

## EFFECT OF COLD MACERATION TIME ON ŽILAVKA WINES COMPOSITION UTJECAJ HLADNE MACERACIJE NA KEMIJSKI SASTAV VINA ŽILAVKA

Stanka HERJAVEC<sup>1\*</sup>, Ana JEROMEL<sup>1</sup>, Tihomir PRUSINA<sup>2</sup>, Luna MASLOV<sup>1</sup>

<sup>1</sup>Department of viticulture and enology, Faculty of Agriculture, Svetošimunska 25, 10 000 Zagreb, Croatia

<sup>2</sup>Faculty of Agriculture, Biskupa Čule 10, 88000 Mostar, Bosnia and Herzegovina

Corresponding author: e-mail: herjavec@agr.hr; tel. ++38512393807; fax: ++3851293834

Manuscript received: November 16, 2007; Reviewed: November 7, 2008; Accepted for publication: November 8, 2008

### ABSTRACT

Controlling skin contact conditions is vital to obtain high quality white wines and it has become a standard procedure in many white wine-production areas. The aim of this work was to investigate the changes in composition and sensory properties of Žilavka wines obtained with different cold skin contact time. Results indicate that cold maceration positively influence the quality of Žilavka wines. The results pointed out significant increase in dry extract, ash, total phenol and decrease in tartaric acid and higher alcohol content in Žilavka wines obtained by 10 or 20 hours cold maceration period. Best organoleptic quality of Žilavka wines was obtained by cold maceration at 10 °C during 20 hours period.

Keywords: Cold maceration, Žilavka, higher alcohols, sensory properties

### SAŽETAK

Postupak kontrolirane maceracije grožđa uvelike može utjecati na poboljšanje kvalitete bijelog vina te je u nekim zemljama postao standardni tehnološki postupak u procesu proizvodnje. Cilj ovoga rada bio je istražiti utjecaj različite duljine maceracije na kemijski sastav i senzorna svojstva vina Žilavka. Rezultati ukazuju da je hladna maceracija pozitivno utjecala na kvalitetu vina Žilavka. Kod vina dobivenih hladnom maceracijom utvrđeno je signifikantno povećanje sadržaja suhog ekstrakta, pepela i ukupnih fenola te smanjenje vinske kiseline u odnosu na kontrolu. Najbolju senzorno ocjenjeno je vino Žilavka dobiveno hladnom maceracijom na 10 °C u duljini od 20 sati.

Ključne riječi: Hladna maceracija, Žilavka, viši alkoholi, senzorna svojstva

## DETALJNI SAŽETAK

Postupak kontrolirane maceracije grožđa uvelike može utjecati na poboljšanje kvalitete bijelog vina te je u nekim zemljama postao standardni tehnološki postupak u procesu proizvodnje. U praksi najčešće se koriste klasična (temperature od 20-25 °C) i hladna (temperature od 5-10 °C) maceracija. Klasična maceracija može dovesti do povećane ekstrakcije fenolnih spojeva što je povezano sa izraženijom astringencijom i trpkocim vina. Nasuprot hladna maceracija uvjetuje intenzivniju ekstrakciju aromastih spojeva dok je izdvajanje nepoželjnih fenolnih frakcija svedeno na najmanju moguću mjeru. Cilj ovoga rada bio je istražiti utjecaj različite duljine maceracije na kemijski sastav i senzorna svojstva vina Žilavka. Tretmani su obuhvaćali kontrolnu varijantu, varijantu maceracije na 10 °C u trajanju od 10 sati i maceracije na 10 °C u trajanju od 20 sati. Kemijska analiza dobivenih vina obuhvatila je osnovne kemijske spojeve, organske kiseline, ukupne fenole i više alkohole. Provedena je i senzorna analiza dobivenih vina pomoću metode 100 pozitivnih bodova te metodom redosljeda. Dobiveni rezultati ukazali su na razlike u koncentracijama pojedinih viših alkohola, ukupnih fenola te organskih kiselina. Rezultati senzornog ocjenjivanja pokazali su da je hladna maceracija pozitivno utjecala na kakvoću vina Žilavka te je kao najbolje ocijenjeno vino dobiveno maceracijom na 10 °C u trajanju od 20 sati.

## INTRODUCTION

Skin contact can be defined as a prefermentative process applied to wine elaboration: the skin of crushed and destemmed white grapes are macerated in their own juice at controlled conditions (time and temperature). Controlling skin contact conditions is vital to obtain high quality white wines [4]. In this sense, maceration has been widely investigated and it has even become a standard procedure in many white wine-production areas [16]. In practice, the most frequently applied are "classic" (skin contact at 20 – 25 °C) and cold (skin contact at 5 – 10 °C) maceration treatments [9, 17]. Classic maceration can increase extraction of phenolic compounds connected with increase wine astringency and bitterness [16, 19]. On the contrary, cold maceration leads to increased extraction of aromatic compounds from berry skin cells while undesirable additional extraction of phenolic fraction is reduced to the highest possible degree [5, 7]. The low maceration temperature inhibit the activity of oxidative enzymes, what is of crucial importance since cold maceration treatment can be performed without the addition of sulphur dioxide which increases solubility, and as a consequence, extraction of undesirable phenols

from the berry [7, 19]. In white wine studies, increased pH, color and phenolic compounds and decreased titrable acidity were reported to result from longer skin contact times. Lateryon [11] examined compositional differences in Chardonnay must and wine produced following skin contact for six hours at 15,5 °C, 20 °C and 24 °C. She found lower acidity, higher pH, higher total nitrogen, deeper color and higher phenolic levels with increased skin contact temperature. The wine aroma depends on many factors, such as climate, grape variety, yeast and wine-making techniques. In white wine-making, skin contact during must preparation is a pre-fermentative process for improving fruity and flowery attributes of wines but in some cases may cause more astringent and bitter taste [16]. In several reports quality was inferior in wines of pomace contact longer than 12 hours. Singleton and co-workers [19] concluded that fruitiness and general quality were generally harmed by appreciable skin contact. Ramey et al. [16] found that the skin maceration of Chardonnay significantly improves aroma quality and wine structure without increasing bitterness and astringency. On the contrary, Test et al. [21] found no significant increase in fruity aroma of Chardonnay wine due to the skin contact. The aim of this work was to investigate the changes in wine composition and sensory properties of Žilavka wine which would arise due to different cold skin contact time at skin contact temperature of 10 °C.

## MATERIALS AND METHODS

Žilavka white wine grapes from the wine region of Herzegovina, vineyard Blizanci, were harvested during 2006 season, destemmed and crushed. Must obtained by separation of liquid fraction from solid cluster parts right after grape crashing [without maceration] was used as a first, control treatment. Second treatment was cold maceration at 10 °C in duration of 10 hours while third treatment was cold maceration at 10 °C in duration of 20 hours. Each of mentioned treatments was performed in 3 repetitions. Must from control treatment was treated with 80 mg/L SO<sub>2</sub> and sediment for 24 hours at 12°C. Grape mashes cold maceration treatments were treated with 80 mg/L SO<sub>2</sub> after the maceration process finished (right before pressing). Alcoholic fermentation of all treatments was performed in controlled temperature conditions of 18°C with the addition of selected wine yeast Uvaferm CEG. Two racking were carried out to clarify the wines before bottling. The samples of all treatments were chemically analysed just after second racking and two months afterwards tested by sensorial evaluation. The common analyses of basic wine components were analyzed by O.I.V. methods [13]. Total phenols were determined spectrophotometrically with Folin

– Ciocalteu reagent [6] following the method of Slinkard and Singleton [20] using UV/VIS spectrophotometer at wavelength of 280 nm. Organic acids were determined by HPLC method [23]. The wines were subjected to sensory evaluation by the 100-point O.I.V. / U.I.O.E method, and by ranking method with a panel of 7 judges. The determination of statistical significance was done according to [1]. One-way analysis of variance and Least Significant Difference (LSD) comparison test were used to statistically interpret mean differences in mean values if any, at 95 % and 99% accuracy level.

## RESULTS AND DISCUSSION

Results in Table 1,2 confirm previous study by [8] that skin maceration resulted in a decrease of total acidity and an increase in pH values in both musts and wines. According to [17] and numerous other authors these changes are linked to the liberation of potassium from the skins and the resulting partial salification of tartaric acid what is also confirmed in our study where only tartaric acid concentration in the both cold maceration treatments was significantly lower compared to the control treatment. The majority of phenolic compounds in wine originates from the grapes (mostly from seed, skin and stems, while less from juice), and only a small part is produced as a yeasts metabolism product (volatile phenols). Consequently, maceration duration and temperature can significantly influence the final total phenol concentration in obtained wines [15]. Results presented in Table 2. show that control treatment wines have the lowest total phenol concentrations and, moreover, that concentration in wines obtained by cold maceration is increasing proportionally to maceration time what is in accordance to literature data [7, 9]. Significant increase in dry extract and ash concentrations in both cold maceration treatment wines was noted compared to control wine. Between cold maceration treatment longer skin contact time significantly influenced the dry extract

and total phenol concentrations (table 2). Succinic acid is only present in trace quantities in the berries of *Vitis vinifera* cultivars. However, succinic acid is a normal by-product of alcoholic fermentation. [10]. Coulter et al. [3] indicated that succinic acid participates in the vinosity of wine and gives fermented drinks the special taste they all have in common. Among the factors that can influence the production of succinic acid are yeast strain, fermentation temperature, aeration, must clarity and composition (sugar concentration, nutrient content, pH, titratable acidity),[3]. Even thou skin contact treatment have influence on must composition our result did not show any changes in succinic acid concentration between tested wines what is in accordance with previous reports [21]. Contrary to them Palomo et al. [14] reported that skin contact wines had significantly higher concentrations of succinic acid compared to control wine. Higher alcohol data are given in table 3. They are principally yeast-produced compounds rather than grape compounds, but would be expected to vary according to initial must composition. According to [9] higher alcohol concentration below 300 mg/l contribute to desirable aroma complexity of wine, but when these concentrations exceed 400 mg/l, these compounds are regarded as a negative quality factor. As shown in table 3 all tested wines had relatively high concentration of total higher alcohols but also there was a significant difference between them. Wines made with cold maceration at 10 °C for 20 hours contained significantly the lowest amount of total higher alcohols compared to other treatments. There was no significant change in 1-propanol concentration, three other higher alcohols decreased at longer skin contact time, while methanol displayed a quite linear increase. This significant methanol increase is undoubtedly due to increase activity of natural pectin methylesterase connected with skin contact duration. Results by [14] showed that Albillo wines made from skin macerated must also had higher concentrations of

**Table 1:** Chemical composition of Žilavka must  
**Tablica 1:** Kemijski sastav mošta Žilavke

	Control treatment Kontrolna varijanta	Cold maceration 10 °C/10 hours Hladna maceracija 10 °C/10 sati	Cold maceration 10 °C/20 hours Hladna maceracija 10 °C/20 sati
Sugar (g/l) Šećer	218	215	215
Total acidity (g/l)* Ukupna kiselost	6,5	6,2	6,2
pH	3,1	3,2	3,2

\*as tartaric acid, kao vinska kiselina

**Table 2:** Mean values of basic chemical composition and total phenols concentrations of Žilavka wines  
**Tablica 2:** Srednje vrijednosti osnovnog kemijskog sastava i koncentracije ukupnih fenola vina Žilavka

Compounds Sastojak	Control treatment Kontrolna varijanta	Cold maceration 10 °C/10 hours Hladna maceracija 10 °C/10 sati	Cold maceration 10 °C/20 hours Hladna maceracija 10 °C/20 sati	LSD
Alcohol (vol%) Alkohol	13,2	13,1	13,1	n.s.
Residual sugar (g/l) Residualni šećer	1,4	2,0	2,0	n.s.
Dry extract (g/l) Suhi ekstrakt	17,6 <sup>Aa</sup>	19,8 <sup>Bb</sup>	20,4 <sup>Cc</sup>	5%=0,11 1%=0,16
Total acidity (g/l)* Ukupna kiselost	5,7 <sup>Aa</sup>	5,5 <sup>Bb</sup>	5,5 <sup>Bb</sup>	5%=0,08 1%=0,12
Tartaric acid (g/l) Vinska kiselina	2,2 <sup>Aa</sup>	1,7 <sup>Bb</sup>	1,6 <sup>Bb</sup>	5%=0,22 1%=0,41
Malic acid (g/l) Jabučna kiselina	2,5	2,4	2,4	n.s.
Citric acid (g/l) Limunska kiselina	0,27	0,30	0,30	n.s.
Lactic acid (g/l) Mliječna kiselina	0,1	0,1	0,1	n.s.
Succinic acid (g/l) Jantarna kiselina	0,5	0,5	0,5	n.s.
Volatile acidity (g/l)** Hlapiva kiselost	0,36	0,40	0,39	n.s.
pH	3,2	3,3	3,3	n.s.
Ash (g/l) Pepeo	1,9 <sup>Aa</sup>	2,1 <sup>Bb</sup>	2,2 <sup>Cb</sup>	5%=0,09 1%=0,13
Total phenols (mg/l) Ukupni fenoli	289,14 <sup>Aa</sup>	316,13 <sup>Bb</sup>	365,11 <sup>Cc</sup>	5%=17,32 1%=24,29

Note: Different letters beside the mean of a compound denote a significant difference among treatments (A, B, C for 5%; a, b, c for 1%).

Naznaka: Različita slova pored sastojka označavaju signifikantnu različitost (A,B,C za 5%, a,b,c, za 1%).

n.s.: not significant /nesignifikantno, as tartaric acid /kao vinska kiselina, \*\*as acetic acid/kao octena kiselina

methanol than a control wines, since methanol is derived from demethylation of skin pectins. The concentration of higher alcohols in Albillo wines tested generally declined with skin-contact time, probably as a result of blockage of the Ehrlich mechanism-the main pathway for the formation of these compounds-due to increased levels of nitrogenous substances in musts what is in accordance with results presented in our work. Ramey et al. [16] and Herjavec et al. [8]. also found decrease in some higher alcohol concentrations due to skin contact treatment. At the contrary Baumes et al. [2] noted significant increase of isoamyl alcohol in Chenin macerated wines and investigation carried by [18] showed significant increase of isoamyl alcohol, 2-phenylethanol and isobutanol connected with skin contact process. Acetaldehyde is one of the most important sensory carbonyl compounds formed during vinification and mainly originates from

yeast metabolism during alcoholic fermentation [12]. Cold maceration had no effect on the concentration of this compound what is confirmed in work published by [18]. Contrary results are shown by [14] where significant decrease in acetaldehyde concentration was noted due to longer skin contact time. Esters are one of the major components of wine aroma, providing delicate odors and ethyl acetate is the most abundant one occurring in wine with concentrations between 50 and 200 mg/l. Concentrations of ethyl acetate contribute significantly to the volatile character of „acetic nose“ and levels of 150 to 200 mg/l impart spoilage character to wine. But in very low concentrations (50-80 mg/l) ethyl acetate contributes to the olfactory complexity and has a significant influence on the wine quality [17]. Ethyl acetate concentration in all tested wines was very low and there was no significant differences noted. By contrast results published by [14]

Table 4: Sensory evaluation of Žilavka wine by 100 point method (O.I.V./U.I.O.E)  
 Tablica 4: Senzorna ocjena vina Žilavka metodom 100 pozitivnih bodova(O.I.V./U.I.O.E)

	Treatments/Tretmani		
	Control treatment Kontrolna varijanta	Cold maceration 10 °C/10 hours Hladna maceracija 10 °C/10 sati	Cold maceration 10 °C/20 hours Hladna maceracija 10 °C/20 sati
Total score Ukupan zbroj	82,0	81,2	84,4

Table 5: Sensory evaluation of Žilavka wine by ranking method (odor quality)  
 Tablica 5: Senzorna ocjena vina Žilavka metodom redoslijeda (kakvoća mirisa)

Treatments/Tretmani	Order/redoslijed	Rank total/Ukupni zbroj
Cold maceration 10 °C/20 hours Hladna maceracija 10 °C/20 sati	1	7**
Control Kontrola	2	17
Cold maceration 10 °C/10 hours Hladna maceracija 10 °C/10 sati	3	18

Note: any rank total outside 10-18 range is significant at the P < 5 %; 8-20 at P < 1 %.

Naznaka: svaka vrijednost izvan ranga 10-18 je signifikantna na nivou 5%, 8-20 na nivou 1%

Table 6: Sensory evaluation of Žilavka wine by ranking method (taste quality)  
 Tablica 6: Senzorna ocjena vina Žilavka metodom redoslijeda (kakvoća okusa)

Treatments /Tretmani	Order /Redoslijed	Rank total/Ukupni zbroj
Cold maceration 10 °C/20 hours Hladna maceracija 10 °C/20 sati	1	8*
Control Kontrola	2	16
Cold maceration 10 °C/10 hours Hladna maceracija 10 °C/10 sati	3	18

Note: any rank total outside 10-18 range is significant at the P < 5 %; 8-20 at P < 1 %.

Naznaka: svaka vrijednost izvan ranga 10-18 je signifikantna na nivou 5%, 8-20 na nivou 1%

and partly by [8] showed significant decline of ethyl acetate by skin-contact treatment.

Sensory evaluation by the ranking method and the 100 point method shown, that significantly the best general quality had the wines obtained by cold maceration at 10 °C during 20 hours time period. These wines were characterized by the more pronounced varietal flavours (odor quality), and intensity and complexity of the taste. This is probably connected with increase mouthfeel/palate fullness likely due to increased phenol and polysaccharides concentrations [22] and lower amount of higher alcohols. However, it is important to notice that cold maceration during 10 hours time period didn't have much effect on Žilavka wines what is shown in Tables 4,5,6.

## CONCLUSION

Results of this study indicate that cold maceration positively influence the quality of Žilavka wines and confirmed the compatibility of on tested treatments with this grape variety. The results pointed out significant increase in dry extract, ash and total phenol and decrease in tartaric acid content in Žilavka wines obtained by 10 or 20 hours cold maceration treatment period. Best organoleptic quality of Žilavka wines was obtained by cold maceration at 10 °C during 20 hours time period.

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