9%), while the  
313 lowest observed was for Pinot noir yogurt (+11.4%).

314 Fortified yogurts had higher values of syneresis compared to the control during storage due  
315 to the addition of GSF and statistically differences were found between yogurt types  
316 ( $p < 0.001$ , except on the 1<sup>st</sup> day) whereas no differences were found with respect to storage  
317 time ( $p > 0.05$ ). The IDF present in GSF causes a rearrangement of the matrix gel, which  
318 was previously observed by García-Pérez et al. (2005) and Tseng and Zhao (2013).  
319 Chardonnay yogurt exhibited the highest value at each sampling time, while Pinot noir  
320 exhibited the lowest.

321

### 322 **TPC and RSA of yogurt**

323 As expected, all fortified yogurts exhibited a high and statistically significantly increase in  
324 the total phenolic content compared to the control yogurt (about 38%, 54% and 66% for  
325 Moscato, Chardonnay and Pinot noir respectively) at each sampling time (Table 3).

326 The TPC was stable generally during storage for all samples and only Moscato yogurt  
327 showed statistically differences during the storage time ( $p < 0.05$ ). The DPPH• values  
328 indicated that all FYs had higher antiradical activity compared to the control. The RSA did  
329 not decrease significantly during storage for FYs, whereas it changed significantly in the  
330 control yogurt ( $p < 0.05$ ). The RSA control value was lower on the 21<sup>st</sup> day of sampling than  
331 for day 0, with a reduction of 75%. Similar studies (Karaaslan et al. 2011; Tseng and Zhao,  
332 2013) stated that the RSA dropped during storage in yogurt containing 10% of red grape  
333 extract and yogurt containing 3%, 2% and 1% of red wine grape pomace. As expected, in  
334 our work, yogurt containing Pinot noir grape skin flour exhibited the highest RSA during  
335 all storage times, whereas there was no statistically significant difference between yogurt  
336 containing Moscato and Chardonnay.

337

338

339



### 340 **Sugar and organic acid contents**

341 The glucose values were higher in FYs compared to the control due to the addition of GSF  
342 (Table 3) and were very different at each time of sampling ( $p<0.001$ ). The control and FY  
343 containing Pinot noir were also significantly different during storage (respectively  $p<0.01$   
344 and  $p<0.001$ ). The glucose content dropped during storage in the control, with a reduction  
345 of 38% between 0 and 21 days of storage ( $p<0.01$ ). The glucose content of FY containing  
346 Pinot noir increased on the 1<sup>st</sup> day (10.62 g/L) and remained approximately the same until  
347 the 14<sup>th</sup> day (10.67 g/L), followed by a decrease at the last sampling time (10.29 g/L). This  
348 trend could be explained by the dissolution of glucose from GSF into yogurt. Changes in  
349 the glucose contents of Moscato and Chardonnay yogurts were not significant during the  
350 storage time ( $p>0.05$ ). As expected, the lactose content decreased during storage in all  
351 yogurts. Lactose content at the beginning of storage was approximately 36 g/L in FY,  
352 while at the end it was approximately 33 g/L. Fructose was observed in all FYs, and the  
353 highest content was found in Pinot noir yogurt, followed by Chardonnay and Moscato  
354 yogurts. As expected, the content of lactic acid increased during storage in all yogurts, and  
355 by a higher percentage in the control yogurt than in FY. As a consequence, large  
356 statistically significant differences were found at each sampling time among yogurt type  
357 ( $p<0.001$ ). Citric acid content was similar among FYs but slightly different from control  
358 yogurt ( $p<0.05$ ) and storage did not affect its content in the yogurts ( $p>0.05$ ). Malic and  
359 tartaric acids are the most important organic acids of grape and they were found in all FYs.  
360 FY containing Pinot noir exhibited the lowest content of tartaric acid during storage (1.72-  
361 2.05 g/L), while FY containing Moscato and Chardonnay showed similar values, except at  
362 0 and 14<sup>th</sup> day of storage. During storage, highly significant differences were observed in  
363 the malic acid contents of Moscato and Chardonnay yogurts ( $p<0.001$ ), which exhibited a  
364 decreasing trend, while that of Pinot noir did not change during storage and had the highest

365 values at each sampling time (0.48-0.51 g/L). The lowest values were found in Moscato  
366 yogurt (0.15-0.19 g/L). Butyric, propionic and acetic acids were not found in any yogurt.

367

### 368 **Profiles of phenolic compounds**

369 A total of nine compounds were identified and quantified: gallic acid, protocatechuic acid,  
370 procyanidin B1 (PB1), 2,3,4-trihydroxybenzoic acid (THA), catechin, vanillic acid,  
371 epicatechin, rutin and quercitrin (Table 4). None of these phenolic compounds were  
372 detected in control yogurt. In yogurt containing Moscato and Chardonnay GSF, gallic acid,  
373 protocatechuic acid, catechin, epicatechin, rutin and quercitrin were detected, while all  
374 phenolic compounds except for epicatechin were detected in yogurt containing Pinot noir  
375 GSF.

376 Statistically significant differences were found between yogurt types with respect to gallic  
377 acid, while there were no statistically significant differences within each yogurt type  
378 during storage.

379 Moscato FY exhibited the highest gallic acid content (3.6-4.2  $\mu\text{g/g}$ ), followed by FY  
380 containing Chardonnay and Pinot noir. Protocatechuic acid was detected in all types of  
381 FYs, and its content did not change significantly during storage ( $p>0.05$ ). The only  
382 significant difference for protocatechuic acid content was found on the 14<sup>th</sup> day, in which  
383 reporting levels of protocatechuic acid decreased in the following order: Moscato >  
384 Chardonnay > Pinot noir. PB1 and THA were only detected in Pinot noir yogurt and their  
385 contents did not change during storage ( $p>0.05$ ). The PB1 content ranged from 26 to 30  
386 mg/g. Catechin was the predominant polyphenol in all fortified yogurts, with the highest  
387 levels in Moscato yogurt on the first day (19.3  $\mu\text{g/g}$ ) and Chardonnay yogurt on day 0  
388 (22.9  $\mu\text{g/g}$ ). Its content did not change significantly during storage ( $p>0.05$ ). On the 1<sup>st</sup>, 7<sup>th</sup>  
389 and 14<sup>th</sup> day of storage, statistically significant differences in catechin content were found  
390 between the yogurt types. Yogurts containing Moscato and Chardonnay exhibited higher

391 levels of catechin compared to yogurt containing Pinot noir. Epicatechin was present at  
392 similar levels in Moscato and Chardonnay yogurts. During the storage of these yogurts, the  
393 epicatechin content did not change significantly ( $p>0.05$ ). According to Karaaslan et al.  
394 (2011), the catechin concentration was higher than epicatechin in yogurt to which grape  
395 callus extract had been added (*Vitis vinifera* cv. Merlot).

396 Vanillic acid was exclusively detected in Pinot noir yogurt, in which its content did not  
397 change significantly during storage ( $p>0.05$ ).

398 Rutin was detected in all three FYs, with higher values in Pinot noir (1<sup>st</sup> and 14<sup>th</sup> day) than  
399 in Chardonnay and Moscato yogurts ( $p<0.001$ ) and its content did not change significantly  
400 during the storage of the three yogurts ( $p>0.05$ ).

401 A higher content of quercitrin was found at day 21 in Chardonnay yogurt with respect to  
402 Moscato and Pinot noir yogurts ( $p<0.05$ ). At days 14 and 21, the Pinot noir yogurt was  
403 characterized by the lowest amount of quercitrin (respectively 4.7 and 4.6  $\mu\text{g/g}$ ). Quercitrin  
404 content did not change significantly during the storage, except for the Chardonnay yogurt  
405 for which a slight increase in the quercitrin level was observed at day 21. This could be due  
406 to an increase in compound solubilisation into the yogurt, due to its ability to be extracted  
407 into water.

408

#### 409 **Analysis of volatile compounds**

410 A total of 48 compounds were found in control and FYs, which corresponded to 10 ketones  
411 (2-pentanone; 2,3-pentanedione (diacetyl); 2-heptanone; acetoin; 6-methyl-5-hepten-2-one;  
412 3-hydroxy-2-pentanone; 2-nonanone; 6-methyl-3,5-heptadien-2-one; 2-undecanone; 2-  
413 tridecanone), four aldehydes (nonanal; benzaldehyde; 4-methylbenzaldehyde; dodecanal),  
414 12 alcohols (isobutanol; 1-pentanol; 3-methyl-1-butanol; 1-hexanol; 2-hexen-1-ol; 1-octen-  
415 3-ol; 1-octanol; 1-nonanol; benzyl alcohol; phenylethyl alcohol; 1,4-butanediol; 1-  
416 dodecanol), 11 acids (acetic acid; isobutyric acid; butanoic acid; methacrylic acid;

417 pentanoic acid; hexanoic acid; 2-ethyl-hexanoic acid; heptanoic acid; octanoic acid;  
418 nonanoic acid; benzenecarboxylic acid), one ester ( $\beta$ -phenylethyl acetate), two lactones ( $\gamma$ -  
419 caprolactone;  $\delta$ -decalactone), three furan derivatives (2-pentyl-furan; furfural; 2-  
420 furanmethanol), four terpenoids (limonene; *cis*-linalool oxide; linalool;  $\alpha$ -terpineol) and  
421 one phenol (phenol). Table 5 displays the sums of all of the volatile compounds in each of  
422 these chemical classes. Carbonyl compounds, such as aldehydes and ketones, are the major  
423 volatile compounds responsible for the desirable flavour of yogurt (Cheng, 2010). Their  
424 content is affected by the symbiotic relationship that occurs between *S. thermophilus* and  
425 *Lb. bulgaricus* that are added as starter cultures (Routray and Mishra, 2011). As reported in  
426 Table 5, ketones were the most abundant compounds observed, and their values increased  
427 significantly during storage in all three FYs ( $p < 0.001$ ). Highly statistically significant  
428 differences ( $p < 0.001$ ) were found between yogurt type at sampling days 0 and 21. On the  
429 21<sup>st</sup> day of storage, the contents of ketones found in control and yogurt containing Pinot  
430 noir, 1153.28 and 1092.65  $\mu\text{g}/\text{kg}$ , respectively, were lower compared with those found in  
431 white grape varieties. The ketone contents of yogurts containing Moscato and Chardonnay  
432 were not significantly different. The ketone content increased at a rate of 11% (control),  
433 23% (Pinot), 47% (Moscato) and 55% in Chardonnay. Of the ketones, 2,3-pentanedione, 2-  
434 heptanone and acetoin were the most abundant (data not shown), and they play an  
435 important role in yogurt flavour, as reported by Routray and Mishra (2011). The most  
436 abundant aldehyde was benzaldehyde. Its content ranged (data not shown) from 2.63  
437 (control at 14<sup>th</sup> day) to 15.89  $\mu\text{g}/\text{kg}$  (Moscato at 14<sup>th</sup> day). Moreover, all FYs demonstrated  
438 higher amounts of these volatile compounds compared to the control. Sánchez-Palomo et  
439 al. (2005) studied the volatile compound contents of the pulp and skin of Muscat grapes,  
440 and reported that benzaldehyde was found in its skin. The same was found in Chardonnay  
441 grape skin and juice by Rosillo et al. (1999). We could confirm a major portion of the  
442 benzaldehyde content is due to the addition of GSF.

443 On the 21<sup>st</sup> day of storage FYs containing Moscato and Chardonnay exhibited higher  
444 amounts of aldehydes compared to the Pinot noir and control yogurts.

445 The amount of alcohols increased during yogurt shelf-life in fortified yogurt, and their  
446 levels were higher in FYs compared to the control. Moscato and Chardonnay showed an  
447 average of ~300 µg/kg of alcohols during storage, which was higher compared to the  
448 alcohol content in Pinot noir yogurt (~140 µg/kg). In FYs containing Moscato, phenylethyl  
449 alcohol was the most abundant alcohol observed, and it ranged from 92.19 µg/kg (21<sup>st</sup> day)  
450 to 157.69 µg/kg (14<sup>th</sup> day). This alcohol was also the most abundant compound found in  
451 Moscato skin flour according to Sánchez-Palomo et al. (2005). The acid content within  
452 yogurt types and sampling time was always highly significantly different ( $p < 0.001$ ), except  
453 for FY containing Moscato ( $p < 0.01$ ). The total acids increased during storage (21<sup>st</sup> day > 0  
454 day) in all yogurts except for Chardonnay. The percentage increase was 90% (control),  
455 24% (Moscato) and 31% for Pinot noir. FY exhibited higher acid values compared to  
456 control yogurt during storage, which is due to the typical acidity of GSF and the microbial  
457 activity of starter microorganisms. On the 21<sup>st</sup> day of storage, FYs containing Moscato and  
458 Pinot noir exhibited the highest acid levels compared to yogurt containing Chardonnay.

459 Esters were represented by  $\beta$ -phenylethyl acetate, which was found in all fortified yogurts.  
460 The amount of this ester was higher in Moscato and Chardonnay (15.62 and 12.32 µg/kg,  
461 respectively), whereas less than 1 µg/kg was found in Pinot and control yogurts.

462 Lactones originate from lipolysis that occurs during yogurt fermentation, in which  
463 unsaturated fatty acids lead to the formation of 4- or 5-hydroxyacids that readily cyclise to  
464  $\gamma$ - or  $\delta$ -lactones (Cheng, 2010). The trend of the total lactones in control and FY containing  
465 Chardonnay was not statistically significant during the storage time ( $p > 0.05$ ). On the 21<sup>st</sup>  
466 day of storage, the highest total lactone content was found in yogurt containing  
467 Chardonnay (4.00 µg/kg), followed by yogurt containing Moscato (2.35 µg/kg).

468 The amount of furan derivatives in samples was significantly higher in FY ( $p<0.001$ )  
469 compared to the control, probably due to the drying and sterilization process used to  
470 prepare grape skin flour before yogurt production.

471 During all sampling times, the highest levels of terpenes were found in Moscato yogurt,  
472 which was expected because Moscato grape is an aromatic variety characterized by  
473 linalool, geraniol and nerol (Sánchez-Palomo et al. 2005). Varietal terpenoids such as  
474 limonene, *cis*-linalool oxide, and  $\alpha$ -terpineol increased in FY containing Moscato skin  
475 flour during storage ( $p<0.001$ ), probably due to release from aromatic grape skin, whereas  
476 they decreased in FY containing Chardonnay.

477

#### 478 **Microbiological analyses**

479 The addition of grape skin flour to yogurt did not affect the survival of starter strains  
480 during storage conditions and both *Streptococcus thermophilus* and *Lactobacillus*  
481 *delbrueckii* subsp. *bulgaricus* survived the addition of flours in all FY. After 21 days, *S.*  
482 *thermophilus* reached a concentration very similar to the control in all three FYs (data not  
483 reported). The final concentration of *S. thermophilus* in control yogurt was 9.33 log  
484 CFU/mL, whereas for FY the average concentration was 9.20 log CFU/mL.

485 The same trend was recorded for *L. bulgaricus*, which, at the end reached a lower  
486 concentration approximately 7.8 log CFU/mL for all yogurt tested compared to *S.*  
487 *thermophilus*. This result was expected, as a different amounts of starter were added to the  
488 product (ratio of 2:1 *S. thermophilus* : *L. bulgaricus*).

489

#### 490 **Liking test**

491 The effect of fortification on overall consumer liking and purchase interest for yogurts is  
492 shown in Figure 1. A significant difference was found in liking among samples based on  
493 appearance ( $F = 22.74$ ;  $p<0.0001$ ), odour ( $F = 42.80$ ;  $p<0.0001$ ), taste ( $F = 125.46$ ;

494  $p < 0.0001$ ), flavour ( $F = 72.84$ ;  $p < 0.0001$ ), texture ( $F = 40.50$ ;  $p < 0.0001$ ), overall liking ( $F$   
495  $= 102.04$ ;  $p < 0.0001$ ), and purchase interest ( $F = 54.98$ ;  $p < 0.0001$ ). The control sample was  
496 acceptable and exhibited the highest scores for its appearance, odour, taste, flavour and  
497 texture. In general, the results for the fortified yogurts distinguished them from each other.  
498 Both of them had a low liking score that never reached the central value of the scale (5 =  
499 neither like nor dislike). The Moscato yogurt was disliked more, with a very low mean  
500 liking score, especially for taste and flavour. In contrast, Chardonnay was the sample with  
501 the highest mean scores for appearance, flavour and overall liking. Considering the overall  
502 liking, Chardonnay yogurt was significantly better liked than Moscato yogurt. Thus,  
503 samples prepared with Chardonnay reported a generally higher hedonic performance than  
504 samples fortified with Moscato, suggesting a more suitable use in combination with yogurt.  
505 The results for purchase interest were highly correlated to overall liking, ( $r^2 = 0.9996$ ),  
506 demonstrated the key role of liking on declared buying behaviour. Sensory evaluation  
507 results suggested the need of further optimization of prototypes, indicating as Chardonnay  
508 grape skin flour as most suitable for use in this application. In general, the observed low  
509 acceptability for FYs was not surprising because a decrease in liking due to fortification  
510 was expected. Indeed, the addition of bioactive compounds or plant-based phytonutrients  
511 can result in a change in the sensory quality of enriched foods, which can strongly affect  
512 the consumers' acceptance of such foods (Verbeke, 2006). Verbal comments informally  
513 collected by participants after the end of the test, indicated that the fortified yogurts were  
514 perceived as "too sour", "not enough sweet", with "unpleasant flavours", "not  
515 homogeneous", and "grainy/sandy". It is probable that the unpleasant texture was due to  
516 the perception of the grape skin flour particles.

517 It should be taken into account that the mean overall liking score obtained for the control  
518 sample was just above the acceptability limit. Therefore, it can be hypothesized that the  
519 fortification of a more pleasant control yogurt could induce a similar decrease in the liking

520 score, resulting in an overall liking above the acceptability limit (e.g., starting from an  
521 overall liking of eight, a decrease in two points of the liking score would result in a final  
522 overall liking equal to six, which would be higher than the acceptability limit).

523 In the future, it would be interesting to investigate the consumers' acceptance of the  
524 fortified yogurt under informed conditions instead of in a blind test. Indeed, it has been  
525 demonstrated that information regarding the health benefits of grape skin flour fortification  
526 can increase the consumers' acceptance of fortified products (Cheng et al. 2010).

527

## 528 **Conclusion**

529 The feasibility of using grape skin pomace as an ingredient in yogurt production was  
530 evaluated. The addition of grape skin flour to yogurt resulted in a significant increase in the  
531 TPC and RSA with respect to control yogurt. The TPC and RSA values of fortified yogurts  
532 were retained during yogurt storage and no significant changes were observed. Regarding  
533 the differences found between grape cultivars, yogurt containing Pinot noir, a red cultivar,  
534 showed the highest TPC and RSA values. At the same time, phenolic compounds, which  
535 were only found in FY, were not influenced by storage. It is noteworthy that the addition of  
536 grape skin flour did not affect the survival of starter strains during storage. The results  
537 obtained based on acceptance testing suggested that Pinot noir cannot be used for addition  
538 to yogurt due to the production of an undesirable aroma.

539 Results of the liking tests suggested that obtaining a higher preference by consumers will  
540 require decreasing the sour taste perception (by using sweeteners or a different yogurt with  
541 a lower acidity) and improving the texture by using grape skin flour with a lower particle  
542 size.

543 The results obtained in this study demonstrated that grape skin flour could be an alternative  
544 and safe source of antioxidants in the daily diet. Grape skin might be used in dairy



545 applications, in particular for yogurt production, which could be a new way to use grape  
546 by-products.

547

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550

#### 551 **Declaration of interest**

552 The authors report no conflict of interest. The authors alone are responsible for the content  
553 and the writing of this article. All procedures performed in studies involving human  
554 participants were in accordance with the ethical standards of the institutional and/or  
555 national research committee and with the Helsinki Declaration of 1964 and its later  
556 amendments or comparable ethical standards.

557

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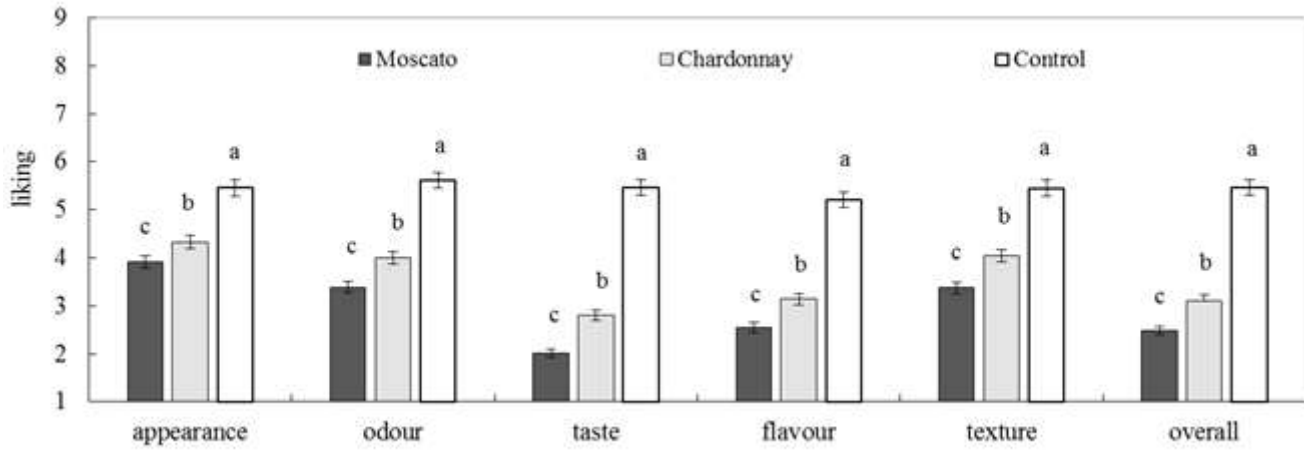
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647 **Fig. 1.** LIKING OF APPEARANCE, ODOUR, TASTE, FLAVOUR, TEXTURE AND  
 648 OVERALL LIKING EXPRESSED BY 256 CONSUMERS FOR THE CONTROL AND  
 649 FORTIFIED YOGURTS.



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651 Means within a sensory modality with different letters are significantly different; Fisher's

652 test,  $P \leq 0.05$ ; error bars are standard deviations of means

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666 **Table 1.** CHEMICAL COMPOSITION OF GRAPE SKIN FLOUR AND RESULTS OF  
 667 ANALYSIS OF VARIANCE WITH DUNCAN'S TEST

<b>Chemical parameters</b>	<b>Moscato</b>	<b>Chardonnay</b>	<b>Pinot noir</b>	<i>Significance</i>
Protein <sup>‡</sup>	93.5±3.7 <sup>b</sup>	97.0±0.3 <sup>c</sup>	88.3±1.1 <sup>a</sup>	**
Fat	50.1±1.6 <sup>c</sup>	41.0±1.1 <sup>b</sup>	23.2±1.1 <sup>a</sup>	***
Carbohydrates	271.4±0.4 <sup>a</sup>	326.8±1.6 <sup>b</sup>	501.2±3.8 <sup>c</sup>	***
Moisture	57.9±0.5 <sup>c</sup>	45.2±1.1 <sup>b</sup>	20.8±0.9 <sup>a</sup>	***
Ash	45.9±0.6 <sup>b</sup>	63.9±0.2 <sup>c</sup>	20.9±0.7 <sup>a</sup>	***
IDF	390.9±0.5 <sup>c</sup>	346.3±3.9 <sup>b</sup>	285.0±1.5 <sup>a</sup>	***
SDF	90.2±1.7 <sup>c</sup>	81.5±1.1 <sup>b</sup>	62.9±0.5 <sup>a</sup>	***
TDF	481.0±1.2 <sup>c</sup>	426.2±0.12 <sup>b</sup>	345.5±3.5 <sup>a</sup>	***

668 <sup>‡</sup>The results are reported as g/kg of dry weight and represented as means ± standard deviation

669 IDF – insoluble dietary fibre; SDF – soluble dietary fibre; TDF – total dietary fibre)

670 <sup>a-c</sup> Different letters within a column are significantly different (P < 0.05)

671 \*\*\* P<0.05; \*\*\*\* P<0.01

672

673 **Table 2.** CHEMICAL COMPOSITION OF CONTROL AND FORTIFIED YOGURTS  
 674 AND RESULTS OF ANALYSIS OF VARIANCE WITH DUNCAN'S TEST

	<b>Moscato</b>	<b>Chardonnay</b>	<b>Pinot noir</b>	<b>Control</b>	<i>Significance</i>
Protein <sup>‡</sup>	246.5±9.4 <sup>b</sup>	216.5±3.5 <sup>a</sup>	208.4±4.8 <sup>a</sup>	260.4±8.1 <sup>c</sup>	***
Fat	242.9±3.8 <sup>c</sup>	236.5±1.3 <sup>b</sup>	214.4±2.8 <sup>a</sup>	311.3±3.4 <sup>d</sup>	***
Carbohydrates	461.3±1.6 <sup>b</sup>	488.2±5.0 <sup>c</sup>	528.3±5.5 <sup>d</sup>	365.9±8.6 <sup>a</sup>	***
Moisture	839.1±0.6 <sup>c</sup>	829.9±0.2 <sup>b</sup>	827.1±0.4 <sup>a</sup>	858.0±1.2 <sup>d</sup>	***
Ash	57.0±0.5 <sup>ab</sup>	58.0±1.5 <sup>b</sup>	55.3±1.1 <sup>a</sup>	61.8±1.3 <sup>c</sup>	***

675 <sup>‡</sup>The results are reported as g/kg of dry weight and represented as means ± standard deviation

676 <sup>a-d</sup> Different letters within a column are significantly different (P< 0.05)

677 \*\*\* P<0.01

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686 **Table 3.** PHYSICO-CHEMICAL PARAMETERS OF CONTROL AND FORTIFIED  
 687 YOGURTS DURING STORAGE AND RESULTS OF ANALYSIS OF VARIANCE  
 688 WITH DUNCAN'S TEST

Parameter <sup>‡</sup>	Days	Control	Moscato	Chardonnay	Pinot noir	Significance
pH	0	4.59±0.02 <sup>cB</sup>	4.22±0.02 <sup>eA</sup>	4.26±0.01 <sup>eA</sup>	4.24±0.01 <sup>cA</sup>	***
	1	4.52±0.13 <sup>cB</sup>	4.12±0.01 <sup>dA</sup>	4.15±0.01 <sup>dA</sup>	4.13±0.01 <sup>bA</sup>	***
	7	4.30±0.02 <sup>bC</sup>	4.07±0.01 <sup>cA</sup>	4.09±0.10 <sup>cA</sup>	4.12±0.10 <sup>bB</sup>	***
	14	4.00±0.04 <sup>aB</sup>	3.90±0.02 <sup>bA</sup>	3.92±0.01 <sup>bA</sup>	3.96±0.02 <sup>aAB</sup>	**
	21	4.00±0.01 <sup>aD</sup>	3.86± <sup>aA</sup>	3.88±0.01 <sup>aB</sup>	3.93± <sup>aC</sup>	***
	Significance	***	***	***	***	
Acidity (lactic acid %)	0	0.72±0.03 <sup>aA</sup>	0.90± <sup>aB</sup>	0.89±0.01 <sup>aB</sup>	0.92±0.01 <sup>aB</sup>	***
	1	0.79± <sup>bA</sup>	0.96± <sup>bC</sup>	0.94±0.01 <sup>aB</sup>	0.97± <sup>bC</sup>	***
	7	0.89± <sup>cA</sup>	1.00±0.02 <sup>cB</sup>	1.01±0.03 <sup>bB</sup>	1.00±0.01 <sup>cdB</sup>	**
	14	0.99±0.01 <sup>d</sup>	1.04±0.01 <sup>d</sup>	1.06±0.04 <sup>b</sup>	0.99±0.01 <sup>bc</sup>	ns
	21	0.99± <sup>dA</sup>	1.07±0.01 <sup>eC</sup>	1.04±0.02 <sup>bBC</sup>	1.02±0.01 <sup>dB</sup>	**
	Significance	***	***	**	**	
Syneresis (%)	0	32.73±0.31 <sup>A</sup>	45.49±0.39 <sup>C</sup>	49.60±0.35 <sup>D</sup>	43.05±0.28 <sup>B</sup>	***
	1	32.34±0.91 <sup>A</sup>	46.92±1.99 <sup>C</sup>	50.86±2.21 <sup>D</sup>	42.87±1.11 <sup>B</sup>	**
	7	33.38±0.25 <sup>A</sup>	46.39±0.58 <sup>C</sup>	48.33±0.32 <sup>D</sup>	43.21±0.63 <sup>B</sup>	***
	14	34.03±0.35 <sup>A</sup>	46.03±0.57 <sup>C</sup>	48.43±1.27 <sup>D</sup>	43.34±0.19 <sup>B</sup>	***
	21	32.82±0.18 <sup>A</sup>	45.82±0.33 <sup>C</sup>	48.15±0.67 <sup>D</sup>	43.13±0.42 <sup>B</sup>	***
	Significance	ns	ns	ns	ns	
TPC (µg GAE.g <sup>-1</sup> )	0	9.38 ± 0.04 <sup>A</sup>	12.88 ± 0.60 <sup>Bab</sup>	13.96 ± 0.66 <sup>B</sup>	15.83 ± 1.13 <sup>C</sup>	***
	1	9.17 ± 0.05 <sup>A</sup>	13.30 ± 0.42 <sup>Bb</sup>	14.43 ± 0.11 <sup>C</sup>	15.14 ± 0.10 <sup>D</sup>	***
	7	9.30 ± 0.13 <sup>A</sup>	12.23 ± 0.20 <sup>Ba</sup>	13.60 ± 0.73 <sup>C</sup>	15.09 ± 0.58 <sup>D</sup>	***
	14	9.40 ± 0.01 <sup>A</sup>	12.21 ± 0.07 <sup>Ba</sup>	13.94 ± 0.01 <sup>C</sup>	14.61 ± 0.08 <sup>D</sup>	***
	21	9.35 ± 0.17 <sup>A</sup>	12.94 ± 0.46 <sup>Bab</sup>	14.37 ± 0.46 <sup>C</sup>	15.25 ± 0.51 <sup>D</sup>	***
	Significance	ns	*	ns	ns	
RSA (-i%)	0	20.21 ± 3.31 <sup>Ac</sup>	23.35 ± 1.12 <sup>A</sup>	23.98 ± 1.64 <sup>A</sup>	30.79 ± 2.80 <sup>B</sup>	*
	1	13.37 ± 0.50 <sup>Abc</sup>	22.60 ± 1.99 <sup>B</sup>	25.29 ± 3.11 <sup>BC</sup>	29.04 ± 0.76 <sup>C</sup>	**
	7	12.31 ± 0.42 <sup>Abc</sup>	18.88 ± 4.29 <sup>AB</sup>	18.95 ± 4.68 <sup>AB</sup>	28.24 ± 1.76 <sup>B</sup>	*
	14	12.18 ± 0.55 <sup>Aab</sup>	17.62 ± 5.28 <sup>A</sup>	18.61 ± 1.80 <sup>AB</sup>	28.86 ± 1.18 <sup>B</sup>	*
	21	11.53 ± 0.61 <sup>Aa</sup>	18.97 ± 1.54 <sup>B</sup>	20.67 ± 1.11 <sup>B</sup>	25.31 ± 0.68 <sup>C</sup>	***
	Significance	*	ns	ns	ns	
Glucose (g.L <sup>-1</sup> )	0	1.53±0.08 <sup>cA</sup>	4.94±0.11 <sup>B</sup>	7.13±0.01 <sup>C</sup>	10.13±0.05 <sup>dD</sup>	***
	1	1.33±0.12 <sup>bcA</sup>	5.20±0.09 <sup>B</sup>	7.46±0.27 <sup>C</sup>	10.62±0.06 <sup>cdD</sup>	***
	7	1.27±0.11 <sup>abA</sup>	4.95±0.09 <sup>B</sup>	7.21±0.03 <sup>C</sup>	10.57±0.01 <sup>cdD</sup>	***
	14	1.07±0.13 <sup>aA</sup>	4.78±0.31 <sup>B</sup>	7.16±0.20 <sup>C</sup>	10.57±0.10 <sup>cdD</sup>	***
	21	1.11±0.13 <sup>aA</sup>	4.90±0.23 <sup>B</sup>	7.25± <sup>C</sup>	10.29±0.13 <sup>bdD</sup>	***
	Significance	**	ns	ns	***	
Lactose (g.L <sup>-1</sup> )	0	41.37±0.47 <sup>dB</sup>	36.40±0.14 <sup>dA</sup>	36.02±0.10 <sup>cA</sup>	36.24±0.13 <sup>cA</sup>	***
	1	41.15±0.40 <sup>dB</sup>	35.42±0.26 <sup>cA</sup>	35.30±0.09 <sup>cA</sup>	36.18±0.35 <sup>cA</sup>	***
	7	39.71±0.10 <sup>cdD</sup>	34.53±0.14 <sup>bc</sup>	33.86±0.09 <sup>bA</sup>	34.19±0.24 <sup>bB</sup>	***
	14	37.52±0.21 <sup>bb</sup>	33.66±0.35 <sup>aA</sup>	32.41±0.97 <sup>aA</sup>	33.47±0.39 <sup>aA</sup>	**
	21	35.85±0.66 <sup>ab</sup>	33.32±0.19 <sup>aA</sup>	33.15±0.04 <sup>abA</sup>	33.62±0.18 <sup>aB</sup>	***
	Significance	***	***	***	***	
Fructose (g.L <sup>-1</sup> )	0	nd	7.32±0.04 <sup>aA</sup>	8.70±0.16 <sup>aB</sup>	12.36±0.22 <sup>aC</sup>	***
	1	nd	7.75±0.09 <sup>bA</sup>	9.32±0.03 <sup>bB</sup>	13.13±0.17 <sup>bC</sup>	***
	7	nd	7.91±0.02 <sup>bA</sup>	9.51±0.13 <sup>bB</sup>	13.26±0.11 <sup>bC</sup>	***
	14	nd	7.92±0.18 <sup>bA</sup>	9.47±0.11 <sup>bB</sup>	13.20±0.30 <sup>bC</sup>	***
	21	nd	8.24±0.03 <sup>cA</sup>	9.85±0.14 <sup>cB</sup>	13.23±0.02 <sup>bC</sup>	***
	Significance	ns	***	***	***	
Pyruvic acid (g.L <sup>-1</sup> )	0	0.05±0.01 <sup>c</sup>	0.04±	0.05±	0.04±0.01	ns
	1	0.05± <sup>c</sup>	0.05±	0.04±0.01	0.04±0.01	ns
	7	0.04± <sup>b</sup>	0.05±	0.04±0.01	0.05±0.01	ns
	14	0.02± <sup>aA</sup>	0.04± <sup>B</sup>	0.04±0.01 <sup>B</sup>	0.04±0.01 <sup>B</sup>	**
	21	0.02± <sup>aB</sup>	0.04± <sup>B</sup>	0.04±0.01 <sup>B</sup>	0.04±0.01 <sup>B</sup>	*
	Significance	***	ns	ns	ns	
Lactic acid (g.L <sup>-1</sup> )	0	11.48±0.10 <sup>aD</sup>	8.67±0.02 <sup>aC</sup>	8.46±0.06 <sup>aB</sup>	8.22±0.02 <sup>aA</sup>	***
	1	11.70±0.10 <sup>aC</sup>	9.29±0.04 <sup>bA</sup>	9.49±0.11 <sup>bB</sup>	9.18±0.13 <sup>bA</sup>	***
	7	13.63± <sup>bD</sup>	10.22±0.08 <sup>cB</sup>	10.51±0.01 <sup>cC</sup>	9.86±0.08 <sup>cA</sup>	***

	14	14.50±0.37 <sup>cC</sup>	10.55±0.21 <sup>dB</sup>	10.55±0.34 <sup>cB</sup>	9.80±0.16 <sup>cA</sup>	***
	21	15.63±0.24 <sup>dD</sup>	11.11±0.01 <sup>eB</sup>	11.38±0.02 <sup>dC</sup>	10.65±0.09 <sup>dA</sup>	
	<i>Significance</i>	***	***	***	***	
Citric acid (g.L <sup>-1</sup> )	0	1.99±0.10 <sup>B</sup>	1.76±0.08 <sup>A</sup>	1.75±0.09 <sup>A</sup>	1.74±0.07 <sup>A</sup>	*
	1	1.97±0.10 <sup>B</sup>	1.75±0.07 <sup>A</sup>	1.78±0.08 <sup>A</sup>	1.78±0.09 <sup>A</sup>	*
	7	2.00±0.09 <sup>B</sup>	1.76±0.08 <sup>A</sup>	1.77±0.08 <sup>A</sup>	1.74±0.06 <sup>A</sup>	*
	14	1.89±0.04 <sup>B</sup>	1.74±0.05 <sup>AB</sup>	1.72±0.12 <sup>A</sup>	1.71±0.09 <sup>A</sup>	ns
	21	2.01±0.06 <sup>B</sup>	1.75±0.07 <sup>A</sup>	1.77±0.08 <sup>A</sup>	1.76±0.06 <sup>A</sup>	**
	<i>Significance</i>	ns	ns	ns	ns	
Tartaric acid (g.L <sup>-1</sup> )	0	nd	2.59±0.01 <sup>bC</sup>	2.51±0.04 <sup>bB</sup>	2.01±0.02 <sup>cdA</sup>	***
	1	nd	2.25±0.01 <sup>a</sup>	2.09±0.24 <sup>a</sup>	2.05±0.09 <sup>d</sup>	ns
	7	nd	2.68±0.02 <sup>bB</sup>	2.79±0.20 <sup>bB</sup>	1.89±0.06 <sup>bcA</sup>	***
	14	nd	2.55±0.20 <sup>bB</sup>	2.85±0.03 <sup>bC</sup>	1.72±0.10 <sup>aA</sup>	ns
	21	nd	2.74±0.11 <sup>bB</sup>	2.67±0.23 <sup>bB</sup>	1.77±0.08 <sup>abA</sup>	***
	<i>Significance</i>		**	**	**	
Malic acid (g.L <sup>-1</sup> )	0	nd	0.19± <sup>bA</sup>	0.32±0.01 <sup>cB</sup>	0.50±0.01 <sup>C</sup>	***
	1	nd	0.19± <sup>bA</sup>	0.31±0.01 <sup>cB</sup>	0.51±0.02 <sup>C</sup>	***
	7	nd	0.16±0.01 <sup>aA</sup>	0.28±0.01 <sup>bB</sup>	0.49±0.01 <sup>C</sup>	***
	14	nd	0.15± <sup>aA</sup>	0.28±0.01 <sup>bB</sup>	0.48±0.02 <sup>C</sup>	***
	21	nd	0.17±0.02 <sup>aA</sup>	0.27±0.01 <sup>aB</sup>	0.51±0.03 <sup>C</sup>	***
	<i>Significance</i>	ns	**	***	ns	

689 ‡The results are represented as means ± standard deviation

690 <sup>a-c</sup>Values in each column having different lowercase letters are significantly different at P< 0.05 within storage time. Values in each row  
691 having different capitals letters are significantly different at P< 0.05 within yogurt type.

692 \* P< 0.05; \*\* P< 0.01; \*\*\* P<0.001; ns not significant

693 nd: not detected; where not specified, standard deviation are less than 0.01

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709 **Table 4.** PHENOLIC COMPOUNDS OF CONTROL AND FORTIFIED YOGURTS  
710 DURING STORAGE AND RESULTS OF ANALYSIS OF VARIANCE WITH  
711 DUNCAN'S TEST

	Days	Moscato	Chardonnay	Pinot noir	Significance
Gallic acid <sup>‡</sup>	0	3.6±0.5 <sup>B</sup>	2.7±0.2 <sup>AB</sup>	1.5±0.1 <sup>A</sup>	*
	1	4.2± <sup>C</sup>	2.6±0.2 <sup>B</sup>	1.7±0.1 <sup>A</sup>	**
	7	3.9±0.3 <sup>C</sup>	2.7± <sup>B</sup>	1.6±0.2 <sup>A</sup>	**
	14	3.8± <sup>C</sup>	2.6± <sup>B</sup>	1.6±0.1 <sup>A</sup>	***
	21	4±0.3 <sup>C</sup>	2.7±0.2 <sup>B</sup>	1.7±0.1 <sup>A</sup>	**
	<i>Significance</i>	ns	ns	ns	
Protocatechuic acid	0	1.1±0.3	1.2±	0.7±0.1	ns
	1	1.5±0.1	1.2±0.1	1.1±0.4	ns
	7	1.2±0.9	1.2±0.1	0.8±0.1	ns
	14	1.4±0.2 <sup>B</sup>	1.2±0.1 <sup>AB</sup>	0.8± <sup>A</sup>	*
	21	1.1±0.2	1.1±0.3	1.2±0.1	ns
	<i>Significance</i>	ns	ns	ns	
Procyanidin B1	0	nd	nd	2.6±0.1	--
	1	nd	nd	2.7±0.1	--
	7	nd	nd	2.9±0.4	--
	14	nd	nd	2.9±	--
	21	nd	nd	3.0±0.2	--
	<i>Significance</i>	--	--	ns	
2,3,4-trihydroxybenzoic acid	0	nd	nd	1.7±0.1	--
	1	nd	nd	2.2±0.1	--
	7	nd	nd	2.3±0.3	--
	14	nd	nd	2.4±0.1	--
	21	nd	nd	1.7±0.6	--
	<i>Significance</i>	--	--	ns	
Catechin	0	17.9±1.5	22.9±3.4	5.1±0.2	ns
	1	19.3±0.1 <sup>B</sup>	18.8±0.6 <sup>B</sup>	5.3±0.6 <sup>A</sup>	**
	7	18.8±0.1 <sup>B</sup>	18.1±1.2 <sup>B</sup>	6.6±3.1 <sup>A</sup>	***
	14	18.0±0.1 <sup>B</sup>	19.0±0.7 <sup>B</sup>	7.0±0.3 <sup>A</sup>	***
	21	16.1±1.7	17.2±0.1	6.7±0.3	ns
	<i>Significance</i>	ns	ns	ns	
Vanillic acid	0	nd	nd	3.5±0.3	--
	1	nd	nd	3.4±0.1	--
	7	nd	nd	3.1±0.5	--
	14	nd	nd	2.9±0.2	--
	21	nd	nd	3.3±0.2	--
	<i>Significance</i>	--	--	ns	
Epicatechin	0	0.3±	0.4±	nd	--

	1	0.4±	0.3±	nd	--
	7	0.3±	0.3±	nd	--
	14	0.3±	0.3±	nd	--
	21	0.3±	0.3±	nd	--
	<i>Significance</i>	ns	ns	--	
	0	3.1±0.1	3.7±0.4	5.3±1.0	ns
	1	3.9± <sup>A</sup>	3.4±0.1 <sup>B</sup>	5.6±0.1 <sup>C</sup>	***
	7	3.7±0.7	3.4±0.1	5.1±1.0	ns
	14	4.0±0.1 <sup>B</sup>	3.3± <sup>A</sup>	5.2±0.1 <sup>C</sup>	***
	21	4.3±0.3	4.1±0.1	5.0±0.4	ns
	<i>Significance</i>	ns	ns	ns	
	0	6.3±0.6 <sup>AB</sup>	9.9±1.2 <sup>abB</sup>	4.6±1.0 <sup>A</sup>	*
	1	8.4±0.6 <sup>B</sup>	8.9±0.5 <sup>abB</sup>	4.9±0.2 <sup>A</sup>	**
	7	7.7±1.4	8.6±0.5 <sup>a</sup>	4.5±0.9	ns
	14	8.9± <sup>B</sup>	8.8±0.1 <sup>aB</sup>	4.7±0.1 <sup>A</sup>	***
	21	9.3±2.3 <sup>AB</sup>	11.4±0.4 <sup>bB</sup>	4.6±0.5 <sup>A</sup>	*
	<i>Significance</i>	ns	*	ns	

712 ‡The results are reported as µg/g and represented as means ± standard deviation

713 <sup>a-c</sup>Values in each column having different lowercase letters are significantly different at P< 0.05 within storage time. Values in each row

714 having different capitals letters are significantly different at P< 0.05 within yogurt type.

715 \* P< 0.05; \*\* P< 0.01; \*\*\* P<0.001; ns not significant

716 nd: not detected; where not specified, standard deviation are less than 0.1

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730 **Table 5.** VOLATILE COMPOUNDS OF CONTROL AND FORTIFIED YOGURTS  
 731 DURING STORAGE AND RESULTS OF ANALYSIS OF VARIANCE WITH  
 732 DUNCAN'S TEST

	Days	Control	Moscato	Chardonnay	Pinot noir	Significance
$\Sigma$ Ketones <sup>‡</sup>	0	1030.51±36.98 <sup>bB</sup>	927.48±20.49 <sup>aA</sup>	914.32±11.02 <sup>bA</sup>	887.81±28.12 <sup>aA</sup>	***
	1	995.20±38.40 <sup>abC</sup>	969.05±14.49 <sup>bBC</sup>	899.04±50.88 <sup>bAB</sup>	881.72±54.39 <sup>aA</sup>	*
	7	1017.23±43.42 <sup>ab</sup>	857.83±100.24 <sup>a</sup>	912.81±61.76 <sup>b</sup>	978.09±17.87 <sup>b</sup>	ns
	14	893.43±75.08 <sup>aB</sup>	898.02±12.06 <sup>abB</sup>	761.96±56.57 <sup>aA</sup>	959.24±5.74 <sup>bB</sup>	**
	21	1153.28±109.31 <sup>cA</sup>	1367.59±49.81 <sup>cB</sup>	1425.31±25.26 <sup>cB</sup>	1092.65±14.77 <sup>cA</sup>	***
	Significance	*	***	***	***	
$\Sigma$ Aldehydes	0	9.87±0.66 <sup>cA</sup>	18.43±2.15 <sup>abC</sup>	23.69±2.61 <sup>cD</sup>	14.92±0.53 <sup>B</sup>	***
	1	5.60±0.27 <sup>aA</sup>	17.51±0.51 <sup>aB</sup>	23.28±1.34 <sup>cD</sup>	19.94±0.31 <sup>C</sup>	***
	7	5.91±0.31 <sup>aA</sup>	20.75±2.02 <sup>bcB</sup>	23.30±0.57 <sup>cB</sup>	18.31±6.74 <sup>B</sup>	**
	14	7.70±1.50 <sup>bA</sup>	22.51±0.47 <sup>cD</sup>	19.84±1.91 <sup>bc</sup>	15.34±0.79 <sup>B</sup>	***
	21	8.28±1.02 <sup>bcA</sup>	16.95±1.05 <sup>aC</sup>	15.59±0.54 <sup>aC</sup>	13.23±1.83 <sup>B</sup>	***
	Significance	***	*	***	ns	
$\Sigma$ Alcohols	0	55.52±1.00 <sup>cA</sup>	263.87±34.02 <sup>bc</sup>	332.55±22.25 <sup>bd</sup>	115.79±3.42 <sup>bcB</sup>	***
	1	27.04±6.05 <sup>aA</sup>	224.23±5.97 <sup>aB</sup>	310.12±9.11 <sup>abD</sup>	267.21±3.68 <sup>dC</sup>	***
	7	38.51±9.66 <sup>bA</sup>	336.39±21.52 <sup>cC</sup>	311.27±23.85 <sup>abC</sup>	98.54±19.98 <sup>abB</sup>	***
	14	21.78±5.56 <sup>aA</sup>	365.00±8.29 <sup>cD</sup>	287.43±25.08 <sup>aC</sup>	87.73±6.64 <sup>aB</sup>	***
	21	49.05±4.35 <sup>bcA</sup>	346.83±9.32 <sup>cC</sup>	413.31±18.42 <sup>cD</sup>	127.19±15.58 <sup>cB</sup>	***
	Significance	***	***	***	***	
$\Sigma$ Acids	0	71.01±7.53 <sup>aA</sup>	231.68±18.40 <sup>aB</sup>	284.10±29.23 <sup>bc</sup>	210.70±0.78 <sup>aB</sup>	***
	1	72.24±13.01 <sup>aA</sup>	219.25±20.83 <sup>aC</sup>	173.78±14.20 <sup>aB</sup>	177.86±8.45 <sup>aB</sup>	***
	7	115.16±3.05 <sup>bA</sup>	288.38±15.67 <sup>bc</sup>	210.47±14.20 <sup>aB</sup>	276.20±41.93 <sup>bc</sup>	***
	14	144.20±8.09 <sup>cA</sup>	298.44±42.52 <sup>bB</sup>	179.12±9.39 <sup>aA</sup>	351.33±9.24 <sup>cC</sup>	***
	21	134.60±26.78 <sup>bcA</sup>	287.25±2.29 <sup>bc</sup>	201.87±31.62 <sup>aB</sup>	277.07±8.54 <sup>bc</sup>	***
	Significance	***	**	***	***	
Esters	0	0.75±0.12 <sup>bB</sup>	13.58±0.82 <sup>bd</sup>	10.09±0.20 <sup>C</sup>	0.56±0.03 <sup>bA</sup>	***
	1	0.22±0.01 <sup>abA</sup>	11.36±0.15 <sup>aC</sup>	10.41±0.26 <sup>B</sup>	0.48± <sup>abA</sup>	***
	7	0.18±0.01 <sup>abA</sup>	17.62±0.92 <sup>dC</sup>	11.06±0.08 <sup>B</sup>	0.45±0.09 <sup>aA</sup>	***
	14	0.13±0.01 <sup>aA</sup>	21.52±1.03 <sup>cC</sup>	10.33±0.98 <sup>B</sup>	0.48±0.03 <sup>abA</sup>	***
	21	0.29±0.02 <sup>bA</sup>	15.62±0.72 <sup>cC</sup>	12.32±2.53 <sup>B</sup>	0.56±0.02 <sup>bA</sup>	***
	Significance	***	***	ns	*	
$\Sigma$ Lactones	0	1.17±0.03 <sup>A</sup>	2.93±0.17 <sup>bcB</sup>	4.09±0.67 <sup>C</sup>	1.24±0.08 <sup>aA</sup>	***
	1	1.18±0.10 <sup>A</sup>	2.54±0.35 <sup>abB</sup>	3.52±0.19 <sup>C</sup>	1.09±0.07 <sup>aA</sup>	***
	7	1.09±0.15 <sup>A</sup>	3.34±0.27 <sup>bc</sup>	3.86±0.13 <sup>C</sup>	2.42±0.93 <sup>bB</sup>	***
	14	1.21±0.29 <sup>A</sup>	3.81±0.27 <sup>bd</sup>	3.16±0.13 <sup>C</sup>	2.23±0.10 <sup>bB</sup>	***
	21	1.13±0.20 <sup>A</sup>	2.35±0.05 <sup>aB</sup>	4.00±1.06 <sup>C</sup>	1.02±0.03 <sup>aA</sup>	***
	Significance	ns	***	ns	**	
$\Sigma$ Furan derivatives	0	12.08±0.76 <sup>cA</sup>	98.88±10.06 <sup>abC</sup>	85.82±3.84 <sup>bB</sup>	112.45±5.18 <sup>D</sup>	***
	1	4.34±0.08 <sup>aA</sup>	89.77±6.05 <sup>abC</sup>	97.77±7.25 <sup>cC</sup>	84.25±5.36 <sup>B</sup>	***
	7	4.22±0.07 <sup>aA</sup>	110.08±9.68 <sup>bc</sup>	85.91±6.21 <sup>bB</sup>	96.61±13.26 <sup>BC</sup>	***

	14	3.78±0.19 <sup>aA</sup>	124.11±2.28 <sup>cD</sup>	79.16±4.10 <sup>bB</sup>	94.80±0.28 <sup>C</sup>	***
	21	5.07±0.33 <sup>ba</sup>	87.65±5.55 <sup>aC</sup>	62.67±4.80 <sup>ab</sup>	100.05±15.02 <sup>C</sup>	***
	<i>Significance</i>	***	***	***	ns	
Σ Terpenoids	0	32.02±2.55 <sup>A</sup>	49.79±2.22 <sup>aB</sup>	66.85±6.71 <sup>cC</sup>	31.03±3.06 <sup>A</sup>	***
	1	32.87±3.33 <sup>A</sup>	46.46±3.46 <sup>aB</sup>	33.75±2.32 <sup>ba</sup>	33.24±2.09 <sup>A</sup>	***
	7	30.69±2.82 <sup>A</sup>	51.73±3.06 <sup>aB</sup>	27.35±1.42 <sup>abA</sup>	44.89±13.31 <sup>B</sup>	**
	14	25.42±4.06 <sup>A</sup>	58.48±2.48 <sup>bc</sup>	24.40±2.21 <sup>aA</sup>	31.43±1.61 <sup>B</sup>	***
	21	28.98±0.64 <sup>A</sup>	64.91±2.98 <sup>cB</sup>	31.10±0.62 <sup>ba</sup>	28.97±1.68 <sup>A</sup>	***
	<i>Significance</i>	ns	***	***	ns	

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‡The results are reported as µg/kg and represented as means ± standard deviation

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<sup>a-c</sup>Values in each column having different lowercase letters are significantly different at P < 0.05 within storage time. Values in each row

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having different capitals letters are significantly different at P < 0.05 within yogurt type.

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\* P< 0.05; \*\* P< 0.01; \*\*\* P<0.001; ns not significant

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nd: not detected; where not specified, standard deviation are less than 0.01

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756 Additional data – Volatile compounds (mean  $\pm$  standard deviation;  $\mu\text{g}/\text{kg}$ ) of control  
 757 and fortified yogurts during storage  
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Control	Days				
	0	1	7	14	21
<b>Ketones</b>					
2-Pentanone	25.22 $\pm$ 0.56	27.73 $\pm$ 2.90	39.84 $\pm$ 3.19	31.77 $\pm$ 7.71	41.57 $\pm$ 6.19
2,3-Pentanedione	133.14 $\pm$ 1.79	85.14 $\pm$ 2.04	138.75 $\pm$ 0.22	129.01 $\pm$ 12.70	145.45 $\pm$ 12.81
2-Heptanone	277.04 $\pm$ 34.15	323.24 $\pm$ 15.32	304.49 $\pm$ 22.48	253.44 $\pm$ 25.54	416.85 $\pm$ 75.73
Acetoin	489.06 $\pm$ 5.14	447.60 $\pm$ 22.27	423.50 $\pm$ 17.39	379.04 $\pm$ 29.96	425.44 $\pm$ 27.13
6-Methyl-5-hepten-2-one	nd	0.08 $\pm$ 0.01	nd	nd	nd
3-Hydroxy-2-pentanone	42.19 $\pm$ 2.02	47.08 $\pm$ 2.39	48.49 $\pm$ 2.39	42.02 $\pm$ 3.75	54.44 $\pm$ 5.66
2-Nonanone	56.68 $\pm$ 1.94	56.39 $\pm$ 2.02	54.31 $\pm$ 1.94	50.84 $\pm$ 3.67	62.27 $\pm$ 5.78
6-Methyl-3,5-heptadien-2-one	nd	nd	nd	nd	nd
2-Undecanone	6.43 $\pm$ 0.16	7.08 $\pm$ 0.10	6.93 $\pm$ 0.23	6.51 $\pm$ 1.09	6.46 $\pm$ 0.26
2-Tridecanone	0.76 $\pm$ 0.01	0.85 $\pm$ 0.03	0.91 $\pm$ 0.11	0.79 $\pm$ 0.13	0.80 $\pm$ 0.06
$\Sigma$ Ketones	1030.51 $\pm$ 36.98	995.20 $\pm$ 38.40	1017.23 $\pm$ 43.42	893.43 $\pm$ 75.08	1153.28 $\pm$ 109.31
<b>Alddehydes</b>					
Nonanal	1.12 $\pm$ 0.26	0.35 $\pm$ 0.08	1.18 $\pm$ 0.09	2.52 $\pm$ 0.40	1.91 $\pm$ 0.59
Benzaldehyde	5.84 $\pm$ 0.67	2.70 $\pm$ 0.10	2.99 $\pm$ 0.09	2.63 $\pm$ 0.10	2.90 $\pm$ 0.33
4-Methyl-benzaldehyde	2.61 $\pm$ 0.25	2.27 $\pm$ 0.12	1.64 $\pm$ 0.25	2.46 $\pm$ 1.06	3.39 $\pm$ 0.75
Dodecanal	0.29 $\pm$ 0.06	0.28 $\pm$ 0.19	0.09 $\pm$ 0.08	0.10 $\pm$ 0.01	0.09 $\pm$ 0.01
$\Sigma$ Alddehydes	9.87 $\pm$ 0.66	5.60 $\pm$ 0.27	5.91 $\pm$ 0.31	7.70 $\pm$ 1.50	8.28 $\pm$ 1.02
<b>Alcohols</b>					
Isobutanol	0.10 $\pm$ 0.06	0.53 $\pm$ 0.57	nd	0.93 $\pm$ 0.10	nd
1-Pentanol	7.90 $\pm$ 0.81	1.63 $\pm$ 0.17	2.01 $\pm$ 0.33	1.53 $\pm$ 0.10	1.67 $\pm$ 0.06
3-Methyl-1-butanol	27.19 $\pm$ 0.62	16.49 $\pm$ 5.88	26.60 $\pm$ 8.23	12.04 $\pm$ 4.83	39.31 $\pm$ 4.12
1-Hexanol	12.22 $\pm$ 0.18	5.36 $\pm$ 0.16	5.17 $\pm$ 0.33	4.31 $\pm$ 0.40	5.40 $\pm$ 0.14
2-Hexen-1-ol	nd	nd	nd	nd	nd
1-Octen-3-ol	1.54 $\pm$ 0.06	0.73 $\pm$ 0.13	0.82 $\pm$ 0.09	0.81 $\pm$ 0.02	nd
1-Octanol	0.44 $\pm$ 0.01	0.24 $\pm$ 0.05	0.28 $\pm$ 0.01	0.32 $\pm$ 0.01	0.29 $\pm$ 0.03
1-Nonanol	0.34 $\pm$ 0.01	0.33 $\pm$ 0.07	0.42 $\pm$ 0.06	0.37 $\pm$ 0.05	0.39 $\pm$ 0.03
Benzyl alcohol	0.75 $\pm$ 0.15	0.18 $\pm$ 0.03	0.29 $\pm$ 0.03	0.15 $\pm$ 0.01	0.25 $\pm$ 0.04
Phenylethyl alcohol	4.56 $\pm$ 0.84	0.61 $\pm$ 0.04	0.66 $\pm$ 0.14	0.31 $\pm$ 0.04	0.95 $\pm$ 0.15
1,4-Butanediol	0.18 $\pm$ 0.03	0.15 $\pm$ 0.04	0.18 $\pm$ 0.02	0.14 $\pm$ 0.02	0.18 $\pm$ 0.03
1-Dodecanol	0.30 $\pm$ 0.10	0.78 $\pm$ 0.34	2.08 $\pm$ 0.88	0.87 $\pm$ 0.28	0.62 $\pm$ 0.12
$\Sigma$ Alcohols	55.52 $\pm$ 1.00	27.04 $\pm$ 6.05	38.51 $\pm$ 9.66	21.78 $\pm$ 5.56	49.05 $\pm$ 4.35
<b>Acids</b>					
Acetic acid	16.56 $\pm$ 2.49	19.63 $\pm$ 3.19	40.21 $\pm$ 4.12	43.84 $\pm$ 9.17	54.77 $\pm$ 14.02
Isobutyric acid	0.42 $\pm$ 0.02	0.52 $\pm$ 0.12	0.59 $\pm$ 0.03	0.38 $\pm$ 0.04	0.30 $\pm$ 0.04
Butanoic acid	19.32 $\pm$ 1.63	20.86 $\pm$ 3.99	28.68 $\pm$ 1.04	34.47 $\pm$ 4.68	33.61 $\pm$ 5.19
Methacrylic acid	nd	0.42 $\pm$ 0.08	0.18 $\pm$ 0.07	2.09 $\pm$ 0.47	0.18 $\pm$ 0.17
Pentanoic acid	2.28 $\pm$ 0.31	2.07 $\pm$ 0.50	3.7 $\pm$ 0.61	1.33 $\pm$ 0.31	0.74 $\pm$ 0.10
Hexanoic acid	24.33 $\pm$ 7.79	21.34 $\pm$ 3.93	31.05 $\pm$ 0.93	50.70 $\pm$ 7.49	33.63 $\pm$ 5.39
2-Ethyl-hexanoic acid	3.65 $\pm$ 1.18	2.76 $\pm$ 0.12	3.13 $\pm$ 0.94	1.29 $\pm$ 0.48	0.80 $\pm$ 0.16
Heptanoic acid	1.87 $\pm$ 0.60	1.99 $\pm$ 0.63	2.13 $\pm$ 0.33	1.63 $\pm$ 0.17	1.14 $\pm$ 0.12
Octanoic acid	nd	nd	nd	nd	nd
Nonanoic acid	1.66 $\pm$ 0.31	1.11 $\pm$ 0.45	2.20 $\pm$ 0.69	4.00 $\pm$ 0.11	2.77 $\pm$ 0.71
Benzenecarboxylic acid	0.91 $\pm$ 0.05	1.53 $\pm$ 0.11	3.22 $\pm$ 0.90	4.47 $\pm$ 2.73	6.66 $\pm$ 2.65
$\Sigma$ Acids	71.01 $\pm$ 7.53	72.24 $\pm$ 13.01	115.16 $\pm$ 3.05	144.20 $\pm$ 8.09	134.60 $\pm$ 26.78
<b>Esters</b>					
$\beta$ -Phenylethyl acetate	0.75 $\pm$ 0.12	0.22 $\pm$ 0.01	0.18 $\pm$ 0.01	0.13 $\pm$ 0.01	0.29 $\pm$ 0.02
<b>Lactones</b>					
$\gamma$ -Caprolactone	0.19 $\pm$ 0.01	0.18 $\pm$ 0.02	0.22 $\pm$ 0.01	0.16 $\pm$ 0.01	0.22 $\pm$ 0.03
$\delta$ -Decalactone	0.98 $\pm$ 0.04	1.00 $\pm$ 0.11	0.87 $\pm$ 0.15	1.05 $\pm$ 0.29	0.92 $\pm$ 0.17
$\Sigma$ Lactones	1.17 $\pm$ 0.03	1.18 $\pm$ 0.10	1.09 $\pm$ 0.15	1.21 $\pm$ 0.29	1.13 $\pm$ 0.20
<b>Furan derivatives</b>					
2-Pentyl-furan	nd	0.81 $\pm$ 0.04	0.83 $\pm$ 0.04	0.73 $\pm$ 0.07	0.89 $\pm$ 0.08
Furfural	10.24 $\pm$ 0.82	1.51 $\pm$ 0.04	1.36 $\pm$ 0.05	1.15 $\pm$ 0.02	1.86 $\pm$ 0.01
2-Furanmethanol	1.84 $\pm$ 0.07	2.02 $\pm$ 0.07	2.03 $\pm$ 0.15	1.91 $\pm$ 0.22	2.32 $\pm$ 0.32
$\Sigma$ Furan derivatives	12.08 $\pm$ 0.76	4.34 $\pm$ 0.08	4.22 $\pm$ 0.07	3.78 $\pm$ 0.19	5.07 $\pm$ 0.33
<b>Terpenes</b>					
Limonene	31.67 $\pm$ 2.53	32.23 $\pm$ 3.45	29.32 $\pm$ 3.03	24.92 $\pm$ 3.94	28.62 $\pm$ 0.78
<i>cis</i> -Linalool oxide	nd	nd	nd	nd	nd
Linalool	0.24 $\pm$ 0.03	0.19 $\pm$ 0.01	0.16 $\pm$ 0.01	0.13 $\pm$ 0.01	0.15 $\pm$ 0.01
$\alpha$ -Terpineol	0.11 $\pm$ 0.01	0.46 $\pm$ 0.10	1.21 $\pm$ 0.21	0.36 $\pm$ 0.15	0.21 $\pm$ 0.15
$\Sigma$ Terpenes	32.02 $\pm$ 2.55	32.87 $\pm$ 3.33	30.69 $\pm$ 2.82	25.42 $\pm$ 4.06	28.98 $\pm$ 0.64

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Moscatto	Days				
	0	1	7	14	21
<b>Ketones</b>					
2-Pentanone	24.61±1.74	24.50±1.27	22.13±4.23	22.30±1.60	90.23±3.96
2,3-Pentanedione	196.44±1.76	202.76±13.26	187.05±35.09	177.53±8.34	85.89±2.24
2-Heptanone	219.40±13.81	222.21±5.47	202.44±21.59	223.79±5.03	666.91±49.77
Acetoin	391.72±1.84	427.77±17.67	351.74±34.14	369.94±2.42	371.33±5.11
6-Methyl-5-hepten-2-one	5.73±0.78	4.05±0.25	7.34±0.65	8.14±0.56	10.00±1.28
3-Hydroxy-2-pentanone	26.49±0.52	28.40±1.05	26.83±2.62	31.55±1.65	44.61±1.24
2-Nonanone	52.43±3.57	49.81±1.68	48.43±2.61	51.60±0.52	87.51±7.00
6-Methyl-3,5-heptadien-2-one	1.85±0.21	1.50±0.10	2.69±0.18	2.99±0.16	2.87±0.16
2-Undecanone	8.13±0.63	7.40±0.15	8.40±0.19	9.35±0.48	7.58±0.24
2-Tridecanone	0.68±0.06	0.65±0.02	0.79±0.14	0.83±0.03	0.66±0.07
Σ Ketones	927.48±20.49	969.05±14.49	857.83±100.24	898.02±12.06	1367.59±49.81
<b>Alddehydes</b>					
Nonanal	4.32±0.46	3.78±0.13	2.99±0.16	2.47±0.25	1.56±0.51
Benzaldehyde	11.74±1.36	10.59±0.13	14.56±1.57	15.89±0.71	9.36±0.60
4-Methyl-benzaldehyde	2.00±0.29	2.45±0.36	2.45±0.41	3.16±1.17	5.03±0.32
Dodecanal	0.37±0.05	0.68±0.14	0.75±0.12	0.99±0.25	1.00±0.24
Σ Alddehydes	18.43±2.15	17.51±0.51	20.75±2.02	22.51±0.47	16.95±1.05
<b>Alcohols</b>					
Isobutanol	3.11±0.35	1.43±0.04	4.62±1.53	3.02±0.11	7.09±0.32
1-Pentanol	13.03±2.50	9.61±0.75	13.28±1.87	16.08±1.64	21.58±0.42
3-Methyl-1-butanol	43.60±9.38	42.54±11.55	69.12±15.89	51.66±7.45	50.67±1.35
1-Hexanol	58.43±10.89	48.64±5.74	76.13±8.09	89.04±2.98	117.92±3.91
2-Hexen-1-ol	7.12±0.87	5.79±0.33	8.84±0.85	9.39±0.70	11.27±0.72
1-Octen-3-ol	18.08±4.00	11.55±1.18	22.02±1.46	26.95±1.98	37.21±0.50
1-Octanol	1.47±0.16	1.13±0.04	1.98±0.04	2.51±0.02	2.44±0.10
1-Nonanol	0.77±0.10	0.72±0.05	1.38±0.11	1.94±0.04	1.55±0.08
Benzyl alcohol	4.50±0.32	3.96±0.39	5.20±0.34	6.37±0.70	3.72±0.13
Phenylethyl alcohol	113.09±6.61	97.91±13.19	133.31±10.66	157.69±7.37	92.19±2.40
1,4-Butanediol	0.41±0.07	0.31±0.03	0.20±0.05	0.31±0.06	0.52±0.07
1-Dodecanol	0.24±0.07	0.64±0.22	0.31±0.06	0.04±0.04	0.67±0.03
Σ Alcohols	263.87±34.02	224.23±5.97	336.39±21.52	365.00±8.29	346.83±9.32
<b>Acids</b>					
Acetic acid	76.20±1.72	73.24±7.23	95.60±2.72	101.64±11.31	115.33±3.56
Isobutyric acid	3.75±0.71	4.12±0.29	4.23±0.30	4.12±1.00	4.73±0.17
Butanoic acid	34.83±3.36	35.62±1.44	41.77±1.79	41.04±7.38	39.45±2.20
Methacrylic acid	nd	nd	0.69±0.05	0.28±0.06	0.20±0.07
Pentanoic acid	4.47±0.10	5.33±0.12	4.90±0.84	4.17±0.73	6.49±0.13
Hexanoic acid	74.86±9.96	65.88±6.90	91.36±2.64	93.67±17.25	70.42±1.92
2-Ethyl-hexanoic acid	14.52±0.98	15.56±1.56	13.33±4.91	11.50±0.54	20.64±0.35
Heptanoic acid	10.47±0.76	10.35±2.49	10.83±3.16	8.68±0.35	11.94±0.92
Octanoic acid	nd	nd	nd	0.03±0.03	0.02±0.01
Nonanoic acid	9.00±4.04	5.76±1.70	17.72±1.00	19.73±2.11	11.26±0.33
Benzenecarboxylic acid	3.58±0.51	3.38±1.51	7.94±1.68	13.59±3.00	6.79±1.13
Σ Acids	231.68±18.40	219.25±20.83	288.38±15.67	298.44±42.52	287.25±2.29
<b>Esters</b>					
β-Phenylethyl acetate	13.58±0.82	11.36±0.15	17.62±0.92	21.52±1.03	15.62±0.72
<b>Lactones</b>					
γ-Caprolactone	2.02±0.08	1.73±0.15	2.14±0.16	2.52±0.12	1.74±0.05
δ-Decalactone	0.91±0.12	0.81±0.20	1.21±0.13	1.29±0.16	0.61±0.04
Σ Lactones	2.93±0.17	2.54±0.35	3.34±0.27	3.81±0.27	2.35±0.05
<b>Furan derivatives</b>					
2-Pentyl-furan	26.58±1.04	31.95±1.25	31.30±0.37	35.59±1.03	40.36±4.05
Furfural	68.78±9.12	54.96±6.25	75.36±9.63	84.71±2.64	41.68±1.37
2-Furanmethanol	3.52±0.13	2.86±0.08	3.42±0.27	3.80±0.21	5.61±0.37
Σ Furan derivatives	98.88±10.06	89.77±6.05	110.08±9.68	124.11±2.28	87.65±5.55
<b>Terpenes</b>					
Limonene	31.01±0.82	30.80±2.68	28.24±1.85	28.41±0.67	36.67±1.64
cis-Linalool oxide	5.77±0.48	4.36±0.69	6.88±0.47	9.50±0.74	8.87±0.77
Linalool	8.61±0.82	7.62±0.29	11.03±0.42	13.61±0.46	14.26±0.66
α-Terpineol	4.40±0.11	3.68±0.20	5.58±0.72	6.96±0.71	5.11±0.05
Σ Terpenes	49.79±2.22	46.46±3.46	51.73±3.06	58.48±2.48	64.91±2.98

Chardonnay	Days				
	0	1	7	14	21
<b>Ketones</b>					
2-Pentanone	21.9±1.11	22.37±1.85	26.89±2.2	22.77±2.68	67.92±3.28
2,3-Pentanedione	174.17±15.86	176.45±4.45	204.49±23.85	162.18±17.65	137.78±6.87
2-Heptanone	200.9±4.25	242.35±16.84	229.92±11.28	203.64±24.36	678.19±28.18
Acetoin	433.38±23.28	361.8±22.37	356.35±19.59	289.78±10.99	391.27±10.47
6-Methyl-5-hepten-2-one	2.98±0.28	2.63±0.16	2.29±0.43	2.52±0.16	2.49±0.07
3-Hydroxy-2-pentanone	25.81±1.23	29.92±1.99	31.48±1.42	25.83±1.08	45.06±1.76
2-Nonanone	46.92±2.54	53.83±3.58	51.46±3.26	46.38±1.95	91.68±1.86
6-Methyl-3,5-heptadien-2-one	0.55±0.01	0.59±0.04	0.6±0.03	0.59±0.01	0.67±0.06
2-Undecanone	6.99±0.23	8.29±0.36	8.38±0.19	7.59±0.2	9.29±0.52
2-Tridecanone	0.73±0.08	0.8±0.04	0.96±0.08	0.67±0.05	0.96±0.12
∑ Ketones	914.32±11.02	899.04±50.88	912.81±61.76	761.96±56.57	1425.31±25.26
<b>Alddehydes</b>					
Nonanal	6.72±2.18	5.73±0.48	7.27±0.1	2.35±0.13	3.08±0.74
Benzaldehyde	14.2±0.74	15.16±0.81	13.15±0.7	14.73±1.79	8.78±0.15
4-Methyl-benzaldehyde	2.4±0.27	2.2±0.06	2.88±0.19	2.66±0.24	3.38±0.13
Dodecanal	0.36±0.01	0.19±0.05	nd	0.1±0.02	0.34±0.18
∑ Alddehydes	23.69±2.61	23.28±1.34	23.3±0.57	19.84±1.91	15.59±0.54
<b>Alcohols</b>					
Isobutanol	3.27±0.51	2.54±0.39	4.17±0.62	3.25±0.16	5.89±1
1-Pentanol	9.75±0.17	10.53±1.14	7.72±1.72	8.47±1.04	10.6±0.56
3-Methyl-1-butanol	66.71±9.65	52.71±0.82	58.66±9.58	61.48±20.3	129.6±7.99
1-Hexanol	134.19±2.73	136.63±8.06	121.44±14.43	112.36±7.81	135.28±2.19
2-Hexen-1-ol	11.42±1.11	12.92±1.18	13.11±0.82	10.6±0.98	12.55±0.21
1-Octen-3-ol	7.35±1.3	5.63±.37	4.93±0.22	6.85±1.2	6.31±0.1
1-Octanol	1.58±0.12	1.64±0.1	1.55±0.1	1.62±0.13	1.72±0.16
1-Nonanol	0.72±0.17	0.78±0.04	1.14±0.06	1.23±0.06	1.55±0.2
Benzyl alcohol	9.02±2.11	6.69±0.28	7.35±0.41	7.39±0.81	8.51±2.1
Phenylethyl alcohol	88.16±7.5	79.21±2.42	90.31±3.09	73.44±8.3	100.12±27.56
1,4-Butanediol	0.37±0.06	0.2±0.02	0.19±0.01	0.12±0.02	0.14±0.14
1-Dodecanol	nd	0.65±0.09	0.69±0.17	0.62±0.19	1.02±0.3
∑ Alcohols	332.55±22.25	310.12±9.11	311.27±23.85	287.43±25.08	413.31±18.42
<b>Acids</b>					
Acetic acid	94.59±4.88	68.71±10.31	77.67±3.88	76.04±5.84	100.91±10.02
Isobutyric acid	4.26±0.13	3.81±0.34	4.01±0.05	2.91±0.47	4.21±0.31
Butanoic acid	42.45±7.99	31.54±2.04	31.69±2.63	29.2±3.31	34.05±2.33
Methacrylic acid	nd	nd	1.7±0.02	nd	1.23±1.95
Pentanoic acid	5.23±0.9	2.68±0.14	4.58±1.15	1.89±0.34	2.51±0.65
Hexanoic acid	95.05±8.77	54.47±1.59	73.47±8.51	58.5±0.9	44.86±15.24
2-Ethyl-hexanoic acid	13.56±2.3	1.36±0.22	1.09±0.03	0.97±0.08	2.93±1.56
Heptanoic acid	11.3±0.73	2.66±0.27	2.75±0.34	1.81±0.16	2.06±0.26
Octanoic acid	nd	nd	nd	nd	nd
Nonanoic acid	12.43±4	4.04±0.35	8.88±0.97	2.39±0.2	2.4±0.16
Benzenecarboxylic acid	5.23±0.78	4.51±0.25	4.64±1.61	5.39±2.65	6.73±4.93
∑ Acids	284.1±29.23	173.78±14.2	210.47±14.2	179.12±9.39	201.87±31.62
<b>Esters</b>					
β-Phenylethyl acetate	10.09±0.2	10.41±0.26	11.06±0.08	10.33±0.98	12.32±2.53
<b>Lactones</b>					
γ-Caprolactone	2.91±0.44	2.28±0.12	2.52±0.12	2.17±0.13	2.87±0.79
δ-Decalactone	1.18±0.24	1.24±0.08	1.34±0.09	0.99±0.06	1.13±0.27
∑ Lactones	4.09±0.67	3.52±0.19	3.86±0.13	3.16±0.13	4±1.06
<b>Furan derivatives</b>					
2-Pentyl-furan	14.36±3.95	20.46±1.84	16.73±1.36	16.64±0.67	9.04±7.13
Furfural	67.85±0.62	74.03±5.3	65.94±4.98	59.65±3.53	49.09±0.5
2-Furanmethanol	3.61±0.26	3.28±0.15	3.24±0.13	2.87±0.09	4.54±1.95
∑ Furan derivatives	85.82±3.84	97.77±7.25	85.91±6.21	79.16±4.1	62.67±4.8
<b>Terpenes</b>					
Limonene	62.61±6.41	30.37±2.14	24.14±1.45	21.07±2.49	27.17±1.14
cis-Linalool oxide	0.96±0.08	0.91±0.08	0.87±0.01	0.86±0.01	1.11±0.15
Linalool	2.32±0.41	1.86±0.07	1.75±0.07	1.71±0.21	2.07±0.28
α-Terpineol	0.96±0.03	0.6±0.04	0.59±0.1	0.76±0.11	0.76±0.32
∑ Terpenes	66.85±6.71	33.75±2.32	27.35±1.42	24.4±2.21	31.1±0.62

Pinot noir	Days				
	0	1	7	14	21
<b>Ketones</b>					
2-Pentanone	28.84±2.17	22.64±1.63	37.55±2.93	30.58±0.67	46.38±3.79
2,3-Pentanedione	213.37±13.23	174.54±3.09	220.45±36.44	228.16±5.74	169.34±19.14
2-Heptanone	231.10±6.59	233.45±23.16	234.25±13.66	235.49±3.54	435.23±18.55
Acetoin	335.59±5.60	357.84±21.54	392.83±27.99	368.68±5.72	332.46±19.45
6-Methyl-5-hepten-2-one	2.38±0.13	1.51±0.17	1.61±0.19	1.81±0.07	2.62±0.33
3-Hydroxy-2-pentanone	20.93±0.79	29.45±1.84	30.66±1.30	30.75±1.22	35.06±0.05
2-Nonanone	48.23±0.05	52.73±3.10	52.09±0.22	53.83±1.15	63.75±3.25
6-Methyl-3,5-heptadien-2-one	0.34±0.01	0.57±0.02	0.29±0.05	0.38±0.03	0.36±0.04
2-Undecanone	6.38±0.06	8.18±0.32	7.47±1.11	8.60±0.37	6.78±0.14
2-Tridecanone	0.64±0.01	0.80±0.02	0.89±0.22	0.95±0.08	0.66±0.02
∑ Ketones	887.81±28.12	881.72±54.39	978.09±17.87	959.24±5.74	1092.65±14.77
<b>Alddehydes</b>					
Nonanal	3.19±0.04	2.64±0.14	7.03±5.32	2.17±0.06	1.75±0.11
Benzaldehyde	8.94±0.39	14.88±0.66	9.09±1.60	8.90±0.08	8.24±1.24
4-Methyl-benzaldehyde	2.45±0.05	2.15±0.29	1.87±0.23	3.67±0.80	2.78±0.59
Dodecanal	0.34±0.06	0.27±0.19	0.33±0.05	0.60±0.02	0.46±0.11
∑ Alddehydes	14.92±0.53	19.94±0.31	18.31±6.74	15.34±0.79	13.23±1.83
<b>Alcohols</b>					
Isobutanol	0.92±0.85	1.02±0.46	0.75±0.65	1.26±0.20	1.06±1.01
1-Pentanol	8.20±0.74	6.69±3.13	5.83±1.63	5.67±0.17	7.80±0.83
3-Methyl-1-butanol	41.26±5.75	18.84±3.23	31.38±2.69	22.10±4.27	44.87±7.13
1-Hexanol	38.60±3.44	134.15±6.97	29.23±3.86	25.77±0.12	46.89±5.66
2-Hexen-1-ol	3.44±0.52	12.75±1.15	2.54±0.24	2.63±0.06	3.73±0.17
1-Octen-3-ol	6.35±1.25	5.53±0.34	7.06±4.04	4.07±0.37	6.18±0.80
1-Octanol	0.96±0.07	1.61±0.09	0.97±0.38	0.86±0.11	0.99±0.05
1-Nonanol	0.41±0.01	0.46±0.01	0.66±0.08	0.96±0.08	0.95±0.08
Benzyl alcohol	5.52±0.21	6.59±0.21	9.15±4.19	9.61±0.67	5.31±0.09
Phenylethyl alcohol	9.58±0.74	78.92±2.35	10.51±2.78	14.42±1.05	8.81±0.25
1,4-Butanediol	0.32±0.08	0.19±0.01	0.23±0.09	0.18±0.01	0.26±0.05
1-Dodecanol	0.24±0.23	0.46±0.12	0.22±0.06	0.19±0.03	0.35±0.04
∑ Alcohols	115.79±3.42	267.21±3.68	98.54±19.98	87.73±6.64	127.19±15.58
<b>Acids</b>					
Acetic acid	84.07±6.56	68.19±10.30	112.32±19.88	164.22±8.96	117.58±2.09
Isobutyric acid	1.70±0.21	3.74±0.32	1.33±0.09	1.87±0.08	1.78±0.04
Butanoic acid	32.14±0.98	30.10±3.45	32.11±2.44	45.60±1.48	34.41±2.10
Methacrylic acid	nd	nd	nd	0.79±0.19	0.61±0.12
Pentanoic acid	4.35±0.61	2.87±0.27	5.19±0.38	3.36±0.14	4.11±0.40
Hexanoic acid	56.67±4.34	53.87±1.24	83.55±19.44	86.86±1.35	66.45±2.91
2-Ethyl-hexanoic acid	15.73±1.67	5.63±7.61	15.12±6.20	10.82±4.08	23.59±1.18
Heptanoic acid	8.12±0.69	7.01±1.29	11.41±1.33	11.27±2.22	15.03±1.17
Octanoic acid	nd	nd	nd	nd	nd
Nonanoic acid	5.49±0.07	4.82±1.19	13.20±7.16	15.81±1.31	10.41±1.08
Benzenecarboxylic acid	2.42±0.89	1.65±0.24	1.98±0.19	10.73±0.03	3.11±0.19
∑ Acids	210.70±0.78	177.86±8.45	276.20±41.93	351.33±9.24	277.07±8.54
<b>Esters</b>					
β-Phenylethyl acetate	0.56±0.03	0.48±0.01	0.45±0.09	0.48±0.03	0.56±0.02
<b>Lactones</b>					
γ-Caprolactone	0.57±0.02	0.49±0.01	1.16±0.62	0.81±0.09	0.56±0.01
δ-Decalactone	0.66±0.07	0.60±0.07	1.26±0.36	1.42±0.02	0.45±0.04
∑ Lactones	1.24±0.08	1.09±0.07	2.42±0.93	2.23±0.10	1.02±0.03
<b>Furan derivatives</b>					
2-Pentyl-furan	11.01±1.94	8.06±1.65	11.29±0.99	9.10±1.06	14.33±2.11
Furfural	98.84±3.20	73.11±5.11	82.26±12.88	82.08±0.92	82.09±13.22
2-Furanmethanol	2.59±0.05	3.08±0.27	3.06±0.72	3.62±0.14	3.64±0.30
∑ Furan derivatives	112.45±5.18	84.25±5.36	96.61±13.26	94.80±0.28	100.05±15.02
<b>Terpenes</b>					
Limonene	28.60±3.26	29.83±1.98	42.39±12.67	28.39±1.33	26.38±1.60
cis-Linalool oxide	1.04±0.07	0.91±0.08	0.89±0.11	1.00±0.07	1.11±0.14
Linalool	0.45±0.03	1.85±0.06	0.60±0.24	0.49±0.04	0.46±0.05
α-Terpineol	0.95±0.13	0.65±0.06	1.01±0.29	1.55±0.17	1.02±0.02
∑ Terpenes	31.03±3.06	33.24±2.09	44.89±13.31	31.43±1.61	28.97±1.68