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THE APPLICATION OF SHEET FILTERS IN TREATMENT OF FRUIT BRANDY AFTER COLD STABILISATION

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Considering the common use of sheet filtration for clarification of fruit brandies, the aim of this study was to evaluate the influence of its application on the stability and composition of volatile compounds of apricot brandy after cold stabilisation. Cold stabilisation treatment involved holding of the brandy at -1°C during 24 hours. Five depth filter sheets with the nominal retention rate of 0.3 µm, 0.5-0.7 µm, 0.7-1.0 µm, 1.0-2.0 µm and 2.5-4.0 µm, were tested in the study. It was shown that all assessed filter sheets were efficient in removing chill haze by significantly reducing the content of fatty acid esters (primarily ethyl palmitate and ethyl laurate). Other volatile and aromatic compounds were not significantly influenced by the applied treatments. However, the filter sheets with higher nominal retention rate (> 0.7 µm), had a smaller impact on the sensory characteristics of the apricot brandy. The re-exposure to lower temperatures did not lead to chill haze formation in any sample obtained after sheet filtration.

KEY WORDS: filter sheets, apricot brandy; cold stabilisation; volatile compounds

INTRODUCTION

Fruit spirits are obtained by distillation of fermented fruit juice. Production of this alcoholic beverage can be divided into at least three major distinct stages: (i) raw material preparation and fermentation (ii) distillation (iii), and maturation and stabilisation of the distillate to produce the final, typical fruit brandy. The flavour and aroma constituents of finalised brandy are derived from each successive stage of the production process (1). Both the major and minor components found in brandy are responsible and essential for the total brandy aroma. However, fusel alcohols, fatty acids and their esters are usually more dominant than carbonyl, phenolic, sulphur and nitrogen compounds, and play an important role in the overall aroma profile and quality of brandy (2).

The solubility of the aromatic volatile compounds is decreasing at lower ethanol content (below 45% v/v), as well as at lower temperatures. The consuming temperature for fruit brandies is around 15-18°C. However, these alcoholic beverages are often stored and transported at lower temperatures (below 7°C), which can lead to the formation of cloudiness and haze. Haze can develop in the finished, bottled beverage since its formation may

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be slow. This is the reason why producers are recommended to intentionally encourage forming of haze and then remove it (3). Cloudiness in fruit brandies also often appears when the distillate strength is set to less than 45% v/v (4). It was shown that fatty acid esters, such as ethyl laurate (ethyl dodecanoate), ethyl palmitate (ethyl hexadecanoate), ethyl palmitoleate (ethyl-9-hexadecanoate), are linked to chill haze formation (5). The process which effectively solves the problems connected with the chill haze formation in brandies is called cold stabilisation. It includes thinning of the distillate to the desired alcohol strength, its storage at -5 to -7^{o}C during several days and, finally, filtration (4).

Depending on the nominal retention rate of the filtration material, the filtration process may excessively remove some valuable flavour compounds responsible for the typical character of fruit brandies. There are numerous possibilities for removal of chill haze by filtration such as sheet filters, plate and frame filters, lenticular sheet filters, candle filters, etc. (6). It was reported that chill filtration is causing a significant decrease of ester and terpene compounds, provoking a loss of characteristic grape spirits flavour (7). The chill haze removal from whiskies by application of membrane filtration was shown as flexible, efficient and economic (5). The use of ceramic membranes with pore sizes of 200, 450 and 800 nm for removal of chill haze in apricot brandy has been reported recently (8). It was shown that all tested membranes efficiently reduced the content of fatty acid esters, ensured stability of samples after the re-exposure to lower temperatures, while the best sensory characteristics were saved after filtration through the 800 nm pore size membrane. Information from the industry indicates that sheet filters are the most commonly used filtration systems applied in the finalisation steps of brandy production.

The common apricot (*Prunus armeniaca*) is an edible fruit, the quality of which is determined by a balance of sugar and acidity, as well as a strong and characteristic aroma. It is an excellent source of vitamin A and vitamin C (9). There are many different uses of apricots. It is enjoyed as fresh fruit, but a large portion of the worldwide production is preserved primarily by drying. All fruits for the fresh market are hand-harvested. Apricots are also subjected to different ways of processing, and, therefore, utilised as canned, dried, frozen, etc. Other products made from apricots include wine, brandy, jam, and nectar (10). An average annual production of apricots in Serbia is about 30000 t (11). A significant amount of apricot fruits produced in Serbia is processed into brandy.

Considering the common use of sheet filters for the filtration of fruit brandies, the available literature is very poor and difficult to access. Hence, the aim of this research was to investigate the influence of sheet filtration on stability and volatile compounds of apricot brandy subjected to the cold stabilisation treatment. Furthermore, the sensory characteristics of the treated brandy samples were assessed. In addition to the scientific contribution, the obtained results may also have great practical importance for the industry of strong alcoholic drinks.

EXPERIMENTAL

Samples

Cold stabilisation and filtration experiments were carried out on a Premier (Promont distillery, Novi Sad, Serbia) apricot brandy. Fermentation was conducted at 18^{o}C and pH

3.2, and finished after 18 days. The inoculation was performed with 0.25 g/kg of previously rehydrated commercial yeast *Saccharomyces cerevisiae* (Spiriferm, Erbslöh, Geisenheim, Germany), intended for application in distilleries. The distillation was carried out in a 500-L distillation unit Arnold Holstein SH 1200, equipped with a 4-floor rectification column. The distillate was set to the alcohol strength of 42% v/v.

Cold stabilisation and sheet filtration experiments

Apricot brandy was kept in a 1000-L stainless steel vessel at -1°C during 24 hours. Filtration experiments were carried out in a conventional sheet filter (filter press) (Stainless Steel Layer Filter - ENO 20, Arnold Holstain, Markdorf, Germany) equipped with a centrifugal pump and 20 filter sheets (20 x 20 cm). A volume of 200 L of the brandy was used for each filtration cycle. The following depth filter sheets were used in this study: BECO Steril 60, BECOPAD 270, BECOPAD 350, BECOPAD 450 (Begerow. Langenlonsheim, Germany) and SEITZ K150 (Pall Corporation, New York, USA) with the nominal retention rate of 0.3 μm, 0.5-0.7 μm, 0.7-1.0 μm, 1.0-2.0 μm and 2.5-4.0 μm, respectively. The used depth filter sheets were composed of diatomaceous earth (Kieselguhr) and perlite (the filtration active substances) and cellulose as the matrix material. Further small quantities of special resins are used to ensure the wet strength of filter sheets. The experiments were carried out under the differential pressure of around 1 bar, which was achieved by the use of a centrifugal pump operated at a frequency of 38 Hz. The obtained flow rates, depending on the used filter sheets, were in the range of 10-20 L/min.

Analytical methods

The determination of the concentrations of methanol, ethanol and higher alcohols was performed by gas chromatography using a gas chromatograph Agilent 7890A equipped with flame ionization detector (FID) and a split/splitless injector. A capillary column HP-INNOWax (polyethylene glycol; 30 m x 250μm i.d., with 0.25 μm film thickness) was used for the separation of components. A sample volume of 1 μL was injected directly into the column. The detector and injector temperatures were 280°C and 220°C, respectively. The GC oven temperature program was: 35°C (5 min) to 240°C (2 min) at the rate of 5°C/min. The carrier gas was helium at a 150 mL/min flow, while the flow speed of hydrogen and air was 30 mL/min and 400 mL/min, respectively.

Other volatile components (aromatic compounds) of the apricot brandy were determined after a pre-treatment of the samples. This included Soxhlet extraction during 2 hours, according to the following procedure: a volume of 50 mL of the brandy sample was mixed with 50 mL of distilled water, 50 mL of methylene chloride (a solvent) and 200 μL of methyl undecanoate (1mg/mL, an internal standard), while the injection volume was 1 μL. The extract was concentrated to the volume of 1 mL by vacuum evaporation (2 hours at 20 ºC), and subjected to the GC analysis on the same apparatus. The separation was carried out using an HP-5 (5% phenyl methyl siloxan) capillary column, 50 m x 320μm i.d., with 1.05 μm film thickness. The detector and injector temperatures were 300°C and 250°C, respectively. GC oven temperature was programmed from 50°C to 300°C at a rate

of 2°C/min. The carrier gas was helium (45 mL/min), while the flow speed of hydrogen was 30 mL/min and that of air 400 mL/min.

Sensory analysis

Sensory analysis of apricot brandy samples (before and after cold stabilisation and filtration) was carried out according to the Buxbaum model of positive ranking (12). Sensory evaluation was performed by a panel of 5 qualified testers who rated up 4 sensorial experiences (colour, clearness, odour, taste) to a maximum of 20 points.

Statistical analysis

Statistical analyses were conducted using STATISTICA 10.0 (Statsoft, 2011). The analysis of variance (ANOVA) at the 95% confidence level was applied for expressing the statistical significance of the differences between average values for parameters analysed. Different letters (a, b, c, etc.) were used for marking significantly different values.

RESULTS AND DISCUSSION

Keeping the brandy at -1°C during 24 hours caused the formation of a clearly visible and stable haze. After the cold stabilisation the brandy requires treatment by filtration in order to remove the formed haze. It is important to apply filtration process which will be effective in removal of cloudiness, but, at the same time, will ensure the minimal impact on the sensory characteristics. The analysis of variance (ANOVA) was used to determine the statistically significant differences ($p<0.05$) between the concentration of the brandy volatile compounds, before (control sample) and after cold stabilisation and filtration through the different sheets (Table 1). In this study, 28 different volatile compounds were identified by using the GC-FID analysis. The applied treatments did not have significant influence on the content of alcohols (methanol, propanol, etc.), aldehydes (acetaldehyde, furfural, etc.) and, especially important, on the content of terpenes. The only exception was nerol, an important constituent of apricot brandy aroma, which was completely removed after the filtration on BECO Steril 60 and BECOPAD 270. The concentration of fatty acid esters, which contribute to the fruity and flowery aroma of brandies (13), has undergone the most significant changes. The use of BECO Steril 60 and BECOPAD 270 sheets for filtration of the brandy after cold stabilisation caused complete removal of ethyl laurate. Furthermore, the concentration of this ester was significantly ($p<0.05$) decreased also after the filtration on the filter sheets with higher nominal retention rate (BECOPAD 350, BECOPAD 450 and SEITZ K150). This trend was even more pronounced in terms of ethyl palmitate content. This aromatic ester was completely removed from the brandy after filtration on all sheets applied, except for SEITZ K150, where its concentration decreased bellow 1 mg/L. Regarding the status of the other important components of the brandy flavour, it is also important to emphasize the considerable decrease ($p<0.05$) in the content of ethyl decanoate and ethyl pentadecanoate. These results are in

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agreement with those obtained in the research where the cold stabilisation of brandy was followed by membrane filtration (8).

Table 1. Influence of sheet filtration on the concentration of volatile compounds of apricot brandy

Volatile compound	Control sample	BECO Steril 60	BECOPAD 270	BECOPAD 350	BECOPAD 450	SEITZ K150
			[mg/L 100° EtOH]			
Acetaldehyde	67 ± 2^{a}	61 ± 2^{a}	64 ± 2^{a}	66 ± 3^{a}	68 ± 3^{a}	67 ± 1^{a}
Ethyl acetate	851 ± 16^{a}	742 ± 6^{a}	771 ± 14^{a}	828 ± 11^{a}	830 ± 20^{a}	848 ± 18^{a}
Methanol	8040 ± 36^{a}	7750 ± 60^{a}	7867 ± 28^{a}	8030 ± 41^{a}	8120 ± 28^{a}	8055 ± 12^{a}
1-Propanol	656 ± 8^{a}	648 ± 3^{a}	655 ± 8^{a}	658 ± 4^{a}	655 ± 18^{a}	663 ± 14^{a}
1-Butanol	124 ± 8^{a}	121 ± 4^{a}	123 ± 7^{a}	125 ± 6^{a}	124 ± 3^{a}	128 ± 6^{a}
Isobutanol	503 ± 26^{a}	487 ± 8^{a}	499 ± 13^{a}	495 ± 4^{a}	512 ± 16^{a}	508 ± 20^{a}
3-methyl-1-butanol	965 ± 34^{a}	933 ± 7^{a}	938 ± 3^{a}	962 ± 13^{a}	972 ± 26^{a}	964 ± 16^{a}
1-Pentanol	9 ± 2^{a}	7 ± 1^{a}	9 ± 1^{a}	10 ± 1^{a}	9 ± 2^{a}	9 ± 3^{a}
1-Hexanol	72 ± 4^{a}	70 ± 1^{a}	70 ± 3^{a}	73 ± 8^{a}	75 ± 1^{a}	73 ± 3^{a}
Furfural	14 ± 2^{a}	14 ± 3^{a}	13 ± 1^{a}	14 ± 3^{a}	14 ± 4^{a}	15 ± 1^{a}
Benzaldehyde	26 ± 1^{a}	23 ± 2^{a}	24 ± 1^{a}	25 ± 5^{a}	25 ± 1^{a}	26 ± 3^{a}
1-Nonanol	48 ± 1^{a}	48 ± 2^{a}	49 ± 1^{a}	52 ± 6^{a}	48 ± 3^{a}	49 ± 1^{a}
			[mg/L brandy sample]			
Ethyl lactate	44 ± 6^{a}	38 ± 2^{a}	43 ± 7^{a}	42 ± 7^{a}	45 ± 2^{a}	44 ± 8^{a}
Ethyl isovalerate	0.9 ± 0.2^{a}	0.8 ± 0.1^{a}	0.9 ± 0.2^{a}	0.8 ± 0.3^{a}	0.9 ± 0.3^{a}	0.9 ± 0.1^{a}
Isoamyl acetate	4 ± 2^{a}	4 ± 1^{a}	3 ± 1^{a}	4 ± 1^{a}	4 ± 2^{a}	4 ± 2^{a}
Ethyl hexanoate	0.5 ± 0.4^{a}	0.4 ± 0.1^{a}	0.6 ± 0.1^{a}	0.5 ± 0.3^{a}	0.6 ± 0.2^{a}	0.5 ± 0.3^{a}
Linalool	13 ± 4^{a}	11 ± 3^{a}	12 ± 2^{a}	12 ± 3^{a}	13 ± 1^{a}	13 ± 4^{a}
1-Phenylethanol	1.3 ±0.4^{a}	1.0 ± 0.4^{a}	1.3 ± 0.1^{a}	1.3 ± 0.1^{a}	1.3 ± 0.2^{a}	1.4 ± 0.3^{a}
Ethyl octanoate	4.9 ± 0.6^{c}	2.0 ± 0.2^{a}	2.8 ± 0.4^{a}	3.6 ± 0.3^{b}	4.8 ± 0.4^{c}	5.3 ± 0.8^{c}
α-terpineol	8 ± 2^{a}	6 ± 3^{a}	8 ± 3^{a}	8 ± 1^{a}	9 ± 4^{a}	9 ± 2^{a}
Nerol	0.9 ± 0.2^{a}	-	-	0.8 ± 0.2^{a}	1.3 ± 0.5^{a}	1.0 ± 0.3^{a}
β-citronellol	0.5 ± 0.1^{a}	0.5 ± 0.3^{a}	0.7 ± 0.5^{a}	0.5 ± 0.1^{a}	0.6 ± 0.3^{a}	0.5 ± 0.2^{a}
Geranyol	1.6 ± 0.8^{a}	1.5 ± 0.4^{a}	1.4 ± 0.1^{a}	1.8 ± 0.3^{a}	1.6 ± 0.2^{a}	1.8 ± 0.6^{a}
Ethyl decanoate	11 ± 4^{b}	3 ± 1^{a}	6 ± 3^{a}	8 ± 1^{ab}	7 ± 2^{ab}	8 ± 3^{ab}
Ethyl 2-trans-4-cis-decadienoate	1.3 ± 0.2^{a}	1.0 ± 0.4^{a}	1.2 ± 0.3^{a}	1.6 ± 0.2^{a}	1.3 ± 0.1^{a}	1.4 ± 0.4^{a}
Ethyl laurate	3.5 ± 1.4^{c}	-*	-	1.7 ± 0.2^{a}	2.9 ± 0.4^{b}	2.8 ± 0.8^{b}
Ethyl pentadecanoate	1.7 ± 0.2^{b}	0.3 ± 0.2^{a}	0.6 ± 0.4^{a}	1.6 ± 0.6^{b}	1.7 ± 0.3^{b}	1.5 ± 0.5^{b}
Ethyl palmitate	3.3 ± 0.2^{b}	-	-	-	-	0.5 ± 0.2^{a}

Data given in the table present mean values ± 2 standard deviation from three replications (n=3)
Different letters within the same row mean significant differences (P<0.05).
*not detected

The brandy samples were re-exposed to the lower temperatures (3-4^{o}C) after performing the sheet filtration. The chill haze was not observed in any of the samples tested. This confirms the adequacy of the cold stabilisation process. As the next major goal of the applied brandy processing was to preserve its original flavour, the changes of which have occurred in the content of volatile compounds created the need for the evaluation of

their influence on the sensory characteristics of the brandy. For this purpose, the Buxbaum model of positive ranking was carried out. It was shown that there exist sensorial differences between the filtered samples and basic brandy (Table 2). These differences are most pronounced in terms of odour experience. The filtration on BECO Steril 60 and BECOPAD 270 gave the brandy with the lowest overall sensory acceptability. There was no significant difference between the sensory characteristics of samples obtained after the filtration on BECOPAD 350, BECOPAD 450 and SEITZ K150 and control sample. The highest score (19.0) in the sensory analysis of treated brandies was given to the sample filtered on SEITZ K150, and it was shown to be the most similar to the control (untreated) sample. Considering the fact that the filtration on BECOPAD 350, BECOPAD 450 and SEITZ K150 gave similar results in terms of sensory acceptability of the brandy, and that they were efficient in removing the chill haze causes, they all can be used equally effectively in the industry. The parameter which can be taken into account when choosing among these three filter sheets is the filter capacity, which is larger when the sheets with higher nominal retention rate are applied.

Table 2. Sensory analysis of the apricot brandy samples

Sample	Sensorial experiences				
	Colour (max 2 pts)	**Clearness (max 2 pts)**	**Odour (max 4 pts)**	**Taste (max 12 pts)**	**Total (max 20 pts)**
Control	2	2	3.7	11.5	19.2[b]
BECO Steril 60	2	2	2.9	10.8	17.7[a]
BECOPAD 270	2	2	3.0	11.2	18.2[a]
BECOPAD 350	2	2	3.5	11.3	18.8[b]
BECOPAD 450	2	2	3.5	11.2	18.7[b]
SEITZ K150	2	2	3.6	11.4	19.0[b]

Data given in the table present mean values from the marks assigned by a panel of five testers
Different letters mean significant differences ($P<0.05$).

CONCLUSION

The results of this study showed that the tested filter sheets were efficient in removing chill haze formed due to the exposure of the apricot brandy to the lower temperatures (-1°C). This is primarily related to the decrease in the content of ethyl palmitate and ethyl laurate, the main causes of chill haze in alcoholic spirits. All applied filtration treatments gave the brandy which was stable after the re-exposure to lower temperatures. The filtration on BECOPAD 350, BECOPAD 450 and SEITZ K150, filter sheets with higher nominal retention rate, had the least impact on the sensory characteristics of the apricot brandy, which recommends them as the best filtration solution.

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ПРИМЕНА ПЛОЧАСТИХ ФИЛТЕРА У ОБРАДИ ВОЋНИХ РАКИЈА НАКОН ПОСТУПКА ХЛАДНЕ СТАБИЛИЗАЦИЈЕ

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С обзиром на честу употребу плочастих филтера за бистрење воћних ракија, циљ овог рада је био да се испита утицај примене овог вида филтрације на стабилност и састав испарљивих једињења ракије од кајсија која је претходно подвргнута поступку хладне стабилизације. Хладна стабилизација је подразумевала складиштење ракије на температури од -1^{o}C у току 24 сата. У раду је коришћено пет филтер плоча са номиналним опсегом задржавања од 0,3 μm, 0,5-0,7 μm, 0,7-1,0 μm, 1,0-2,0 μm и 2,5-4,0 μm. Све тестиране филтер плоче су се показале ефикасним у отклањању магличастог замућења насталог услед излагања ракије ниским температурама, пре свега захваљујући значајном смањењу садржаја естара масних киселина (пре свега етил-палмитата и етил-лаурата). Примењени поступци филтрације нису довели до значајнијих промена у садржају осталих детектованих испарљивих једињења. Међутим, употреба филтер плоча са вишим номиналним опсегом задржавања (већи од 0,7 μm), имала је мањи утицај на сензорне карактеристике ракије од кајсије. Поновно излагање филтрираних ракија ниским температурама није довело до формирања магличастог замућења ни код једног узорка.

Кључне речи: филтер плоче; ракија од кајсија; хладна стабилизација; испарљива једињења

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