



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



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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 $72.3 \pm 2.08 \text{ g} \cdot \text{L}^{-1}$ in orange, $101 \pm 1.78 \text{ g} \cdot \text{L}^{-1}$ in mango, $66.1 \pm 4.02 \text{ g} \cdot \text{L}^{-1}$ in cherry and $98.2 \pm 7.88 \text{ g} \cdot \text{L}^{-1}$ in banana with maximal productivities of $0.4 \pm 0.0 \text{ g} \cdot \text{L}^{-1} \cdot \text{h}^{-1}$, $1.0 \pm 0.1 \text{ g} \cdot \text{L}^{-1} \cdot \text{h}^{-1}$, $1.7 \pm 0.2 \text{ g} \cdot \text{L}^{-1} \cdot \text{h}^{-1}$ and $1.0 \pm 0.1 \text{ g} \cdot \text{L}^{-1} \cdot \text{h}^{-1}$, respectively. Evaluation of total antioxidant activity by FRAP demonstrated fruit wines potential for the development of foods and formulations with functional properties, attaining $22.6 \pm 0.46 \text{ mmol} \cdot \text{L}^{-1}$ for orange, $7.14 \pm 0.77 \text{ mmol} \cdot \text{L}^{-1}$ for mango, $28.0 \pm 1.84 \text{ mmol} \cdot \text{L}^{-1}$ for cherry and $9.54 \pm 0.89 \text{ mmol} \cdot \text{L}^{-1}$ 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 properties was established with PLSR2 between analytical and sensory data, which allowed an insight of chemical composition impact in consumer perceived quality.

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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

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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), α -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)-s-triazine ($\geq 98\%$), Iron (III) chloride ($>97\%$) and Sodium Acetate ($\geq 99\%$), all from Sigma–Aldrich. For GC-FID the following standards were used: acetaldehyde ($\geq 99.5\%$), methyl acetate ($\geq 99.9\%$), 1-propanol ($\geq 99.9\%$), 2-methyl-1-propanol ($\geq 99.8\%$), 2-methyl-1-butanol ($\geq 98\%$), 3-methyl-1-butanol ($\geq 99.8\%$), 2,3-butanediol, levo ($\geq 99.0\%$), 2,3-butanediol, meso ($\geq 99.0\%$), 2-phenylethanol ($\geq 99.0\%$) from (Fluka) and ethyl acetate (99.8%), methanol ($\geq 99.8\%$), diethyl succinate (99.0%) from (Sigma–Aldrich). For GC–MS calibration: 1-hexanol ($\geq 99.9\%$), Z-3-hexenol ($\geq 98\%$), 1-octanol ($\geq 99.5\%$), 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%), methyl alcohol ($\geq 99.0\%$), 2-phenoxyethanol (98.0%), ethyl butyrate (99.0%), 3-methylbutyl acetate ($\geq 99.0\%$), ethyl hexanoate ($\geq 99.9\%$), Z-3-hexenyl acetate ($\geq 98\%$), ethyl octanoate ($\geq 99.0\%$), ethyl 3-hydroxybutyrate (99.0%), ethyl decanoate ($\geq 99.0\%$), benzyl acetate ($\geq 99.0\%$), linalool (97%), terpinen-4-ol ($\geq 99.0\%$), citronellol (95%), nerol (97%), geraniol (98%), eugenol (99%), 4-vinylguaiacol ($\geq 98\%$), 4-vinylphenol (12%), acetovanillone (98%), zingerone ($\geq 96\%$), 3,4,5-trimethoxyphenol (97%), 3-methyl + 2-methylbutyric acids (99%), octanoic acid ($\geq 99.5\%$), methoxyfuranol ($\geq 97\%$), furaneol ($\geq 98\%$), γ -decalactone ($\geq 98\%$), 2-methyltetrahydrothiophen-3-one ($\geq 97\%$), 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 α -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⁻¹ as mobile phase at

0.5 mL·min⁻¹ 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⁻¹ NaOH. Alcoholic fermentation was conducted in Erlenmeyer flasks with glycerol lock, ensuring anaerobic conditions and CO₂ exhaustion. Musts were inoculated with lyophilized oenological yeast Lalvin QA23 (Lallemand), incubated with temperature control, orbital agitation of 150 min⁻¹ and monitored by weight loss measurement, equivalent to CO₂ production and exhaustion, for stationary phase determination. Ethanol concentration (C_{EtOH}) was quantified by HPLC, and productivity (P) was calculated dividing C_{EtOH} 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⁻¹ mobile phase at a 0.7 mL·min⁻¹ flow. Organic acids were measured using a Jasco 870-UV detector (210 nm wavelength) and ethanol was

Table 1
Brix degree (°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

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