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Evolution of quality parameters during red wine dealcoholization by osmotic distillation

Loredana Liguori [a], Paola Russo [b,*], Donatella Albanese [a], Marisa Di Matteo [a]

[a] Dipartimento di Ingegneria Industriale, Università di Salerno, via Ponte don Melillo, 84084 Fisciano (SA), Italy
[b] Dipartimento di Ingegneria Chimica Materiali Ambiente, Università di Roma "La Sapienza", via Eudossiana 18, 00184 Roma, Italy

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ABSTRACT

Osmotic distillation technique was used for the total dealcoholization of a red wine (Aglianico grape variety) up to 0.19 vol.%. The dealcoholization process was performed in subsequent cycles which gave rise wine samples at different alcoholic degrees. The effect of processing on the main chemical and physical properties of Aglianico wine was evaluated. Among wine samples, no significant differences ($p < 0.05$) of oenological parameters such as pH, total acidity were found. Similarly, the total phenolic, flavonoids and tartaric esters content and the composition of organic acids did not show significant differences ($p < 0.05$) during the process. On the contrary, colour intensity and tonality of wine samples changed significantly when the alcohol reduction was over the 6.5 vol.%. Finally, the total dealcoholized wine showed properties similar to Aglianico wine except for the volatile compounds, which decreased over 98%. Hence, flavour enrichment may be required to produce a pleasurable and delicious non alcoholic beverage from wine.

1. Introduction

Techniques for producing low and reduced alcoholic strength beverages have been developed over the last years in order to satisfy consumer demand for healthier products.

Decreasing alcohol consumption is a worldwide trend in order to achieve healthier life styles, and it could be advisable for wine producers when external factors, such as global warming and winemaking practices, cause an increasing alcohol content in wines. Accordingly, consumers can complain about these beverages that are getting too heavy and strong to drink together with harmful effects of alcohol on health. Besides, in some countries, winemakers have to pay taxes when alcohol content in wine is over 14.5 vol.% (Massot, Mietton-Peuchot, Peuchot, & Milisic, 2008).

Technologies for reducing alcohol in wines can be classified according to the stage of wine production in which they are typically applied; that is pre-, concurrent or post-alcoholic fermentation (Schmidtke, Blackman, & Agboola, 2012). Several methods to reduce the concentration of fermentable sugars in juice are: early grape harvest, juice dilution, or arrested fermentation that involve some defects in wine such as the need of pasteurization treatment and thus the potential loss of volatile compounds. Recent methods of pre-fermentation strategies are focused on technologies that, using enzymes (glucose oxidase), minimize loss or alteration of desirable organoleptic qualities and off-flavour development. Another practice is the use of selected or novel yeast strains alternative to *Saccharomyces cerevisiae* in order to lower ethanol production during fermentation of wine grapes. One of the drawbacks with novel or wild yeast species is the potential off-flavour development and the loss or alteration of desirable sensorial parameters (Heard, 1999).

Distillation under vacuum, extraction using supercritical carbon dioxide (Pickering, 2000), spinning cone column (Belisario-Sanchez, Taboada-Rodriguez, Marin-Iniesta, & Lopez-Gomez, 2009), membrane processes such as reverse osmosis (Labanda, Vichi, Llorens, & Lopez-Tamames, 2009), pervaporation (Takács, Vataia, & Korány, 2007) and osmotic distillation (Bocca, Piubelli, Stassi, Carbognin, & Ferrarini, 2010; Diban, Athes, Bes, & Souchon, 2008; Hogan, Canning, Peterson, Johnson, & Michaels, 1998; Liguori, Attanasio, Albanese, & Di Matteo, 2010; Liguori, Russo, Albanese, & Di Matteo, 2012; Lisanti, Gambuti, Genovese, Piombino, & Moio, 2012; Varavuth, Jiraratananon, & Atchariyawut, 2009) are used as post-vinification treatments.

Among membrane processes, osmotic distillation (OD) is proposed as an emerging and promising technique to reduce the ethanol content in beverages. Because of operating conditions (room temperature and atmospheric pressure), OD avoids thermal damage to aroma volatile compounds and assures low energy consumption (Varavuth et al., 2009). Osmotic distillation is a membrane separation process which involves the transport of

* Corresponding author. Tel.: +39 06 44585565; fax: +39 06 44585451.
E-mail addresses: paola.russo@uniroma1.it, parusso@unisa.it (P. Russo).

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volatile components from an aqueous solution (feed) into another liquid solution (stripping agent) capable of absorbing these components. The driving force of the process is the vapor pressure difference of volatile components across the membrane, which is usually microporous and hydrophobic. The mass transfer involves the ethanol evaporation from the feed stream at the membrane surface, the diffusion through the membrane pores and, the condensation into the stripping agent on the opposite side of the membrane (Gostoli, 1999). The main advantages of OD are the following: (i) ethanol has higher volatility and diffusivity than the main components of the wine, (ii) the vapor pressure of volatile components is low and, so is their flux through the membrane and (iii) their solubility in hydroalcoholic solutions is substantially higher than in pure water. As a consequence, the mass transfer rate of these components from wine to stripping agent is low. Furthermore, because the vapor pressure of water in wine is nearly identical to that of pure water, there is virtually no transfer of water from stripping stream into the wine (Hogan et al., 1998). In fact a water flux from stripping stream into the wine was measured when pure water was used as stripping agent; on the contrary, an opposite water flux (from feed to the stripper) occurred using as stripping agent salt solutions at concentration >7 wt.% NaCl (Michaels, 1993) and equal to 40 wt.% $CaCl_2$ (Varavuth et al., 2009).

Osmotic distillation used for wine dealcoholization was investigated in literature: Diban et al. (2008) studied the effect of partial dealcoholization on ethanol and aroma compounds in wine. Varavuth et al. (2009) investigated the best stripping agent for ethanol removal, the analysis of mass transfer coefficients and the loss of aroma compounds during the process. Few papers were focused on the changes of chemical and physical properties of wine during the dealcoholization process (Liguori et al., 2012; Lisanti et al., 2012). These authors studied the evolution of wine properties as consequence of partial dealcoholization which, according to Commission Regulation (EC) No.606/2009, consists of a reduction of the actual alcoholic strength not more than 2 vol.% and an alcoholic strength of the final product not less than 8.5 vol.% (Commission Regulation EC, 2009). The literature lacks of papers investigating the total dealcoholization (final alcohol content lower than 0.5 vol.%) of wine. This latter could be used for the development of a healthy beverage that contains the wholesome properties of wine against atherosclerosis and heart disease, without the negative effect of alcohol (Shrikhande, 2000).

Hence, the objective of this work was to evaluate the effect of total dealcoholization by means of OD on the main properties of a red wine. It is well known (Ronald, 2008; Singleton, 1992) that the quality of red wine was affected by several chemico-physical parameters such as alcohol content, pH, total acidity, total phenolic content, colour, flavonoids and tartaric esters content, organic acids composition and volatile compounds, thus the changes in the mentioned parameters were investigated during the dealcoholization process.

2. Materials and methods

2.1. Materials

Red wine from cv. *Aglianico* grape variety grown in Campania region (year 2009) was used in dealcoholization process.

The hollow fiber membrane module, 1×5.5 minimodule (Liqui-Cel) was used. Its characteristics were: polypropylene membrane, 1800 cm^2 surface area, 42 μm thickness and 14 cm length, 40% porosity, 0.03 μm membrane pore diameter. It consisted of 2300 fibers with dimensions: 11.5 cm length, 220 μm inner diameter and 300 μm outer diameter.

All reagents used for chemical analysis were analytical grade by Sigma Aldrich.

2.2. Experimental setup and dealcoholization conditions

A lab scale plant equipped with membrane module was set up (Fig. 1): feed and stripping streams were fed into the module in counter-current and circulated through tube and shell side, respectively. Wine and hydroalcoholic solution (ranging from 0.7 to 13.0 vol.%) were used as feed stream while water as stripping agent. The temperature of inlet streams was set at 20 °C by a thermostatic water bath while that of outlet streams was monitored by K-type thermocouples. Feed pressure was measured by a manometer, whereas flow rates by flow meters. Dealcoholization tests were carried out at feed and stripping flow rates of 70 and 140 ml/min, respectively. These conditions were chosen according to previous study (Liguori et al., 2012), where the effect of operating conditions on OD was investigated. The feed (0.5 L) and stripping agent (1 L), with an initial volume ratio between stripping and feed streams equal to 2, were recycled during the trials. The process was performed in cycles: at the end of each cycle the

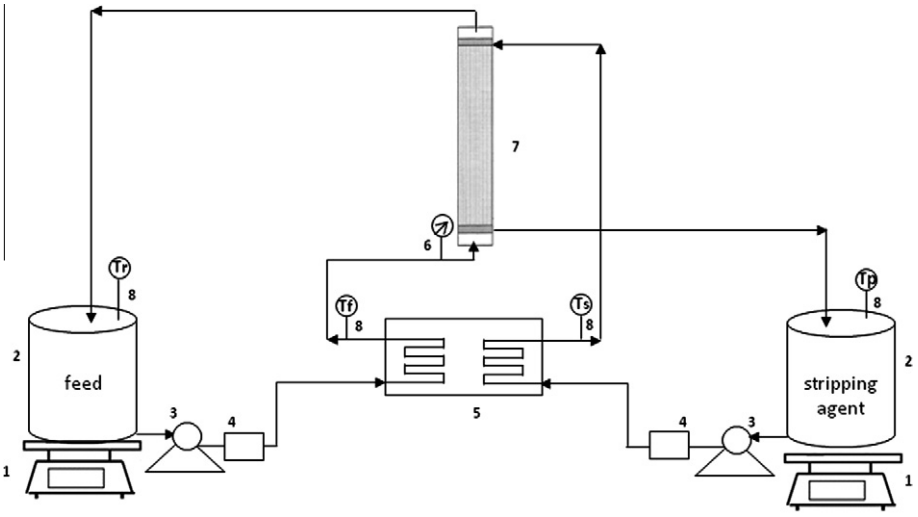

Fig. 1. Experimental setup of dealcoholization plant: (1) stirrer, (2) feed and stripping tanks, (3) pumps, (4) flow meter, (5) thermostatic bath, (6) pressure gauge, (7) membrane module, (8) thermocouples in feed (T_f), stripping (T_s), retentate (T_r) and permeate streams (T_p).

stripping agent was renewed and the retentate (V, i.e. dealcoholized wine collected at the end of each cycle) was codified as follows: V_1, V_2, V_3, V_4, V_5. The cycle times (60 min for the 1st and 2nd cycle and 45 min for the 3rd, 4th and 5th cycle) were set up on the basis of results of dealcoholization kinetic tests, performed starting by hydroalcoholic solutions at different alcohol content (0.7–13.0 vol.%). The dealcoholization ended when an alcohol content lower than 0.5 vol.% was reached in retentate.

2.3. Analyses

Alcohol content, pH and total acidity were measured according to the OIV Compendium of International Methods of Analysis of Wine and Musts (2007).

The amount of phenolic compounds in wine samples was determined with the Folin–Ciocalteu reagent (Singleton & Rossi, 1965). Wine samples were diluted 10-fold and 1 mL was transferred into a 100 mL volumetric flask where 5 mL of Folin–Ciocalteu reagent and 15 mL of Na_2CO_3 (20 %w/v) were added. The solutions were stored in the dark and absorbances were measured at 765 nm after 2 h of reaction. Total phenols were expressed as gallic acid equivalents (mg/L of GAE). Gallic acid standard solutions were prepared at a concentration ranging from 100 to 500 mg/L.

Colour parameters were evaluated by spectrophotometric measurements, which were made on diluted wine samples (10-fold) using a 1 cm optical path. Absorbances were measured at 420, 520, 620 nm, by Perkin Elmer UV/VIS Spectrometer equipped with Lambda Bio 40 software.

According to Glories method (1984), the colour intensity (CI) and the tonality (T) were expressed as follows:

$$IC = A_{420} + A_{520} + A_{620} \quad (1)$$

$$T = \frac{A_{420}}{A_{520}} \quad (2)$$

Flavonols and tartaric esters contents were determined by spectrophotometry. In particular, 0.5 mL of wine sample was diluted to 5 mL by ethanol solution (10 vol.%). Then 0.25 mL aliquot of each diluted sample was subsequently added to 0.25 mL of 0.1% HCl in 95% ethanol, and 4.55 mL of 2% HCl. Each sample was vortexed and allowed to stand for 15 min. The absorbance of each sample was measured in a 1 cm quartz cuvette at 320 and 360 nm which correspond to tartaric esters and flavonols content. The calibration curves were constructed using diluted solutions of quercetin (in 95% ethanol) and caffeic acid (in 10% ethanol). Total flavonols and tartaric esters content were expressed respectively as quercetin (mg/L) and caffeic acid (mg/L) equivalents. (Cliff, King, & Schlosser, 2007).

Organic acids were determined by ion exchange chromatography. The apparatus (Dionex Corporation, USA) was equipped with an ED 50 electrochemical detector, Ionpac AS11 column (250 × 4 mm) and Ionpac AS11 Guard (50 × 4 mm). All wine samples were diluted (20-fold) and then filtered (Millex-gv, 0.22 μm pore size filters, Millipore, USA). Standards of the organic acids were prepared from 1 g/L stock solution and diluted to the required concentration before use. Acquisition and integration of chromatograms were performed with Peaknet G4G1T0 Dionex Corp. software. The elution phase used was bidistilled water (E1) and NaOH 100 mM (E2) for a total running time of 25 min, using the following gradient: from 93% E1 at time 0 to 65% E1 at 20 min, then to 93% E1, in 5 min. The flow rate was 0.5 ml/min. The procedure was in accordance with Albanese, Cinquanta, and Di Matteo (2007).

The procedure for the extraction of the aroma compounds was according to Cocito, Gaetano, and Delfini (1995). Wine or dealcoholized wine sample (100 mL) was put into a 200 mL spherical flask and extracted with dichloromethane (15 mL) by means of three successive ultrasound treatments at 20 °C for 10 min. After separation, the organic layer was dried on anhydrous sodium sulphate and transferred to a vacuum flask. The organic layers obtained by the three extractions were collected in the same flask. The extracts were concentrated to a final volume of about 100 μl in a vacuum rotatory evaporator, and then by a gentle stream of nitrogen. The identification and determination of the volatile compounds were made by GC–MS (Trace MS plus, Thermo Finnigan, USA) and by GC–FID (HP 6890, Agilent), both equipped with a capillary column (Supelcowax 10; 60 m × 0.25 mm × 0.25 μm, Supelco, USA). The gas chromatographic conditions were in accordance with Albanese, Attanasio, Cinquanta, and Di Matteo (2012). The identification of volatile compounds was carried out by comparison of the mass spectrum with those reported in NIST and Wiley libraries. The semi-quantitative results were obtained by the ratio of peak area of the identified volatile compounds and 2-octanol, as internal standard.

2.4. Statistical analysis

Dealcoholization trials and analytical measurements were carried out in triplicate and mean values and standard deviation values were reported. Monofactorial variance analysis was used to determine significant differences ($p < 0.05$) among Aglianico (control) wine and dealcoholized wine samples by Analysis Lab software.

3. Results and discussion

3.1. Dealcoholization kinetics

In the dealcoholization of beverages by OD, the selective removal of ethanol arises from the establishment of vapour pressure difference across the wall of the membrane. In order to fix the optimum exchange time of alcohol from feed to stripping stream, dealcoholization kinetic tests were carried out for each cycle of the process up to obtain an alcohol content lower than 0.5 vol.%. Hydroalcoholic solutions at decreasing concentrations of ethanol (13.0, 6.0, 3.0, 1.2, 0.7 vol.%) were used. The temporal profiles of alcoholic concentration in feed and stripping streams starting by hydroalcoholic solution at 13.0 vol.% (20 °C) were shown in Fig. 2a. During 100 min of the process, a decreasing ethanol concentration in hydroalcoholic solution occurred and a corresponding moderate alcoholization of water, with an ethanol loss in feed stream gradually smaller approaching an equilibrium condition. In particular after 60 min, the alcoholic concentration in hydroalcoholic solution was 6.0 vol.%. Going on the process (within 100 min), the ethanol concentration slightly decreased, reaching 4.7 vol.%, hence, 60 min was chosen as the cycle time.

Afterwards, the alcohol reduction was monitored in a hydroalcoholic solution at 6.0 vol.% (Fig. 2b). Similarly to the previous cycle, a percentage reduction of about 50% of the ethanol concentration after 60 min was observed.

Starting by an alcohol content of 3.0 vol.%, the ethanol reduction was of about 50% after 45 min and the two streams approach the equilibrium in 70 min (Fig. 2c). Therefore, the 3rd cycle time was chosen shorter than previous ones, equal to 45 min.

When hydroalcoholic solutions at 1.2 and 0.7 vol.% were used, an ethanol reduction (in percentage) similar to that of 3rd cycle was observed, then the duration of the two subsequent cycles was chosen equal to 45 min (data not reported).

3.2. Wine dealcoholization

The process parameters established by dealcoholization kinetic tests were applied for the production of a total dealcoholized wine.

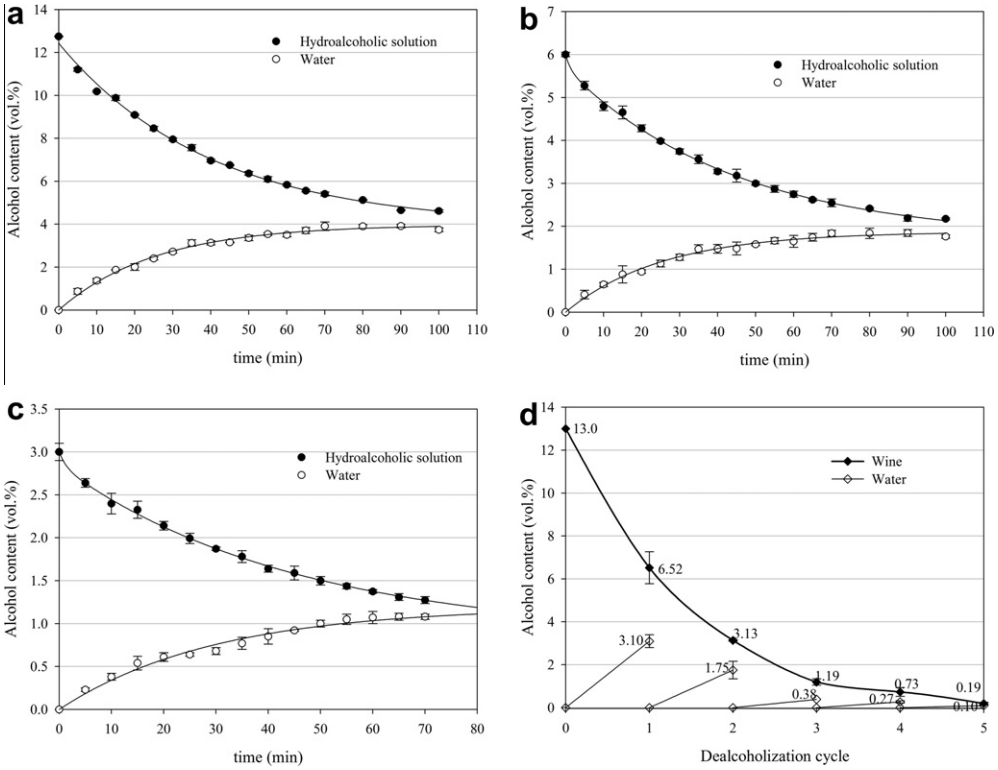

Fig. 2. Alcohol content in feed and stripping streams during dealcoholization of hydroalcoholic solutions at different initial concentrations (a) 13.0 vol.%, (b) 6.0 vol.%, (c) 3.0 vol.% and of (d) Aglianico wine.

The evolution of alcohol content in wine and water during the process was shown in Fig. 2d. The initial alcohol content (13 vol.%) of Aglianico wine (V_0) decreased progressively, and it was halved (−6.5 vol.%) after the 1st cycle (V_1); a further reduction of about 50% (−9.9 vol.%) was achieved after the 2nd cycle (V_2). In subsequent cycles (3rd -V_3 and 4th -V_4) the alcohol decrease was −11.8 and −12.3 vol.%, respectively, whereas at the end of 5th cycle (V_5), the final alcohol content was 0.19 vol.% (−12.8 vol.%). Correspondingly, the stripping stream, from zero alcohol content at the beginning of each cycle, was enriched in ethanol up to a concentration equal to about half of the percentage loss observed in the wine (Fig. 2d).

It is worth noting that in the case of wine a similar behaviour to that obtained for hydroalcoholic solutions was found, suggesting that the presence of other components in wine (i.e. aroma compounds) did not have a significant influence on the kinetics of dealcoholization.

3.3. Dealcoholization effect on Aglianico wine

Aglianico wine (V_0) and dealcoholized wine samples at different alcoholic concentrations (V_1, V_2, V_3, V_4, V_5) were compared in terms of the main chemical and physical parameters.

The change in pH, total acidity, total phenolics, flavonols, tartaric esters content of Aglianico wine during the dealcoholization process is reported in Table 1. It is worth noting that no significant differences ($p < 0.05$) in pH and total acidity were observed among samples. Moreover, data obtained were in accordance with the results reported by Lisanti et al. (2012) for partially dealcoholized red wine samples.

The quantification of phenolic compounds in wine is an important issue because of their influence on the colour, astringency and antioxidant activity of wine. Moreover, they contribute to the healthy properties of wines with reduced alcohol content, without the negative effect of alcohol consumption.

The total phenolic content (Table 1) in Aglianico wine (V_0) was 2932.5 GAE mg/L, in agreement with Sicilian red and Aglianico wines, ranging from 2280 to 3630 GAE mg/L (Di Majo, La Guardia, Giammanco, La Neve, & Giammanco, 2008; Liguori et al., 2012).

In our study, the amount of phenolic compounds did not change significantly ($p < 0.05$) during the dealcoholization process (Table 1); similar behaviour was observed for red wines from three grape varieties (Merlot, Aglianico and Piedirosso) dealcoholized at different levels (ethanol removal of −2, −3 and −5 vol.%) (Lisanti et al., 2012). No data are available on wines with a reduced alcohol content over − 5 vol.%, by osmotic distillation.

The flavonols and tartaric esters concentrations were measured in Aglianico wine equal to 102.5 mg/L and 192.8 mg/L, respectively (Table 1). Similar values for Cabernet Franc, Merlot, and Pinot Noir wines were found by Mazza, Fukumoto, Delaquis, Girard, and Ewert (1999) in the range of 76–157 mg/L and 117–226 mg/L for flavonols and tartaric esters, respectively. The dealcoholization process did not change the amount of these compounds significantly ($p < 0.05$) (Table 1).

The evolution of colour intensity and tonality of wine samples during the dealcoholization process was evaluated and results are reported in Table 1. Not significantly different values ($p < 0.05$) were detected for V_0 and V_1 samples. Alike, Lisanti et al. (2012) observed that the dealcoholization process did not affect the chromatic characteristics of wines for alcohol reduction up to − 5 vol.%. On the contrary, for ethanol reduction higher than −10 vol.% (V_2–V_5 samples), a significant increase ($p < 0.05$) in colour intensity with respect to V_0 and V_1 samples was observed. These results can be justified by a different solubility of wine pigments as function of alcohol concentration or the formation of a more coloured pigment during the dealcoholization process, due

Table 1
Wine chemical parameters (mean values ± standard deviation) during the dealcoholization process.

Sample	Alcohol content (vol.%)	pH	Total acidity (g/L)	Total phenolics (GAE mg/L)	Flavonols (quercitin mg/L)	Tartaric esters (caffeic acid mg/L)	Colour intensity	Tonality
V_0	13.00	3.30 ± 0.03^a	5.61 ± 0.10^a	2932.5 ± 263.2^a	102.5 ± 0.9^a	192.8 ± 14.4^a	1.01 ± 0.01^a	0.84 ± 0.01^{cd}
V_1	6.52	3.27 ± 0.03^a	5.70 ± 0.09^a	2960.9 ± 282.5^a	104.1 ± 6.9^a	201.1 ± 1.2^a	1.04 ± 0.01^a	0.84 ± 0.02^{cd}
V_2	3.13	3.18 ± 0.10^a	5.73 ± 0.12^a	2950.5 ± 301.9^a	100.0 ± 0.1^a	200.3 ± 7.2^a	1.24 ± 0.06^b	0.89 ± 0.07^{bd}
V_3	1.19	3.25 ± 0.06^a	5.45 ± 0.15^a	2942.3 ± 180.3^a	113.9 ± 3.0^a	202.4 ± 3.3^a	1.44 ± 0.16^c	0.96 ± 0.07^{ab}
V_4	0.73	3.27 ± 0.05^a	5.50 ± 0.05^a	2948.3 ± 241.7^a	109.4 ± 5.9^a	196.5 ± 10.7^a	1.45 ± 0.07^c	1.00 ± 0.10^a
V_5	0.19	3.28 ± 0.04^a	5.55 ± 0.13^a	3024.7 ± 88.5^a	112.4 ± 2.2^a	203.2 ± 3.2^a	1.40 ± 0.04^c	0.94 ± 0.03^{abc}

Different letters indicate significant differences ($p < 0.05$) among the samples.

Table 2
Organic acids composition (mg/L; mean values ± standard deviation) of Aglianico and dealcoholized wine samples.

Sample	Lactic acid	Acetic acid	Succinic acid	Malic acid	Tartaric acid	Ossalic acid	Ascorbic acid	Citric acid
V_0	2434.7 ± 23.6^a	553.4 ± 2.9^a	151.8 ± 1.1^a	645.0 ± 1.4^a	1697.8 ± 1.2^a	327.6 ± 1.5^a	29.2 ± 0.8^a	546.6 ± 3.4^a
V_1	2282.6 ± 190.0^a	548.5 ± 11.2^a	130.0 ± 25.3^a	640.7 ± 43.4^a	1680.8 ± 56.6^a	328.4 ± 9.9^a	27.2 ± 0.6^a	565.0 ± 4.6^a
V_2	2329.0 ± 129.2^a	542.7 ± 26.8^a	136.8 ± 25.3^a	669.8 ± 53.2^a	1702.8 ± 14.6^a	341.6 ± 7.1^a	27.1 ± 1.4^a	589.1 ± 16.4^a
V_3	2447.2 ± 57.4^a	538.7 ± 26.6^a	141.1 ± 29.0^a	734.8 ± 17.5^a	1830.2 ± 109.1^a	343.3 ± 8.5^a	31.8 ± 2.6^a	613.2 ± 56.8^a
V_4	2333.5 ± 60.1^a	548.3 ± 55.3^a	122.1 ± 22.3^a	629.0 ± 27.9^a	1731.1 ± 39.0^a	349.5 ± 2.1^a	30.9 ± 0.3^a	604.3 ± 1.5^a
V_5	2231.5 ± 276.7^a	547.2 ± 26.4^a	138.5 ± 0.7^a	568.5 ± 175.3^a	1693.4 ± 220.6^a	355.3 ± 15.5^a	28.9 ± 0.8^a	619.0 ± 10.3^a

Different letters indicate significant differences ($p < 0.05$) among the samples.

to the oxygen intake and to the loss of SO_2 (Hermosin Gutierrez, 2003).

Organic acids can affect pH values dramatically and also have implications on biological stability, sensory properties and the colour of the wine (Ronald, 2008). The chromatographic screening of organic acids showed a quali-quantitative profile in Aglianico wine, according to Clarke and Bakker (2004) for red wines: tartaric and lactic acids were in amounts greater than 1500 ppm, while malic, citric, oxalic and acetic acids in lower concentrations; small quantities of succinic acid and ascorbic acid (<200 ppm) were found (Table 2). As expected, no significant changes ($p < 0.05$) were observed in organic acids during the dealcoholization process, being compounds with high boiling points, and therefore not involved in the OD mechanism, at room temperature.

Volatile compounds of Aglianico wine (V_0) were reported in Table 3. A total of 42 compounds were identified in the aroma fraction of control wine, which were quantified and classified in the following seven chemical classes: alcohols, ethyl esters, acids, sulphur compounds, phenols, ketones, lactones and aldehydes.

Herein, we analyzed the volatile profile of wine at different alcoholic concentrations (Table 3).

Higher alcohols or fusel alcohols were quantitatively the largest group (~90 wt.%) of the volatile compounds identified in control wine. Higher alcohols may be present in healthy grapes, but seldom occur in significant amount. They also donate a herbaceous odour in certain wines. Quantitatively, the most important higher alcohols are the straight-chain alcohols: isoamyl alcohols (3-methyl-1-butanol and 2-methyl-1-butanol) were found as the most abundant (85 wt.%) followed by 2-phenylethanol (14 wt.%) and isobutyl alcohol (0.7 wt.%). 2-phenylethanol is the most important phenol-derived higher alcohol. Most straight-chain higher alcohols have a strong pungent odour. At low concentrations (0.3 g/L or less), they generally add an aspect of complexity to the bouquet. At higher levels, they increasingly overpower the fragrance.

After the 1st dealcoholization cycle (−6.5 vol.%), alcohols class suffered a deep loss (58%) than other aromatic classes, due to the great loss (66%) of isoamyl alcohols. In the subsequent cycles, isoamyl alcohols underwent a further reduction (95% in the 2nd cycle up to 99% in the 5th cycle). The 2-phenylethanol suffered a more gradual decrease, which became higher with greater alcohol removal up to disappear in totally dealcoholized wine. In V_5, only some alcohol compounds remained in small amount less than 0.21 mg/L. The behaviour of alcohol compounds seems to be due to different values of the vapour pressure: i.e. the 2-propanol has got a high vapour pressure (33.15 mmHg at 20 °C) in comparison with 2,3-butanediol and 2-hexanol (respectively 0.26 and 2.07 mmHg at 20 °C) (Perry & Green, 2007).

As previously reported, there are no paper in literature about the total dealcoholization of wine by OD; so only the results at the end of the 1st cycle can be compared with other data.

As regards isoamyl alcohols, similar results were found by Diban et al. (2008), Varavuth et al. (2009) for model solutions and by Lisanti et al. (2012) for red wines.

For 2-phenylethanol and benzyl alcohol, our results are also in agreement with those obtained by other researchers (Diban et al., 2008; Lisanti et al., 2012). The unchanged amount of this compound after the 1st cycle may be justified by a retention effect of red wine due to π–π interactions resulting from the high content of polyphenols (Rodriguez-Bencomo et al., 2011). The strength of these interactions could be dependent on the alcohol concentration, stronger for alcohol content higher than about 7 vol.% and thus justify the significant reduction ($p < 0.05$) of 2-phenylethanol after the 2nd and subsequent cycles.

Esters are the second most abundant group (~7 wt.%) in the Aglianico wine. These compounds have two distinct origins in wine: enzymatic esterification during the fermentation process and chemical esterification during long-term aging. The same esters may be synthesized in either way (Ribereau-Gayon, Glories, Maujean, & Dubourdieu, 2006). Monoethyl and diethyl succinate were found in major concentrations. Ethyl acetate was found in Aglianico wine at 0.02 mg/L. This compound adds complexity to the aroma of wines at low levels, but it can give an unpleasant odour (vinegary) to the wine at concentrations higher than 150 mg/L (Mallouchos, Komaitis, Koutinas, & Kanellaki, 2002). Esters concentration became lower with the alcohol concentration decrease: the percentage loss was just about 10% in the 1st cycle where ethyl lactate and monoethyl succinate amount remained almost unchanged; on the contrary, ethyl hexanoate, ethyl octanoate, β-phenylethyl acetate, ethyl acetate and isoamyl acetate were lost.

Table 3
Volatile compounds content (mg/L; mean values ± standard deviation) in Aglianico and dealcoholized wine samples.

No	Ret. time (min)	Compounds (mg/L)*	V_0	V_1	V_2	V_3	V_4	V_5
Alcohols								
7	20.85	Isoamyl alcohols	265.90 ± 56.47^{a}	89.95 ± 1.77^{b}	11.16 ± 0.46^{c}	2.95 ± 0.32^{d}	0.10 ± 0.03^{e}	0.21 ± 0.03^{f}
31	43.86	2-Phenylethanol	43.63 ± 1.44^{a}	40.36 ± 5.78^{ab}	29.25 ± 7.54^{bc}	27.24 ± 1.67^{c}	17.84 ± 1.38^{c}	n.d.
11	26.80	3-Ethoxy-1-propanol	0.02 ± 0.01^{a}	0.01 ± 0.01^{a}	n.d.	n.d.	n.d.	n.d.
14	29.70	2-Propanol	0.08 ± 0.03	n.d.	n.d.	n.d.	n.d.	n.d.
16	32.41	2,3-Butanediol	0.47 ± 0.02^{a}	0.32 ± 0.01^{b}	0.25 ± 0.05^{b}	0.05 ± 0.01^{c}	0.03 ± 0.01^{c}	0.07 ± 0.01^{c}
17	33.40	3-Methyl-2-hexanol	0.24 ± 0.03^{a}	0.14 ± 0.01^{b}	0.08 ± 0.02^{c}	0.12 ± 0.01^{bc}	0.01 ± 0.01^{d}	0.01 ± 0.01^{d}
18	33.52	2-Hexanol	0.11 ± 0.01^{a}	0.11 ± 0.01^{a}	0.16 ± 0.04^{a}	0.16 ± 0.02^{a}	0.02 ± 0.01^{b}	0.02 ± 0.01^{b}
27	40.48	1-Propanol	0.35 ± 0.01^{a}	0.31 ± 0.01^{b}	0.27 ± 0.01^{b}	0.19 ± 0.01^{c}	0.14 ± 0.01^{d}	0.07 ± 0.01^{e}
4	16.09	Isobutyl alcohol	2.28 ± 1.06 ^{a}	0.32 ± 0.03 ^{b}	0.22 ± 0.01 ^{c}	0.07 ± 0.01 ^{d}	n.d.	n.d.
30	42.68	Benzyl alcohol	0.44 ± 0.10^{a}	0.48 ± 0.01 ^{a}	0.49 ± 0.02^{a}	0.30 ± 0.16^{a}	0.09 ± 0.01^{b}	n.d.
		Total	313.53 ± 59.19	131.99 ± 7.62	41.72 ± 8.14	31.62 ± 2.20	18.23 ± 1.42	0.38 ± 0.06
Esters								
8	21.63	Ethyl hexanoate	0.04 ± 0.00	n.d.	n.d.	n.d.	n.d.	n.d.
10	25.84	Ethyl lactate	0.11 ± 0.02^{a}	0.08 ± 0.01^{ac}	0.06 ± 0.01 ^{b}	0.07 ± 0.01 ^{bc}	0.01 ± 0.01^{d}	n.d.
12	28.70	Ethyl octanoate	0.05 ± 0.01	n.d.	n.d.	n.d.	n.d.	n.d.
24	36.70	Diethyl succinate	12.17 ± 3.76^{a}	10.12 ± 1.33^{a}	4.51 ± 0.50^{b}	4.23 ± 0.17^{b}	2.11 ± 0.28^{c}	1.45 ± 0.13^{d}
33	46.92	Diethyl malate	0.64 ± 0.06 ^{a}	0.61 ± 0.04^{a}	0.65 ± 0.02^{a}	0.37 ± 0.02^{b}	0.36 ± 0.07^{bc}	0.16 ± 0.13^{c}
42	55.68	Monoethyl succinate	11.71 ± 4.76^{ab}	10.99 ± 2.92^{a}	6.15 ± 1.17^{b}	2.65 ± 0.24^{c}	1.86 ± 0.15^{d}	0.92 ± 0.12^{e}
28	41.04	β-Phenylethyl acetate	0.18 ± 0.29	n.d.	n.d.	n.d.	n.d.	n.d.
2	9.38	Ethyl acetate	0.02 ± 0.01	n.d.	n.d.	n.d.	n.d.	n.d.
5	18.22	Isoamyl acetate	0.05 ± 0.02	n.d.	n.d.	n.d.	n.d.	n.d.
1	5.69	Isopropyl acetate	0.04 ± 0.03^{a}	0.04 ± 0.01^{a}	n.d.	n.d.	n.d.	n.d.
15	31.74	Ethyl-3-hydroxybutyrate	0.04 ± 0.01^{a}	0.04 ± 0.01^{a}	0.05 ± 0.03^{a}	0.05 ± 0.01^{a}	n.d.	n.d.
21	35.44	1,6 Diethyl hexanedioate	0.04 ± 0.01^{a}	0.02 ± 0.01^{a}	n.d.	n.d.	n.d.	n.d.
41	54.19	β-Phenylethyl formate	0.22 ± 0.23^{a}	0.12 ± 0.01^{a}	n.d.	n.d.	n.d.	n.d.
		Total	25.30 ± 9.22	22.05 ± 4.34	11.44 ± 1.73	7.38 ± 0.44	4.34 ± 0.50	2.54 ± 0.39
Acids								
6	19.41	4-Hydroxybenzoic acid	0.03 ± 0.01	0.02 ± 0.01	n.d.	n.d.	n.d.	n.d.
13	29.5	Acetic acid	0.43 ± 0.21^{a}	0.13 ± 0.01^{b}	0.10 ± 0.01^{c}	n.d.	n.d.	n.d.
20	30.35	Butanoic acid	0.03 ± 0.01^{a}	0.03 ± 0.01^{a}	0.04 ± 0.01^{a}	n.d.	n.d.	n.d.
23	36.43	Hexanoic acid	0.16 ± 0.03 ^{a}	0.07 ± 0.01 ^{b}	0.06 ± 0.01 ^{b}	0.08 ± 0.01 ^{b}	n.d.	n.d.
29	41.64	Heptnaoic acid	0.47 ± 0.03^{a}	0.49 ± 0.01^{a}	0.37 ± 0.06^{b}	0.35 ± 0.02^{b}	0.10 ± 0.01^{c}	0.09 ± 0.01^{c}
35	47.33	Octanoic acid	0.90 ± 0.22 ^{ab}	0.93 ± 0.06 ^{a}	0.66 ± 0.04^{b}	0.49 ± 0.03 ^{c}	0.42 ± 0.03 ^{c}	0.15 ± 0.03^{d}
38	52.35	5-Oxotetrahydrofuran-2-carboxylic acid	2.66 ± 1.72^{ab}	1.91 ± 0.23^{b}	1.24 ± 0.13^{a}	1.14 ± 0.08^{a}	1.06 ± 0.43^{ac}	0.77 ± 0.09^{c}
39	52.46	Decanoic acid	0.19 ± 0.06^{a}	0.16 ± 0.01^{a}	0.09 ± 0.02^{b}	0.08 ± 0.00^{b}	0.12 ± 0.05^{ab}	0.02 ± 0.01^{c}
		Total	4.87 ± 2.27	3.72 ± 0.32	2.56 ± 0.29	2.14 ± 0.14	1.70 ± 0.52	1.03 ± 0.14
Sulphur compounds								
25	38.06	Methionol	0.39 ± 0.02 ^{a}	0.37 ± 0.02 ^{ab}	0.31 ± 0.05 ^{b}	0.39 ± 0.01 ^{a}	0.07 ± 0.01 ^{c}	0.08 ± 0.01 ^{c}
3	13.64	Methanethiol	0.07 ± 0.02^{ab}	0.09 ± 0.01^{a}	0.05 ± 0.01^{b}	0.04 ± 0.01^{b}	0.04 ± 0.01^{b}	0.01 ± 0.01^{c}
		Total	0.47 ± 0.04	0.46 ± 0.03	0.36 ± 0.06	0.43 ± 0.02	0.11 ± 0.02	0.10 ± 0.01
Ketones and lactones								
9	23.91	Acetoin	0.05 ± 0.01^{a}	0.05 ± 0.01^{a}	0.05 ± 0.02^{a}	0.06 ± 0.01^{a}	n.d.	n.d.
22	36.22	γ-Butyrolactone	0.86 ± 0.05^{a}	0.89 ± 0.09 ^{a}	0.84 ± 0.02^{a}	0.70 ± 0.03^{b}	0.18 ± 0.02^{c}	0.02 ± 0.01^{d}
26	38.61	1-Hydroxy-2-propanone	0.03 ± 0.01 ^{a}	0.03 ± 0.01 ^{a}	n.d.	n.d.	n.d.	n.d.
34	47.21	Pantolactone	0.35 ± 0.05^{a}	0.05 ± 0.01 ^{b}	0.08 ± 0.01 ^{c}	0.03 ± 0.01 ^{b}	0.02 ± 0.01 ^{b}	0.01 ± 0.01 ^{b}
36	49.97	3-Methyl-2-pentanone	0.12 ± 0.02^{a}	0.06 ± 0.04^{a}	0.08 ± 0.02^{a}	0.10 ± 0.02^{a}	n.d.	n.d.
40	53.14	Phenylacetone	0.03 ± 0.01^{a}	0.02 ± 0.01^{a}	0.03 ± 0.01^{a}	0.03 ± 0.01^{a}	0.03 ± 0.01^{a}	n.d.
		Total	1.44 ± 0.14	1.10 ± 0.16	1.09 ± 0.07	0.93 ± 0.06	0.24 ± 0.04	0.03 ± 0.02
Phenols								
32	45.47	2-Methoxyphenol	0.05 ± 0.04	n.d.	n.d.	n.d.	n.d.	n.d.
37	51.03	2-Ethylphenol	0.10 ± 0.01^{a}	0.05 ± 0.01^{b}	0.03 ± 0.00^{b}	0.04 ± 0.02^{b}	n.d.	n.d.
		Total	0.15 ± 0.05	0.05 ± 0.01	0.03 ± 0.00	0.04 ± 0.02	0.0	0.0
Aldehydes								
19	34.27	Acetaldehyde	0.03 ± 0.01^{ab}	0.03 ± 0.01^{ab}	0.03 ± 0.01^{ab}	0.04 ± 0.01^{a}	0.01 ± 0.01^{b}	n.d.

Different letters indicate significant differences ($p < 0.05$) among the samples.
* The concentration is expressed by the ratio of peak area of the identified volatile compounds and 2-octanol as internal standard.

Increasing the level of dealcoholization, esters loss grew up to 90% in V_5 sample, with a residual amount of esters equal to 2.54 mg/L in comparison with 25.30 mg/L in V_0.

In dealcoholization tests reported in literature (Diban et al., 2008; Lisanti et al., 2012; Varavuth et al., 2009), similar esters loss (over 50%) were found.

Despite of alcohols, some esters reduction could be explained by means of their hydrophobic character (expressed by partition coefficient between octanol and water, $\log K_{ow}$), and hence by means of their affinity to the membrane, coupled to their significant volatility that allow them to easily pass through the membrane pores. These results are in agreement with Diban et al. (2008) but in contrast with Lisanti et al. (2012). In fact, esters with high value of partition coefficient such as ethyl octanoate ($\log K_{ow} = 3.81$), ethyl hexanoate ($\log K_{ow} = 2.83$) and isoamyl acetate ($\log K_{ow} = 2.26$) disappeared after the 1st dealcoholization cycle.

The fatty acids have been described as giving rise to fruity, cheesy, fatty and rancid notes. Although, C_6–C_{10} fatty acids are usually related to the appearance of negative odours, they are important for aromatic equilibrium in wines because they oppose the hydrolysis of the corresponding esters, and their presence plays

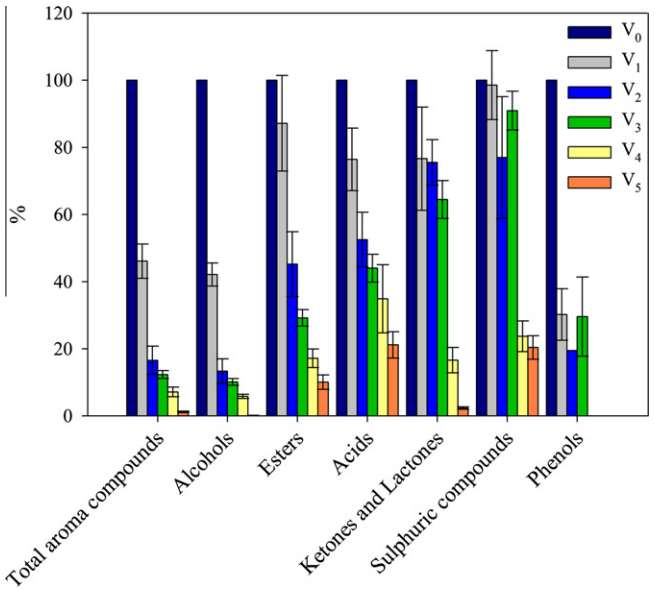

Fig. 3. Total aroma (%) and chemical classes (%) during the dealcoholization process with respect to the control wine.

an important role in the complexity of the aroma (Boidron, Chatonnet, & Pons, 1988). These compounds represented about 1.5 wt.% of the overall volatile compounds identified; a large amount of 5-oxotetrahydrofuran-2-carboxylic acid, followed by octanoic, eptanoic and acetic acid were detected in wine (Table 3). Results showed higher loss (over 50% of the initial concentration) of acetic and hexanoic acids, while butanoic, heptanoic and octanoic acids concentration remained almost unchanged. As for alcohols, the different losses observed during the dealcoholization process can be related to the different vapour pressure values (i.e. acetic acid = 12.02 mmHg and heptanoic acid = 0.002 mmHg, at 20 °C) (Perry & Green, 2007).

Ketones and lactones group (0.4 wt.% of all volatile compounds) was constituted by six compounds: four ketones and two lactones. Many ketones are produced during fermentation, but few appear to have sensory significance. Among the ketones identified in control wine, acetoin (3-hydroxy-2-butanone) has a sugary, butter-like character. Its sensory significance in table wines, in which it occurs at low concentrations, is doubtful. About lactones, γ-butyrolactone is the most abundant found in control wine; pantolactone has a pleasant aroma with spicy and caramel attribute (Selli et al., 2004).

As showed in Table 3, at the end of 1st cycle, pantolactone showed a higher loss than other compounds, while 1-hydroxy-2-propanone, phenylacetone, acetoin and γ-butyrolactone remained almost unchanged. The latter, corresponding to the 60 wt.% of this class, remained unchanged up to the 2nd cycle; afterwards decreased significantly ($p < 0.05$).

These aroma are high-boiling compounds, and their solubility in water is relatively high with consequent low volatility (Clarke & Bakker, 2004).

Only five compounds belonging to sulphur, phenolic, aldehydic compounds were identified and detected in small quantities (Table 3). After the 1st cycle, 2-methoxyphenol disappeared while 2-ethylphenol and acetaldehyde decreased when the alcohol reduction became dramatically high (4th cycle). On the contrary, the sulphur compounds presented a percentage loss (80%) lower than phenols and aldehydes in the total dealcoholized wine (V_5). The loss of these volatile compounds can be considered not negative since they are responsible for off-flavour in wine.

Finally, the percentage of total aroma and chemical classes with respect to the control wine during the dealcoholization process was shown in Fig. 3. Total volatile compounds decreased about 50% and 80% after the 1st and the 2nd cycle, respectively. At the end of the process, a minimum amount of volatiles (about 1.2% of the initial one) remained in totally dealcoholized wine. The trend of the chemical classes highlighted a different percentage decrease. Esters, ketones and lactones showed minimum losses during the first three cycles, while the acids seemed to be the volatile compounds less influenced by the total dealcoholization process. In conclusion, the aroma loss mechanism depends on the synergism between the chemical and physical properties (chemical structure, boiling point, vapour pressure, water solubility and hydrophobicity) of the volatile compounds and their interaction with the wine matrix, that changes during the process.

4. Conclusions

A total dealcoholized wine (0.19 vol.%) was obtained by osmotic distillation technique. The effect of the dealcoholization on wine is negligible for the main chemical and physical properties investigated (total phenols, flavonols, tartaric esters, organic acids), which did not show significant differences ($p < 0.05$) compared to the control wine. The results on colour intensity and tonality of wine samples pointed out an increase for deeper alcohol reduction (higher than −6.5 vol.%) during the dealcoholization process. The volatile fraction of wine decreased significantly together with the dealcoholization level. Therefore, future work should be focused on flavour enrichment, by adding specific aroma compounds or wine volatiles recovered from stripping stream, to make a delicious non alcoholic beverage from wine.

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