Slovak Journal of Food Sciences

Potravinarstvo Slovak Journal of Food Sciences
vol. 14, 2020, p. 1191-1199
https://doi.org/10.5219/1472
Received: 30 September 2020. Accepted: 3 December 2020.
Available online: 28 December 2020 at www.potravinarstvo.com

ISSN 1337-0960 (online)

A CASE STUDY COMPARING DISTILLATION TECHNOLOGIES FOR PLUM PALINKA PRODUCTION

Péter Korzenszky, Gábor Barátossy, László Székely, Gábor Géczi

ABSTRACT

Palinka production has a long tradition in Hungary and the neighboring countries. Previously, the fruit distillate was produced exclusively using the traditional Pot-Still Double Distillation (PSDD) technology. This distillation method means, in practice, a simple fractional distillation repeated twice. However, in other industries, such as the petroleum industry or the pharmaceutical industry, a continuous, so-called repeated distillation procedure is used (RCDS – Rectification Column Distillation Systems). In the production of palinka, the latter procedure has gained more and more ground in recent years, thus displacing the traditional technology. In the territory of today's Hungary, there are more than 16,000 registered private palinka distillers. However, based on public databases, it is not possible to know the proportion of the two different palinka making processes used in palinka production. The two processes differ to a large degree. The amount of hearts obtained using the continuous operation plate rectification column (RCDS) is lower, while its alcohol content is very high: 75 – 90 vol%, depending on the fruit. On the other hand, when using the traditional pot-still double distillation (PSDD) method, the amount of hearts is higher, but its alcohol content is lower (60 – 70%). The continuous procedure, also called single-stage, is faster. This is one of the reasons for its popularity because it makes production more economical. The objective of our research was to find out whether a significant difference could be detected between the two plum palinkas produced using the two different distillation technologies, based on current legal requirements. Our research also included sensory testing to determine whether consumers could distinguish between the products manufactured in different ways. Our analyses were carried out in 2019 in the accredited laboratory of the National Food Chain Safety Office and among the students and staff of the Gödöllő campus of Szent István University.

Keywords: plum palinka, distillation, pot-still double distillation, rectification column

INTRODUCTION

According to the current regulation, only alcoholic beverages prepared from fruit produced in Hungary, by fermentation and distillation and with an alcohol content between 37.5% and 86% (V/V%) can be called palinkas **(László, Hodúr and Csanádi, 2016; Panyik, 2018)**. The name palinka can also be used for beverages produced in four Austrian provinces from apricots. However, distillation is also used in other countries to produce alcoholic products: brandy, spirit schnaps, obstbrand (fruit-based), vodka (corn, potatoes), whiskey (grain), borovička (juniper), cachaca (sugar cane juice), rum (cane sugar), tequila (blue agave), mezcal (agave), poitin (barley malt), baijiu (sorghum, rice). Alcoholic drinks, made by fermentation and distillation from fruits, mainly from plums, are very popular in Europe, primarily in Slovakia (*Slivovica*), the Czech Republic (*Slivovice*), Poland (*Sliwowica*), Serbia (*Prepečenica and Sljivovica*), Romania (*Tuica*) and Hungary (*Szilvapálinka*) **(Portugal, et al., 2016; Śliwińska et al., 2016; Satora and Tuszyński, 2008; Zheng et al., 2014)**.

Due to the technology, the quality of palinka is influenced by three well-distinguishable stages: the fruit itself (its variety, state of ripeness, and date of harvest); the mashing procedure, and, finally, the distillation technology. Storage and consumption habits may also be influencing factors, but experts agree that the three areas listed are dominant. In the course of our research, distillation technology has been analyzed. Distillation is a separation technology process in which the volatile components that enter the vapor phase during the evaporation are separated from the liquid phase. This is followed by the condensation of the generated vapors and re-liquefaction. The composition of the condensate formed after recooling the vapors that formed during the distillation of the mash is different than that of the mash since the mash does not contain only volatile compounds. The different components are found in higher concentrations in the distillate than in the mash, depending on their volatility. Distillation has a dual purpose during the brewing of palinka. On the one hand, the extraction of the alcohol content of the mash, and on the other hand, the separation of undesirable volatile components present in the mash from the precious hearts, by including them in the heads and the tails **(Nagygyörgy, 2010)**.

The traditional Hungarian method is considered to be the double distillation performed in pots, and this is commonly called pot-still technology. By definition, pot-still technology is brewing in an apparatus that has a pot with a volume of no more than 1,000 liters. The pot-still technology begins with the distillation of the mash that has a relatively low alcohol content (2 to 10%). The first distillate (brute alcohol) has an alcohol content of 15 to 30%, depending on the fruit and the apparatus. The second step is the refining of the brute alcohol, that is, the increase of the alcohol concentration to 60 – 70%. At the same time, the heads and the tails are also separated. PSDD (Figure 1a) in the breweries is usually carried out in two separate pots, mainly due to excise regulation and economic operation reasons. However, technically, the second distillation can be carried out in the same pot, but this is most typical of home brewing.

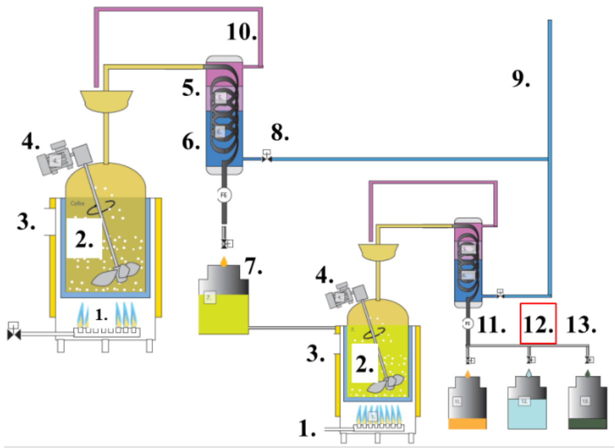
The other technology, which is gaining more and more ground due to Austrian and German influences, is continuous distillation based on column or tower apparatuses (RCDS). In the case of the RCDS technology, multiple distillations can occur in the distillation column. The operating principle is based on having the upward flowing vapors containing volatile substances meet a downward liquid stream while ensuring an adequate exchange of heat and material. The meeting of the vapors and the reflux liquid takes place on the plates in the column. Going up, the alcohol and other volatile component content of the vapor increases and, after condensation of the vapor, a distillate with an ethyl alcohol concentration of 70 to 90% can be obtained. (Figure 1b). In tower systems, depending on the design, 4 to 6 rectifications are carried out, but the separation of the heads and the tails are realized here as well in the final distillate (condensate) **(Nagygyörgy, 2010; Balcerek et al., 2017, Géczi, Korzenszky and Nagygyörgy, 2018)**.

From a palinka brewing point of view, one of the most important points of distillation is the sharp separation of the heads. According to the theory, the amount of the heads is appropriate if the highest possible amount of aroma, characteristic of the fruit, is transferred to the hearts, but unfavorable components, mainly ethyl acetate, are only included in the hearts in such small amounts that they do not cause sensory faults. After the separation of the heads, distillation is continued by the collection of the hearts. The ethyl alcohol content of the vapor decreases continuously and, at the same time, the concentration of the necessary aromas decreases as well, and if distillation is continued, the condensate will have an undesirable, unpleasant sour taste. So the characteristics of the heart are influenced not only by the distillation apparatus but also by the selection of the heads and tails cut points. Palinka suitable for consumption can then be obtained by rest and dilution with softened water **(Nagygyörgy, 2010)**.

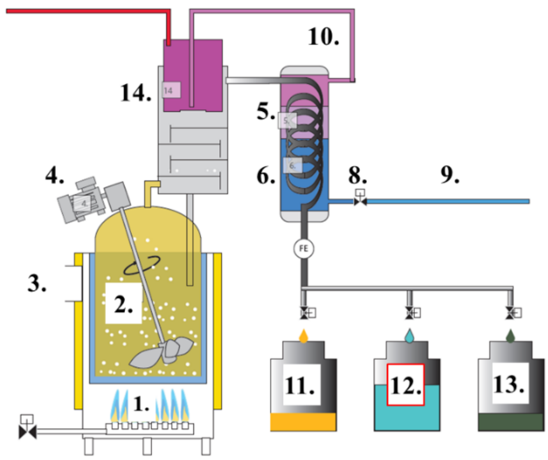
To improve the quality of palinka, research into palinka distillation is ongoing, with the number of papers increasing significantly in recent years, although being still less than the amount of scientific publications investigating the quality of beer and wine products.

The determination of the cut points is clearly of great importance, and a numerical method to predict cut-points was developed by **Gössinger et al. (2012)** of Austria for apple distillates. For the more accurate identification of the cut points, research has been focusing on the determination of the volatile components characteristic of the fruit. **Rodriguez-Solana et al. (2018)** performed the analysis of fig distillates popular in Mediterranean countries. 130 volatile compounds were identified in the fig distillates, including, as common constituents, ethyl decanoate, ethyl octanoate, and ethyl dodecanoate, the aldehydes benzaldehyde and furfural, the monoterpene limonene, and the norisoprenoid β-damascenone. Knowledge of the volatile components also allows for objective control of the market, which is a part of the fight against counterfeiting as well.

Claus and Berglund (2005) showed in the case of a tower system technology that the plate number and dephlegmation (reflux) play significant roles. The concentrations of ethanol and related compounds, such as methanol, acetaldehyde, ethyl acetate, 1-propanol, and



a)



b)

Figure 1 The two typical distillation technologies.
Note: a) Schematic representation of the pot-still technology (PSDD); b) Schematic representation of the rectification column technology (RCDS). 1 – heating; 2 – mashing; 3 – flue gas discharge; 4 – stirring; 5 – condensation; 6 – cooling; 7 – first distillation; 8 – flow measurement; 9 – cooling media IN; 10 – condenser outlet; 11 – heads; 12 – hearts; 13 – tails; 14 – distillation tower.

isoamyl alcohol in the final product were affected by the plate number.

The effect of dephlegmation was also investigated by **Rodríguez-Bencomo et al. (2016).** The results showed that a high reflux ratio at the beginning of the distillation is a good way to reduce the amount of unpleasant compounds in the hearts. **Nagygyörgy (2016)** paid great attention to the research of the effect of dephlegmation. It was determined by him that dephlegmation is primarily determined by the surface of the dephlegmator, the temperature of the vapor tube, and the intensity of heating. In addition to research into cut points and components that determine advantageous quality, there has been a strong focus on studies investigating the legal background of palinka production and the effects of changes in it **(Zsótér and Molnár, 2015)**. **Deák et al. (2010)** stressed the importance of ethyl carbamate detection in their article. Ethyl carbamate (EC, urethane, $C_2H_5OCONH_2$) is a genotoxic carcinogen and is regularly found in fermented foods, including alcoholic beverages **(Monakhova, Kuballa, Lachenmeier, 2012).** The target value for ethyl carbamate according to the recommendation of the European Food Safety Authority (EFSA) is 1 mg.L^{-1}. For a rapid determination of the components, **Śliwińska et al. (2016)** drew attention to the suitability of the electronic nose.

The importance of the cut points and product components is not disputed by anyone, and many professionals see technology development as the solution. A pervaporation membrane technology for the production of apple palinka was developed by **Molnár, Márki and Vatai (2016)**, which is energetically more efficient than the traditional pot-still technology.

García-Llobodanin et al. (2008) examined the concentration and quality of the raw material placed in the still and, regarding pear distillates, found that using natural pear juice does not offer any benefit when compared to using concentrated pear juice from Blanquilla variety pears.

Balcerek et al. (2017) investigated the effect of pot-still and rectification column distillation technologies on the hearts, namely the distribution of the volatile matter content and the concentrations of undesirable compounds (methanol, hydrogen cyanide, ethyl carbamate) in plum palinka. Irrespective of the distillation method used, the heads contained mainly aliphatic aldehydes, acetals, and esters, as well as higher alcohols (1-propanol, 2-methyl-1-propanol, 1-butanol, 2-methyl-1-butanol, and 3-methyl-1-butanol). Increasing the alcohol concentration in the hearts from 70% to 90% resulted in the gradual decrease in the concentration of all detected volatile compounds. Compared to the pot-still technology, single-stage distillation (tower system) resulted in hearts with lower concentrations of acetaldehyde and benzaldehyde. In the case of the pot-still technology, a statistically significant increase in the amount of methanol and ethyl carbamate in the hearts was observed.

Technological, as well as tourism issues, are discussed by **Harcsa (2017a)** and **Harcsa (2017b)**, in his research aimed at the comparison of the energy consumptions of the two different systems (pot-still and tower).

Kovács et al. (2018) stressed that the quality of fruit-based alcohol content varies from year to year; therefore, it is necessary to identify the vintage of distilled alcoholic beverages. They found that three ingredients are associated with the vintage, regardless of the type of fruit: propanol, butanol, and ethyl propionate.

It was mentioned in the introduction that some of the most popular distillates in neighboring countries are made from plums, and based on this it is understandable that plum palinka is a popular research subject. **Jakubíková et al. (2019)** showed differences between the origin and harvest time of raw materials by examining the phenol and anisole compounds of plum palinkas. **Satora et al. (2017)** examined the quality of distillates prepared from four varieties of plums and found that the aroma of the samples is determined by six compounds (ethyl dodecanoate, benzyl acetate, methyl cinnamate, 1-heptanol, α-terpineol and benzothiazol).

Pecić et al. (2012) found that the ageing of Serbian plum palinka in wooden casks not only improves organoleptic characteristics, but also changes the total polyphenol content and contributes to other important health properties, such as the increase in their antioxidant capacity.

As early as 1980, **Ismail, Williams and Tucknott (1980)**, identified 54 constituents based on the gas chromatographic and mass spectrometric analysis of volatile compounds in the distillate of fermented plum leaves, including 11 hydroxy compounds, 1 acid, 30 esters, 4 carbonyl compounds, 3 lactones, and 5 acetals. Thirty of these compounds had not been previously reported in fermented plum products. Benzaldehyde, linalool, methyl cinnamate, and y-decalactone, based on gas chromatographic odor assessment, are believed to contribute significantly to the plum character of this beverage. However, in 1982, **Velíšek et al. (1982)** pointed out that, in the Czechoslovakia of the time, when examining the volatile taste components of plum palinka, higher aliphatic aldehydes (nonanal and some others), 2-undecanone, benzaldehyde, damascenone, benzyl acetate, ethyl phenylacetate, phenyl ethyl acetate, ethyl 3-phenyl propionate, methyl cinnamate, and ethyl cinnamate were the most significant contributors to the typical plum palinka aroma.

Sádecká et al. (2016) used synchronous fluorescence spectroscopy, combined with principal component analysis (PCA) and linear discriminant analysis (LDA), to distinguish plum distillates according to their geographical origins. A total of 14 Czech, 12 Hungarian, and 18 Slovak plum distillate samples were used.

Jurica et al. (2016) investigated the occurrence of phthalates during the production of plum distillates and in the plum distillate final product manufactured by registered producers in five European countries using gas chromatography-mass spectrometry (GC-MS). As the distillation process progressed, a decreasing trend was observed for the mean values of diethyl phthalate (DEP), diisobutyl phthalate (DiBP), and di-n-butyl phthalate (DBP).

Satora and Tuszyński (2008) found that homemade Polish plum palinka usually contained more ethanol (64.7 – 72.5 vol%), methanol (5.59 – 8.74 g.L^{-1} AA) and butanol (32 – 335 mg. L^{-1} AA) and less isobutanol (406 – 491 mg. L^{-1} AA). The results showed that plum palinkas produced in the Łacko area are characterized by similar and original chemical compositions, which come mainly from spontaneous fermentation, as well as the traditional production technology.

The importance of plum palinka in Hungary is demonstrated by the fact that in 2013 it was included on the list of Hungarikums. Hungarikums are products, foods, or values that represent Hungarian traditions with their properties, uniqueness, specialty, and quality **(Harcsa, 2018)**.

Fodor, Hlédik and Totth (2011) demonstrated that the popularity of palinka is on the rise, but consumers are choosing well-known brand names. The role of the regions can also be observed in Hungary in connection with plum palinka, indicating a certain quality: plum palinka of Békés and Szatmár **(Kassai et al., 2016)**. Food safety is becoming increasingly important among consumers. According to a survey of Slovak researchers, in Slovakia, 84% of respondents buy higher-quality foods **(Nagyová et al., 2019)**.

A large scale questionnaire survey was conducted by **Szegedyné Fricz et al. (2017)** to evaluate consumer behavior. Their conclusion was, among other things, that a significant proportion of consumers do not have even the slightest knowledge regarding the production of palinka.

Scientific hypotheses

Hypothesis No. 1: The instrumentally measurable content values, based on legal obligations, of final products prepared by various distillation technologies from the same batch of raw materials with identical preparation procedures are different.

Hypothesis No. 2: The final products prepared by various distillation technologies from the same batch of raw materials with identical preparation are different based on sensory evaluation.

MATERIAL AND METHODOLOGY

Materials

The raw material used in the study was an 18 Brix% (g sugar/100 g mash) mash made from President (*Prunus domestica President*) and Stanley (*Prunus domestica Stanley*) plums harvested in mid-September 2018. President plum is a slightly elongated, very large (45 – 55 g), hard, freestone, sweet variety with a purple-red skin, yellow flesh, and a pleasant taste. Stanley plum is one of the most widespread varieties in the world, it is large (diameter 34 – 36 mm), its peel is dark blue, strongly waxy, the flesh is yellowish-green, tasty, juicy, freestone. (Figure 2) Due to the unusually warm weather, the ripening period of the two varieties was longer, making it possible to harvest the required amount of both varieties at the same time at their optimal stage of ripeness.

Preparation

The mashing procedure took place in Bózsva, at the premises of Tiszta Gyümölcs Kft. According to the planned program, 500 kg of pitted plums were fermented at 18 °C in a stainless-steel tank that could be cooled and was equipped with a stirrer for 21 days. At the same time when the precooled raw material was placed in the tank, Lallyzim HC pectin breakdown mixture was added to the raw material. The pH was adjusted (pH = 3.2) with a 10% solution of citric acid, and inoculation was performed by the addition of Uvaferm 228 yeast and Uvavital yeast nutrient.

After the fermentation of the mash was over, the raw material was homogenized by stirring, and then it was divided into two parts. Tower system (RCDS) distillation was carried out at the same site in Bózsva, using the German-made Christian Carl equipment of Tiszta Gyümölcs Kft., and the plum palinka thus obtained was marked I. The location of the pot-still technology was the brewery of the Veresegyház Palinka House, where the distillation was carried out using the Czech-made Kovodel Janca s.r.o. double-still (PSDD) equipment and the final product thus obtained was marked II.

Due to the various distillation processes, the products were available with different alcohol concentrations. In the case of the pot-still technology, the alcohol content of the hearts was 66.9 vol%, while in the case of the column technology it was 84.4 vol%.

The final products were prepared by adjusting the alcohol concentration to the planned value of 44 vol% with the same distilled water.

This way, 24.1 liters of product were obtained during pot-still distillation and 23.5 liters using the tower technology. Analytical tests were performed in February 2019, while sensory evaluations took place in October 2019, when the plum palinka was 1 year old.

President Stanley

Figure 2 The plums included in the study (*Prunus domestica*).

Table 1 The parameters measured in the tests and their measurement methods.

Parameter	Measurement method	Measurement technique	Limit of quantification	Regulatory limit value
Actual alcohol	IR-1:2000	Anton Paar, Alcolyzer	1.00%vol.	37.5% – 86%
Acetaldehyde	Regulation (EC) No 2870/2000*	GC	3.7 mg.L^{-1}	no limit value
Allyl alcohol	Regulation (EC) No 2870/2000*	GC	2.0 mg.L^{-1}	no limit value
Methanol	Regulation (EC) No 2870/2000*	GC	2.0 mg.100cm^{-3}	1200 g.hL^{-1} AA
Propanol	Regulation (EC) No 2870/2000*	GC	1.6 mg.L^{-1}	no limit value
2-Butanol	Regulation (EC) No 2870/2000*	GC	0.7 mg.L^{-1}	no limit value
2-Methyl-1-propanol	Regulation (EC) No 2870/2000*	GC	1.3 mg.L^{-1}	no limit value
n-Butanol	Regulation (EC) No 2870/2000*	GC	1.9 mg.L^{-1}	no limit value
2-Methylbutanol	Regulation (EC) No 2870/2000*	GC	0.8 mg.L^{-1}	no limit value
3-Methylbutanol	Regulation (EC) No 2870/2000*	GC	2.0 mg.L^{-1}	no limit value
Volatile matter	Regulation (EC) No 2870/2000*	Calculated value	2.0 mg.100cm^{-3}	min.: 200 g.hL^{-1}AA
Ethyl acetate	Regulation (EC) No 2870/2000*	GC	1.7 mg.L^{-1}	no limit value
Acetal	Regulation (EC) No 2870/2000*	GC	2.9 mg.L^{-1}	no limit value
Total hydrogen cyanide	MSZ 9589-12:2013	titrimetry	1 mg.100cm^{-3}	7 g.hL^{-1}AA
Ethyl carbamate	CEN/TC 275/WG 13:2012	GC-MS	0.41 mg.L^{-1}	no limit value

Note: *Method III.2 of **Regulation (EC) No 2870/2000** for alcoholic beverages (distillate).

Analytical test

Laboratory analysis of samples was performed by a blind test in the testing laboratory of the Directorate of Oenology and Alcoholic Beverages of the National Food Chain Safety Office, accredited under reg. no. NAH-1-1673/2015, under current legal regulations. 15 parameters were tested for both samples with multiple replicates. The tested parameters, measurement methods, descriptions of the measurement techniques, limits of quantification, and regulatory limit values are given in the following table (Table 1). Sampling and sample preparation was carried out by qualified personnel.

Organoleptic tests were carried out according to Hungarian Standard MSZ 9600:2016 Guidelines for sensory analysis of spirit drinks.

Organoleptic test

Tests were carried out among the students and staff of the Gödöllő campus of Szent István University, in groups of 15 – 25 people, on 18 occasions in October 2019, in the Food Technology and Machines Laboratory of the university. All participants were occasional palinka consumers who participated in the study voluntarily. Test conditions (room temperature, palinka temperature) were the same in all cases so that external parameters did not affect the results.

During the organoleptic tests, samples were marked only with labels suitable for distinction („I" and „II"), test subjects did not know which mark meant what. For the sensory examination, samples with a volume of 2 cl were provided. A total of 341 people participated in the sensory examinations, all of them only once.

In each case, during the sensory evaluation, respondents had to answer the question: „Do you feel a difference between the two samples?" Those who did feel a difference, had to answer the question: „Which palinka sample tasted better?" The questionnaire included a minimal number of demographic questions regarding the gender and age of the respondents.

Statistical analysis

Statistical analysis was performed based on the data obtained during the laboratory tests. The difference in the variance of the two-sample series was checked by the *F*-test. Since there was no significant difference between them two-tailed independent sample *t*-test was used to determine if there is a significant difference between any of the examined parameters. We say that the difference is significant if the corresponding *p*-value $p < 0.05$ holds.

Questionnaires of the sensory examinations were summarized using Excel. As a further analysis, we examined whether the choice of palinka of our sample population depends on gender or age. To this end, contingency tables were applied. The dependencies were examined by using Pearson's χ^2-test, furthermore, Goodman, and Kruskal's λ value was calculated to measure the strength of the association.

Our data analysis was performed using the data analysis module (Analysis ToolPak) of Excel and IBM SPSS 25.

RESULTS AND DISCUSSION

A test report was compiled on the measurement results of the different plum palinka samples analyzed in the accredited testing laboratory. Available data were processed and evaluated using the statistical methods described.

The following table lists the 15 parameters tested by the laboratory and the average values of 10 measurement series for both samples (Table 2). For the anonymous identification of the samples during the laboratory tests, marks of I and II were used. Sample I marked the final product of the tower distillation apparatus, while sample II meant the plum palinka sample made with the traditional pot-still technology.

Based on the statistical analysis it can be stated that our hypothesis No.1 must be rejected because no significant differences ($p < 0.05$) could be detected between the two samples in the statutory palinka quality parameters of the final products prepared from the same batch of raw material and the same preparation using various distillation technologies.

Table 2 Statistical results.

Measured parameter	Expected value of sample I	Expected value of sample II	p value of *F*-test	p value of *t*-test
Actual alcohol [%]	44.08 ±0.23	44.00 ±0.34	0.2670	0.5461
Acetaldehyde [mg.L^{-1}]	69.1 ±4.56	64.3 ±5.14	0.7301	0.0506
Allyl alcohol [mg.L^{-1}]	<LOQ*	<LOQ*	–	–
Methanol [mg.100cm^{-3}]	807.1 ±51.43	803 ±53.05	0.9278	0.8696
Propanol [mg.L^{-1}]	920.2 ±108.7	831.8 ±68.88	0.1902	0.0540
2-Butanol [mg.L^{-1}]	6.82 ±2.29	8.02 ±0.39	0.3781	0.1400
2-Methyl-1-propanol [mg.L^{-1}]	179.2 ±13.30	192.3 ±14.13	0.8594	0.0580
n-Butanol [mg.L^{-1}]	4.5 ±0.31	4.5 ±0.27	0.6959	1
2-Methylbutanol [mg.L^{-1}]	149.7 ±9.74	158.7 ±9.46	0.9316	0.0623
3-Methylbutanol [mg.L^{-1}]	637.8 ±40.1	643.4 ±40.5	0.9731	0.7717
Volatile matter [mg.100cm^{-3}]	480.4 ±30.08	484.4 ±29.74	0.9734	0.7799
Ethyl acetate [mg.L^{-1}]	131.1 ±9.41	138.6 ±5.12	0.0846	0.0515
Acetal [mg.L^{-1}]	37.0 ±9.23	44.6 ±5.92	0.2021	0.0522
Total hydrogen cyanide [mg.100cm^{-3}]	<LOQ*	<LOQ*	–	–
Ethyl carbamate [mg.L^{-1}]	<LOQ*	<LOQ*	–	–

Note: <LOQ* – Limit Of Quantification.

The actual alcohol content values of the plum palinka samples included in the study met the minimum value of 37.5% specified in **Regulation (EC) No 110/2008**, based on the measurements the alcohol content of the distillates were 44.08 ±0.23% and 44.0 ±0.34%, respectively.

The presence of large amounts of acetaldehyde can lead to headaches and nausea. In the samples tested by us, acetaldehyde was detected in amounts of 69.1 ±4.56 and 64.3 ±5.14 mg.L^{-1}, respectively. This is in contrast to the scientific results of **Balcerek et al. (2017)** who obtained significantly different values when comparing the two technologies.There is no limit value for this component in the regulation.

Allyl alcohol was present in the samples tested in concentrations below the limit of quantification of 2 ±0.2 mg.L^{-1}.

Methanol is converted by our bodies to formaldehyde, which is toxic and can cause blindness or even death in large quantities. In the case of the samples tested, it could be detected in amounts of 807.1 ±51.43 mg.100cm^{-3} and 803 ±53.05 mg.100 cm^{-3}, while its legal limit value is 1.200 g.hL^{-1} absolute alcohol in the case of plum distillates.

Fusel alcohols, such as propanol, butanol, and methyl butanol, are formed during fermentation, and they have a solvent odor and taste. Depending on their quantities, they may cause opacification during reconstitution. The esters of these higher alcohols with organic acids are pleasant, desirable aroma components in the distillate. Propranol and butanol are suitable for determining vintage **(Kovács et al., 2018)**, however, no difference between the technologies was found. There are no specific limit values in the regulation for the individual components. However, the limit value for the volatile matter content, which is associated with it, is set at 200 g.hL^{-1} of absolute alcohol. In the case of the plum palinka samples included in the study, the volatile matter content values were 480.4 ±30.08 and 484.4 ±29.74 mg.100cm^{-3}, respectively.

The amounts of the components ethyl acetate and acetal are not legally regulated.

Research has shown that in the case of omitted or poorly performed pitting, more hydrogen cyanide can be present in the palinka than the permissible amount, which is detrimental to health. The legal upper limit is 7 g.hl^{-1} absolute alcohol. In the case of the samples analyzed by us, the amount of hydrogen cyanide was below the limit of quantification of 1 ±0.1mg.100cm^{-3} in all cases.

Carcinogenic ethyl carbamate may form from components added as unsuitable yeast nutrients **(Deák et al., 2010; Monakhova, Kuballa and Lachenmeier, 2012)**. The regulation does not set a limit value for it, but based on the recommendation of EFSA, it is proposed that it is kept below a value of 1 mg.L^{-1}. The presence of this component could not be detected in the samples analyzed. The limit of quantification for ethyl carbamate was 0.41 ±0.041 mg.L^{-1}.

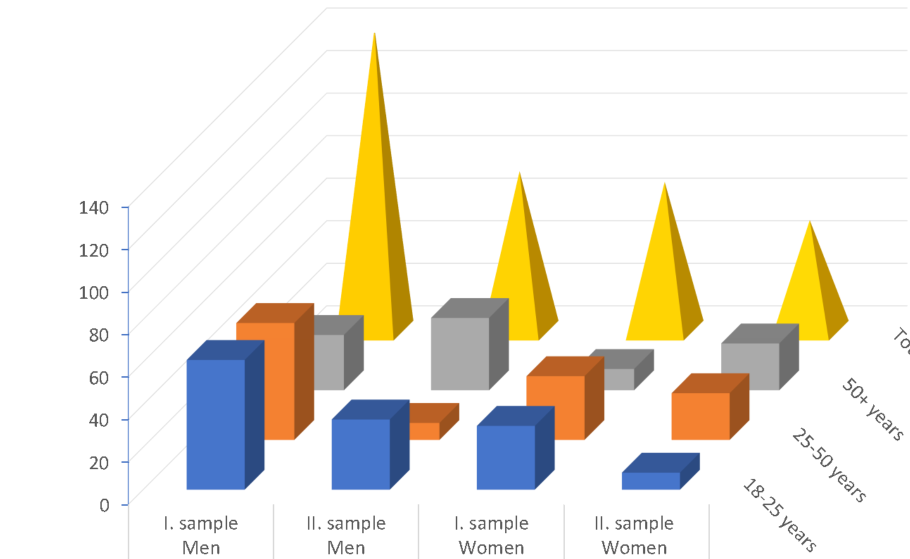
Emphasizing the importance of the testing of 15 components, the presence of propanol specifically indicates the presence of the fruit raw material, while its absence can be an indicator of counterfeiting, for example, by grain alcohol.

Legal regulations are always aimed at producing safe and healthy food. It is necessary to measure the components of the distillate and to set limit values, as exceeding them can either initiate severe irreversible processes in the human body or even cause death. Even though there are no specified limit values for fusel alcohols, however, it has been mentioned that their excessive presence gives the distillate a pungent odor and an unpleasant aftertaste. Our analyses revealed that, in the case of the samples tested by us, each batch complied with the legal requirements, i.e., the foods were safe.

However, based on previous research by **Satora et al. (2017)**, **Ismail et al. (1980)**, **Velíšek et al. (1982)**, measurements that meet the legal requirements do not include components that determine the character of plums.

Results of sensory tests

The opinions of consumers were tested by sensory analyses and this was not difficult since it was also found by **Fodor, Hlédik and Totth (2011)** that the popularity of palinka is increasing. We have 341 questionnaires completed in October 2019 at our disposal on the sensory comparison of plum palinkas produced in 2018.



	I. sample Men	II. sample Men	I. sample Women	II. sample Women
18-25 years	61	33	30	8
25-50 years	55	8	30	22
50+ years	26	34	10	22
Total	142	75	70	52

Figure 3 Results of the palinka test.

64% of respondents were male, 36% were female; in terms of age distribution, 34% were between 25 and 50 years old, 39% were between 18 and 25 years old, typically college students, while 27% were over 50 years old. The results of the questionnaire survey are shown in Figure 3.

As a result of the study it can be stated that in the comparative organoleptic study of the one-year-old products, 99.4% of the respondents (339 people) distinguished between the two palinka samples. There were only two people who found no difference between the samples. 63% of those who found a difference marked plum palinka I tastier, while 37% chose sample II. To examine that the choice depends on gender or age contingency tables (or crosstabs) were used. In terms of gender the p-value for Pearson's χ^2-test was $p = 0.141$, thus it can be stated that no difference was found between the choice pattern of the different genders. On the other hand, in the case of age groups for the p-value of Pearson's χ^2-test $p < 0.001$ holds, therefore the preference of palinka does depend on age, but this dependence is weak since the Goodman and Kruskal's λ value equals to 0.157. Although the pool of respondents cannot be considered representative, it can be stated that the sample produced using the pot-still technology (sample II) was chosen by the age group over 50, while the age group 25 – 50 found the sample prepared using the column technology tastier. According to our results, we can say as well, that young adults don't really have a clear preference of palinka with respect to the technology it was produced. A similar conclusion, that consumers are unaware of production technology processes, was reached by **Szegedyné Fricz et al. (2017)**. In Table 3 we present the corresponding contingency table. In this table, Count means the number of persons in a specific category while Expected Count shows the number of persons in the same category in the case when there would be no association between age and palinka choice. Too high difference between these values indicates that in this certain category age has an effect on palinka choice. Based on the answers given to question 1 during the sensory examinations, it can be stated that our hypothesis No. 2 can be retained, that is, the final products prepared using different distillation technologies from the same batch of raw material and the same preparation differ based on organoleptic evaluation. The answers to the second question do not explain the reason for the discrimination but confirm the difference. Naturally, another question arises as to the product made by which technology is found to be tastier by consumers, but the present case study does not examine this issue and our previous research **(Géczi, Korzenszky and Nagygyörgy, 2018)** has shown that during the relaxation and maturation of palinka, processes take place that influences the quality of palinka and the perception of being the tastier technology highly depends on the "age" of the product.

Taking into account the differences in sensory examinations, in order to compare the technologies, it would be necessary to measure the components characteristic of the product and to compare the degree of dephlegmation analyzed by **Nagygyörgy (2010)** and **Nagygyörgy (2016)**.

Table 3 Contingency table for age groups and palinka.

			palinka I	palinka II	Total
Age	18 – 25	Count	91	41	132
		Expected Count	82.5	49.5	132
	25 – 50	Count	85	30	115
		Expected Count	71.9	43.1	115
	50<	Count	36	56	92
		Expected Count	57.5	34.5	92
Total		Count	212	127	339
		Expected Count	212	127	339

CONCLUSION

Based on the measurement results it can be stated that there is no significant difference in the legally specified and tested 15 content values between the two plum distillates produced from the mash prepared from the same raw material and the same preparation technology but using two various distillation technologies. No differences could be detected between the final products prepared using the pot-still technology (PSDD) and the rectification column technology (RCDS) with accredited instrumental analyses, so our No.1 hypothesis was rejected.

However, in the case of organoleptic analyses, participants in the study made a clear distinction between the samples tested. In each case, they found one of the samples tastier than the other. It can be stated that the final products made using the two different distillation technologies can be clearly distinguished from each other based on organoleptic tests, but the analytical tests could not support this result. To support this, we consider it necessary to perform analytical determination of further components specific to plums. Legally prescribed qualification parameters generally do not include components specific to distillates made from the individual fruits, such as benzaldehyde, methyl cinnamate, and y-decalactone in the case of plums. Regardless, our hypothesis No. 2 was accepted. Consequently, we are developing new measurement and evaluation methods to investigate the cause of the difference.

As a result of our tests, it can be stated that the analytical parameters of the legal requirements are not suitable for the comparison of palinkas produced using various technologies.

REFERENCES

Balcerek, M., Pielech-Przybylska, K., Patelski, P., Dziekońska-Kubczak, U., Strąk, E. 2017. The effect of distillation conditions and alcohol content in ‘heart' fractions on the concentration of aroma volatiles and undesirable compounds in plum brandies. *J. Inst. Brew,* vol. 123, no. 3, p. 452-463. https://doi.org/10.1002/jib.441

Claus, M. J., Berglund, K. A. 2005. Fruit brandy production by batch column distillation with reflux. *Journal of Food Process Engineering,* vol. 28, no. 1, p. 53-67. https://doi.org/10.1111/j.1745-4530.2005.00377.x

Deák, E., Gyepes, A., Stefanovits-Bányai, É., Dernovics, M. 2010. Determination of ethyl carbamate in pálinka spirits by liquid chromatography–electrospray tandem mass spectrometry after derivatization. *Food Research International,* vol. 43, no. 10, p. 2452-2455. https://doi.org/10.1016/j.foodres.2010.09.014

Fodor, M., Hlédik, E., Totth, G. 2011. Consumer Opinions and Preferences on the Market of Palinka *Élelmiszer, Táplálkozás és Marketing,* vol. 8, no 1-2. p. 41-47. (in Hungarian)

García-Llobodanin, L., Ferrando, M., Güell, C., López, F. 2008. Pear distillates: influence of the raw material used on final quality. *Eur. Food Res. Technol.,* vol. 228, p. 75-82. https://doi.org/10.1007/s00217-008-0908-9

Géczi, G., Korzenszky, P., Nagygyörgy, L. 2018. Analytical and Sensory Testing of Palinkas Made with Different Distillation Technologies. *Hungarian Agricultural Research,* vol. 27, no. 3, p. 4-9.

Gössinger, M., Eitner, C., Vogl, K. 2012. Impact of several processing parameters on important parameters of counter-current distillation of apple mash. *Mitteilungen Klosterneuburg, Rebe und Wein, Obstbau und Früchteverwertung,* vol. 62, no. 1, p. 45-55.

Harcsa, I. M. 2018. From the „just for the naked public good" drink to the Hungaricum; the faith of the pálinka. (in Hungarian) *Régiókutatás Szemle,* vol. 1, 12 p. https://doi.org/10.30716/RSZ/2018/1/4

Harcsa, I. M. 2017a. Energy demand for pálinka production and some practical issues of waste treatment. *Economic and Regional Studies*, vol. 10, no. 3, p. 82-95. https://doi.org/10.2478/ers-2017-0027

Harcsa, I. M. 2017b. Increasing Palinka Recognition with tourism and gastronomy. *Applied Studies in Agribusiness and Commerce,* vol. 11, no. 3-4, p. 37-44. https://doi.org/10.19041/APSTRACT/2017/3-4/6

Ismail, H. M. M., Williams, A. A., Tucknott, O. G. 1980. The flavour components of plum. *Z Lebensm Unters Forch,* vol. 171, p. 24-27. https://doi.org/10.1007/BF01044413

Jakubíková, M., Sádecká, J., Hroboňová, K. 2019. Classification of plum brandies based on phenol and anisole compounds using HPLC. *Eur Food Res Technol,* vol. 245, p. 1709-1717. https://doi.org/10.1007/s00217-019-03291-3

Jurica, K., Brčić Karačonji, I., Lasić, D., Vukić Lušić, D., Anić Jurica, S., Lušić, D. 2016. Determination of phthalates in plum spirit and their occurrence during plum spirit production. *Acta Alimentaria*, vol. 45, no. 1, p. 141-148. https://doi.org/10.1556/066.2016.45.1.17

Kassai, Z., Káposzta, J., Ritter, K., Dávid, L., Nagy, H., Farkas, T. 2016. The Territorial Significance Of Food Hungaricums: The Case Of Pálinka, *Romanian Journal of Regional Science*, vol. 10, no. 2, p. 64-84.

Kovács, A. G., Szöllősi, A., Szöllősi, D., Panyik, I. A., Nagygyörgy, L., Hoschke, Á., Nguyen, Q. D. 2018. Classification and Identification of Three Vintage Designated Hungarian Spirits by Their Volatile Compounds. *Periodica Polytechnica Chemical Engineering,* vol. 62, no. 2, p. 175-181. https://doi.org/10.3311/PPch.11078

László, Z., Hodúr, C., Csanádi, J. 2016. "Pálinka": Hungarian Distilled Fruit. In Oliveira, J., Kristbergsson, K. *Traditional foods*. New York, USA : Springer, p. 313-318. ISBN 978-14899-76482. https://doi.org/10.1007/978-1-4899-7648-2_24

Molnár, M. A., Márki, E., Vatai, G. 2016. Preparation of apple spirit by ceramic pervaporation membrane. *Acta Alimentaria,* vol. 45, no. 4, p. 551-557. https://doi.org/10.1556/066.2016.45.4.12

Monakhova, Y. B., Kuballa, T., Lachenmeier, D. W. 2012. Rapid Quantification of Ethyl Carbamate in Spirits Using NMR Spectroscopy and Chemometrics. *International Scholarly Research Network Analytical Chemistry*, 5 p. https://doi.org/10.5402/2012/989174

MSZ 9600:2016. *Guidelines for sensory analysis of spirit drinks.*

Nagygyörgy, L. 2010. *Basics of palinka making*. Budapest, Hungary : Wessling International Research and Educational Centre, 130 p. ISBN 978-9630803229.

Nagygyörgy, L. 2016. The effect of dephlegmation on distillate composition. *Journal of Food Investigation* vol. 62, no. 4, p. 1261-1275.

Nagyová, Ľ., Andocsová, A., Géci, A., Zajác, P., Palkovič, J., Košičiarová, I., Golian, J. 2019. Consumers´ Awareness of Food Safety. *Potravinarstvo Slovak Journal of Food Sciences*, vol. 13, no. 1, p. 8-17. https://doi.org/10.5219/1003

Panyik, G. 2018. *Palinka Brewing - Making soaked brandy and liqueur*. Budapest, Hungary : Cser Kiadó. 116 p. ISBN 978-9632785462. (in Hungarian)

Pecić, S., Veljović, M., Despotović, S., Leskošek-Čukalović, I., Jadranin, M., Tešević, V., Nikšić, M., Nikićević, N. 2012. Effect of maturation conditions on sensory and antioxidant properties of old Serbian plum brandies. *Eur. Food Res. Technol.,* vol. 235, p. 479-487. https://doi.org/10.1007/s00217-012-1775-y

Portugal, C. B., Alcarde, A. R., Bortoletto, A. M., Paron de Silva, A. 2016. The role of spontaneous fermentation for the production of *cachaça*: a study of case. *Eur. Food Res. Technol.,* vol. 242, p. 1587-1597. https://doi.org/10.1007/s00217-016-2659-3

Regulation (EC) No 110/2008 of the European Parliament and of the Council of 15 January 2008 on the definition, description, presentation, labelling and the protection of geographical indications of spirit drinks and repealing Council Regulation (EEC) No 1576/89. OJ L 39. 13.2.2008, p. 16-54.

Regulation (EC) No 2870/2000 of 19 December 2000 laying down Community reference methods for the analysis of spirits drinks. Official Journal L 333, 29/12/2000 P. 0020 - 0046

Rodríguez-Bencomo, J. J., Pérez-Correa, J. R., Orriols, I., López F. 2016. Spirit Distillation Strategies for Aroma Improvement Using Variable Internal Column Reflux. *Food Bioprocess Technol.,* vol. 9, p. 1885-1892. https://doi.org/10.1007/s11947-016-1776-0

Rodríguez-Solana, R., Galego, L. R., Pérez-Santín, E., Romano, A. 2018. Production method and varietal source influence the volatile profiles of spirits prepared from fig fruits (*Ficus carica* L.). *Eur. Food Res. Technol.,* vol. 244, p. 2213-2229. https://doi.org/10.1007/s00217-018-3131-3

Sádecká, J., Jakubíková, M., Májek, P., Kleinová, A., 2016. Classification of plum spirit drinks by synchronous fluorescence spectroscopy. *Food Chemistry,* vol. 196, p. 783-790. https://doi.org/10.1016/j.foodchem.2015.10.001

Satora, P., Kostrz, M., Sroka, P., Tarko, T. 2017. Chemical profile of spirits obtained by spontaneous fermentation of different varieties of plum fruits. *Eur. Food Res. Technol.,* vol. 243, p. 489-499. https://doi.org/10.1007/s00217-016-2762-5

Satora, P., Tuszyński T. 2008. Chemical characteristics of Śliwowica Łącka and other plum brandies. *J. Sci. Food Agric.,* vol. 88, no. 1, p. 167-174. https://doi.org/10.1002/jsfa.3067

Śliwińska, M., Wiśniewska, P., Dymerski, T., Wardencki, W., Namieśnik, J. 2016. Evaluation of the suitability of electronic nose based on fast GC for distinguishing between the plum spirits of different geographical origins. *Eur. Food Res. Technol.,* vol. 242, p. 1813-1819 https://doi.org/10.1007/s00217-016-2680-6

Szegedyné Fricz, Á., Szakos, D., Bódi, B., Kasza, G. 2017 Palinka: consumer knowledge, preferences, consumption habits, marketing opportunities. *Gazdálkodás,* vol. 61, no. 2, p. 158-170.

Velíšek, J., Pudil, F., Davídek, J., Kubelka, V. 1982. The neutral volatile components of Czechoslovak plum brandy. *Z Lebensm. Unters. Forch.*, vol. 174, p. 463-466. https://doi.org/10.1007/BF01042726

Zheng, J., Liang, R., Huang, J., Zhou, R .P., Chen, Z. J., Wu, C. D., Zhou, R. Q., Liao, X. P. 2014. Volatile Compounds of Raw Spirits from Different Distilling Stages of *Luzhou-flavor* Spirit. *Food Science and Technology Research*, vol. 20, no. 2, p. 283-293. https://doi.org/10.3136/fstr.20.283

Zsótér, B., Molnár, A. 2015. Examining the effects of changes in legislation (2010) related to the production of brandy in practice - production conditions, tax obligations. *Jelenkori társadalmi és gazdasági folyamatok*, vol. 10, no. 2, p. 35-52. https://doi.org/10.14232/jtgf.2015.2.35-52 (in Hungarian)

Acknowledgments:

We wish to express our appreciation to Bózsvai Bibendum Pálinka Distillery and Veresi Pálinka House for their participation. We would also like to thank National Food Chain Safety Office (Budapest, Hungary) for analytical tests of pálinka.

Contact address:

*Péter Korzenszky, Szent István University, Faculty of Mechanical Engineering, Department of Agriculture and Food Machinery, Páter K. u. 1., 2100, Gödöllő, Hungary, Tel.: +3628522000,
E-mail: korzenszky.peter.emod@szie.hu
ORCID: https://orcid.org/0000-0001-6295-8474

Gábor Barátossy, National Food Chain Safety Office, Budaörsi út 141-145. 1118, Budapest, Hungary,
Tel.: +361346-0949,
E-mail: BaratossyG@nebih.gov.hu
ORCID: https://orcid.org/0000-0001-6317-2772

László Székely, Szent István University, Institute of Mathematics and Basic Science, Department of Mathematics, Páter K. u. 1., 2100, Gödöllő, Hungary, Tel.: +3628522000,
E-mail: szekely.laszlo@szie.hu
ORCID: https://orcid.org/0000-0003-2608-8365

Gábor Géczi, Szent István University, Faculty of Mechanical Engineering, Institute of Process Engineering, Department of Enviromental and Building Engineering, Páter K. u. 1., 2100, Gödöllő, Hungary, Tel.: +3628522000,
E-mail: geczi.gabor@szie.hu
ORCID: https://orcid.org/0000-0002-0909-7131

Corresponding author: *