

Maceration affects mineral composition and pH of wines

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Summary: Mineral composition of wines is affected by many ecological and technological factors. These variables are mostly discussed focusing on certification of origin and human health. This paper gives data on possible changes in mineral composition and pH of wines due to 4 hours skin maceration (1st trial) and fermentation sur marc (2nd trial). Experimental site is situated on acidic sandy soil in middle east Hungary. The variety collection was established in 2003 with own rooted planting material in 3x1 m spacing, trained for single curtain training system. Mineral composition of wines was determined with ThermoFischer Scientific iCAP 6300 ICP-OES, pH was measured with pH10pen (VWR International) in field, and with SevenEasy™ pH meter (Mettler Toledo) in laboratory. 1st trial with 9 PIWI white wine grape varieties pointed to demonstrate effects of skin maceration, which is abundantly used to elevate aroma content. 2nd trial with the use of ‘Medina’ red PIWI grape variety aimed to demonstrate effects of double pasta skin maceration in rose and red wine technology. Data of 1st trial show, that K, Cu, Mn and P respective increase with skin maceration, despite Fe show considerable decrease. Data of 2nd trial show, that K, Mg, Mn, P, Sr and B increase with longer skin contact and higher fermentation temperature, despite Fe and Ba decrease with this technology. In regard to pH, data show, that skin maceration and fermentation sur marc increase K content with about 30-70% respectively, withstanding that pH also increase with a considerable 0,4-0,5 value. K content can be higher in the end product, despite to earlier higher level of tartrate formation. An attention should be driven to lower level of tartaric acid and consequently higher pH characteristic for wines produced with the application of skin maceration or fermentation sur marc.

Keywords: wine, maceration, mineral composition, pH

Introduction

Mineral composition of wines is mostly taken in point of view in connection with terroir, certification of geographical origin and limits of heavy metals. Effects of production technology and wine technology is seldom discussed (Pohl, 2007). This work aims to give some basic data to facilitate producers to have an approximate information on possible effects of skin maceration (1st trial) and fermentation sur marc (2nd trial) on pH and changes in mineral composition of wine. Tartrate formation in wines is basically in correlation with potassium and acidic content of the wine. Most of this precipitation takes place under alcoholic fermentation, and besides acidic and potassium content, is highly affected by clearness on the wine, alcohol content and temperature. Mechanism between levels of mineral and acidic content, tartaric formation and pH is a very important technological question. This topic in Hungary was broadly studied by Ferenczi (1966), but he could not publicate a concise work on this complex mechanism.

Critical changes in pH are seldom emphasized despite its critical significance. Focus should be primarily driven to K content, tartrate formation and pH. Demonstrated double marc fermentation technology is abundantly used to produce two distinct quality product with the use of one harvested item.

Optimum levels on mineral take up of grape in Hungary was published by (Szőke, 1997; Eifert et al., 1985) on bases of leaf sample analyses of long term field trials. Optimum levels of mineral take up can be correctly evaluated only when corresponding data on biological production, quality and quantity are given.

Mineral composition of wines is affected by geological origin of the territory, biological bases both rootstock and scion and technological elements including plant protection (Füzi and Holb, 2007; Csikászné, 2008; Kocsis, 2010; Taksonyi et al., 2010; Holb and Füzi, 2015), climatic factors, natural and antropogenic pollution (Eschnauer, 1982; Murányi, 2005), processing of the grape, skin maceration, lies, time of decantation, clarification with bentonite, barrels (Pohl, 2007). It is clear to see, that bottled wine could be comprehended the most contaminated sample in characterisation of a terroir (soil, sub soil, technology/-ies, vintage, cultivar, cultural background).

Eschnauer (1988, 1998) based on quantity categorises elements in wine in following groups. Main elements: 10-1000 mg/l, trace elements: 1 mg/l or below; ultra-trace elements: 1 µg/l or below (ppb range). Murányi (2005) ads, that range of 1-10 mg/l is missing, for the sake of Na, Mn and Fe. Eschnauer (1988, 1998) aims to clarify, that source of the elements in wine is very important, for example when geographical origin or effects of terroir is discussed.

Evaluation of technological trials, like fertigation, foliar and/or soil application of mineral nutrition/ manure also presuppose definition of possible sources. It is hard to distinguish even in case of incorporated element content whether it comes from the sub soil or from atmospheric pollution.

Eschnauer (1988, 1998) distinguishes primary element content of the must/ wine coming from the grape. That, taken up by the plant, mostly affected by the soil, manure and plant protection. In optimal case mostly this fraction forms the total metallic content of the wine, however it is not exclusive, and it is very difficult to define. Secondary concentration is not in relation with natural uptake, and could be divided in two groups: 1st group is that of natural sources, like emissions of sea sides and volcanic activities; 2nd group is of anthropogenic sources, either of direct or indirect activities, like industrial production (melting industry, motorways), technological measures (grape production technology, wine technology) or wine adulteration.

Murányi (2005) summarises mostly important factors affecting metallic content of wines as follows: a) growing site (soil and pollutions of the surrounding territory); b) elements of applied growing and wine making technology, of which sur marc maceration of red wines increase metallic content of the wine (Würdig et al., 1989), clarification can increase or decrease concentration of certain metals (Postel et al, 1987; Gorinstein, 1984); c) vintage: it is well known, that due to confined mineral take up of the plant in arid years, metallic content of the wines will be consequently lower (Würdig et al., 1989) STB).

Potassium in wines can reach 370-1160 mg/l Eschnauer, 1988; Zee et al. 1983; Dyer-Ansher, 1977). In Hungary 550-1800 mg/l (Diófási et al., 1983; Szitha et al., 1985) was measured. Mostly determined by the amount coming from the grape, slightly affected by lies/yeast and formation of potassium hydrogen tartrate ($K_2C_4H_4O_6$) in fermentation (Würdig et al., 1989; Eperjesi et al., 1998; Frenzy, 1966) up to 5-20% compared to the must. Interesting to notice that blue fining, and use of potassium disulphite in technology can increase, while at use of bentonite (+20 mg/l) (Postel et al, 1987) and perlite (+60-90 mg/l) increase was detected (Postel et al., 1986). Excessive use of potassium in plant nutrition can be a reason for its higher level in wine, however this could entail other plant physiological disorders like bunch stem necrosis by defecting K/Mg-ratio. According to Dyer-Ansher (1977) red wines usually contain more potassium.

Magnesium in wines is between 60–140 mg/l (Eschnauer, 1988). In Hungarian wines 65–95 was published (Papp et al., 1979). Its presence in wines compared to must, does not decrease (Würdig et al., 1989; Eperjesi et al., 1998), but even increase (Postel et al, 1987; Szitha et al., 1985; Lőrincz et al., 1986), which can be an effect of fining with bentonite (Postel et al, 1987). White wines, and wines of arid vintages usually contain less magnesium (Würdig et al., 1989).

Calcium content of wines is usually between 70-120 mg/l (Eschnauer, 1988). Its presence is critical from the point of wine stability, since it forms precipitate with certain

acids of the wine, of which solubility negatively correlate with increasing alcoholic content (Ferenzy, 1966). Turbidity resulting from this criterion must be taken in count over 80 mg/l calcium content (Madau et al., 1990; Goranov, 1979). Since fining of wines with bentonite can increase its presence (Postel et al, 1987) with about 10–15%, and application of calcium carbonate in deacidification can entail a 50–100% increase (Dyer-Ansher, 1977), calcium level of the must is normally lower than that of the wine. Two other alkali earth metals can be found in wines in a much lower concentration. **Strontium** between 0,12–6,5 mg/l, **barium** between 0,04–0,41g/l was measured (Thiel et al, 1998).

Iron content of wines is normally around 4-15 mg/l, but in certain cases can be much higher (Eschnauer, 1988; Szitha et al., 1985; Papp et al., 1979; Ferenzy-László, 1978). Higher concentration can entail spoilage of the wine through formation and precipitation of ferric phosphate (white braking) or ferric tannates (black braking) or blue braking, when Fe(III) forms precipitate with colour compounds. This can otherwise occur at a much lower 2–3 mg/l iron level already (Würdig et al., 1989). Its presence does not change significantly in the technology, however compared to iron level of the must (2–6 mg/l) it must be highlighted, that most of its fraction can come from the technology instrumentation.

Copper in wines is normally between 0,1–0,5 mg/l (Eschnauer, 1988; Green et al., 1997; Suturovic-Marjanovic, 1998). Red wines usually contain more than white wines (Martin-Ostapczuk, 1992). It is normal, if the copper content of the must is some fold higher, than that of the wine. High amount can come from fungicides of the plant protection. This fraction normally is reduced by the fermentation and sediments with the yeast (Frenzy, 1966). Any other increment resulting from weaknesses of the technology is to be eliminated, because copper can cause considerable turbidity (Würdig et al., 1989).

Manganese in wines can reach a 0,5–5 mg/l level (Eperjesi et al., 1998; Eschnauer, 1988; Szitha et al., 1985; Lőrincz et al., 1986). It seems to be an interesting result, that in case of American direct producers a higher 6,5 mg/l level on average was published (Eschnauer, 1982). Würziger (1964) found more manganese in red wines, while Tuzson (1964) found higher level in case of white wines. Manganese content of the must is normally somewhat lower, than that of the wine (Szitha et al., 1985). Technological treatments usually does not lower its concentration (Ferenzy-László, 1978).

Materials and methods

Experimental site is located 47°35'25,58" north latitude/ 21°38'24,15east altitude. The variety collection of the University of Debrecen was established in Pallag on immune sandy soil, by 3m between row and 1m between stock spacing trained for single curtain stock form, with the use of European own rooted planting material. Five stocks of each cultivar represent one experimental block. This paper reports data on

interspecific varieties listed in *Table 1*. Nutrition is carried out on the basis of the specific nutrient demand of the grape (Kozma, 1993) giving out 310 kg NPK (effective material) manure on yearly bases in two phases corresponding to a 10 t/ha production level. Farmyard manuring is carried out every four years. Dripping irrigation system is built out in the plantation.

Table 1. Varieties in the trial (Csepregi-Zilai, 1988)

	Parentage		Ennoblers	Year of qualification
<i>Refrén</i>	<i>Gloria Hungariae</i> x <i>Seibel 5279</i> (<i>Ferl</i>)	PIW 1	<i>Füri J. mts., 1964</i>	2005
<i>Csillám</i>	<i>S.V. 12375</i> x <i>Csaba gyöngye</i>	PIW 1	<i>Kozma Pál és mts. 1966</i>	1997
<i>Kunleány</i>	(<i>V. amurensis</i> x <i>V. vinifera</i>) F2 x <i>A fuz Ali</i>	PIW 1	<i>Tamássy I. x Koleda I., 1960</i>	1975
<i>Göcseji zamatos</i>	<i>Egerl</i> (<i>S.V. 12286</i>) x <i>Medoc noir</i>	PIW 1	<i>Csizmazia J. és Bereznai L., 1957</i>	2005
<i>Vértes csillaga</i>	<i>Egerl</i> (<i>S.V. 12286</i> selection) x <i>Medoc noir</i>	PIW 1	<i>Csizmazia L.- Bereznai L. 1963</i>	
<i>Generosa</i>	<i>Ezerjő</i> x <i>Piros Tramini</i>	intraspécif	<i>Biró Károly 1951</i>	2004
<i>Bianca</i>	<i>Eger2</i> x <i>Bouvier</i> (<i>S.v. 12375</i> seedling)	PIW 1	<i>Csizmazia L.- Bereznai L. 1963</i>	1982
<i>Aletta</i>	<i>Muscat Ottonel</i> x <i>S.V. 12375</i> (<i>E.2</i>)	PIW 1	<i>Csizmazia L.- Bereznai L. 1975</i>	2009
<i>Viktória gyöngye</i>	<i>S.V. 12375</i> x <i>Csaba gyöngye</i>	PIW 1	<i>Kozma Pál és mts. 1966</i>	1995
<i>Medina</i>	<i>Eger 1</i> (<i>S.V. 12286</i> selection) x <i>Medoc noir</i>	PIW 1	<i>Csizmazia J. x Bereznai L., 1959</i>	1984

Table 2. Basic quality parameters of cultivars in trials

	pH	BRIX %	Mm'	date
<i>Refrén</i>	3,19	19,9	18	2013.08.26
<i>Csillám</i>	2,56	21	19	2013.09.13
<i>Kunleány</i>	2,78	22,5	20,1	2013.09.13
<i>Göcseji zamatos</i>	2,77	20,2	18	2013.09.13
<i>Vértes csillaga</i>	2,75	19,1	17	2013.09.13
<i>Generosa</i>	3,29	23	20,5	2013.09.27
<i>Bianca</i>	3,12	22,2	20	2013.08.26
<i>Aletta</i>	2,8	19,7	17,5	2013.09.13
<i>Viktória gyöngye</i>	3,57	23,4	20,7	2013.09.27
<i>Medina</i>	3,34	21,3	19	2011.08.29

Table 3. Wine technology of 'Medina' grape

Started 2011.08.29 with 60kg of 'Medina' grape, BRIN: 21.3%; 3.34pH:				
	Fast processing	4 hours soaking	Fermentation sur marc in cold (12°)	Fermentation on mild temperature (25°)
	mixed after smashing			
	direct extraction after smashing	4 hours soaking after smashing	mixed half (30kg) separated after smashing	
starting quantity	15 l must	15 l must	15 kg sur marc	15 kg sur marc
sulfiting before fermentation	30mg/l sulfuration	30mg/l sulfuration	30mg/l sulfuration	30mg/l sulfuration
inoculation	same culture			
1st racking	2012.10.25	2012.10.25	2012.10.25	2012.10.25
2012.10.25	50 mg/l sulfuration	50 mg/l sulfuration	50 mg/l sulfuration	50 mg/l sulfuration

Vine technology

Trials are to be discussed in technological order, not on basis timing of their set up.

First trial (2013) based on resistant white wine cultivars pointing estimation of effect of soaking (maceration) on mineral element content and pH of the wine. Crushing of the bunches was followed by addition of 50mg/l sulphurous acid and Everzyme liquid (InterkerWine) to facilitate pectin degradation. (This step was not highly/ generally necessary, but makes the technology safer.) About half of the potential

must was separated directly after crashing. BRIX%, sugar degree of the must and pH was measured after processing. Fermentation was started uniformly with *Sacharomyces cerevisiae* (InterkerWine) on about 17–20 °C temperature. After 2 days, by the start of safe fermentation, items were put to 12 °C cooling chamber (uniformly adapted technology of the Institute). After the fermentation ended Everzyme arom

(InterkerWine) was added to each item. In the end 50 mg/l sulphurous acid was added, together with a 500 mg/l bentonite (BW200; InterkerWine) primarily to halt the enzymes. In the end alcoholic content, extracts, sugar content, acidic content and pH was measured. After one year in the bottle pH was measured again together with element content.

Harvest parameters of white wine varieties is detailed in *table 2*.

Second trial was established in (2011) with the use of 'Medina', a resistant (PIWI) red wine variety. Order of the technology trial is detailed in *table 3*.

Focus should be primarily driven to K content, tartrate formation and pH. Demonstrated double marc fermentation technology is abundantly used to produce two distinct quality product, with the use of one harvested item.

Sample preparation and analysis

Wine samples were diluted ten-fold with 5 (m/V) % nitric acid (69% VWR ARISTAR® for trace analysis) before analysis. As deionized water type-1 grade (Milli-Q® water purification system, Merck-Millipore, France) water was used. All samples were analyzed in duplicates.

The analysis was made by a ThermoFischer Scientific iCAP6300 ICP-OES instrument equipped with CETACASX-520 autosampler and a Meinhard-type concentric nebulizer attached to a cyclonic spray chamber. All measurements were made in axial-mode, analytical wavelengths are summarized in *Table XY*.

Quantification of desired elements was made by external calibration. As a stock solution a multielement solution was made from monoelement standards (1000 mg/L in 2% nitric acid, Scharlau, Scharlab, Spain) and to construct calibration curves appropriate dilutions were made from that.

The pH was measured with pH10pen (VWR International) in field, and with SevenEasy™ pH meter (Mettler Toledo) in laboratory, in case of ready wines.

Results and discussion

Concerning the technological order of winification, data on effect of skin maceration is discussed as first trial (table 4).

Data clearly show, that potassium content of wines increase due to skin maceration. A 4 hours' double pasta maceration resulted an approximate 30% increase in potassium content on average. This phenomenon showed uniformity on the range of cultivars, however in case of 'Viktoria gyöngye' an almost twofold outstanding was registered.

In case of Fe, an about 25% decrease was experienced due to skin maceration on average. In this concern 'Viktoria

gyöngye' appeared to be an exception again with its Fe-data showing no significant difference.

Cu, Mn and P showed 15, 27, and 30% increase due to 4 hours' double pasta skin maceration respectively (Table 5). A serious deviation of data on Cu content should be noted, dominantly thanks to 'Csillám' variety. This is especially the variety, which results the increase of the average data on Mn content with its twofold outstanding value.

Following technological point regarding the contact with the grape skin is fermentation. 2nd trial with 'Medina' grape resembles an example from the point alteration of element composition and pH.

In this trial element composition was measured two times for the sake of detection of possible later changes. Data clearly describe, that K level is increasing with longer skin contact and temperature in case of fermentation sur marc. The same increasing tendency stands for Mg,

Table 4. Data on element composition of white wine varieties in respect to the application of skin maceration

Average (mg/l)	Rebéra'		Csillám'		Kunleány'		Gölcseji szamósa'		Vértes csillaga'			
	Fast processing	Skin maceration	Fast processing	Skin maceration	Fast processing	Skin maceration	Fast processing	Skin maceration	Fast processing	Skin maceration		
K	473,238	619,622	383,747	962,836	485,236	966,514	303,236	546,338	383,270	694,538		
Mg	43,926	39,877	46,873	45,387	42,338	41,087	38,823	36,780	28,588	38,347		
Na	8,617	9,263	4,873	5,036	4,886	3,177	3,373	4,388	4,779	3,780		
Ca	46,368	27,887	44,473	44,827	33,836	38,373	40,967	33,343	27,398	42,833		
Fe	2,889	2,244	2,848	2,363	1,922	0,973	1,684	0,983	1,878	1,439		
Cu	0,069	0,056	0,148	0,144	0,029	0,060	0,014	0,048	0,040	0,033		
Mn	0,373	0,570	0,476	0,978	0,459	0,678	0,428	0,493	0,399	0,463		
P	132,331	124,427	48,928	184,389	48,677	84,138	79,787	118,878	98,348	118,858		
Ba	0,517	0,513	0,561	0,543	0,523	0,519	0,526	0,523	0,517	0,518		
pH	at processing	2,19		2,36		2,78		2,77		2,73		
	after fermentation	*	*	2,739	2,700	3,140	3,478	3,080	3,120	3,160	3,480	
	2015.02.04	*	*	2,640	3,250	3,190	2,480	3,630	3,390	3,040	3,330	
Average (mg/l)	Generosa'		Blanca'		Alta'		Viktória gyöngye'		Fast processing		Skin maceration	
	Fast processing	Skin maceration	Fast processing	Skin maceration	Fast processing	Skin maceration	Fast processing	Skin maceration	at processing	85/0%	at processing	85/0%
K	261,064	628,284	364,083	493,980	262,788	450,303	789,882	1126,856	486,128	38,828	780,338	33,787
Mg	34,980	48,856	37,480	62,080	42,437	43,112	37,388	38,833	38,823	21,480	42,803	28,388
Na	3,477	3,036	8,298	3,397	3,486	3,489	4,640	3,883	3,337	31,780	3,526	28,388
Ca	38,000	37,233	23,480	17,388	37,338	33,838	34,778	42,434	33,860	38,847	33,752	28,086
Fe	2,499	1,830	3,180	0,873	3,432	2,233	1,680	1,718	2,778	22,743	3,242	28,332
Cu	0,026	0,042	0,064	0,020	0,026	0,048	0,039	0,064	0,032	48,732	0,062	52,587
Mn	0,412	0,583	0,348	0,292	0,442	0,498	0,424	0,428	0,437	12,342	0,380	28,627
P	186,241	139,842	118,183	147,442	63,837	86,288	171,184	198,867	84,223	43,273	122,718	28,864
Ba	0,016	0,027	0,014	0,017	0,019	0,013	0,008	0,008	0,027	47,880	0,028	52,488
pH	at processing	2,79		3,12		2,40		3,17		2,88		
	after fermentation	*	*	3,16	3,33	3,31	3,38	*	*	3,08	3,46	3,39
	2015.02.04	3,00	3,49	*	3,67	3,82	3,30	3,34	4,09	3,38	3,87	3,50

*missing data from technical reason

Table 5. Data on element composition and pH of 'Medina' wines in respect to differentiated fermentation process

	Fast processing, cold fermentation		4 hours maceration, cold fermentation		Cold fermentation sur marc		Tempered fermentation sur marc	
mg/l	2013.10	2014.03	2013.10	2014.03	2013.10	2014.03	2013.10	2014.03
K	382,62	787	687,61	423,8	638,77	948	782,38	642
Mg	49,69	66,08	31,33	68,33	68,32	184,5	80,08	186
Na	5,85	8,3	7,3	6,86	3,03	3,47	3,84	4,12
Ca	28,88	63,79	37,08	38,23	38,49	47,7	33,6	43,3
Fe	1,27	1,66	1,23	2,89	0,3	1,22	0,31	1,89
Cu	0,09	0,1985	0,11	0,082	0,07	0,0203	0,07	0,086
Mn	0,39	0,496	0,79	0,737	1,29	1,28	1,26	1,28
P	63,18	31,5	66,44	46,79	118,8	136,8	139,76	149
Ba	0,02	0,077	0,02	0,0885	0,03	0,039	0,03	0,029
B	2,73	2,613	3,12	2,495	3,33	2,829	3,32	2,77
Sr	0,45	0,432	0,39	0,494	0,36	0,41	0,35	0,448
Zn	0,2	0,6888	0,2	0,6888	0,2	0,3383	0,27	0,478
pH	at processing			3,34				
	2012.12.17	3,1	3,3		3,44		3,54	
	2015.02.04	3,1	3,31		3,52		3,6	

however more characteristic differences were measured in fermentation sur marc (both temperature) in case of 2nd measurement 2014.03. An interesting data could be, a considerably higher level of Fe, which was detected in rose wines. The same could stand for Ba, but data are not coherent. Concentration of Mn, P and Sr is increasing by skin maceration with about 100%. A minor increase was measured in case of B.

A very important parameter in wine technology is pH. An emphasized point of this paper is to highlight interaction of skin maceration with increasing K content and increasing pH. Data show, that skin maceration and fermentation sur marc could increase K content with about 30–70% respectively, withstanding that pH also increase with a considerable 0.4–0.5 value. Corresponding to literature cited (Ferenczi,

1966; Kállay, 2010) it is clear to state, that basic change of pH in winification takes place in fermentation affected by increasing alcoholic content and turbidity, followed by cold stabilization, when decreasing temperature entails crystallization of tartrates. K content can be higher in the end product, despite to earlier higher level of tartrate formation. Attention should be driven to lower level of tartaric acid and consequently higher pH characteristic for wines produced with the application of skin maceration or fermentation sur marc.

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