LWT - Food Science and Technology 62 (2015) 1043-1052

Contents lists available at ScienceDirect

# LWT - Food Science and Technology

journal homepage: www.elsevier.com/locate/lwt

## Systematic approach for the development of fruit wines from industrially processed fruit concentrates, including optimization of fermentation parameters, chemical characterization and sensory evaluation



LWI

Eduardo Coelho<sup>a</sup>, Mar Vilanova<sup>b</sup>, Zlatina Genisheva<sup>a</sup>, José M. Oliveira<sup>a</sup>, José A. Teixeira<sup>a</sup>, Lucília Domingues<sup>a, \*</sup>

<sup>a</sup> CEB – Centre of Biological Engineering, University of Minho, 4710-057, Braga, Portugal <sup>b</sup> Misión Biológica de Galicia – CSIC, PO Box 28, 38080 Pontevedra, Spain

#### ARTICLE INFO

Article history: Received 9 October 2014 Received in revised form 5 February 2015 Accepted 15 February 2015 Available online 24 February 2015

Keywords: Alcoholic fermentation Mathematical modelling Antioxidant activity Chemical–sensory correlation

#### ABSTRACT

This work presents an optimized approach alongside with the mathematical models describing the production of fruit wines, using fruit concentrates as an alternative to attain the desired ethanol yields and enhance organoleptic and functional properties. Box-Behnken design was used for modeling and optimization of ethanol yield and productivity in banana, orange, cherry and mango concentrates fermentations. Optimization allowed ethanol yields of 72.3  $\pm$  2.08 g·L<sup>-1</sup> in orange, 101  $\pm$  1.78 g·L<sup>-1</sup> in mango, 66.1  $\pm$  4.02 g·L<sup>-1</sup> in cherry and 98.2  $\pm$  7.88 g·L<sup>-1</sup> in banana with maximal productivities of 0.4  $\pm$  0.0 g·L<sup>-1</sup>·h<sup>-1</sup>, 1.0  $\pm$  0.1 g·L<sup>-1</sup>·h<sup>-1</sup>, 1.7  $\pm$  0.2 g·L<sup>-1</sup>·h<sup>-1</sup> and 1.0  $\pm$  0.1 g·L<sup>-1</sup>·h<sup>-1</sup>, respectively. Evaluation of total antioxidant activity by FRAP demonstrated fruit wines potential for the development of foods and formulations with functional properties, attaining 22.6  $\pm$  0.46 mmol·L<sup>-1</sup> for orange, 7.14  $\pm$  0.77 mmol·L<sup>-1</sup> for mango, 28.0  $\pm$  1.84 mmol·L<sup>-1</sup> for cherry and 9.54  $\pm$  0.89 mmol·L<sup>-1</sup> for banana wines. Characterization of aroma active compounds was performed by GC–MS and sensory evaluation by trained panelists. All fruit wines had good acceptance with cherry wine presenting the highest overall preference, followed by orange, mango and banana wines. Correlation between chemical and sensory groperties was established with PLSR2 between analytical and sensory data, which allowed an insight of chemical composition impact in consumer perceived quality.

© 2015 Elsevier Ltd. All rights reserved.

## 1. Introduction

Fruit is one of the main sources of diversity for food formulations. Besides providing flavors, aromas and colors, some are also rich in dietary fiber, vitamins and phenolic compounds, with functional properties advantageous for food design (Müller, Gnoyke, Popken, & Bröhm, 2010). However, fruit possess limited shelf-life, causing product losses and spoilage, which can be amplified by quality regulation, where pieces that do not fulfill the desired morphological requisites are not suitable for direct distribution (Gustavsson, Cederberg, Sonesson, Otterdijk, & Meybeck, 2011). Alcoholic fermentation is highly acknowledged in the beverage industry, generating less perishable value added products, such as wine and beer (Caplice & Fitzgerald, 1999). Besides conservation, fermentation has impact on secondary metabolites, transforming organoleptic properties and differentiating products (Ribéreau-Gayon, Glories, Maujean, & Dubourdieu, 2006). Furthermore, alcoholic fermentation can generate added-value products by further processing, such as vinegars, spirits and food ingredients. One concern regarding alcoholic beverages is their health impact, being the type of beverage and patterns of consumption extremely important when focusing consumer concerns. Patterns of excessive consumption are widely acknowledged by their strong negative effects on human and public health (Room, Babor, & Rehm, 2005). On the other hand, beneficial effects of moderate drinking have been reported, such as lower risk of cardiovascular diseases (Artero, Artero, Tarín, & Cano, 2015), lower risk of type 2 diabetes (Koppes, Dekker, Hendriks, Bouter, & Heine, 2005) and reducing cognitive function

<sup>\*</sup> Corresponding author. Tel.: +351 253 604402; fax: +351 253 604429. *E-mail address:* luciliad@deb.uminho.pt (L. Domingues).

losses (Neafsey & Collins, 2011). Recent efforts have been made to create alcoholic beverages from fruit, as recently reported for fruit wastes re-valorization (Isitua & Ibeth, 2010). Moreover, experimental approaches for the production of cherry (Sun et al., 2013) and orange (Santos, Duarte, Carreiro, & Schwan, 2013) spirits were recently reported, focusing on the beverages sensory quality. Some of these works included strategies such as enzymatic hydrolysis. sucrose addition or post fermentation distillation to compensate low fermentable sugar concentration and attain the desired ethanol yield. As an alternative, this work resorts to fruit concentrates for sugar concentration and increase of ethanol yield, concentrating also taste, aroma and functional features to generate a wine grade product, suitable for multiple applications. A systematic approach was carried out focusing on mathematical modeling and optimization of fermentation parameters to maximize ethanol production and productivity. Fruit wines were characterized to establish chemical-sensorial correlations and assess acceptability. Their functional potential was evaluated, with quantification of antioxidant activity, providing further added-value to fruit wines as food grade products.

## 2. Materials and methods

## 2.1. Chemicals

The following chemicals were used for the standards: citric acid monohydrate (99.5%) (Merck), absolute ethanol (99.5%) (Panreac), L(-)- Malic Acid (99%) (Acros Organics),  $\alpha$ -D-Glucose (96%) (Aldrich Chemistry), D(-)- Fructose (99%) (Acros Organics), D(+)-Saccharose (99%) (Fisher Scientific) and Iron (II) Sulfate Heptahydrate (99%) (Acros Organics). For the FRAP assay: 2,4,6-Tris(2-pyridyl)-striazine (>98%), Iron (III) chloride (>97%) and Sodium Acetate (≥99%), all from Sigma-Aldrich. For GC-FID the following standards were used: acetaldehyde ( $\geq$ 99.5%), methyl acetate ( $\geq$ 99.9%), 1-propanol (≥99.9%), 2-methyl-1-propanol (≥99.8%), 2-methyl-1butanol (298%), 3-methyl-1-butanol (299.8%), 2,3-butanediol, levo (299.0%), 2,3-butanediol, meso (299.0%), 2-phenylethanol  $(\geq 99.0\%)$  from (Fluka) and ethyl acetate (99.8%), methanol (>99.8%), diethyl succinate (99.0%) from (Sigma-Aldrich). For GC-MS calibration: 1-hexanol (≥99.9%), Z-3-hexenol (≥98%), 1octanol ( $\geq$ 99.5%), furfuryl alcohol ( $\geq$ 98%), isobutyl acetate  $(\geq 98.5\%)$ , 2-phenylethyl acetate  $(\geq 99.0\%)$ , fenchol  $(\geq 99.0\%)$ , borneol (>95.0%), trans-furan linalool oxide and cis-furan linalool oxide ( $\geq$ 97.0%), propanoic acid ( $\geq$ 99.5%), isobutyric acid ( $\geq$ 99.5%), butyric acid ( $\geq$ 99.5%), hexanoic acid ( $\geq$ 98.0%), decanoic acid  $(\geq 98.0\%)$ , benzaldehyde  $(\geq 99.0\%)$  from Fluka, 3-ethoxy-1-propanol (97%), benzyl alcohol (≥99.0%), 2-phenoxyethanol (98.0%), ethyl butyrate (99.0%), 3-methylbutyl acetate (>99.0%), ethyl hexanoate (>99.9%), Z-3-hexenyl acetate (>98%), ethyl octanoate (>99.0%), ethyl 3-hydroxybutyrate (99.0%), ethyl decanoate (>99.0%), benzyl acetate (>99.0%), linalool (97%), terpinen-4-ol (>99.0%), citronellol (95%), nerol (97%), geraniol (98%), eugenol (99%), 4-vinylguaiacol (≥98%), 4-vinylphenol (12%), acetovanillone (98%), zingerone 3,4,5-trimethoxyphenol (97%), 3-methyl + (>96%). 2methylbutyric acids (99%), octanoic acid (≥99.5%), methoxyfuraneol ( $\geq$ 97%), furaneol ( $\geq$ 98%),  $\gamma$ -decalactone ( $\geq$ 98%), 2methyltetrahydrothiophen-3-one (>97%), 2-(methylthio)ethanol (99%), methionol (98%), 6-methyl-5-hepten-2-one (99%) from Sigma–Aldrich, isopulegol I (>85.0%) from TCI, myrcenol ( $\geq$ 90.0%) from Ventós and  $\alpha$ -terpineol ( $\geq$ 98.0%) from Merck.

## 2.2. Characterization of fermentable sugars in the fruit concentrates

Fermentable sugars were quantified by HPLC using a Varian Metacarb 87H column,  $H_2SO_4$  5 mmol·L<sup>-1</sup> as mobile phase at

0.5 mL·min<sup>-1</sup> and oven temperature of 35 °C to prevent sucrose hydrolysis. Sugars were measured using a Jasco RI-1530 detector and quantified with the proper calibration curves. Total fermentable sugar concentration was calculated by sum of fermentable sugars concentration, namely sucrose, glucose and fructose.

## 2.3. Fruit mashes preparation

Four whole, non-clarified, industrial fruit concentrates were used, kindly provided by Frulact S.A. (Maia, Portugal) with °Brix, pH and processing presented in Table 1.

### 2.4. Alcoholic fermentations

Musts were prepared diluting fruit mash with sterile water to the desired initial °Brix ( $B_i$ ), followed by pH correction to 4.5 using 5 mol·L<sup>-1</sup> NaOH. Alcoholic fermentation was conducted in Erlenmeyer flasks with glycerol lock, ensuring anaerobic conditions and CO<sub>2</sub> exhaustion. Musts were inoculated with lyophilized oenological yeast Lalvin QA23 (Lallemand), incubated with temperature control, orbital agitation of 150 min<sup>-1</sup> and monitored by weight loss measurement, equivalent to CO<sub>2</sub> production and exhaustion, for stationary phase determination. Ethanol concentration (C<sub>EtOH</sub>) was quantified by HPLC, and productivity (P) was calculated dividing C<sub>EtOH</sub> by stationary phase entry time.

## 2.5. Factorial design

Ethanol yield and productivity were mathematically modeled using Box -Behnken design, to evaluate dependent variables (ethanol concentration ( $C_{EtOH}$ ) and productivity (P)) response to fermentation parameters, namely must initial °Brix ( $B_i$ ), temperature (T) and inoculum concentration ( $C_{inoc}$ ). Box-Behnken design was outlined with 3 independent variables and triplicates in the central point, generating the experiments represented in Table 2, where the independent variables are expressed in dimensional and adimensional parameters. For the optimization, mathematical models were converged for determination of optimal fermentation conditions and responses, using StatGraphics Plus software (Version 5.1, Statistical Graphics corp.). After optimization, a validation assay was conducted to determine models accuracy.

#### 2.6. Chemical characterization of fruit wines

### 2.6.1. Ethanol concentration and organic acid composition

Ethanol and organic acids were measured by HPLC, using a Varian Metacarb 87H column using  $H_2SO_4$  5 mmol·L<sup>-1</sup> mobile phase at a 0.7 mL·min<sup>-1</sup> flow. Organic acids were measured using a Jasco 870-UV detector (210 nm wavelength) and ethanol was

Table 1

Brix degree ( ${}^{\circ}B$ ), initial pH and fruit mash processing steps, of the fruit concentrates used for must preparations and fermentation.

Mash	°B (°Brix)	Initial pH	Processing
Comminuted Orange	40.0	3.8 ± 0.1	Whole crunched, heated, chilled and packed
Mango puree	28.0	3.8 ± 0.1	Mashed, fine sieved, concentrated, pasteurized and packed
Sour Cherry puree	32.0	3.4 ± 0.3	Mashed, fine sieved, concentrated, pasteurized and packed
Banana puree	31.5	4.4 ± 0.2	Peeled, mashed, acidified, homogeneized, deaerated, concentrated, pasteurized and packed

#### Table 2

Box-Behnken experimental planning included process parameters studied (initial Brix degree ( $B_i$ ), Temperature (T) and Inoculum concentration ( $C_{inoc}$ )), expressed in terms of adimensional and corresponding dimensional values (between brackets).

Experiment	B <sub>i</sub> (°Brix)		T (°C)	$C_{inoc} (g \cdot L^{-1})$	
	Mango	Cherry and orange	Banana	All fruits	All fruits
1	-1 (14.0)	-1 (14.5)	-1 (14.3)	-1 (18.0)	0 (0.6)
2	1 (24.0)	1 (24.0)	1 (24.3)	-1 (18.0)	0 (0.6)
3	-1 (14.0)	-1 (14.5)	-1 (14.3)	1 (32.0)	0 (0.6)
4	1 (24.0)	1 (24.0)	1 (24.3)	1 (32.0)	0 (0.6)
5	-1 (14.0)	-1 (14.5)	-1 (14.3)	0 (25.0)	-1 (0.3)
6	1 (24.0)	1 (24.0)	1 (24.3)	0 (25.0)	-1 (0.3)
7	-1 (14.0)	-1 (14.5)	-1 (14.3)	0 (25.0)	1 (0.9)
8	1 (24.0)	1 (24.0)	1 (24.3)	0 (25.0)	1 (0.9)
9	0 (19.0)	0 (19.5)	0 (19.3)	-1 (18.0)	-1 (0.3)
10	0 (19.0)	0 (19.5)	0 (19.3)	1 (32.0)	-1 (0.3)
11	0 (19.0)	0 (19.5)	0 (19.3)	-1 (18.0)	1 (0.9)
12	0 (19.0)	0 (19.5)	0 (19.3)	1 (32.0)	1 (0.9)
13	0 (19.0)	0 (19.5)	0 (19.3)	0 (25.0)	0 (0.6)
14	0 (19.0)	0 (19.5)	0 (19.3)	0 (25.0)	0 (0.6)
15	0 (19.0)	0 (19.5)	0 (19.3)	0 (25.0)	0 (0.6)

measured using a Jasco RI-1530 detector. Proper standards were used for quantification.

#### 2.6.2. Major volatile compounds

Major volatiles were analyzed after adding 410  $\mu$ g of 4-nonanol (internal standard) to 5 mL of sample. A Chrompack CP-9000 gas chromatograph equipped with a split/splitless injector, a flame ionization detector (FID) and a capillary column, coated with CP-Wax 57CB (50 m × 0.25 mm; 0.2  $\mu$ m film thickness, Chrompack), was used. Injector and detector temperatures were set to 250 °C. Oven temperature was initially held at 60 °C, for 5 min, then programmed to rise from 60 °C to 220 °C, at 3 °C min<sup>-1</sup>, and maintained at 220 °C for 10 min. The carrier gas was helium 4 × (Praxair) at an initial flow rate of 1 mL min<sup>-1</sup> (125 kPa at the head of the column). Analyses were performed by injecting 1  $\mu$ L of sample in the split mode (15 mL min<sup>-1</sup>). Quantification of major volatiles was performed using Star-Chromatography Workstation version 6.41 (Varian) software, taking into account the detector response factor for each analyte and comparing retention times with those of pure standards.

#### 2.6.3. Minor volatile compounds

Minor volatiles were analyzed by GC-MS after extraction of 8 mL of sample with 400 µL of dichloromethane, spiked with 3.28 µg of 4nonanol (IS). A gas chromatograph Varian 3800 with a 1079 injector and an ion-trap mass spectrometer Varian Saturn 2000 was used.  $1 \,\mu$ L injections were made in splitless mode (30 s) in a Varian Factor Four VF-Wax ms column (30 m  $\times$  0.15 mm; 0.15  $\mu$ m film thickness). Carrier gas was helium  $4 \times$  (Praxair) at a constant flow of 1.3 mL·min<sup>-1</sup>. The detector was set to electronic impact mode with an ionization energy of 70 eV, mass acquisition range from 35 m/z to 260 m/z and acquisition interval of 610 ms. Oven temperature was initially set to 60 °C for 2 min and then raised from 60 °C to 234 °C at 3 °C min  $^{-1}$ , raised from 234 °C to 250 °C at 10 °C min  $^{-1}$  and maintained at 250 °C for 10 min. Injector temperature was maintained at 250 °C during analysis and the split flow was maintained at 30 mL min<sup>-1</sup>. Compounds were identified using MS Workstation version 6.9 (Varian) software, by comparing their mass spectra and retention indexes with those of pure standards. Minor compounds were quantified in terms of 4-nonanol equivalents.

## 2.6.4. Antioxidant activity

Antioxidant activity was evaluated using Ferric Reducing Antioxidant Power (FRAP) assay. 10  $\mu$ L of sample were mixed, in a 96 well microplate, with 290  $\mu$ L of freshly prepared FRAP reagent. FRAP

reagent was prepared by mixing a 10 mmol·L<sup>-1</sup> 2,4,6-tris-(1-pyridyl)-5-triazine (TPTZ) solution (made with 40 mmol·L<sup>-1</sup> HCl) with a 20 mmol·L<sup>-1</sup> FeCl<sub>3</sub> solution and 300 mmol·L<sup>-1</sup> acetate buffer (pH 3.6) in a volumetric proportion of 1:1:10. After mixing, samples

#### 2.7. Sensory analysis of fruit wines

## 2.7.1. Sensory evaluation

Sensory analysis was carried out by seven trained panellists from Rias Baixas A.O.C. (Galicia, Spain), two male and five female, ages between 40 and 50 years. All judges were experienced tasters and have previously participated in similar studies. Judging was performed in a professional-standard room in agreement with the ISO Norm 8589 (1988). Evaluation was carried out in two sessions. In the first, evaluation was carried out using the QDA methodology, to establish fruit wines descriptors. One training period of 1 h was carried out, where judges generated visual, olfactory and gustatory descriptive terms to define the fermented fruit samples. In the second session, a sample volume of 30 mL of was evaluated in taster glasses at 12 °C. During the analysis, judges scored the intensity of each attribute using a 9-point scale, where 9 indicated a very high intensity. The relative frequency (F), relative intensity (I) and geometric mean (GM) of the different descriptors were calculated for each sample. Geometric mean was calculated as the square root of the product between relative intensity and relative frequency.

were incubated at 37 °C followed by determination of the absorbance at 593 nm. FRAP activity was expressed as concentration of Fe<sub>2</sub>SO<sub>4</sub> equivalents, supported by the proper calibration curve.

$$GM/(\%) = \sqrt{I \times F} \times 100$$

*I* corresponds to the sum of the intensities given by the panel for a descriptor, divided by the maximum possible intensity for this descriptor; and F is the number of times that the descriptor was mentioned divided by the maximum number of times that it could be mentioned.

Descriptors were classified for each sample using the *GM* according to the International Organization for Standardization – ISO Norm 11035 (1994), which allowed the elimination of the descriptors whose geometric means were relatively low. This method allowed taking into account descriptors rarely mentioned but very important in terms of perceived intensity, and descriptors with a low perceived intensity but often mentioned.

## 2.7.2. Data analysis

Sensory and instrumental data were analyzed using XLstat-Pro. Relative intensity (I) and Geometric mean (GM) data were statistically analyzed using multivariate techniques. To show the relationship between sensory variables and volatile families, Partial Least Squares Regression (PLSR2) was applied. PLSR2 shows the relationship between X data (volatile compounds) and Y data (sensory descriptor). The X data are actively used in estimating the latent variables to ensure that the first components are those that are most relevant for predicting the Y variable. This is a data reduction technique since it reduces the X variables to a set of no correlated factors that describe the variation in the data.

### 3. Results and discussion

#### 3.1. Fruit wine production optimization

Fermentations were conducted in order to evaluate ethanol production and productivity as described in 2.4. The resulting data (supplied in Supplementary Table 1) allowed mathematical modeling of fermentation response for each fruit, with the resulting

Table 3

Mathematical models relating ethanol production ( $C_{EtOH}$  (g·L<sup>-1</sup>)) and productivity (P (g·L<sup>-1</sup>h<sup>-1</sup>) with adimensional values for initial °Brix ( $B_i$ ), temperature (T) and inoculum concentration ( $C_{inoc}$ ) for each fruit, resulting from the factorial design.  $\mathbb{R}^2$  is the regression coefficient of the model.

Fruit	Model for ethanol yield	R <sup>2</sup>
Orange	$C_{EtOH} = 60.2886 + 11.5188*Bi - 2.2024*T + 0.1842*C_{inoc} + 0.0088*B_i^2 + 7.6978*B_i*T + 3.9246*B_i*C_{inoc} - 8.6496*T^2 - 1.7994*T*C_{inoc} - 3.3039*C_{inoc}^2 + 0.0088*B_i^2 + 0$	0.921
	$P = 0.4286 + 0.0311*B_{\rm i} + 0.1476*T + 0.0146*C_{\rm inoc} + 0.0104*B_{\rm i}^2 + 0.0741*B_{\rm i}*T + 0.0138*B_{\rm i}*C_{\rm inoc} - 0.0952*T^2 + 0.0025*T*C_{\rm inoc} - 0.0427*C_{\rm inoc}^2 + 0.0104*B_{\rm i}^2 + 0.0741*B_{\rm i}*T + 0.0138*B_{\rm i}*C_{\rm inoc} - 0.0952*T^2 + 0.0025*T*C_{\rm inoc} - 0.0427*C_{\rm inoc}^2 + 0.0104*B_{\rm i}^2 + 0.0741*B_{\rm i}*T + 0.0138*B_{\rm i}*C_{\rm inoc} - 0.0952*T^2 + 0.0025*T*C_{\rm inoc} - 0.0427*C_{\rm inoc}^2 + 0.0104*B_{\rm i}^2 + 0.0741*B_{\rm i}*T + 0.0138*B_{\rm i}*C_{\rm inoc} - 0.0952*T^2 + 0.0025*T*C_{\rm inoc} - 0.0427*C_{\rm inoc}^2 + 0.0104*B_{\rm i}^2 + 0.0741*B_{\rm i}*T + 0.0138*B_{\rm i}*C_{\rm inoc} - 0.0052*T*C_{\rm inoc} - 0.0427*C_{\rm inoc}^2 + 0.0104*B_{\rm i}^2 + 0.0741*B_{\rm i}*T + 0.0138*B_{\rm i}*C_{\rm inoc} - 0.0952*T*C_{\rm inoc} - 0.0427*C_{\rm inoc}^2 + 0.0104*B_{\rm i}^2 + 0.0104*B_{\rm i}^2 + 0.0138*B_{\rm i}*C_{\rm inoc} - 0.0052*T*C_{\rm inoc} - 0.0427*C_{\rm inoc}^2 + 0.0014*B_{\rm inoc}$	0.993
Mango	$C_{EtOH} = 71.0173 + 19.4419*B_i + 2.8077*T + 1.2978*C_{inoc} + 3.4986*B_i^2 - 2.6515*B_i*T + 2.2436*B_i*C_{inoc} - 2.6432*T^2 - 4.3720*T*C_{inoc} - 3.8604*C_{inoc}^2 - 4.3720*T*C_{inoc} + 3.4986*B_i^2 - 2.6515*B_i*T + 2.2436*B_i*C_{inoc} - 3.6432*T^2 - 4.3720*T*C_{inoc} - 3.8604*C_{inoc}^2 - 3.8604*C_{$	0.924
	$P = 1.2244 + 0.0545^{8}B_{i} + 0.3987^{*}T - 0.0169^{*}C_{inoc} - 0.2197^{8}B_{i}^{2} + 0.0292^{*}B_{i}^{*}T + 0.1023^{*}B_{i}^{*}C_{inoc} - 0.2000^{*}T^{2} - 0.0629^{*}T^{*}C_{inoc} - 0.0788^{*}C_{inoc}^{2} - 0.0788^{*$	0.932
Cherry	$C_{EtOH} = 52.2794 + 15.3397*B_i - 0.1941*T - 0.6810*C_{inoc} + 0.1316*B_i^2 + 0.0329*B_i*T - 0.5329*B_i*C_{inoc} + 0.4803*T^2 + 0.3224*T*C_{inoc} + 1.5593*C_{inoc}^2 + 0.5224*T*C_{inoc} + 0.1316*B_i^2 + 0.0329*B_i*T - 0.5329*B_i*C_{inoc} + 0.4803*T^2 + 0.3224*T*C_{inoc} + 0.593*C_{inoc}^2 + 0.5329*B_i*T - 0.5329*B_i*$	0.999
	$P = 1.8671 + 0.1133^{*}B_{i} + 0.8186^{*}T + 0.1646^{*}C_{inoc} - 0.1358^{*}B_{i}^{2} + 0.0103^{*}B_{i}^{*}T + 0.0015^{*}B_{i}^{*}C_{inoc} - 0.1128^{*}T^{2} + 0.0334^{*}T^{*}C_{inoc} + 0.0245^{*}C_{inoc}^{2} + 0.0135^{*}B_{i}^{*}T + 0.0015^{*}B_{i}^{*}C_{inoc} - 0.1128^{*}T^{2} + 0.0034^{*}T^{*}C_{inoc} + 0.0245^{*}C_{inoc}^{2} + 0.0015^{*}B_{i}^{*}T + 0.0015^{*}B_{i}^{*}C_{inoc} - 0.1128^{*}T^{2} + 0.0034^{*}T^{*}C_{inoc} + 0.0245^{*}C_{inoc}^{2} + 0.0015^{*}B_{i}^{*}T + 0.0015^{*}B_{i}^{*}C_{inoc} - 0.1128^{*}T^{2} + 0.0034^{*}T^{*}C_{inoc} + 0.0245^{*}C_{inoc}^{2} + 0.0015^{*}B_{i}^{*}T + 0.0015^{*}B_{i}^{*}$	0.990
Banana	$C_{\textit{EtOH}} = 82.6233 + 13.7804*B_i + 1.7896*T + 0.0526*C_{\textit{inoc}} - 8.3952*B_i^2 + 2.1778*B_i*T - 0.3553*B_i*C_{\textit{inoc}} + 2.7896*T^2 + 0.7632*T*C_{\textit{inoc}} - 4.8687*C_{\textit{inoc}}^2 + 0.7632*T*C_{\textit{inoc}} + 0.8687*C_{\textit{inoc}}^2 + 0.1687*C_{\textit{inoc}} + 0.8687*C_{\textit{inoc}} + 0.8687*C_{\textit{inoc}} + 0.8687*C_{\textit{inoc}}^2 + 0.1681*C_{\textit{inoc}} + 0.1681$	0.933
	$P = 1.2805 - 0.2935*B_{\rm i} + 0.3836*T + 0.1402*C_{\rm inoc} - 0.2386*B_{\rm i}^2 - 0.1109*B_{\rm i}*T - 0.0586*B_{\rm i}*C_{\rm inoc} - 0.0424*T^2 + 0.0022*T*C_{\rm inoc} - 0.0877*C_{\rm inoc}^2 - 0.0424*T^2 + 0.0022*T*C_{\rm inoc} - 0.0424*T^2 + 0.0022*T*C_{\rm inoc} - 0.0424*T^2 + 0.0022*T*C_{\rm inoc}^2 - 0.0424*T^2 + 0.0022*T*C_{\rm inoc} - 0.0424*T^2 + 0.0022*T*C_{\rm inoc}^2 - 0.0424*T^2 + 0.0022*T*C_{\rm inoc} - 0.0424*T^2 + 0.044*T^2 + 0.044*T$	0.940

models presented in Table 3. Mathematical models showed proper adjustment to the experimental data, demonstrated by the  $R^2$ values obtained in the range of 0.9-1. Optimal ethanol and productivity values were predicted for each fruit and are presented in Table 4. Optimal conditions determined were within the values studied in the assays. Optimal  $B_i$  was consistent with maximal ethanol production, close to the highest  $B_i$  studied in the experimental designs and inherent to high sugar content in the must. Similar optimal fermentation temperatures for all fruits were obtained, between 22.6 °C and 24.7 °C, with the exception of banana with optimal fermentation temperature of 31.9 °C. This process parameter is highly influenced by the yeast used, which for the microorganism in use is in the range of 14.0 °C-28.0 °C, as indicated by the supplier. However, optimal process temperature can be influenced by the rheological properties of the must, and in the case of banana must, its high viscosity can affect yeast growth and CO<sub>2</sub> diffusion, leading to a deviation in the optimal temperature value. Inoculum concentration values ranged through the entire interval studied, implying a strong influence of the fruit used on the amount of inoculum needed. Overall, response factors predicted were coherent with the expected from the conditions established. To confirm the relations established by the models, a validation assay was conducted in conditions similar to the optimal predicted, adjusting only fermentation temperature to 23.5 °C for orange, mango and cherry fermentations. Fermentations were monitored by CO<sub>2</sub> mass loss to determine fermentation time and calculate productivity, with fermentation profiles presented in Fig. 1. Banana, cherry and mango fermentations presented reduced lag phases, whereas orange presented a longer lag phase which can be related to yeast inhibition by orange must composition. Banana and mango fermentations entered stationary phase at about 100 h of fermentation, cherry at 40 h and orange at 190 h. The relative CO<sub>2</sub>

#### Table 4

Optimal process conditions (Initial Brix (B<sub>i</sub>), Temperature (T) and Inoculum concentration (C<sub>inoc</sub>)) and correspondent responses (C<sub>EtOH</sub>) and Productivity (P), predicted values using mathematical models for maximal ethanol production and for the validation conditions and experimental values obtained in the validation assay (real values). Errors represent standard deviations from fermentation triplicates and standard error for the estimate.

Fruit	Optimal conditions			Optimal responses			
	$B_i/^{\circ}B$	T/°C	$C_{inoc}/g \cdot L^{-1}$	$C_{EtOH}/g \cdot L^{-1}$	$P/g \cdot L^{-1} \cdot h^{-1}$		
Cherry Orange Mango Banana	22.9 24.2 24.0 24.2 Valida	22.6 24.7 23.2 31.9	0.49 0.72 0.83 0.63 nditions	$63.4 \pm 0.63$ 72.0 ± 5.44 94.9 ± 7.46 94.7 ± 5.29 Predicted values/Re	$1.5 \pm 0.1$ $0.5 \pm 0.0$ $1.0 \pm 0.2$ $1.0 \pm 0.2$ al values		
	$B_i/^{\circ}B$	T/°C	$C_{inoc}/g \cdot L^{-1}$	$C_{EtOH}/g \cdot L^{-1}$	$P/g \cdot L^{-1} \cdot h^{-1}$		
Cherry Orange Mango Banana	22.9 24.2 24.0 24.2	23.5 23.5 23.5 31.9	0.49 0.72 0.83 0.63	$\begin{array}{c} 63.0 \pm 0.7/66 \pm 4 \\ 71 \pm 5/72 \pm 2 \\ 95 \pm 7/101 \pm 1.8 \\ 95 \pm 5/98 \pm 8 \end{array}$	$\begin{array}{c} 1.6 \pm 0.1/1.7 \pm 0.2 \\ 0.4 \pm 0.0/0.4 \pm 0.0 \\ 1.0 \pm 0.2/1.0 \pm 0.1 \\ 1.0 \pm 0.2/1.0 \pm 0.1 \end{array}$		

production for all of them was consistent with the expected ethanol yield. Ethanol yield and productivity values obtained are presented in Table 4, alongside with the values predicted by the models for the validation assay conditions.

Ethanol yield and productivity in the validation assay were in good agreement with the responses predicted using the models, reinforcing their validity. Despite similar  $B_i$  for all four fermentations, cherry and orange presented lower ethanol productions than the ones observed for mango and banana. To substantiate these yields, fermentable sugar concentration was measured in each must and is presented in Table 5.

Fermentable sugars concentration in cherry must was lower in comparison with the other fruit, justifying the lower ethanol yield. In orange fermentation the lower ethanol yield can be a direct consequence of the long lag phase observed, probably caused by the presence of inhibitors in the concentrated must. This long lag phase influenced also productivity of orange wine, being largely inferior in comparison with the other fermentations. Nevertheless, orange fermentation allowed the production of a wine with  $72.3 \pm 2.08 \text{ g} \cdot \text{L}^{-1}$  of ethanol, without sugar addition to the must, 0.2 folds superior to the reported (Santos et al., 2013). Mango and banana presented similar fermentation behaviors, in good agreement with the chemical similarities of both musts allowing ethanol yields 1.5 and 0.5 folds superior to ones previously reported (Akubor, Obio, Nwadomere, & Obiomah, 2003; Reddy & Reddy, 2009).

Considering the results, optimization was successfully attained, with the mathematical models in good agreement with the experimental data. Ethanol yield and productivity were maximized in the studied conditions, making the process feasible for industrial implementation. Furthermore, mathematical models described are of upmost importance for selecting process conditions and predicting responses if alternative applications, quality features or subsequent processing steps are desired. Due to the use of fruit



**Fig. 1.**  $CO_2$  production and exhaustion (m<sub>CO2</sub>) during time-course (t) of alcoholic fermentation using optimal conditions predicted by ethanol production models for (--) Orange, (--) Mango, (--) Cherry and (--) Banana.

#### Table 5

HPLC analysis of sugar content in fruit musts (sum of fermentable sugars concentrations) and main organic acids found in fruit wines. Errors represent standard deviation from fermentation triplicates.

Fruit	Fruit musts	Fruit wines			
	Fermentable sugars $(g \cdot L^{-1})$	Citric acid $(g \cdot L^{-1})$	Malic acid $(g \cdot L^{-1})$		
Orange Mango Cherry Banana	$\begin{array}{c} 193 \pm 1.43 \\ 182 \pm 1.30 \\ 127 \pm 1.00 \\ 181 \pm 1.30 \end{array}$	$\begin{array}{c} 41.2 \pm 0.57 \\ 9.15 \pm 0.87 \\ 5.01 \pm 0.25 \\ 4.27 \pm 0.15 \end{array}$	$\begin{array}{c} 0.00 \pm 0.00 \\ 0.87 \pm 0.21 \\ 37.0 \pm 1.46 \\ 4.78 \pm 0.10 \end{array}$		

concentrates, ethanol yields in all fruit wines were superior to the observed in previous works, without the need for sugar addition or other strategies. Ethanol yield was within the ranges observed in grape wines, demonstrating the feasibility of fruit wine production from concentrates.

## 3.2. Characterization of fruit wines

Considering the production of a fermented beverage, with organoleptic and functional value, a characterization of the key compounds was conducted, in order to evaluate fruit wine composition.

#### 3.2.1. Organic acid composition

Among the compounds participating in wine flavor, organic acids may be considered as one of the main contributors to taste. Citric and malic acids are present in most fruit species (Tucker, 1993) and therefore dominant in fruit wines. Organic acids composition of the produced fruit wines is presented in Table 5. Citric acid was mainly found in orange and present in lower concentration in mango, cherry and banana wines. Malic acid was mainly found in cherry, with much lower concentration in banana, residual in mango and null in orange. Organic acids in fruit wines were highly fruit dependent, which is in good agreement with the described for the corresponding fruit, namely high content of citric acid in orange (Kelebek, Selli, Canbas, & Cabaroglu, 2009) and malic acid in cherry fruit (Usenik, Fabcic, & Stampar, 2007). In comparison to cherry and orange, mango and banana wines showed lower concentrations of these organic acids, also coherent with the titrable acidity previously reported for these wines (Akubor et al., 2003: Reddy & Reddy, 2009). The use of fruit concentrates in this work led to higher concentrations of these organic acids, when compared to the naturally found in the fruit, potentiating acidic flavors in the corresponding wines.

## 3.2.2. Antioxidant activity (FRAP)

Antioxidant activity of fruit wines and musts was evaluated in order to assess the impact of alcoholic fermentation on this feature and the functional potential of fruit wines. For this purpose an FRAP analysis was performed with results presented in Table 6. Fruit

#### Table 6

Antioxidant activities in fruit musts and fruit wines produced from concentrates, in comparison with the ones previously reported for the corresponding fruits. Errors represent standard deviations of fermentation triplicates\* adapted from (Fu et al., 2011).

Antioxidant activity	Fruit must $(mmol \cdot L^{-1})$	Fruit wine $(mmol \cdot L^{-1})$	Fruit (mmol·kg <sup>-1</sup> )*
Orange	$24.8 \pm 0.01$	$22.6 \pm 0.46$	$13.4 \pm 0.26$
Mango	$33.6 \pm 0.02$ $5.38 \pm 0.00$	$28.0 \pm 1.84$ 7.14 ± 0.77	$14.6 \pm 0.33$ $4.86 \pm 0.19$
Banana	$10.3 \pm 0.00$	$9.54 \pm 0.89$	$5.33 \pm 0.10$

demonstrated distinctive antioxidant activities, where orange and cherry presented the highest antioxidant activity among the ones tested. High concentrations of phenolic compounds and anthocyanins have been reported for cherry, both responsible for its antioxidant activity (Usenik et al., 2007). Orange is known to contain carotenoids, vitamin C and phenolic compounds (Kelebek et al., 2009), also broadly acknowledged by their high antioxidant activity. Comparing all four fruit, relative antioxidant activities are in accordance with the previously reported (Fu et al., 2011) as shown in Table 6, where banana and mango wines demonstrated lower antioxidant activity than orange and cherry wines, measured by FRAP. Antioxidant activity was highly fruit dependent and the impact of alcoholic fermentation on this feature was low. Furthermore, the utilization of fruit concentrates allowed the production of wines with around 50%–90% higher antioxidant activity than the ones naturally found in the corresponding fruit (Fu et al., 2011) as shown in Table 6, adding further potential to these wines for functional food-grade formulations. Despite not posing as functional foods themselves, fruit wines can be further processed and included in novel formulations, representing an attractive alternative for preservation and delivery of fruit nutritional properties.

#### 3.3. Fruit wines aromatic and sensory characterization

## 3.3.1. Major volatile compounds

Fruit wines were analyzed by GC-FID, in order to quantify major volatiles. Twelve compounds were quantified, with the concentrations presented in Table 7. Alcohols and esters were the main major volatiles found. Despite having low contribution to wine aroma, these compounds contribute to secondary aroma and enhance sensory perception of primary odors (Ribéreau-Gayon et al., 2006). One important compound is methanol, due to its toxic nature at high concentrations, and it was found in all fruit wines. The appearance of this alcohol in wine is related to pectin content in the must (Ribéreau-Gayon et al., 2006), and dependent of the fruit and fractions used, justifying the differences observed. Nevertheless, methanol was below the maximum level of 250 mg $\cdot$ L<sup>-1</sup> established by wine regulation (OIV, 2014) in all fruit wines. Also found in high concentrations were 1-propanol, 3methyl-1-butanol and 2,3-butanediol, products of yeast metabolism, deriving from the anabolic glucose pathway or specific amino acid catabolic pathway (Ribéreau-Gayon et al., 2006). Considering that these secondary metabolites are a direct consequence of the fermentation of specific substrates in the raw material, the concentrations observed can be influenced by the distinct composition of each fruit concentrate. Other compounds considered to have low impact on wine quality (Ribéreau-Gayon et al.,

#### Table 7

Quantification of major compounds in fruit wines by GC-FID, errors represent standard deviation of fermentation triplicates.

	Orange	Mango	Cherry	Banana
	$C(mg \cdot L^{-1})$	$C(mg \cdot L^{-1})$	$C(mg \cdot L^{-1})$	$C(mg \cdot L^{-1})$
acetaldehyde	13 ± 1.4	21 ± 3	7 ± 1.2	5.3 ± 0.7
methyl acetate	$7 \pm 1.1$	$6.9 \pm 1.4$	$0.0 \pm 0.0$	$3.4 \pm 0.3$
ethyl acetate	$13.3 \pm 0.5$	$40 \pm 3$	$18 \pm 1.0$	$66 \pm 38$
methanol	$213 \pm 40$	$109 \pm 31$	$17 \pm 6$	$42 \pm 12$
1-propanol	$116 \pm 4$	87 ± 9	$236 \pm 29$	$193 \pm 50$
2-methyl-1-propanol	$15 \pm 1.4$	45 ± 1.3	25 ± 1.7	$64 \pm 19$
2-methyl-1-butanol	$18.7 \pm 0.8$	$43 \pm 2$	$15 \pm 1.6$	25 ± 7
3-methyl-1-butanol	$70 \pm 1.3$	$164 \pm 12$	$120 \pm 1.2$	$100 \pm 26$
2,3-butanediol, levo	$208 \pm 26$	$245 \pm 82$	$305 \pm 63$	$365 \pm 80$
2,3-butanediol, meso	$65 \pm 12$	93 ± 32	88 ± 21	$119 \pm 27$
diethyl succinate	$0.0 \pm 0.0$	$0.0 \pm 0.0$	$0.0 \pm 0.0$	$1.4 \pm 0.4$
2-phenylethanol	$12 \pm 2$	$46 \pm 11$	8 ± 1.8	8 ± 2

### Table 8

Identification of minor compounds in fruit wines by GC–MS, with correspondent mean concentration (C<sub>mean</sub>), perception thresholds (PT) and reported descriptors. Errors represent standard deviation of fermentation triplicates.

	Banana	Cherry	Mango	Orange	Threshold	Descriptors
	$\overline{C_{\text{mean}}(\mu g \cdot L^{-1})}$	$\overline{C_{\text{mean}}(\mu g \cdot L^{-1})}$	$C_{\text{mean}} (\mu g \cdot L^{-1})$	$C_{\text{mean}} \left( \mu g \cdot L^{-1} \right)$	$PT(\mu g \cdot L^{-1})$	
C <sub>6</sub> -Compounds						
1-hexanol	$159 \pm 59$	$10 \pm 1$	_	$192 \pm 62$	8 000 [1] [2] <sup>b</sup>	
Z-3-hexenol	-	-	-	96 ± 28	400 [1] <sup>b</sup>	Lettuce-like [3]
Alcohols	404 + 146	C9 . E	204 . 75	210 . 57	50 000 [2]b	
1-octanol	$404 \pm 140$	00 ± 5	294 ± 75	$210 \pm 57$ 1053 $\pm 118$	10 000 [2]	– Coconut nuts oily [4]
Furfuryl alcohol	_	_	161 + 44	-	$15\ 000\ [2]$	Hav Moldy [5]
benzyl alcohol	_	382 ± 32	$101 \pm 11$ $17 \pm 4$	56 ± 17	200 000 [6] <sup>a</sup>	Almonds,Bitter [4]
2-phenoxyethanol	_	9 ± 1	_	_	-	_
Esters						
isobutyl acetate	-	59 ± 1	$342 \pm 46$	-	1605 [7] <sup>a</sup>	Banana,fruity,sweet [4]
ethyl butyrate	-	$52 \pm 10$	$365 \pm 115$	$121 \pm 0.6$	20 [1] [2] <sup>b</sup>	Fruity [3], Papaia, Sweet, Butter, Apple [4]
3-memyibulyi acetale	$3762 \pm 1460$ $912 \pm 400$	$1034 \pm 10$ 122 $\pm 8$	$26/4 \pm 8/9$	$293 \pm 62$ 154 ± 46	$30[1][2]^{2}$ $14[8]^{a}$	Apple Fruity Apiseed Sweet [4]
Z-3-hexenvl acetate	512 ± 400	$122 \pm 0$ 15 + 0 3	414 ± 55 -	- -	-	
ethyl octanoate	466 ± 160	$43 \pm 7$	95 ± 13	127 ± 36	5 [8] <sup>a</sup>	Apple,Sweet,Fruity [4]
ethyl 3-hydroxybutyrate	45 ± 15	$4 \pm 1$	$319 \pm 50$	$9\pm0$	20 000 [6] <sup>a</sup>	-
ethyl decanoate	$273 \pm 134$	$17 \pm 2$	$29 \pm 14$	39 ± 11	200 [8] <sup>a</sup>	Fatty acid, fruity, apple, solvent [4]
benzyl acetate	-	36 ± 3	-	-	-	-
2-phenylethyl acetate	$659 \pm 204$	98 ± 9	-	$296 \pm 81$	650 [9]250 [1] [2] <sup>b</sup>	Roses,honey,apple,sweet [4]
Monoterpenic alcohols		20	44 . 10	6725 . 1561		Animond termony [4] Lemon [10]
linalool isopulagol I	_	39 ± 6	$44 \pm 16$	$6/25 \pm 1561$	25.2 [8]	Aniseed, terpene [4] Lemon [10]
fenchol	_	_	- 42 + 6	$184 \pm 40$	- 50 [11] <sup>c</sup>	– muddy [12]
terpinen-4-ol	_	14 + 6	91 + 21	12404 + 3146	-	
myrcenol	_	_	$35 \pm 3$	_	_	-
borneol	_	_	$54 \pm 15$	_	_	_
α-terpineol	-	$12 \pm 4$	$1036 \pm 275$	$3683 \pm 984$	250 [8] <sup>a</sup>	Pine,terpene [4]
citronellol	-	9 ± 2	-	$307 \pm 81$	100 [1] [2] <sup>b</sup>	Citronella [13]
nerol	-	8 ± 2	-	$280 \pm 32$	400-500 [14] <sup>c</sup>	Lime,floral-hyacinth,roses [4]
geraniol Monotormonia ovidos and diols	-	$14 \pm 1$	_	$241 \pm 64$	36 [10]ª	rose-like,citrus-like [3]
trans-furan linalool oxide	_	_	93 ± 11	_	_	_
cis-furan linalool oxide	_	_	$33 \pm 11$ 70 + 22	-93 + 20	_	_
8-hydroxy-6,7-dihydrolinalool	_	_	_	$168 \pm 55$	_	_
E-8-hidrohylinalool	_	11 ± 1	_	$261 \pm 99$	_	-
Z-8-hidrohylinalool	-	$14 \pm 2$	-	$132 \pm 31$	-	-
C13-Norisoprenoids						
3-hydroxy- $\beta$ -damascone	-	$51 \pm 4$	_	-	-	-
$3$ -nydroxy-/, $8$ -dinydro- $\alpha$ -ionone		$12 \pm 2$	- 41 + 12	-	_	-
3-0x0-7 8-dihydro-q-ionol	$50 \pm 17$ 63 + 36	$45 \pm 6$	$\frac{41 \pm 12}{155 + 40}$	$281 \pm 84$	_	_
Volatile phenols	05 ± 50	15 ± 0	155 ± 10	201 ± 01		
eugenol	8205 ± 3027	225 ± 39	_	$236 \pm 76$	6 [8]; 15 [5] <sup>a</sup>	clove-like [5] [3]
4-vinylguaiacol	188 ± 49	$6 \pm 0.4$	330 ± 90	$2890 \pm 977$	130 [5]; 1100 [8] <sup>a</sup>	phenolic, bitter [4]; pharmaceutic-spicy [12]
4-vinylphenol	-	6 ± 1	23 ± 5	$637 \pm 298$	180 [5] <sup>a</sup>	stramonium [5]; pharmaceutic [12]
acetovanillone	86 ± 38	18 ± 3	-	50 ± 22	1000 [6] <sup>a</sup>	-
zingerone	37 ± 16	$12 \pm 4$	_	$282 \pm 91$	-	-
3,4,5-trimetnoxypnenoi	_	6 ± 1	_	_	-	-
propanoic acid	_	_	41 + 15	33 + 10	_	_
isobutvric acid	540 + 189	21 + 3	98 + 19	_	2300 [8] <sup>a</sup>	Sweaty, bitter, vinegar [4]
butyric acid	$408 \pm 154$	7 ± 2	688 ± 187	78 ± 27		
3-methyl + 2-methylbutyric acids	$728 \pm 242$	$30 \pm 3$	$242 \pm 63$	$39 \pm 10$	-	-
hexanoic acid	$1047 \pm 390$	$194 \pm 22$	$732 \pm 148$	$972 \pm 259$	420 [8] <sup>a</sup>	fatty acid, oily, sweaty [4]; green [10]
octanoic acid	2091 ± 797	544 ± 57	918 ± 219	1047 ± 325	500 [8] <sup>a</sup>	fatty acid, oily, sweaty [4]
decanoic acid	545 ± 174	$97 \pm 10$	$22 \pm 6$	_	1 000 <sup>a</sup>	Wax, rancid, soap [4]
methoxyfuraneol	_	_	48 + 10	_	_	
furaneol	_	_	$40 \pm 10$ 1180 + 282	_	— 37 [15] <sup>a</sup>	– Caramel [3]
γ-decalactone	_	23 ± 4	_	_	1000 [2] <sup>b</sup>	
Sulfur compounds						
2-methyltetrahydrothiophen-3-one	-	$20 \pm 2$	-	-	_	
2-(methylthio)ethanol	-	_	$51 \pm 8$	-	-	
methionol	65 ± 21	$4 \pm 1$	$510 \pm 116$	-	-	
Carbonyl compounds				62 1 22		
o-memyi-o-mepten-2-one	_	 364 48	_	- ± 23		– Almond [16]
benzaluenyue		204 I 40			5000 [2]	/ uniona [10]

[1] (Guth, 1997).
[2] (Moreno, Zea, Moyano, & Medina, 2005).
[3] (Czerny et al., 2008).
[4] (Meilgaard, 1975).

- [5] (Boidron, Chatonnet, & Pons, 1988).
- [6] (Goméz-Míguez, Cacho, Ferreira, Vicario, & Heredia, 2007).
- [7] (Ferreira, Ortin, Escudero, Lopéz, & Cacho, 2002).
- [8] (Ferreira, López, & Cacho, 2000).
- [9] (Salo, 1970).
- [10] (Escudero et al., 2004).
- [11] (Guerche, Dauphin, Pons, Blancard, & Darriet, 2006).
- [12] (Boutou & Chatonnet, 2007).
- [13] (Ribéreau-Gayon et al., 2006).
- [14] (Ribéreau-Gayon, Peynaud, Ribéreau-Gayon, & Sudraud, 1975).
- [15] (Kotseridis & Baumes, 2000).
- [16] (Étievant, 1991).
- -not found.
  - <sup>a</sup> Threshold in model solution.
- <sup>b</sup> Threshold in hydroalcoholic solution.
- <sup>c</sup> Threshold in water.

2006), such as acetaldehyde, methyl acetate, ethyl acetate, 2methyl-1-propanol, 2-methyl-1-butanol, ethyl succinate and 2phenylethanol appeared in lower concentrations.

#### 3.3.2. Minor volatile compounds

For a better understanding of fruit wine sensory profile, minor volatiles were analyzed for correlation with sensory data. Fifty seven compounds were identified among all fruit wines, presented in Table 8, where minor compounds were grouped according to chemical type. All fruit wines presented high content in volatile fatty acids, produced by yeast during lipid metabolism (Vilanova & Oliveira, 2012), which contribute to wine aroma equilibrium despite generating fatty or sweat odors. Only hexanoic and octanoic acids were found above perception threshold, except for cherry wine, which can be related with high lipid content in volatile phenols, secondary metabolites of phenolic

acids metabolism during fermentation. The production of volatile phenols is expected from the raw-materials used and the type of compounds produced dependent on fruit composition (Vilanova & Oliveira, 2012). Banana wine presented high content in esters. all of them above the reported perception threshold, with special emphasis on isoamyl acetate. Mango wine also presented high ester content, namely of ethyl hexanoate and ethyl butyrate, possible contributors to the tropical aromas, along with isoamyl acetate (Meilgaard, 1975). Monoterpenic alcohols were also found in high concentrations in mango wine,  $\alpha$ -terpineol and linalol were found above the perception threshold. One of the distinguishing characteristics of mango wine was its considerable lactone content, approximately 32 folds higher than the perception threshold, due to high furaneol concentration. Monoterpenic alcohols were found in greater concentration and diversity in orange wine, namely linalool, geraniol, citronellol and terpinen-4-ol. Some of these relate to citric aroma descriptors

Table 9

Relative Intensity (1), relative Frequency(F) and Geometrical Mean (GM) determined for the descriptors found in fruit wines sensorial characterization by trained panelists.

Descriptors	Banana	a		Mango			Orange	2		Cherry		
	I %	F %	GM%	I %	F %	GM %	I %	F %	GM %	I %	F %	GM %
Visual Analysis												
Yellow	52	100	72	0	0	0	0	0	0	0	0	0
Orange	0	0	0	40	100	63	78	100	88	0	0	0
Cherry	0	0	0	0	0	0	0	0	0	70	100	84
<b>Olfactory</b> Analysis												
Intensity	56	100	75	49	100	70	79	100	89	84	100	92
Quality	57	100	76	79	100	89	57	100	76	78	100	88
Fruity	0	0	0	33	57	44	0	0	0	44	71	56
Apple	22	71	40	0	0	0	0	0	0	0	0	0
Citric	19	71	37	22	57	36	71	100	85	0	0	0
Orange	0	0	0	8	14	11	68	100	83	0	0	0
Mango	0	0	0	32	71	48	0	0	0	0	0	0
Tropical	0	0	0	57	10	76	0	0	0	0	0	0
Orange peel	0	0	0	0	0	0	21	43	30	0	0	0
Cherry	0	0	0	0	0	0	0	0	0	75	100	86
Dry fruit	10	29	17	0	0	0	0	0	0	0	0	0
Banana	13	57	27	0	0	0	0	0	0	0	0	0
Vegetal	0	0	0	0	0	0	10	43	20	0	0	0
Red fruit	0	0	0	0	0	0	0	0	0	37	71	51
Vanilla	0	0	0	0	0	0	0	0	0	22	43	31
Caramel	0	0	0	0	0	0	0	0	0	16	43	26
Gustatory analysis												
Quality	46	100	68	54.0	100	74	57	100	76	62	100	79
Sweet	3	14	7	8	29	15	8	43	18	6	29	14
Salt	3	14	7	3	29	10	2	29	7	2	14	5
Acid	52	100	72	25	100	50	64	100	80	81	100	90
Bitter	22	100	47	38	100	62	79	100	89	52	100	72
Body	35	100	59	54	100	74	38	100	62	46	100	68
Persistence	29	100	54	33	100	58	22	100	47	41	86	60
Astringency	3	43	12	5	29	12	13	57	27	10	57	23
Global Value	48	100	69	54	100	74	56	100	75	75	100	86



**Fig. 2.** Gustatory profile of fruit wines obtained from geometric mean (*GM*) of descriptors for (—) Orange, (---) Mango, (••••) Banana and (—) Cherry, as measured by trained panelists.

and are expected from orange fruit. Cherry wine, also showed distinctive characteristics, namely higher ester diversity, with ethyl butyrate, isoamyl acetate, ethyl octanoate and ethyl hexanoate above perception thresholds. It also presented a much lower content in volatile fatty acids than the other fruit wines, and characteristic content of C13-norisoprenoids. Overall, fruit wines showed high concentrations of aroma active compounds, coherent with previous works (Reddy & Reddy, 2009; Santos et al., 2013; Sun et al., 2013). The use of fruit concentrates and the optimal conditions determined are feasible for the production of fruit wines with satisfactory and characteristic volatile composition.

#### 3.3.3. Sensory analysis

In order to correlate and complement analytical data and further assess organoleptic quality of fruit wines, a sensory evaluation was carried out. Table 9 shows the sensory descriptors identified in the samples and their correspondent means of relative frequency (F) and intensity (I) obtained by the tasting panelists.

The most characteristic olfactory descriptor for each fruit wine was: apple for banana (GM = 40%), tropical for mango (GM = 76%). citric for orange (GM = 85%) and cherry for cherry (GM = 86%). Esters with apple and tropical descriptors were found in banana and mango, and monoterpenic alcohols with citric descriptors were found in orange, which can be directly related to panelist's evaluation. From gustatory analysis, the highest GM for acidity were found for orange and cherry wines, and the lowest for banana wine. Acidity described in the gustatory evaluation for orange and cherry is directly supported by the higher organic acid concentrations quantified in these wines. Cherry wine was the most valued by the tasters and banana wine the one least preferred. Fig. 2 highlights the main fruit wine characteristics, with acidity, bitterness and body as the dominant features in the gustatory evaluation. However, fruit wines showed very distinguished aromatic features as seen in Fig. 3. Overall value and quality of fruit wines was around 70 %-80 %, reinforcing alcoholic fermentation on generating added-value fruit products. Finally, PLSR2 analysis was performed taking into account the volatile families analyzed and sensory descriptors (%GM), as presented in Fig. 4. The first PLSR2 was performed to relate aroma descriptors with volatile compounds (Fig. 4a). The biplot explained 75% of the variation. According to the loading weight, high correlations (more than 90%) were found among dry fruit descriptors and banana with fatty acid compounds. Mango, and tropical were mainly predicted by sulphur compounds. Citric and orange were correlated with C6-alcohols and monoterpenic alcohols and



Fig. 3. Aroma profile, measured by trained panelists of: a) orange, b) banana, c) mango and d) cherry wines, obtained from geometric mean (GM) of the main descriptors.



**Fig. 4.** Partial Least Square Regression (PLSR2) between volatile families and a) sensory aroma descriptors or b) olfactory and gustatory quality and global value.

oxides. Correlation between sensory perception and chemical composition in fruit wines is in good agreement with descriptors reported for each fruit, and furthermore provides information about the contribution of non-described minor volatiles for aroma. Another PLSR2 was performed to relate volatiles with the global value and olfactory and gustatory quality (Fig. 4b), which were positively correlated with carbonyl compounds (gustatory quality 68%, olfactory quality 97% and global value 96%). The biplot explained 95.6% of the variation. The PLSR2 analysis provided an insight of the synergetic effect between volatiles and perceived descriptors. Thus, fruit wines aroma is a direct result of positive and negative effects of the volatile composition and the balance attained. Correlations established allow further tuning of the fermented fruit products, considering the subsequent desired product and application. Nevertheless, fruit wines produced were broadly accepted, demonstrating its viability as a wine grade product.

#### 4. Conclusions

Four fruit wines were successfully produced from industrially processed fruit concentrates. Mathematical models and optimal conditions for ethanol production were determined leading to maximal ethanol concentration in the shortest fermentation time. Alcoholic fermentation did not affect significantly antioxidant activity and fruit wines showed the antioxidant activity expected when taking into account the corresponding raw materials. Fruit wines had good acceptance from trained panelists, demonstrating its suitability as food grade products. Chemical characterization was in good agreement with sensory data and the correlations established were of upmost importance to understand the variables involved in fruit wine acceptability.

## Acknowledgments

Authors would like to acknowledge the financial funding of: FruitVinegarDRINK QREN Project (ref. 23209), Project "BioInd – Biotechnology and Bioengineering for improved Industrial and Agro-Food processes, REF. NORTE-07-0124-FEDER-000028" Cofunded by the Programa Operacional Regional do Norte (ON.2 – O Novo Norte), QREN, FEDER and the FCT Strategic Project Pest-OE/ EQB/LA0023/2013.

### Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.lwt.2015.02.020.

## References

- Akubor, P., Obio, S., Nwadomere, K., & Obiomah, E. (2003). Production and quality evaluation of banana wine. Plant Foods for Human Nutrition, 58, 1–6.
- Artero, A., Artero, A., Tarín, J. J., & Cano, A. (2015). The impact of moderate wine consumption on health. *Maturitas*, 80(1), 3–13.
- Boidron, J., Chatonnet, P., & Pons, M. (1988). Effect of wood aroma compounds of wine. Connaissance de la Vigne et du Vin, 22(4), 275–294.
- Boutou, S., & Chatonnet, P. (2007). Rapid headspace solid-phase microextraction/gas chromatography/mass spectrometric assay for the quantitative determination of some of the main odorants causing off-flavours in wine. *Journal of Chromatography A*, 1141(1), 1–9.
- Caplice, E., & Fitzgerald, G. F. (1999). Food fermentations: role of microrganisms in food production and preservation. *International Journal of Food Microbiology*, 50, 131–149.
- Czerny, M., Christlbauer, M., Christlbauer, M., Fischer, A., Granvogl, M., Hammer, M., et al. (2008). Re-investigation on odor thresholds of key food aroma compounds and development of an aroma language based on odor qualities of defined aqueous odorant solutions. *European Food Research and Technology*, 228(2), 265–273.
- Escudero, A., Gogorza, B., Melús, M. A., Ortín, N., Cacho, J., & Ferreira, V. (2004). Characterization of the aroma of a wine from Maccabeo. Key role played by compounds with low odor activity values. *Journal of Agricultural and Food Chemistry*, 47(8), 3303–3308.
- Étievant, P. (1991). Volatile compounds in foods and beverages. In H. Maarse (Ed.), Wine (pp. 483-546). New York: Marcel Dekker.
- Ferreira, V., López, R., & Cacho, J. (2000). Quantitative determination of the odorants of young red wines from different grape varieties. *Journal of the Science of Food* and Agriculture, 80(11), 1659–1667.
- Ferreira, V., Ortin, N., Escudero, A., Lopéz, R., & Cacho, R. (2002). Chemical characterization of the aroma of Grenade rosé wines: aroma extract dilution analysis, quantitative determination and sensory reconstitution studies. *Journal of Agricultural and Food Chemistry*, 50(14), 4048–4054.
- Fu, L., Xu, B.-T., Xu, X.-R., Gan, R.-Y., Zhang, Y., Xia, E.-Q., et al. (2011). Antioxidant capacities and total phenolic contents of 62 fruits. *Food Chemistry*, 129(2), 345–350.
- Goméz-Míguez, M., Cacho, J., Ferreira, V., Vicario, I., & Heredia, F. (2007). Volatile components of Zalema white wines. *Food Chemistry*, 100(4), 1464–1473.
- Guerche, S. L., Dauphin, B., Pons, M., Blancard, D., & Darriet, P. (2006). Characterization of some mushroom and earthy off-odors microbially induced by the development of rot on grapes. *Journal of Agricultural and Food Chemistry*, 54(24), 9193–9200.
- Gustavsson, J., Cederberg, C., Sonesson, U., Otterdijk, R. V., & Meybeck, A. (2011). Global food losses and food waste, extent, causes and prevention. Rome: FAO.

- Guth, J. (1997). Quantitation and sensory studies of character impact odorants of different white wine varieties. *Journal of Agricultural and Food Chemistry*, 45(8), 3027–3032.
- Isitua, C., & Ibeth, I. (2010). Novel method of wine production from banana (Musa acuminata) and pineapple (Ananas comosus) wastes. *African Journal of Biotechnology*, 9(44), 7521–7524.
- Kelebek, H., Selli, S., Canbas, A., & Cabaroglu, T. (2009). HPLC determination of organic acids, sugars, phenolic compositions and antioxidant capacity of orange juice and orange wine made from a Turkish cv. Kozan. *Microchemical Journal*, 91(2), 187–192.
- Koppes, L. L., Dekker, J. M., Hendriks, H. F., Bouter, L. M., & Heine, R. J. (2005). Moderate alcohol consumption lowers the risk of type 2 diabetes: a metaanalysis of prospective observational studies. *Diabetes Care*, 28(3), 719–725.
- Kotseridis, Y., & Baumes, R. (2000). Identification of impact odorants in Bordeaux red grape juice, in the commercial yeast used for its fermentation, and in the produced wine. *Journal of Agricultural and Food Chemistry*, 48(2), 400–406.
- Meilgaard, M. (1975). Flavor chemistry of beer part II: flavor and threshold of 239 aroma volatiles. MBAA Technical Quarterly, 12(3), 151–168.
- Moreno, J., Zea, L., Moyano, L., & Medina, M. (2005). Aroma compounds as marker of the changes in sherry wines subjected to biological ageing. *Food Control*, 16(4), 333–338.
- Müller, L., Gnoyke, S., Popken, A. M., & Bröhm, V. (2010). Antioxidant capacity and related parameters of different fruit formulations. *LWT- Food Science and Technology*, 43(6), 992–999.
- Neafsey, E. J., & Collins, M. A. (2011). Moderate alcohol consumption and cognitive risk. Neuropsychiatric Disease and Treatment, 7, 465–484.
- OIV. (2014). International code of oenological practices. Paris: Organisation Internationale du Vigne et du Vin.

- Reddy, L. V., & Reddy, O. V. (2009). Production optimization and characterization of wine from Mango (Mangifera Indica Linn.). *Natural Product Radiance*, 8(4), 426–435.
- Ribéreau-Gayon, P., Glories, Y., Maujean, A., & Dubourdieu, D. (2006). Handbook of enology, the chemistry of wine: Stabilization and treatments. Wiley.
- Ribéreau-Gayon, J., Peynaud, E., Ribéreau-Gayon, P., & Sudraud, P. (1975). Traité d'Oenologie: Sciences et Techniques du Vin. Paris: Dunod.
- Room, R., Babor, T., & Rehm, J. (2005). Alcohol and public health. *Lancet*, 365(9458), 519–530.
- Salo, P. (1970). Determining the odor thresholds for some compounds in alcoholic beverages. *Journal of Food Science*, 35(1), 95–99.
- Santos, C. C., Duarte, W. F., Carreiro, S. C., & Schwan, R. F. (2013). Inoculated fermentation of orange juice (*Citrus sinensis* L.) for a production of a citric fruit spirit. *Journal of the Institute of Brewing*, 119(4), 280–287.
- Sun, S. Y., Che, C. Y., Sun, T. F., Lv, Z. Z., He, S. X., Gu, H. N., et al. (2013). Evaluation of sequential inoculation of *Saccharomyces cerevisiae* and *Oenococcus oeni* strains on the chemical and aromatic profiles of cherry wines. *Food Chemistry*, 138(4), 2233–2241.
- Tucker, G. (1993). Introduction. In G. Seymour, J. Taylor, & G. Tucker (Eds.), Biochemistry of fruit ripening (pp. 1–51). London: Chapman&Hall.
- Usenik, V., Fabcic, J., & Stampar, F. (2007). Sugars, organic acids, phenolic composition and antioxidant activity of sweet cherry (*Prunus avium L*.). Food Chemistry, 107(1), 185–192.
- Vilanova, M., & Oliveira, J. M. (2012). Application of Gas chromatography on the evaluation of grape and wine aroma in Atlantic Viticulture (NW Iberian Peninsula). In Dr B. Salih (Ed.), Gas chromatography in plant science, wine technology, toxicology and some specific applications. InTech.