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A survey on wines from organic viticulture from different European countries

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ABSTRACT

A survey was carried out on a thousand wines from organic viticulture from different European countries. Analytical data were collected about the most used quality control parameters (e.g. alcoholic strength, reducing sugars, total acidity and pH, volatile acidity, malic and lactic acid, free and total sulfur dioxide), as well as regarding some compounds harmful for human health, such as ochratoxin A and biogenic amines. The results collected on quality control parameters were generally in agreement with the values normally detectable for conventional wines. Total sulfur dioxide was lower than 110–120 mg/L in the most of the samples and no significant correlation was found between sulfite levels and other parameters. Ochratoxin A (OTA) seemed not a generalized problem for organic wine productions: its concentration was below the European legal limit, in the 95% of the samples analyzed; nevertheless, the risk of OTA pollution seemed higher in certain southern European regions. On the other hand, biogenic amines (BA) appeared a serious problem for organic winemaking and high concentrations were found in many of the analyzed wines. They seemed connected with a bad management of malolactic fermentation, being generally associated with high pHs and volatile acidities.

1. Introduction

It is a common belief that wines from organic viticulture are lower quality wines with respect to conventional ones, both as concerns sensory characteristics and a supposed higher content in compounds harmful for human health (e.g. ochratoxin A or biogenic amines). Nevertheless, these opinions remain mainly based on hypothetical considerations, because of the lack of scientific data reporting results on the analytical and sensory characterization of organic wines, as well as on the comparison between organic and conventional products.

Concerning sensory characters, Moyano, Zea, Villafuerte, and Medina (2009) compared the aroma profile of Fino type sherry wines cultivated conventionally and ecologically; they found that the organically aged wines had a sensory profile similar to that of the conventional Fino, but a lower odor intensity. The same considerations were previously reported by Dupin, Schlich, and Fischer (2000) on German wines: they found that organic products tend to be less aromatic than conventional ones, even if with a lower vegetal character; anyway, the style effect (organic or conventional) was less important than the grapegrowing area where the wines were produced.

Another point of discussion concerning the differentiation between conventional and organic products, is related to the sulfur dioxide levels in organic winemaking.

Prior to 2012, organic wine processing was not regulated in Europe and the levels allowed for total sulfur dioxide in wines from organic viticulture were the same of those authorized for conventional ones; the new Commission Regulation (EC) No 203/2012 (2012), entered into force since August 2012, establishes specific rules for organic winemaking, and lower levels of sulfites with respect to conventional wines. Nevertheless, as far as we know, there are no scientifically published databases reporting the situation about sulfite concentrations in organic wines and their relationship with the other wine quality control parameters. This last consideration is extremely important for organic winemaking, because of the strong impact that SO_2 can have on consumer's perception and health (Prêtet-Lataste, Berger, & Molot, 2006).

The results published about ochratoxin A (OTA) and biogenic amines (BA) are more complete, even if still not exhaustive. Concerning the former, Chiodini, Scherpenisse, and Bergwerff (2006) compared 44 organic and conventional wines from different

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European countries, finding no significant differences regarding OTA concentrations between the two groups. These results were confirmed by other papers (Miceli, Negro, Tommasi, & De Leo, 2003; Ponsone, Combina, Dalcero, & Chulze 2007); according to these results, it seems that organic wine productions have the same risk of OTA pollution with respect to conventional wines. Nevertheless, all these papers cannot be considered as comprehensive surveys, because of the relatively low number of samples generally analyzed.

Very few data on organic wine productions are available also regarding biogenic amines: in such a case, García-Marino, Trigueros, & Escribano-Bailón (2010) detected significantly higher levels of BA in organic wines, than in conventional ones; they affirm that this result is probably due to the fact that malolactic fermentation occurs spontaneously in organic products, as well as to their lower level of SO_2; nevertheless, it must be underlined that only one organic sample was analyzed in this study.

For all these reasons, a compositional survey was carried out on a thousand wines from organic viticulture from different European countries, with the purpose of collecting a wide dataset on wine basic quality control parameters and some compounds involved in health-related aspects (ochratoxin A and biogenic amines). Free and total sulfur dioxide were also determined on all the wines collected, to assess the average levels used in organic winemaking and their relationship with wine quality control parameters.

2. Materials and methods

2.1. Wine samples

Wines were collected during different local and international organic wine competitions and fairs held in Europe: the Competitions Biodivino (Italy, June 2006 and 2007) and Millésime Bio (France, January 2007 and 2008), one of the most important European organic fairs, Biofach (Nürnberg, Germany, February 2007 and 2008) and a local German Competition organized by the German Federal Association of Organic Wine Producers ECOVIN (Germany, June 2006); 971 wines were available, from different countries: Italy ($n = 478$), France ($n = 310$), Germany ($n = 86$), Austria ($n = 54$), Spain ($n = 29$) and Switzerland ($n = 14$); 634 samples were red, 303 white and 34 rosé. Years ranged from 1999 to 2007. Special wine typologies (e.g. sparkling or dessert wines) were not included in the survey. After sampling, wines were stored at 20 °C and analyzed within one month.

2.2. Chemicals

Ochratoxin A analytical standard from *Aspergillus ochraceus*, analytical grade biogenic amines (histamine, ethylamine, agmatine sulfate, 2-phenylethylamine hydrochloride, isopentylamine, 1,3-diaminopropane, putrescine dihydrochloride, cadaverine, tyramine, spermidine, spermine and 1,7-diaminoheptane), monosodium glutamate, 5-(dimethylamino)naphthalene-1-sulfonyl chloride (dansyl chloride – DNSCl), Tris(hydroxymethyl)aminomethane (Tris), HPLC grade methanol, acetone, acetonitrile and toluene were purchased from Sigma–Aldrich (St. Louis, MO, USA); polyethylene glycol, glacial acetic acid (99%), sodium hydrogen carbonate, sodium carbonate and sodium chloride were from Carlo Erba Reagents (Milan, Italy).

2.3. Basic analyses

Basic quality control parameters, alcoholic strength, reducing sugars, dry extract, total acidity and pH, volatile acidity, malic, lactic and tartaric acid, glycerol and sulfates, were determined on the whole samples, by FTIR spectroscopy, using a Winescan™ FT-120 instrument (FOSS, Hillerød, Denmark); all the samples were analyzed two times, and the mean value of the two measurements was considered for data elaboration.

Free and total sulfur dioxide were determined by the European official method, as reported in the Consolidated Version of the Commission Regulation (EC) No 2676/1990 (2005).

2.4. Ochratoxin A

2.4.1. Sample preparation

OTA was determined as reported by Visconti, Pascale, and Centonze (1999); this method has become the official method adopted by the "Organisation Internationale de la Vigne et du Vin" (O.I.V.), as well as by the AOAC International. 10 mL of wine were diluted with 10 mL of a water solution containing polyethylene glycol (10 g/L) and $NaHCO_3$ (50 g/L), mixed vigorously and then filtered through a Whatman grade GF/A glass microfibre filter (purchased from Sigma–Aldrich, St. Louis, MO, USA); 10 mL of the filtered solution were loaded on a OchraTest™ immunoaffinity column (Vicam, Watertown, MA, USA), at a flow rate of about 1 drop/sec. The column was rinsed with an aqueous solution containing NaCl (25 g/L) and $NaHCO_3$ (5 g/L) and then with distilled water, at a flow rate of 1–2 drops/sec. OTA was then eluted with 2 mL of HPLC grade methanol, at a flow rate of 1 drop/sec and collected in a glass vial; the eluted extract was evaporated under nitrogen stream at 50 °C, reconstituted with 250 μL of the HPLC mobile phase and manually injected (100 μL) in the HPLC system.

2.4.2. HPLC analysis

The HPLC system was a Jasco model 880 PU pump (Jasco Co. Ltd., Tokyo, Japan), equipped with a 7125 NS Rheodyne manual injection valve and with a Jasco model FP-1520 fluorescence detector (λ_{ex}: 333 nm, λ_{em}: 460 nm).

Ochratoxin A was separated in isocratic mode, on a 5 μm particle size, 150 mm × 4.6 mm i.d. Discovery C_{18} Column (Supelco, Bellefonte, PA, USA). The mobile phase consisted in a mixture of acetonitrile: water: acetic acid (99:99:2 v/v/v), pumped at a flow rate of 1.0 mL/min.

Quantitative analysis was performed by measuring OTA peak areas and comparing them with a calibration curve. An ochratoxin A stock solution (100 μg/L) was prepared by dissolving the solid standard in toluene–acetic acid (99:1 v/v); diluted OTA solutions were prepared in HPLC mobile phase, at 0.6, 2.0, 6.0, 20.0, 40.0 and 60.0 μg/L and injected for obtaining the calibration curve.

2.5. Biogenic amines

2.5.1. Sample preparation

Biogenic amines were determined by HPLC and precolumn derivatization with dansyl chloride, as reported by Gonzáles-de Llano, Cuesta, and Rodríguez (1998); DNSCl reagent was prepared daily, by dissolving 50 mg of the solid dansyl chloride in 1 mL of HPLC grade acetone. 500 μL of wine were mixed with 100 μL of DNSCl reagent, 175 μL of a 200 g/L $NaCO_3$ solution and 25 μL of internal standard (1,7-diaminoheptane, 1 g/L), in a conical glass tube. Tubes were stirred, wrapped with aluminum foils and placed in a water bath at 40 °C for 30 min. Subsequently, 200 μL of monosodium glutamate (50 g/L) were added and the mixture was kept for 1 h under the same conditions. Samples were filtered on a 0.45 μm pore size nylon membrane (Albet-Hahnemühle, Barcelona, Spain) and 15 μL were directly injected in the liquid chromatograph.

2.5.2. HPLC analysis

HPLC separation of biogenic amines was carried out on a 5 μm particle size, 250 mm × 3.0 mm i.d. PerfectSil Target ODS-3 Column (MZ-Analysentechnik GmbH, Mainz, Germany), conditioned at