Primary studies of the composition of distillate beverages produced from Sorbus Domestica fruits

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Abstract. S. domestica are valuable plants which have been used for both nutritional purposes and in traditional medicine. Recent studies reveal that in the fruits of S. domestica the sugar content and total acidity is similar to quantities in apples (Malus spp.) and pears (Pyrus spp.). Despite the sufficient data obtained S. domestica is not yet so strongly represented in the production of distilled beverages. For the purpose of this study, we obtained beverages by means of three different methods - distillation of fermented juice (A), distillation of fermented crushed fruit mixture (B) and distillation of ethanol-water extract (C). The different fractions obtained during the distillation process were studied and correspondently used to determine the quantities of Alcohol, Esters, Aldehydes, Higher alcohols and Methanol. Concentration of Esters in the fractions is between 3688.0 mg/L to 29.0 mg/L. The quantity of Esters in fractions of Series A and B is three times higher than in Series C. Regarding the Aldehyde and Higher alcohols content is ten times higher in both A and B Series in comparison to Series C.

1 Introduction

Sorbus Domestica is a species belonging to genus Sorbus, spread mainly in Western, Central and Southern Europe, also in Northwestern and Southwestern Africa. Only some species of the genus Sorbus are grown for their fruits, and one of the economically valuable is S. Domestica, which belongs to the subgenus Cormus [1].

Although these trees are very rare and endangered in many European countries, the fruits of S. domestica have been valued since Roman times. The fruits are 2-3 cm apple or pear shaped, with greenish to brownish color. The distillate beverages obtained from the fruits are highly valued due to their specific characteristics [2].

The composition of the beverages depends mainly on the composition of the raw material, as it is known that the sugar content and titrable acidity is similar to the apple’s (genus Malus) and pear’s (genus Pyrus). The fermentation and distillation processes also play a key role to the distilled drink.

Studies in the field show that the dry material in fresh fruits is between 18.1-23.8% and the amount of reducing sugars in the fruits of S. domestica are in the range of 13.9-18.0% [3].

The aim of this article is to study the composition, and precisely the number of total esters, higher alcohols, aldehydes and methanol in distilled beverages obtained from the fruits of the species S. Domestica. The amounts of dry material, titrable acidity and reducing sugars in the fruits were studied as well.

2 Materials and methods

2.1 Experimental part

In order to determine the amounts of dry material of I fruits two extracts were made:
Extract (1) - containing fruits with total weight 53.571 g, stored in refrigerator at 6°C;
Extract (2) - containing fruits with total weight 52.977 g, stored for 7 days at room temperature. As a result, the skin of the fruits darkens and the flesh softens.

The two quantities were distributed in two measuring cylinders with a volume of 100 ml. For each of the variants was performed three-fold exhaust extraction with water at 35°C. To prevent microbiological development in the extracts was added sodium azide to the water. Each of the steps continues until the refractometric reading of the extracts reaches a constant value. After each step, the aqueous extracts were separated, and afterwards blended and filtered. The content of dry material in blended and filtered extracts was determined using a refractometer, and the sugar content by the method of Shool [4]. The dry material and reducing sugars contents are recalculated to the fruit weight.

For the production of distillate beverages (distilled fractions), three themes of fruit processing were implemented:

- Serie A - distilled beverage obtained by fermentation of fruits and distillation of liquid phase. 15 kg of fruits were stored in cooling chamber after that they

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were crushed by shaft crusher. Due to low content of the liquid phase, dechlorinated water is added to the fruit in 1:1 ratio. Crystal cane sugar is added during the mixing both water and crushed fruits, the level of sugars added are approximately as the contained in processed fruits. It was added tartaric acid with concentration of 1g per liter of used water. Obtained fruit mash is inoculated with 0.15 mg/dm³ S. cerevisiae and fermentation were maintained between 18-20°C. During the fermentation diammonium phosphate was added twice at a dose of 50 mg/dm³ and twice a day the cap was pushed down. After 20 days the analysis shown less than 1.5 mg/dm³, fermented juice was drained and distilled.

- Serie B - distilled beverage obtained by fermentation of fruits and distillation of fermented fruit mash. The experiment is identical to Serie A, but 10 kg of fruits were processed. After complete alcoholic fermentation, the mash was subjected to distillation. Serie C - distilled beverage, obtained from extraction of non-fermented fruits with water-alcohol mixture and subsequent distillation. 2 kg of fresh fruits were caused and extracted with 1 dm³ water-alcohol mixture with 50 vol. %, obtained by mixing rectified spirit and dechlorinated water. After 5 days of extraction, the liquid phase is drained, washed three times with total amount of 3 liters water and achieved water-alcoholic extract is put through distillation. All the three distillations were performed by laboratory distiller with indirect heating and equipped with an air reflux condenser. In the course of the distillation process, fractions were collected in all three distillations until 80% of the total alcoholic strength of the distilled product. In Serie C this percent is 85%. In the Table 1 are shown the numbers of fractions collected from each distillation and their volumes.

Per each of the fractions, the amount of ethanol, methanol, higher alcohols, esters and aldehydes was determined.

**Table 1. Number of collected fractions and their volumes.**

<table>
<thead>
<tr>
<th>Fractions collected</th>
<th>Volume of fractions, cm³</th>
<th>Serie A</th>
<th>Serie B</th>
<th>Serie C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>50</td>
<td>50</td>
<td>500</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>500</td>
<td>500</td>
<td>500</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>500</td>
<td>500</td>
<td>300</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>500</td>
<td>500</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>500</td>
<td>500</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>500</td>
<td>300</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>300</td>
<td>100</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

**2.2 Analytical methods**

The ethanol content was determined by measuring the relative density of the distillates [5]. The content of methanol was determined by its oxidation with potassium permanganate and measurement of adsorption after interaction with chromotropic acid in an acidic environment [5]. The content of higher alcohols was determined by the Komarowski-Felenberg method, by measuring the intensity of a color compound formed by interaction with para-dimethyl amino benzaldehyde [5].

The content of aldehydes was determined by the bisulphite method [5], and that of esters, by saponification with sodium [5].

All reagents used in the methods (sulfuric acid, chromotropic acid, para dimethyl amino benzaldehyde, sodium bisulfite, etc.) are class h.z.a.

**3 Results and Discussions**

Table 2 shows the content of dry material, reducing sugars and total acidity, respectively in fresh fruits of *S. domestica* and those after storage.

**Table 2. Content of dry material, reducing sugars, and titratable acidity of *S. domestica* fruits.**

<table>
<thead>
<tr>
<th>Extract</th>
<th>Dry material (%)</th>
<th>Reducing sugars (mg/dm³)</th>
<th>Titratable acidity (g/100g fruit)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh fruits extract (1)</td>
<td>9</td>
<td>62,6</td>
<td>11,685</td>
</tr>
<tr>
<td>Stored fruits extract (2)</td>
<td>7,5</td>
<td>60,6</td>
<td>11,439</td>
</tr>
</tbody>
</table>

Fresh fruits of *S. domestica* are hard and have a yellowish color, shows specific fruity ester like aromas. Storage at room temperature is associated with softening, darkening of their skin, as well as a certain transformation and loss of specific aroma. The data from Table 2 testify that the storage does not let to an increase in the content of reducing sugars. At the same time, is observed a decrease in total acidity of approximately 42.8%. From the above it can be concluded that the storage of fruits before processing does not lead to positives and such should be avoided.

The concentration of ethanol in fractions collected during the distillation mainly depends on initial alcohol content of the distilled product, as well as on the volume of collected fractions. Regardless of the scheme of fruit processing (Series A, B or C) there is no significant changes observed in the dynamics of the ethanol release, and the distillation curves in all three Series are similar.

The collected that a is set out in Fig. 1. Distillation begins with accumulation of fraction 1 (first of the head fractions) per each Serie, in which the alcohol level is higher in A1 - 81.48 % alcohol by volume, followed by C1 - 80.91 vol.%, and with the lowest alcohol content is fraction B1 - 72.97 % by volume. A significant difference in the last fractions (tails) is not seen in Serie A and B, with respectively 44.23 vol.% and 49.61 vol.% both are the last 8th fraction. Serie C has only 5 fractions and the last one has an alcohol content of 23.07 vol. %.

The esters in spirits are main yeast by-product. This could be a reason to explain why in Serie C - obtained without alcoholic fermentation of fruits, the content of esters is lower than their concentration in fractions of Serie A and B.

Another interesting fact is that the distillation with hard particles (Serie B) did not lead to esters concentrations higher than these in fractions of Serie A, obtained by distillation only of liquid phase.
Similar tendency could be observed for concentrations of higher alcohols in the fractions (Fig. 3). Content of higher alcohols in Serie C is lower than these in Serie A and B.

Concerning the aldehydes in the fractions (Fig. 4) there are differences, both in Serie C and between the values in Series A and B. The fruits of S. domestica contain relatively high percentage of seeds. This has led to increased tones of bitter almonds, in the same time the distillation with hard particles contributes to intensify fruit-ester aromas. These organoleptic perceptions of distilled beverages also confirm the significantly higher content of aldehydes in fractions of Serie B, most probably due to the higher content of benzaldehyde contained in the seeds of S. Domestica.

The content of esters and aldehydes in the initial fractions of Series A are significant. The number of esters in A1 and A2 is 3687.992 mg/dm$^3$ and 3252.34 mg/dm$^3$, and the aldehydes respectively 543.75 mg/dm$^3$ and 617.41 mg/dm$^3$ for the respective fractions. In the tail fraction A8, the test compounds were detected as traces with values of 29.034 mg/dm$^3$ Esters and 12.32 mg/dm$^3$ aldehydes.

In Serie B (distilled beverage obtained by fermentation of fruits and distillation of fruit mash) is observed the same tendency of distribution of the quantities of substances in the fractions. The content of esters in the initial (head) fractions is B1 - 2903.208 mg/dm$^3$ and in B2 - 2642.44 mg/dm$^3$, the quantity of aldehydes in the same two fractions is respectively 1545.77 mg/dm$^3$ and 1761.23 mg/dm$^3$. The quantitative analysis of the compounds shows higher values in tails fraction of Serie B than in Serie A (distilled beverage obtained by fermentation of fruits and distillation of liquid phase). The quantity of esters in fractions B7 and B8 is as follows 81.312 mg/dm$^3$ and 63.848 mg/dm$^3$, while the aldehydes are 78.36 mg/dm$^3$ and 51.99 mg/dm$^3$ for the same fractions.

In C Serie which is obtained by extraction of non-fermented fruits of S. domestica with water-alcohol mixture and following distillation, the quantitative values are few times lower of observed Series A and B. It the head fraction C1 - esters are 464.22 mg/dm$^3$ and aldehydes have the amount of 285.96 mg/dm$^3$. On the other hand, the number of esters in the tail fractions is three times higher than in the previous two Series A and B, 116.16 mg/dm$^3$ in C4 and 135.21 mg/dm$^3$ in C5. The aldehydes in those two fractions are 34.66 mg/dm$^3$ and 20.76 mg/dm$^3$.

The quantity of methanol in the distillate fractions (Fig. 5) will mainly depend on its initial amount, formed due to the action of the enzyme - pectin esterase, contained in the fruits themselves. Production of methanol could be also connected with microorganisms producing pectinases such as yeasts, bacteria and fungi [6]. Logically, in Series obtained without fermentation and soaking of fruit in alcohol, the denaturation of the enzyme leads many times lower values (Series C).
It would also be logical to observe a higher methanol content when distillation is with solid particles. The data in fig. 5 show a higher content in the fractions of Serie A than in Serie B. The final concentration of methanol in the beverage should also take into account the fact that in the specific processing schemes dilution was performed by addition of water and sugar.

According to this research, it has become clear that distillates from Serie B show between two- and three-times lower amount of methanol than distillates from Serie A, in the same fractions. For example, the value of methanol in B1 (1975 mg/dm³) compared to A1 (2936 mg/dm³) is 1.5 times lower, and in B8 (204 mg/dm³) is twice lower than in A8 (404 mg/dm³). The distilled fractions of Serie C show values of methanol almost the same as these in last fractions of Series A and B. This is due to the method by which the distilled material was produced. For example, the head fraction C1 contain 415 mg/dm³ of methanol and the tail fraction C2 contain 74 mg/dm³.

4 Conclusions

The results of the present work should rather be considered as a starting point for future in-depth studies on the composition of S. domestica fruits and the possibilities of their use for distillate beverages. The species is wild and respectively untreated with pesticides, as a result of which the alcoholic extraction of the fruits and subsequent distillation is an option that leads to a fine aroma in the distillates and at the same time contributes to low methanol and aldehydes concentrations.

The method with alcoholic fermentation and subsequent distillation, in terms of a finer aroma and unobtrusive aldehyde nuances, will probably be more successful by separating the hard particles after fermentation and distilling only the liquid phase.

References

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