# The Influence of Maceration on the Composition of Some Volatile Compounds and Sensory Properties of Traminer Wines

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## SUMMARY

Traminer wines produced by usual technology for white wines and by maceration for 3 hours with and without pectolytic enzyme addition were investigated for the differences in chemical composition and sensory properties.

Chemical analyses and sensory testing were performed immediately after fermentation and twelve months after bottle aging. The analyses of higher alcohols and esters were done by gas chromatography. The paired sample test and ranking method were used for sensory evaluation. The wines made from grapes macerated with pectolytic enzymes contained the highest concentration of ethyl acetate, acetates of higher alcohols and fatty acids ethyl esters. At the same time the lowest total higher alcohol concentration with exception of 1-propanol was established in the above metioned wines. There was no difference in the concentration of ethyl lactate and diethyl succinate among the wines of all treatments.

After one year period of bottle aging no changes were noticed in the concentration of higher alcohols. The decrease of the acetates of higher alcohols, ethyl esters of fatty acids were noticed in all observed wines while the concentration of ethyl lactate and diethyl succinate significantly increased.

The best evaluated were the wines produced from pectolytic enzymes macerated grapes, while the others were of inferior quality.

#### **KEY WORDS**

pectolytic enzymes, sensory properties, skin contact, volatile compounds

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## Utjecaj maceracije na tvorbu nekih hlapivih komponenti i senzorna svojstva vina Traminac

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## SAŽETAK

Vina Traminca proizvedena uobičajenom tehnologijom za bijela vina te maceracijom od 3 sata sa i bez dodatka enzima analizirana su kako bi se utvrdile razlike u kemijskom sastavu i senzornim svojstvima. Kemijska analiza i senzorno ocjenjivanje provedeni su odmah nakon fermentacije te nakon 12 mjeseci starenja vina u boci. Analiza viših alkohola i estera rađena je plinskom kromatografijom, a vina su senzorno ocijenjena paired sample testom i metodom redoslijeda.

Vina dobivena maceracijom grožđa uz dodatak pektolitičkog enzima sadržavala su najviše koncentracije etil acetata, acetata viših alkohola i etil estera masnih kiselina te najniže koncentracije ukupnih viših alkohola. Izuzetak u tim vinima bio je sadržaj 1-propanola. Razlike u koncentracijama etil laktata i dietil sukcinata između vina svih tretmana nisu postojale.

Nakon starenja vina u boci nisu utvrđene promjene u koncentracijama viših alkohola, dok je zabilježen pad koncentracija acetata viših alkohola i etil estera masnih kiselina a povišenje koncentracija etil laktata i dietil sukcinata.

Najbolje ocijenjena bila su vina dobivena maceracijom uz dodatak pektolitičkog enzima, dok su vina proizvedena bez maceracije bila slabije kvalitete.

KLJUČNE RIJEČI

hlapive komponente, maceracija, pektolitički enzimi, senzorna svojstva

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## INTRODUCTION

Many volatile compounds are present in wine aroma. They can be influenced by several factors such as fermentation temperature, must, yeast strains, vinification technology. Skin contact or maceration of crushed white grapes prior to pressing has become standard procedure in many white wine-producing areas, generally out of belief that flavor components are being extracted from the skins (Ramey et al. 1986). In the past few decades, increasing interest has been shown in the application of enzymes in wine-making. Enzymatic treatments of must and wine have multiple purposes, since they influence wine clarification and filtration, juice yield, color and aroma extraction. Endogenous enzymes of grapes and microorganisms are often neither efficient nor sufficient under wine-making conditions. Nowdays enzymes from exogenous sources, usually with additional glucosidases activity, have been recognized as useful processing aids, because they can reinforce or replace the grape enzymatic activities (Canal-Llauberes, 1993, Fischer 2001). Skin maceration makes the most of the aromatic potential of the grapes and in general it significantly enhances varietal aroma without increasing herbaceuous flavors (Ribereau-Gayon et al. 2000). The greatest potential for the manipulation of juice flavor lies in the treatment of the terpene based cultivars (Gewürztraminer) by commercial enzyme preparation (Binder 1999). In white wine studies, increased pH, color and phenolic compounds and decreased titrable acidity were reported to result from longer skin contact times (Ough, 1969, Ough and Berg, 1971). In several reports quality was inferior in wines of pomace contact longer than 12 hours (Ough, 1969, Ough and Berg, 1971). Singleton and co-workers (1980) concluded that fruitiness and general quality were generally harmed by appreciable skin contact. Arnold and Noble (1979) 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. (1986) found no significant increase in fruity aroma of Chardonnay wine due to the skin contact. Comparing the wines made with and without skin contact Baumes et al. (1989) noticed the significant increase of most volatile compounds in the skin contact wines. Herjavec (1980) reported that skin contact with enzyme addition can cause intensification of Malvazija wine aroma compared to the control wines. The contemporary investigations regarding skin contact are focused mostly on differences in aroma compounds, especially terpene contents. The objective of this work was to investigate the influence of skin contact with pectolityc enzyme addition on the concentration of some fermentation's aroma compounds as well as final wine quality.

## MATERIAL AND METHODS

### Winemaking

Traminer (syn. Gewürztraminer) grapes used in this experiment were grown in wine region of continental Croatia. Harvested grapes were divided into three lots, destemmed and crushed according to the following treatments. The first lot of grapes (control) was pressed immediately after crushing. The second treatment was represented by three hours skin contact while the third one included three hours skin contact with addition of 4 g/100 L pectolytic enzyme (trade mark Endozym Cultivar by AEB - Italy). The temperature was maintained at 16°C during skin contact. Combined free-run and press juices (by pressing to two bars in 150 L basket press) was settled with SO<sub>2</sub> in concentration of 75 mg/L and racked after 27 hours. The musts were inoculated with selected yeasts S. cerevisiae. The temperature during the fermentation did not exceed 18 °C. After completion of alcoholic fermentation, the wines were sulfited with 50 mg/L of SO<sub>2</sub>. All treatments were done in triplicate. Two rackings were carried out to clarify the wines before bottling. Bottles were stored in a cellar at 15 °C. When analyzed, the wines were aged for 1 and 12 months, respectively.

## Chemical analyses

Ethanol, total and volatile acidity were determinate using methods proposed by O. I. V. (1995). Reducing sugar was determined volumetrically by Lane Eynon method (Amerine and Ough, 1974) and pH was measured on the Beckman Expandomatic SS 2 pHmeter.

Volatile compounds analysis was done on gas chromatograph (Hewlet Packard 5890) using FID. Analysis of higher alcohols (1-propanol, isobutanol, isoamyl alcohol and ethyl acetate ) were performed from wine distillate. Temperature programming was as follows: HP 101 Column, 6 min isothermal at 40°C, then linear temperature rise of 15 °C min<sup>-1</sup> to 200 °C.

Analysis of volatile esters, hexanol, and 2-phenyl ethanol were performed from volatile extracts. Wine (500 mL) was taken in a liquid-liquid upward displacement apparatus and extracted for 10 hours with dichloromethan. The extract was dried over anhydrous sodium sulfate, concentrated to 10 mL and stored prior to GC analysis. Temperature programming was: HP FFAP Column, 5 min isothermal at 60 °C, then linear temperature rise of 2.5 °C min<sup>-1</sup> to 190 °C and 20 min isothermal at 190°C. Determination of volatile compounds was done by method of internal standards.

#### Sensory evaluation

Sensory evaluation of wines was carried out by paired sample test as well as by ranking method in two tasting periods: one month after alcoholic fermentation with the panel of 8 judges and twelve months after bottle aging with panel of 7 judges. The determination of statistical significance was done according to Amerine and Roessler 1976.

## **RESULTS AND DISCUSSION**

Skin maceration resulted in a decrease of total acidity and an increase in pH values in both musts and wines. According to Ribereau-Gayon et al. (2000) and numerous other authors these changes are linked to the liberation of potassium from the skins and the resulting partial salification of tartaric acid.

so obvious. Contrary to our results, the same author established also an increase of hexanol in white wines made from macerated grapes. Ramey et al. (1986) found that hexanol concentration decreased with increase of skin contact temperature. Investigations carried out by Herjavec (1980) in Malvazija wines showed that 3 hours skin contact with pectolytic enzyme can cause a lower increase of 1-propanol, which is similar to the presented results.

#### Volatile esters.

One month after alcoholic fermentation significant differences were established in the concentration of acetates among all wines. The wines produced by skin contact with pectolytic enzyme contained significantly the highest concentration of ethyl acetate and acetates of higher alcohols. Comparing

#### Table 1. Chemical composition of Traminer wine one months after fermentation

Compounds	Treatments						
	No skin contact		Skin contact		Skin contact with enzyme		
	Must	Wine	Must	Wine	Must	Wine	
Alcohol %		11.61		11.64		11.91	
Reducing sugars g/L	200	<1.0	202	<1.0	205	<1.0	
Total acidity g/L*	9.84	8.4	8.65	7.7	8.37	7.6	
Volatile acidity g/L**		0.42		0.37		0.39	
рН	3.00	3.10	3.18	3.30	3.20	3.39	

\* as tartaric acid; \*\* as acetic acid

#### Higher alcohols

The results in table 2 show that one month after alcoholic fermentation there were significant differences in higher alcohols among the wines of all treatments . The wines made from the grapes macerated with pectolytic enzyme contained significantly the lowest concentration of isoamyl alcohol, isobutanol and 2-phenyl ethanol. Contrary to this, the concentration of 1-propanol in these wines was significantly higher. There were no changes in concentration of higher alcohols after 12 months period of bottle aging.

We suppose that the lowest concentration of total higher alcohols in the wines made from grapes macerated with enzyme addition, was primarily due to the better clarification of these juices. Klingshirn (1987) concluded that the wines produced of very clear musts contained lower concentrations of isobutanol and isoamyl alcohol. There is a very few data in the literature about the influence of skin contact with enzyme addition on the level of higher alcohols. Baumes et al. (1989) examined volatile compounds in wines produced by skin contact without enzyme addition. They noted significant increase of isoamyl alcohol in Chenin and Bourboulenc macerated wines while in the Chardonnay wines this difference was not to the control, the amount of isoamyl acetate was 70% higher and of 2-phenyl ethyl acetate for 56 %, respectively. Isobuthyl acetate was detected only in the wines produced of the grapes macerated with enzymes. The highest concentration of ethyl esters of fatty acids were detected in the same wines one month after fermentation. There were no significant differences among the wines in the amount of diethyl succinate and ethyl lactate. Generally speaking, the concentration of acetates and ethyl esters of fatty acids decreased in all wines after bottle aging. The most intensive decrease was established by phenyl ethyl acetate and ethyl caprylate. Diethyl succinate and ethyl lactate increased in all wines after one year of bottle aging. Literature data concerning pectolytic enzyme influence on ester concentration changes in wines are not numerous. The greatest part of them discuss the influence of skin contact time and maceration temperature on methanol levels etc. Rapp et al. (1985) have found in white wines no direct influence of skin contact on the level of esters. The same authors concluded that a longer time of skin contact can cause decrease in the concentrations of acetates and ethyl esters of fatty acids. Rapp et al. (1985), Garofolo and Piracci (1994) observed that acetates of higher alcohols and ethyl esters of fatty acids are hydrolyzed during aging and concentrations

Compounds			LDS		
-	_	No skin contact	Skin contact	Skin contact with enzyme	
Higher alcohols					
1-Propanol	1	19.7a	12.0b	26.7c	5% 0.06, 1% 0.09
	2	19.0a	12.3b	25.3c	5% 0.19, 1% 0.28
Isobutanol	1	39.0a	37.5a	32.7b	5% 1.82, 1% 2.22
	2	37.7a	35.3a	31.7b	5% 0.15, 1% 0.22
Isoamyl alcohol	1	277a	264b	218b	5% 2.45, 1% 3.62
-	2	281a	263b	216c	5% 2.14, 1% 3.16
Hexanol	1	0.66a	0.62a	0.52b	5% 0.06, 1% 0.09
	2	0.59a	0.47b	0.53ab	5% 0.07, 1% 0.10
2-Phenyl ethanol	1	38.7a	40.7a	28.0b	5% 2.15, 1% 3.17
	2	38.0a	39.7a	28.3b	5% 2.18, 1% 3.22
$\Sigma$ Higher alcohols	1	375.06	350.47	303.23	,
0	2	376.29	350.77	301.83	
Esters					
Ethyl acetate	1	31a	29a	43b	5% 2.34, 1% 3.45
	2	42A	43A	38B	5% 3.49, 1% 5.15
Isoamyl acetate	1	1.02a	0.66b	3.40c	5% 0.16, 1% 0.24
	2	0.18a	0.13a	0.61b	5% 0.19, 1% 0.28
Isobutyl acetate	1	nd	nd	0.08	
	2	nd	nd	nd	
Phenyl ethyl acetate	1	0.10a	0.06a	0.23b	5% 0.06, 1% 0.09
	2	0.05a	0.02a	0.04a	n.s., n.s.
$\Sigma$ Acetates of higher alcohols	1	1.12	0.72	3.71	,
0	2	0.23	0.15	0.65	
Ethyl butyrate	1	0.37a	0.26b	0.56c	5% 0.06, 1% 0.09
, ,	2	0.13A	0.10A	0.20B	5% 0.10, 1% 0.14
Ethyl caproate	1	0.25a	0.23a	0.56b	5% 0.18, 1% 0.27
7 1	2	0.21Aab	0.17Aa	0.41Bb	5% 0.19, 1% 0.28
Ethyl caprylate	1	0.43a	0.36a	0.87b	5% 0.12, 1% 0.18
	2	0.29a	0.24a	0.55b	5% 0.10, 1% 0.14
$\Sigma$ Ethyl esters of fatty acids	1	1.05	0.85	1.99	<u>,</u> ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,
,,	2	0.63	0.51	1.16	
Ethyl lactate	1	3.1a	2.3a	3.2a	n.s., n.s.
,	1	11.7a	7.1b	12.8a	5% 1.24, 1% 1.83
Diethyl succinate	1	0.19a	0.15a	0.14a	n.s., n.s.
	2	1.7a	1.9a	1.1b	5% 0.23, 1% 0.34

Table 2. Concentration of volatile compounds in Traminer wines in mg/L during period of analysis

1 = one months after alcoholic fermentation; 2 = after 12 months bottle aging

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

tend to decrease. On the other hand, these authors established that concentration of ethyl lactate and diethyl succinate increase throughout aging what is in accordance with our results.

## Sensory properties of wines

Results of wine tasting by paired sample test are given in table 3 and by ranking method in table 4.

In this test the judge is presented with two samples and asked to identify the one with greater aroma intensity. Results presented in table 3 indicates that Traminer wines made by skin contact with pectolytic enzyme one month after fermentation had significantly the best aroma intensity . The differences among wines produced by skin maceration with and without enzyme were not significant.

On the basis of the presented data, in both tasting periods, significantly the best general quality had the wines made from grapes with skin contact and enzyme addition. One months after fermentation the wines made by skin contact with enzyme could be distinguished by their fruitiness and recognizable varietal aroma. The more pronounced fruitness and recognizable varietal aroma could be related with the lower concentration of higher alcohols on the one hand and with increase of acetates and ethyl esters of

Judges	Treatments							
	No skin contact	Skin contact	No skin contact	Skin contact with enzyme	Skin contact	Skin contact with enzyme		
1		+		+		+		
2		+		+				
3	+			+				
4		+		+				
5		+		+				
6		+		+		+		
7		+		+		+		
8	+			+	+			
Total	2	6		8**	1	7		

#### Table 3. Results of sensory evaluation of Traminer wine by paired sample test

\*\* = significant at p < 0.01

Table 4 Results of sensory evaluation of Traminer wine by ranking method

Treatment	1 month afte	er fermentation	12 months after bottle aging		
	Order	Rank total	Order	Rank total	
Skin contact with enzyme	1	9**	1	7**	
No skin contact	2	17	2	10	
Skin contact	3	22	3	17	

1 month after fermentation: any rank total outside 10-22 range is significant at the P<1 %

12 months after bottle aging: any rank total outside 8-20 range is significant at the P< 1 %

fatty acids on the other. More typical varietal property of pectolytic enzymes made Gewürztraminer wines can be connected with ß-glucosidase activity of commercial enzymes we used. Witowski (2001) also reported positive influence of ß-glucosidase on Gewürztraminer aroma. The fruity character and freshness have lost some of their intensity after a year's storage while the varietal aroma was still good and very well pronounced. Christmann and Papargiriou (2000) did not get changes in aroma intensity of wines made after 16 and 40 hours maceration time. Contrary to them, Fischer (2001) reported that skin contact and enzyme addition leads to intensification of typical floral and fruity Gewürztraminer aroma. Schneider (1998) stated that maceration improves stronger extraction of aroma components from grape berries and that during aging period hydrolyzes of fixed compounds liberates volatile components that can participated in formation of aroma profile.

## CONCLUSION

The current study shows positive relation between use of pectolytic enzyme and Gewürztraminer wine quality. The lower concentration of higher alcohols, higher quantities of volatile esters, improvements of aroma intensity and general wine quality in wines made by skin contact with enzyme addition were observed.

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