

# Wine dealcoholization using new method for low temperature nitrogen assisted distillation

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**Abstract.** New InnoSolv llc. device for low temperature nitrogen assisted distillation was used for wine dealcoholization. The method approved by virtue of collaboration of InoSolv Ltd and UFT include evaporation of alcohol and other volatiles at a temperature not exceeding 32 °C in entirely nitrogen atmosphere. The three regime of wine treatment were used, reducing alcohol level of the treated wine respectively up to 9.80 (regime A); 5.85 (regime B) and 2.75 vol.% (regime C) starting from 14.15 vol.% of the initial wine. More than 80.0% of the esters of the initial wine remain in the treated wine in regime A. The volatiles such as esters, higher alcohols and aldehydes as well as some individual specific volatile compounds were determined in the treated wines and in the separated distillates. The presence of suspended particles does not interfere the proper operation of the installation and device can be used for wines during theirs fermentation. The system and method for offer gentle regimes wine treatment and low operating costs thanks to heat pump incorporation. There is not requirement for steam consumption and cooling water compared to alternative methods. The system is fully closed, without venting outside which is substantial for aroma preservation.

## 1 Introduction

Normally the well-ripe grape is the most desired for the winemaking which usually result in full-bodied and aromatic wines. However this is usually associated with a high alcohol content of the wines. Additionally global warming in recent years also can increase grape sugar and respectively alcohol level in the wines [1,2]. Although in some wine-growing areas the alcohol content of the wines is allowed to be even 20% vol. (EO 2019/934) the alcohol excess can lead to the bitterness and balance flaws of the taste, as well as possible health problems. Therefore high wine alcohol level generally is undesirable and can be partially reduced by appropriate viticultural practices [3,4,5,6], oxidative sugar (glucose) reduction [7] or using yeast strains with reduced ability to alcohol production [7,8,9]. But all these methods can contribute to wine alcohol level reduction and when the purpose is low-alcohol or dealcoholized wine production physical removal of alcohol should be used. The increasing consumer interest to the wines with reduced alcohol level and their market potential [10,11,12] leads more and more wine producers to this kind of production. Research shows that the consumer perception of low-alcohol wines is similar to criteria for standard wines [13] and their acceptance can increase if taste of these wines is similar or the same of conventional wines [11]. These requirements are challenging task especially at the dealcoholized wine production because sweet taste of the ethanol and its warming effect is essential part of the taste of the standard wines. Therefore the physical methods for

alcohol reduction are the subject of essential research interest and undergo continuous improvements.

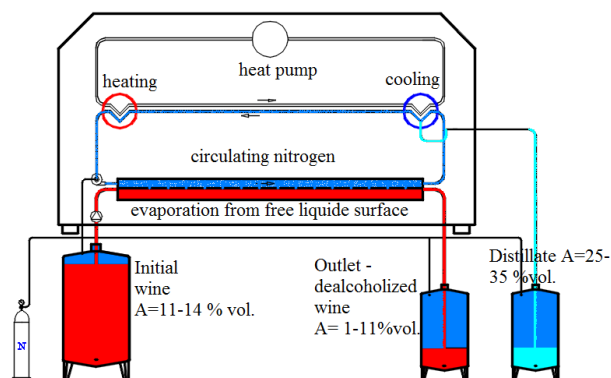
The big group of methods that is used for wine alcohol reduction is membrane based. From this group reverse osmosis is widely used and is the first membrane technology commercially used for wine alcohol reduction [14,15]. Depending of the membrane pore size and the driving force the processes can be divided also in nanofiltration, reverse osmosis, osmotic distillation and prevaporation [16,17,18,19]. Although these method are widely used some disadvantages like high capital investment, some wine aroma losses may occur and addition of second process for separation of the aroma compounds and water from the ethanol may be necessary to incorporate [14,20].

The second widely used technique for wine alcohol reduction is spinning cone [21,22,23] and can also be used and for unfiltered wines. Aroma recovery by pre-evaporating fraction of about 0.9-1.0% of the treated wine at low temperature (26÷28 °C) and high vacuum and fast run of the system are also a part of the advantages of the method. As a disadvantages can be pointed the need of auxiliary equipment (vacuum pump, steam generator, heat exchangers) which makes the initial and operating cost of the system more expensive. In our work new device for low temperature nitrogen assisted distillation was used for wine alcohol reduction using three modes of operation. According to our data a similar treatment method and system has not been used for wine dealcoholization to date.

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## 2 Materials and methods

### 2.1 The used system for wine dealcoholization



**Figure 1.** InnoSolv dealcoholizing system.

The experiments are carried-out on pilot scale equipment for wine dealcoholization created by company InnoSolv plc, Bulgaria – Fig. 1. The method of alcohol removal is distillation meeting OIV requirements for such purposes. In this equipment distillation process in nitrogen assisted one, aiming reducing of wine treatment temperature avoiding oxidation process and keeping of wines native properties as taste, flavor, etc. The nitrogen circulates in close circuit at atmospheric pressure. Initially the nitrogen is heated to increase its removing potential. Then heated nitrogen contacts with wine free surface (wine mirror) for gentle removal of alcohol molecules by means of evaporation. For mirror formation wine is fed in the equipment from inlet wine tank by pump. Dealcoholized wine is collected in outlet tank at alcohol strength between 1 and 11% vol. according dealcoholization parameters settings. After wine mirror the wetted nitrogen pass through cooling process for water, alcohol and other volatiles condensation. The collected distillate has typical strength between 25-35% vol. Dried nitrogen then is recycled back for heating. For this type of dealcoholization process the maximal wine temperature do not exceed 26-28 °C and residence time of wine in equipment is up to few minutes. The working principle of this system is continuous one, fully automated without additional staff requirements. For delivering of heating and cooling process loads heat pump is used. It allows to avoid steam boiler involving for dealcoholization process to work with energy efficient way and to reduce total electricity consumption down to 660 Wh/kg evaporation as pure water. The equipment operates at atmospheric pressure in nitrogen blanket for wines streams and distillates and fully hermitized from surrounding air. Thus there are no losses of valuable aroma compound from the wine.

### 2.2 Samples preparation

The wine made from white grape variety Misket Varnenski (*V.vinifera*) with alcohol content 14,15% vol. was dealcoholized at three mode differing in the speed of the passing through the system respectively led to the three different degree of dealcoholization - up to 9,80

(wine A); 5,85 (wine B) and 2,75 vol.% (wine C). In each run 100 liters of initial wine is pass through the system and dealcoholized wines and distillates were collected in follow volumes (Table 1):

**Table 1.** Sample volumes.

Compound	Initial wine	Dealcoholized wines			Distillates		
		A	B	C	A <sub>d</sub>	B <sub>d</sub>	C <sub>d</sub>
Volume, dm <sup>3</sup>	100.0	76.7	58.8	40.6	20.3	38.2	56.4

In each operation mode after the initial wine is depleted about three liters liquid stay in the system and it is removed and the system is cleaned before every run.

### 2.3 Analysis

The samples (initial wine, wines A, B and C and respectively obtained distillates A<sub>d</sub>, B<sub>d</sub> and C<sub>d</sub>) were analyzed by standard physicochemical analyzes:

- Determination of total acidity (titratable acids) expressed as tartaric acid (Method OIV-MA-AS313-01).
  - Determination of the volatile acids content expressed as acetic acid (Méthode OIV-MA-AS313-02).
  - Determination of the alcohol content of the wine (by Dujardin-salleron ebulliometer).
  - Determination of the content of total phenolic compounds by the FC method (Méthode OIV-MA-AS2-10).
  - A polyphenol index was determined by measuring absorbance at 280 nm in 1 cm cuvette;
- The Folin Chiocalteu (FC) reagent and gallic acid were purchased from Fluka Chemie. All used reagents were chemical grade.
- GC-MS: The static HS-GC-MS analysis was performed in an Agilent 7890A-5975C gas chromatograph-mass spectrometer equipped with an Agilent 7697A headspace auto sampler (Agilent Technologies Inc., Santa Clara, CA, USA). A J&W capillary column HP-5 MS of 30m x 0.250 mm with 0.25 µm film (Agilent Technologies Inc.) was used for the separation. The samples in a 20 mL headspace vials were heated at an equilibrium temperature of 125 °C for 45 min, and the gas phases were injected into the GC-MS for analysis. The injection time was 1.0 min. A low shaker mode of the headspace vial was applied during sample heating. The carrier gas (Helium) was set at a flow rate of 1.0 mL·min<sup>-1</sup>. The inlet temperature was 200 °C with a split ratio of 10:1. The oven temperature programme was as follows: initially set at 60 °C for 5 min, then ramped to 200 °C at 5 °C·min<sup>-1</sup> for 5 min, and finally it was warmed up to 300 °C at 10 °C·min<sup>-1</sup>. The ion source temperature and quadrupole temperature were 230 and 150 °C, respectively. The MSD was operated in full scan mode. All mass spectra were acquired in electron impact mode with 70 eV.

Compounds identification was assigned by comparing their linear retention indices and MS fragmentation patterns with those from the National Institute of

Standards and Technology (NIST'08) and Adams mass spectra library. The estimated LRI were determined using a mixture of a homologous series of aliphatic hydrocarbons from C<sub>8</sub> to C<sub>40</sub> under the same conditions described above.

### 3 Results and Discussion

Three different speeds of wine run were used resulted respectively to 30.74% (mode A), 58.66% (mode B) and 80.57% (mode C) alcohol content reduction of the initial wine (Table 2).

**Table 2.** Initial wine and samples physicochemical analyzes.

sample	Alcohol content	Dry extract	Titratable acidity (TA)	Volatile acidity (VA)	Total phenols (TP)	A <sub>280</sub> (IP)
	vol. %	g/dm <sup>3</sup>	g/dm <sup>3</sup> as tartaric acid	g/dm <sup>3</sup> as acetic acid	mg/dm <sup>3</sup> as gallic acid	
Initial wine	14.15 ± 0.22	20.152 ± 0.23	7.10 ± 0.21	0.24 ± 0.015	275.6 ± 13.8	0.925 ± 0.02
Dealcoholized wines samples:						
A	9.80 ± 0.15 53.12*	25.788 ± 0.32 98.15*	7.49 ± 0.21	0.27 ± 0.015	354.8 ± 20.2 98.15*	1.228 ± 0.03 101.82*
B	5.85 ± 0.15 24.31*	32.308 ± 0.44 94.27*	9.81 ± 0.28	0.30 ± 0.016	454.7 ± 24.6 94.27*	1.540 ± 0.04 97.89*
C	2.75 ± 0.12 7.89*	48.144 ± 0.76 97.00*	14.12 ± 0.38	0.42 ± 0.019	648.2 ± 34.3 96.99*	2.107 ± 0.06 92.48*
Obtained distillates:						
A <sub>d</sub>	32.40 ± 0.46	-	-	0.19 <sup>a</sup> ± 0.010	-	-
B <sub>d</sub>	28.30 ± 0.39	-	-	0.19 <sup>a</sup> ± 0.010	-	-
C <sub>d</sub>	21.70 ± 0.26	-	-	0.15 <sup>a</sup> ± 0.009	-	-

a – In the distillates VA is determined by direct titration with NaOH and expressed as g/l acetic acid;

\* – Calculated in percentage from the initial wine concentration accounting obtained quantities.

Regarding the resulting quantities of wines with reduced alcohol and distillates from each operation mode (Tabl.1) in the treated wines remains respectively 53.12% (wine A), 24.31% (wine B) and 7.89 (wine C) from the amount of alcohol of the initial wine (Table 2). Dry extracts and total phenols remains in the dealcoholized wines as a non-volatile. In the operation mode with the longest treatment of the wine (mode C) in the dealcoholized wine (wine C) the recovery of TP and IP is respectively 95.49% and 92.48%. Wines A, B and C keep about 81.0% from the total acidity and this average 20% reduction can be explained by the partial crystallization of tartaric acid salts as a result of their concentration. Volatile acids as a component of the tail fraction during a conventional distillation keep mainly in the dealcoholized wines.

The acetic acid concentration (Table 3) varies from 204.0 mg/dm<sup>3</sup> to 321.2 mg/dm<sup>3</sup> in the all samples and the initial wine and as a tail fraction more than 50.0% of the initial amount remains in the dealcoholized wines in the A and B mode and 44.13% in the C operation mode.

Unlike acetic acid, the acetaldehyde has a different behavior compared to conventional distillation.

**Table 3.** Volatile compounds in the initial and treated wines and in the distillates (1).

Com-pound	Initial wine	Dealcoholized wines			Distillates		
		A	B	C	A <sub>d</sub>	B <sub>d</sub>	C <sub>d</sub>
acetic acid, mg/dm <sup>3</sup>	246.7 ± 19.7	204.0 ± 24.3 63.45*	234.2 ± 29.1 55.82*	268.1 ± 32.2 44.13*	306.6 ± 42.8 25.24*	321.2 ± 39.6 49.75*	283.7 ± 38.2 64.86*
acetaldehyde mg/dm <sup>3</sup>	36.4 ± 4.1	35.9 ± 6.3 75.65*	47.96 ± 6.8 77.41*	68.3 ± 10.7 76.11*	35.9 ± 6.2 20.04*	31.2 ± 6.2 32.74*	28.8 ± 5.9 44.62*
ethyl-acetate mg/dm <sup>3</sup>	45.2 ± 3.8	40.1 ± 5.3 68.08*	41.5 ± 5.5 53.96*	43.2 ± 5.3 38.82*	49.7 ± 6.0 22.32*	47.8 ± 3.2 40.44*	46.5 ± 4.8 57.93*
Isoamyl alcohol mg/dm <sup>3</sup>	94.4 ± 8.5	99.1 ± 15.0 80.53*	80.3 ± 7.5 49.98*	106.1 ± 14.2 45.61*	92.3 ± 9.6 19.84*	128.9 ± 15.1 52.15*	98.6 ± 13.6 58.89*
amyl alcohol mg/dm <sup>3</sup>	61.9 ± 8.5	71.0 ± 15.0 88.08*	58.6 ± 7.5 55.68*	69.5 ± 14.2 45.62*	60.5 ± 9.6 19.84*	84.4 ± 15.1 52.15*	71.1 ± 14.6 64.86*
Isobuty alcohol mg/dm <sup>3</sup>	57.85 ± 8.4	44.33 ± 9.3 58.77*	49.2 ± 7.1 49.97*	39.5 ± 6.8 27.70*	56.5 ± 8.4 19.84*	48.0 ± 8.4 31.67*	66.5 ± 8.8 64.84*
n-butan 1-ol mg/dm <sup>3</sup>	4.55 ± 8.4	4.2 ± 9.3 71.38*	3.9 ± 7.1 49.99*	5.1 ± 6.8 45.61*	5.9 ± 8.4 26.54*	6.2 ± 8.4 52.13*	5.2 ± 8.8 64.85*

\* – Calculated in percentage from the initial wine concentration accounting obtained quantities of wines with reduce alcohol and distillates.

Although it has a high evaporation coefficient [24] and in batch distillation it is concentrated in the heads during the low temperature distillation more than 75.0% of it quantity remains in the treated wines. Although the system works entirely in an inert atmosphere and at a temperature not higher than 32 °C, with longer exposure, approximately about 10 and 20% increase in the amount of acetaldehyde is observed, respectively in B and C mode. This still remains a much lower value compared to conventional distillation, where the amount of acetaldehyde in the distillate can rise up to 50%. In the classical batch distillation, an additional amount of esters can also be formed but this is not observed with the ethylacetate contents in any of the treatment regimes. The concentration of ethyl acetate is almost the same in all samples and as an amount, except for run A it is distributed between the dealcoholized wine and the resulting distillate.

Regarding the concentrations of the main higher alcohols, it can be concluded that as a less volatile amyl alcohols pass into the distillate to a lesser extent, and even with long-term dealcoholization (mode C), more than 45.0% of their initial amount is preserved in the wines. In regime A it is even over 80.0%. With the same modes of treatment 27.70% of isobutyl alcohol remains in the dealcoholized wine for mode C and 58.77% for mode A. This can change the ratio between amyl alcohols and isobutyl alcohol, for example, and if this effect is repeated, it can be used as a marker to recognize the dealcoholization method used.

The concentration of hexa-, nona- and decanal which aroma are described as grassy, unripe fruit, hay-like, fatty citrus orange peel remains relatively unchanged in the dealcoholized wines and initial wines regardless of

the mode used (Table 4). Average 80.0% of these aldehydes remain in wine A, 50.0% in wine B and 45.0% in wine C.

**Table 4.** Volatile compounds in the initial and treated wines and in the distillates (2).

Compound	Initial wine	Dealcoholized wines			Distillates		
		A	B	C	A <sub>d</sub>	B <sub>d</sub>	C <sub>d</sub>
n-Hexanal mg/dm <sup>3</sup>	1.02 ± 0.14	1.04 ± 0.20 78.20*	0.92 ± 0.11 53.04*	1.14 ± 0.14 45.38*	0.65 ± 0.08 12.94*	0.86 ± 0.12 32.21*	0.76 ± 0.12 42.02*
n-Nonanal mg/dm <sup>3</sup>	0.042 ± 0.005	0.043 ± 0.007 78.51*	0.036 ± 0.007 49.90*	0.048 ± 0.005 45.59*	0.027 ± 0.003 12.91*	0.036 ± 0.007 32.15*	0.032 ± 0.006 42.27*
n-Decanal mg/dm <sup>3</sup>	0.495 ± 0.09	0.535 ± 0.07 82.90*	0.421 ± 0.07 50.01*	0.556 ± 0.05 45.60*	0.316 ± 0.05 12.96*	0.417 ± 0.06 32.18*	0.371 ± 0.05 42.27*
Isoamyl acetate mg/dm <sup>3</sup>	9.63 ± 1.11	8.98 ± 1.21 71.57*	9.45 ± 1.13 57.73*	5.45 ± 0.87 23.00*	11.07 ± 1.13 23.36*	9.93 ± 0.98 39.42*	11.60 ± 1.09 67.97*
Ethylbutanoate mg/dm <sup>3</sup>	0.214 ± 0.09	0.201 ± 0.03 72.04*	0.182 ± 0.03 50.00*	0.241 ± 0.04 45.72*	0.297 ± 0.04 28.17*	0.283 ± 0.04 50.52*	0.246 ± 0.04 64.83*
Ethyl hexanoate mg/dm <sup>3</sup>	0.321 ± 0.09	0.291 ± 0.06 69.53*	0.273 ± 0.03 50.01*	0.321 ± 0.06 40.60*	0.446 ± 0.07 28.20*	0.425 ± 0.07 50.58*	0.340 ± 0.06 59.74*
Ethyl octanoate mg/dm <sup>3</sup>	0.028 ± 0.004	0.027 ± 0.005 72.38*	0.021 ± 0.005 43.69*	0.025 ± 0.005 35.88*	0.036 ± 0.005 26.02*	0.042 ± 0.005 55.95*	0.033 ± 0.005 64.94*
Ethyl decanoate mg/dm <sup>3</sup>	0.043 ± 0.005	0.049 ± 0.006 86.07*	0.037 ± 0.005 49.99*	0.042 ± 0.006 39.01*	0.057 ± 0.007 18.80*	0.050 ± 0.007 50.52*	0.043 ± 0.006 64.85*
Phenyl ethanol mg/dm <sup>3</sup>	0.973 ± 0.156	0.894 ± 0.006 70.47*	0.755 ± 0.005 45.63*	1.207 ± 0.006 4202*	1.250 ± 0.007 26.08*	1.388 ± 0.007 54.40*	1.118 ± 0.006 64.80*
Linalool mg/dm <sup>3</sup>	1.064 ± 0.152	0.926 ± 0.147 66.76*	0.662 ± 0.098 36.61*	0.808 ± 0.094 30.83*	1.653 ± 0.216 31.54*	1.723 ± 0.221 61.87*	1.507 ± 0.166 79.88*
Linalool oxide mg/dm <sup>3</sup>	0.117 ± 0.082	0.089 ± 0.064 58.34*	0.073 ± 0.068 36.69*	0.102 ± 0.091 35.39*	0.192 ± 0.018 33.31*	0.187 ± 0.017 61.05*	0.159 ± 0.011 76.65*
a-Terpinolol mg/dm <sup>3</sup>	0.214 ± 0.026	0.186 ± 0.032 66.66*	0.133 ± 0.030 36.54*	0.163 ± 0.024 30.92*	0.348 ± 0.049 33.01*	0.390 ± 0.050 69.62*	0.295 ± 0.041 77.75*
Nerol mg/dm <sup>3</sup>	0.950 ± 0.114	0.827 ± 0.091 66.75*	0.611 ± 0.005 37.84*	0.721 ± 0.073 30.82*	1.545 ± 0.230 33.02*	1.394 ± 0.241 56.07*	1.309 ± 0.183 77.72*

\* – Calculated in percentage from the initial wine concentration accounting obtained quantities of wines with reduce alcohol and distillates.

The concentration of isoamyl acetate is similar in the initial wine, wine A and B but significantly lower in wine C in which remain only 23.0% from the whole quantity. Probably the higher isoamyl acetate volatility and longer evaporation are the reasons this compound to pass in distillate C. Although ethyl esters (ethyl buta-, hexa-, octa- and decanoate) are also highly volatile at the same operation mode (mode C) on average about 40.0% of them are retained in the dealcoholized wine. Due to the concentration of the volume these esters keep a similar concentration in the starting wine and in the three wines with reduced alcohol level. These compounds have pleasant spicy aroma and very low threshold concentration and their persistence is essential for wine aroma. The distribution of phenyl ethanol among the samples is similar to that of ethyl esters.

The terpenes are essential part of aroma not only for the wines but also for the distillates obtained from Misket (Muscat) grape varieties [25]. Usually under the influence of the high temperature during distillation the terpenes undergo changes and the amount of linalool and linalool-oxide usual are increased [26]. In the used nitrogen assisted low-temperature distillation an increase in the amount of linalool and linalool-oxide was observed only with the longest operation mode (regime C) and this increase is up to 10,0% relative to the quantity in the initial wine. Also no significant decrease in the amount of nerol is observed in none of the operating modes.

## 4 Conclusions

The used device for low temperature nitrogen assisted distillation in the used three operation mode give recovery of the most volatiles, dry extract and total phenols. Although the method does not use boiling of the treated wine the difference of the evaporation of some volatiles suggest their different degree of concentration in resulted wines and distillates compare to the ethanol. No loss of desired volatiles and no significant formation of unwanted compounds are observed in the used operation modes. Preconditioning of the used nitrogen and additional working section are our next improvement of the installation.

This work was supported by the project “Strengthening the research excellence and innovation capacity of University of Food Technologies—Plovdiv, through the sustainable development of tailor-made food systems with programmable properties”, part of the European Scientific Networks National Programme funded by the Ministry of Education and Science of the Republic of Bulgaria (agreement № Д01–288/07.10.2020).

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