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Chemical and sensory characterization of plum spirits obtained from cultivar Čačanska Rodna and its parent cultivars

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Abstract: During the two-year research, a comparative analysis of the contents of 24 major volatile compounds and sensory characteristics of the plum spirits produced by spontaneous alcoholic fermentation of the plum mashes with or without stones from cultivar Čačanska Rodna and its parent cultivars – Stanley and Požegača were carried out. The plum spirits obtained from Čačanska Rodna cultivar contain lower amounts of methanol, 1-hexanol, ethyl acetate and acetaldehyde and higher amounts of 3-methyl-1-butanol and 2-phenyl-ethanol than the spirits from its parent cultivars. Contents of 1-propanol, 1-butanol and hexanoic acid in the spirits obtained from Čačanska Rodna cultivar were lower than the contents in the spirits from Požegača and higher than those in the spirits obtained from Stanley. The sensory quality of plum spirits, in addition to the cultivar, has been significantly influenced by the presence of stones in the mash during processing of plums into spirits. In processing plums with stones, the best-graded plum spirit was from Požegača (17.88), whereas in processing without stones, the best was Čačanska Rodna spirit (17.78). The spirits obtained from Stanley cultivar had the lowest sensory grades regardless of the processing method.

Keywords: plum cultivars; destoning; spontaneous fermentation; spirits; volatile compounds; sensory characteristics.

INTRODUCTION

The old plum cultivar Požegača have been traditionally used for the production of supreme quality plum spirits in Serbia,[1] and in Europe as well[2–4] (the characteristics of plum cultivars for plum spirit production are presented in the

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Supplementary material to this paper). Although with a small fruit, it was also an indispensable raw material for the production of prunes. Nowadays, the area under this cultivar has been reduced in all plum growing European countries, as it is extremely susceptible to plum pox virus that causes premature fruit drop, decrease of total yield and fruit quality.[5,6]

In many plum-growing countries, the cultivars tolerant to plum pox virus, among which is the cultivar Stanley, have been introduced in the production. Plum cultivar Stanley was created in 1912 in the USA and it has been grown in Serbia since 1956. Similar to Požegača, it has been used for fresh market, for freezing, as well as for the production of prunes, jam and plum spirit. Due to its large fruit size, it is particularly suitable for drying, although with no harmonic ratio between sugar and acid contents as in Požegača.[5]

The cultivar Čačanska Rodna was developed from the cross of Stanley and Požegača cultivars at the Fruit Research Institute in Čačak. It was released and named in 1975, and protected in 1991. The main objective of breeding was the creation of a cultivar which would be tolerant to plum pox virus such as Stanley, and enable the production of similar quality (in terms of colour and taste), though larger prunes compared to Požegača.[7] Plum cultivar Čačanska Rodna has become significantly present in Serbian plum orchards in the last quarter of the century, as well as in some European countries, United States and New Zealand.[5,6]

Due to the decreased production of prunes in Serbia today, market surpluses of the fruits of both Stanley and Čačanska Rodna cultivars have been increasingly used as a raw material for spirit production.[5] In our earlier studies, the quality of plum spirit from the cultivar Čačanska Rodna was compared to the quality of plum spirits from other cultivars created in Čačak, intended primarily for fresh consumption and drying.[8] Also, in our previous work we considered the compliance of the content of the most important components in the Čačanska Rodna plum spirits, obtained from the top-quality fruits originating from different localities, with legislation.[9] However, there were no comparative studies of the characteristics of plum spirits obtained from plum cultivar Čačanska Rodna and its parent cultivars – Stanley and Požegača to this date. The aim of this work was to comparatively examine the influence of these cultivars on the content of the major volatile compounds and sensory characteristics of monovarietal plum spirits produced in the same manner (from the plum mashes with or without stones).

EXPERIMENTAL

Experimental details related to the plum fruits used, plum spirits production, the employed chemicals and statistical analysis are given in the Supplementary material to this paper.

GC analysis of volatile compounds

GC analysis of volatile compounds in the plum spirits was performed according to previously reported procedures.[10,11] Details are briefly described in the Supplementary material.

Sensory analysis

For sensory analysis of the plum spirits, the Buxbaum method was used.[3,12] Additional brief details are given in the Supplementary material.

RESULTS AND DISCUSSION

Contents of volatile compounds in plum spirits

The contents of the analysed volatile compounds in g hL^{-1} of absolute alcohol (a. a.) and the ratios of a certain higher alcohols in the twelve plum spirits produced from three plum cultivars are shown in the Supplementary material, Tables S-I (processing fruits with stones) and S-II (processing fruits without stones).

Zhang *et al.*[13] and Satora *et al.*[14] found that methanol contents in plum spirits vary significantly depending on the cultivars used. The contents of toxic methanol were considerably lower ($p \leq 0.001$) in the plum spirits from Čačanska Rodna (668.67 to 715.03 g hL^{-1} a. a.) than in spirits produced in the same manner and in the same year from its parent cultivars (709.30 to 939.37 g hL^{-1} a. a.). Also, they were almost two times lower than the maximum content (1200 g hL^{-1} a. a.) prescribed by EU regulations.[15] Our earlier studies have shown that the plum spirits with low methanol content (275 to 715 g hL^{-1} a. a.) can be obtained from Čačanska Rodna.[8,9] Interestingly, the Požegača plum spirits contained methanol in the similar range as the spirits from the same cultivar produced traditionally in Poland[3] and in ex-Yugoslavia.[8,16]

Higher alcohols are the most common aroma volatiles in the plum spirits, characterized by pleasant or unpleasant aroma depending on the type and their concentration.[1] The contents of higher alcohols in plum spirits vary significantly depending on the plum cultivar.[8] The contents of 3-methyl-1-butanol and 2-phenylethanol were significantly higher ($p \leq 0.001$) in Čačanska Rodna spirits than in those produced in the same way (with or without stones) and in the same year from parent cultivars, while it was contrary for the content of 1-hexanol. The concentrations of 1-propanol and 1-butanol were significantly different ($p \leq 0.001$) in monovarietal plum spirits produced in the same manner, within the same year. Their concentrations in Čačanska Rodna plum spirits were always between the values found in spirits from parent cultivars (higher than in Stanley plum spirit, and lower than in Požegača plum spirit). For other higher alcohols (2-butanol, 2-methyl-1-propanol and 2-methyl-1-butanol) as well as for the total content of higher alcohols no such regularities have been observed. These results confirm our previous findings regarding differences in the content of certain higher alcohols in Čačanska Rodna and Požegača plum spirits.[8]

Yeasts form most of the analysed higher alcohols during fermentation from the corresponding amino acids and/or sugars.[17] Among higher alcohols having direct precursors in amino acids, 3-methyl-1-butanol was predominant in all plum spirits, followed by 2-methyl-1-propanol, 2-methyl-1-butanol and 2-phe-

nylethanol. Fruits of the cultivar Čačanska Rodna had larger share of leucine and phenylalanine, in the total amino acids, than its parent cultivars,[18] which could explain the significantly higher contents of 3-methyl-1-butanol and 2-phenylethanol in Čačanska Rodna plum spirits. Because of 2-butanol dual (yeast and bacterial) origin, differences in 2-butanol contents in monovarietal plum spirits can probably be explained by the existence of subtle differences in the composition of the microflora during spontaneous alcoholic fermentation of plum mashes.

Among the higher alcohols analysed, only 1-hexanol does not derive from amino acids and/or sugars. In cognac production, content of 1-hexanol in wine distillates is considered a cultivar characteristic.[19] Based on the results obtained, the same goes for the monovarietal plum spirits. Contents of 1-hexanol in plum spirits made from Požegača are in accordance with the contents that are found in spirits of the same cultivar produced in ex-Yugoslavia,[8,16] and are considerably lower than the contents found in Požegača spirits produced in Poland.[2,3]

Ratios of certain higher alcohols may be useful for the closer characterization of monovarietal plum spirits. By analysis of variance it was found that higher alcohols ratios vary considerably ($p \leq 0.001$) in plum spirits depending on the plum cultivar used for processing. The highest regularity was observed for 2-phenylethanol/1-hexanol ratio, *i.e.*, its value was always greater in Čačanska Rodna plum spirits (1.24–2.71) than in those from the parent cultivars (0.27––1.28). Ratios of other higher alcohols were similar in Čačanska Rodna and Stanley plum spirits and most often higher than the same ratios in Požegača plum spirits. Their values in Požegača plum spirits were in compliance with the values in the spirits of the same cultivar produced in Poland, applying spontaneous mash fermentation as well.[2,3]

Among the analysed esters, only ethyl acetate contents in Čačanska Rodna plum spirits were always significantly lower ($p \leq 0.001$) than the contents in spirits of Stanley and Požegača. Differences in the content of ethyl acetate in the experimentally produced spirits are likely the result of subtle differences in the compositions of bacterial and yeast microflora in spontaneously fermented mashes of Čačanska Rodna and its parent cultivars.

In the produced plum spirits, ethyl acetate is followed by ethyl lactate. Regardless of the significant differences in the contents of this ester in monovarietal spirits, there were no regularities in these differences that can be attributed to plum cultivar. In Stanley plum spirits, it was observed that the content of ethyl lactate is several times greater in the second year than in the first. It may be caused by differences in the size of lactic acid bacteria population in fruits, which may arise due to different climatic conditions.[20] Depending on the concentration, both ethyl acetate and ethyl lactate have a positive or negative effect on the quality of plum spirit.

Tables S-I and S-II show that in some years there are no significant differences in the content of ethyl esters of fatty acids, diethyl succinate and isoamyl acetate in the monovarietal plum spirits produced. Furthermore, in the cases where a statistically significant difference was observed, there is no regularity in differences of these esters contents between plum spirits from Čačanska Rodna and its parent cultivars. It is most likely that these differences occur due to the existence of subtle differences in the microflora, which may occur in the spontaneous mash fermentations of different plum cultivars.[21] It should be noted that contents of these esters (characterized by pleasant fruity odours) in all produced monovarietal spirits were within the range for spirits obtained by spontaneous fermentation of Požegača and Čačanska Rodna plums previously presented in our earlier study[8] and studies of other authors.[1,2,14,16]

The contents of hexanoic acid in Čačanska Rodna spirits were in most cases significantly ($p \leq 0.01$ and $p \leq 0.001$) higher than in Stanley spirits and lower than in Požegača spirits. However, plum cultivar rarely affected the occurrence of significant differences in the contents of octanoic acid, decanoic acid and the total fatty acids in monovarietal spirits. Compared with the data reported in the literature for traditional Požegača plum spirits,[16] the contents of volatile fatty acids were 2–3 times lower in experimental samples produced from the same cultivar. Because volatile fatty acids are typical congeners of tail fraction, these differences are mainly due to earlier start of cutting between heart and tail fractions during re-distillation in experimental plum spirits production compared to traditional re-distillation.

In the same year, plum cultivars significantly ($p \leq 0.01$ and $p \leq 0.001$) influenced the contents of acetaldehyde in plum spirits produced in the same manner. This finding is in agreement with our previously reported results,[8] and are most likely the result of differences in the monovarietal plum mashes microflora.[21] Contents of acetaldehyde in the Čačanska Rodna spirits was most frequently lower than in the spirits from parent cultivars, except for the spirits obtained from plums without stones in year 2012 when the opposite was found. Experimentally produced Požegača plum spirits contained between 10.53 and 17.07 g hL^{-1} a. a. acetaldehyde, similar to Požegača plum spirits from Poland (between 9.2 and 17.5 g hL^{-1} a. a.).[3]

Significant differences ($p \leq 0.001$) in the benzaldehyde content were observed only in monovarietal plum spirits obtained from mashes with stones. Regardless of the cultivar, higher the stone ratio in fruits (see Table S-III), higher is the content of benzaldehyde in spirits. This is understandable, since most of the benzaldehyde in spirits derives from the amygdalin, cyanogenic glycoside present in the fruit stones.[22] In monovarietal spirits produced from mashes without stones, no statistically significant differences in the content of benzaldehyde have been observed. In this case, benzaldehyde is formed by degradation of the cyano-

genic glycoside – prunasin, present at a considerably smaller concentration in the plum fruits than amygdalin.[23]

The concentrations of 1-propanol, 2-butanol, 2/3-methyl-1-butanol, 1-hexanol, ethyl acetate, ethyl lactate, acetaldehyde and benzaldehyde in all monovarietal plum spirits were significantly lower than their limit concentrations above which they exhibit a negative impact on the sensory characteristics.[3,24]

Sensory analysis

The results of sensory evaluations of produced plum spirits are shown in Fig. 1, both for the processing plums with stones (W) and without stones (WO).

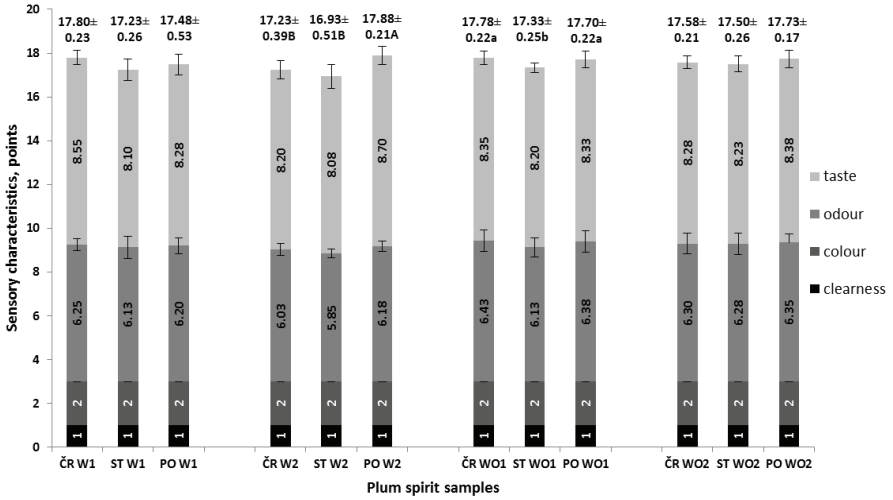

Fig. 1. Sensory analysis of monovarietal plum spirits obtained from plums with stones and without stones ČR – Čačanska Rodna, ST – Stanley, PO – Požegača, W – processing with stones, WO – processing without stones, 1 – Year 2011; 2 – Year 2012.

According to the levels of sensory ranking, which were in most cases > 17.00, the freshly distilled monovarietal spirits were considered to have no defects, forming a good basis for maturation and production of plum spirits of very good and superior quality.[1] Pecić *et al.*[25] have found that plum cultivars strongly influence the sensory characteristics of old Serbian plum spirits. Our results, however, show that differences in sensory characteristics of freshly monovarietal plum spirits can be statistically significant in one year, and yet not significantly pronounced in another. In other words, statistically significant difference ($p < 0.05$) in sensory characteristics was determined only among monovarietal spirits produced with stones in year 2012 (W2), and produced without stones in year 2011 (WO1). In these cases it was noted that: *i*) In the processing of plums with stones – plum spirits obtained from Požegača were assessed by a significantly higher

score than the spirits of Čačanska Rodna and Stanley cultivars, among which there was no significant difference; *ii*) in the processing of plums without stones – no significant difference was found between the spirits of Čačanska Rodna and Požegača, both with significantly higher score than the spirit of Stanley cultivar.

Cluster analysis

As results of the cluster analysis, appropriate dendrograms were obtained. Based on the content of volatile compounds, all experimentally produced plum spirits (12 samples) can be grouped into three main clusters (Fig. 2). The first cluster includes all plum spirits produced from the fruits of the Čačanska Rodna cultivar. Results indicate that, according to the contents of analysed volatile compounds, Čačanska Rodna spirits were different from those produced from parent cultivars. In the third cluster, which contains plum spirits with the highest contents of methanol and ethyl lactate, two subclasters were observed: in the first were ST W2 and ST WO2, and the second subclaster included plum spirit from Požegača (PO W1).

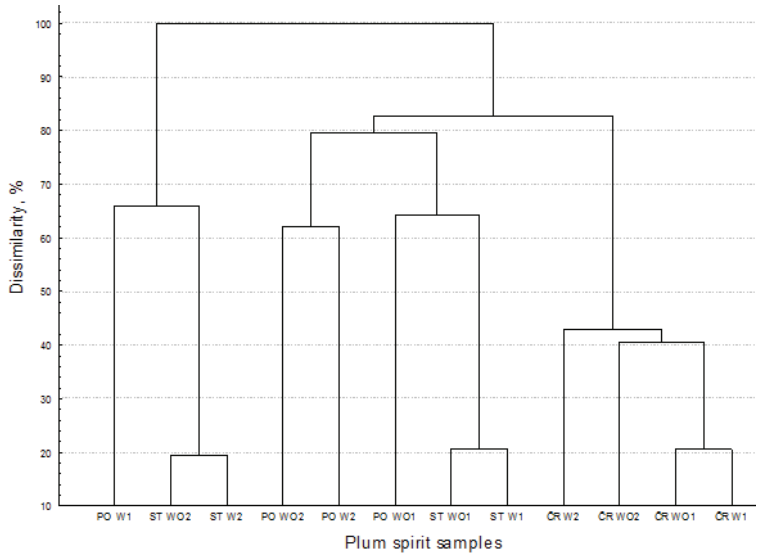

Fig. 2. Dendrogram obtained by the cluster analysis for all 12 plum spirits based on the contents of 24 volatile components ČR – Čačanska Rodna, ST – Stanley, PO – Požegača; W – processing with stones, WO – processing without stones; 1 – Year 2011, 2 – Year 2012.

Dendrogram based on the cluster analysis of sensory grades of the plum spirits produced (Fig. 3) differs from the dendrogram based on the content of volatile compounds.

The spirit samples are more grouped in clusters based on the processing method (with or without stones) than on the cultivar used for their production.

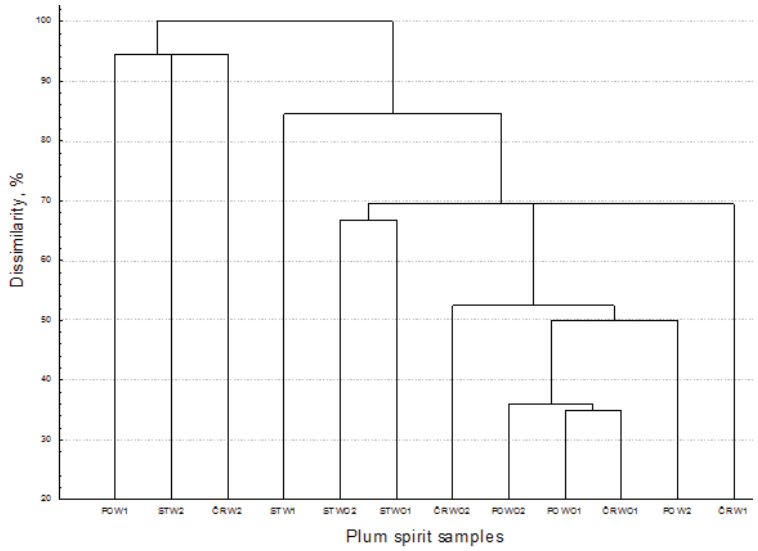

Fig. 3. Dendrogram obtained by the cluster analysis based on the sensory characteristics of 12 plum spirits; ČR – Čačanska Rodna, ST – Stanley, PO – Požegača; W – processing with stones, WO – processing without stones; 1 – Year 2011, 2 – Year 2012.

Cluster 1 contained all samples produced without stones, but also the two spirits produced with stones (PO W2 and ČR W1) in which the presence of stones in mashes did not show a negative impact on the sensory characteristics of the spirit. All the plum spirits of cluster 1 were distinguished by the pleasant fruity odour; sensory grades in this cluster ranged from 17.33 to 17.88. The spirits in other clusters were characterized with fruity odour, although less pronounced, and partially masked by the stone-like ton: cluster 2 (ST W1) and cluster 3 (ST W2, ČR W2 and PO W1). Such sensory characteristics are common for plum spirits obtained by spontaneous alcoholic fermentation of mashes with stones. Because of heavy odour, sensory grades of the plum spirits in this group ranged from 16.93 to 17.48.

CONCLUSION

Based on the content of volatile compounds from the spirit obtained, the fruits of the plum cultivar Čačanska Rodna can be regarded as a suitable raw material for the production of quality plum spirit. Plum spirits produced from the Čačanska Rodna cultivar are distinguished by the content of some important volatile compounds from spirits made in the same manner from the parent cultivars (Stanley and Požegača). The significant differences in sensory characteristics of the monovarietal spirits is not influenced by the cultivar only, but also by the processing method and the production year. In the years with significant differences in the sensory assessments of monovarietal plum spirits, it was est-

ablished that the Čačanska Rodna plum spirits could be of similar sensory characteristics as the spirit of the Stanley cultivar (in the processing with stones), *i.e.*, with similar characteristics as spirits from Požegača (in the processing without stones).

SUPPLEMENTARY MATERIAL

Additional data are available electronically from http://www.shd.org.rs/JSCS/, or from the corresponding author on request.

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И З В О Д

ХЕМИЈСКА И СЕНЗОРНА КАРАКТЕРИЗАЦИЈА ШЉИВОВИЦА ДОБИЈЕНИХ ОД СОРТЕ ЧАЧАНСКА РОДНА И ЊЕНИХ РОДИТЕЉСКИХ СОРАТА

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Током двогодишњих испитивања, анализирани су садржаји 24 важније испарљиве компоненте и сензорне карактеристике шљивовица произведених од спонтано ферментисаних кљукова шљиве са коштицама или без њих сорте чачанска родна и њених родитељских сората – стенлеја и пожегаче. Сорта шљиве значајно утиче на хемијски састав шљивовица. Шљивовице добијене од сорте чачанска родна садржале су мање метанола, 1-хексанола, етилацетата и ацеталдехида, а више 3-метил-1-бутанола и 2-фенилетанола него шљивовице произведене од родитељских сората. Садржаји 1-пропанола, 1-бутанола и хексанске киселине у ракијама од сорте чачанска родна били су нижи од садржаја у шљивовицама од сорте пожегача, а виши него у шљивовицама добијеним од сорте стенлеј. Поред сорте, на сензорни квалитет шљивовица је значајно утицало и присуство коштица у кљуку током прераде шљива у ракију. При преради шљива са коштицама, најбоље је оцењена шљивовица сорте пожегача (17,88), док је при преради без коштица највишу оцену добила шљивовица сорте чачанска родна (17,78). Шљивовице сорте стенлеј добиле су најниже сензорне оцене, без обзира на начин прераде. На основу добијених резултата може се закључити да је сорта чачанска родна погодна сировина за производњу квалитетне ракије шљивовице.

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