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A high-quality berry vodka (Berrovka): Fabrication and investigation by gas chromatography–mass spectrometry

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In this study, a high-quality berry vodka, termed *Berrovka*, was produced using a mixture of berries – strawberries, raspberries, cherries, currants and gooseberries – cultivated and harvested in a household setting and processed into jam. The fermentation products of the mixed-berry jam waste were subsequently distilled, and the resulting distillates were analysed using gas chromatography–mass spectrometry (GC–MS). Analytical conditions for GC–MS were selected and optimised to facilitate the qualitative and quantitative assessment. Ethanol was identified as the primary fermentation product, as anticipated. Several volatile by-products, including acetaldehyde, methanol, propanol, isobutanol, butanol, and others, were identified. The analysis of sequential distillation fractions indicated a marked decrease in acetaldehyde concentration as distillation progressed. Concentrations of propanol, isobutanol, and isoamyl alcohol exhibited a gradual decline, whereas methanol levels remained relatively constant throughout the distillation process. These findings highlight the potential for producing a high-quality distilled spirit from household berry jam waste, with controlled levels of fermentation by-products through optimised distillation.

Keywords: berry vodka, 'Berrovka', various berries, jams, bio-wastes, distillation, gas chromatography, mass spectrometry

INTRODUCTION

Since ancient times, the production, consumption and trade of alcoholic beverages have played a significant role in the economies of many European cities [1]. Alcohol production and sales generated a substantial revenue for municipal authorities and were often employed as a means to alleviate economic hardship. Spirits and beers, in various forms, were commonly used not only in culinary applications but also in medicine and veterinary practice. A wide range of raw materials, including honey, sugar, cherries, apples, plums, raspberries, and other berries, have historically been utilised in alcoholic beverage production [1, 2]. For example, in different regions, non-grape fruits such as blueberries, hawthorn, goji berries, *Rosa roxburghii*, apricots, and others – with distinct sensory characteristics and potential health benefits – have been used in the production of fruit wines [3, 4]. The cultural and traditional significance of rakia, a spirit distilled from fermented fruits and berries, is particularly notable in the Western Balkans, as documented in Ref. [5]. In some cases, alcoholic beverages have been made from rare and region-specific berries, such as the edible fruits of karonda (*karonda*), a thorny evergreen shrub from the Apocynaceae family that grows in arid and semi-arid regions [6]. Regardless of the raw material, traditional flavours in alcoholic

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beverages are most often achieved through fermentation processes involving yeast, which play a crucial role in developing the desired taste profile [7].

Anjos et al. [8] evaluated honey-based spirits, focusing on their physicochemical and sensory properties. At various times, the production of high-proof alcoholic beverages from juniper berries has been documented across different countries [9–12]. More recently, it has been demonstrated that juniper berries, when used as a raw material for spirit production, are rich in carbohydrates, lipids, organic acids and phenolic compounds, which contribute to their antioxidant properties [13]. Notably, a study [14] suggested that an optimised blueberry-based alcoholic beverage may possess potential health benefits. However, there is a notable inconsistency in the reported use of bog bilberries in alcoholic beverage production, indicating a need for further research in this area [15]. Several studies have also shown that less common berries can be used to produce alcoholic beverages of varying strengths. For example, gooseberries, black currants, black elderberries and juneberries have been harvested, juiced and fermented to create fruit-based alcohols [16–19].

The agro-food industry is currently recognised as one of the largest global generators of waste [20]. A significant portion of this waste is produced during the transformation of raw materials, such as fruits, berries, vegetables and dairy products, into processed goods, including jams, sauces, canned foods, dairy products (e.g. cheese and yogurt) and beverages (both alcoholic and non-alcoholic) [20]. Recent scientific efforts have increasingly focused on reducing agricultural and food waste, reusing it as a secondary resource, extracting valuable bioactive compounds, and developing innovative technologies for efficient recycling [21–25]. To the best of our knowledge, this work presents the first successful production and evaluation of high-quality berry vodka made from waste jam derived from a mixture of strawberries, raspberries, cherries, currants and gooseberries. The distillates obtained from the fermented jam were analysed using gas chromatography–mass spectrometry (GC–MS) to determine the chemical composition and assess the quality of the final product.

EXPERIMENTAL

Reagents and characterisation

To prepare stock standard solutions the following reagents were used: methanol (99.9%, Merck), acetaldehyde (99.5%, Sigma-Aldrich), 1-propanol (99.7%, Sigma-Aldrich), isobutanol (99.5%, Merck), 1-butanol (HPLC, Eurochemicals), isoamyl alcohol (98.0%, Merck), acetic acid (99.7%, Sigma-Aldrich), ethanol (96.3%, Vilnius degtinė). In the analysis process, the chromatographic vials (2 ml capacity), analytical balance (Kern.), variable volume automatic pipettes (1000, 200 µl) and volumetric flasks (10 ml) were used. For gas chromatography and mass spectrometry analysis, a Perkin-Elmer Clarus 580S chromatography equipment and a PerkinElmer Clarus 560S quadrupole mass spectrometer were used. Capillary ZB-WAXplus column (30 m long, 0.32 mm internal diameter, stationary phase layer thickness 1 µm) was used for chromatographic analysis.

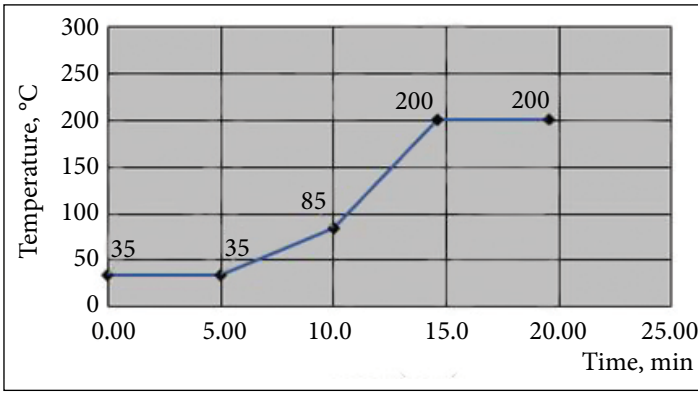
Gas chromatographic–mass spectrometric analysis conditions

Gas chromatographic analysis was performed using an oven temperature program, starting at 35°C and ramping up to 200°C. The temperature profile over time is presented in Fig. 1.

The injector temperature was set to 200°C, and helium (He) was used as the carrier gas at a flow rate of 1.4 ml/min. A split/splitless inlet with a split flow configuration was employed, using a split ratio of 1:30. Each sample injection had a volume of 0.5 µl, and the total run time for a single analysis was 19.6 min.

Electron ionisation (EI) was used for mass spectrometry. The interface between the gas chromatograph and mass spectrometer was maintained at 200°C. For qualitative and quantitative analysis, the mass spectrometer operated in the scan mode, detecting ions in a m/z range of 30.00 to 400.00. The scanning period covered 1.0 to 19.6 min. However, to avoid overloading the detector with the solvent signal, data acquisition was paused between 5.1 and 6.2 min.

For ethanol quantification, the scan range was set from 1.0 to 19.6 min, excluding the solvent signal. In the quantitative analysis of impurity alcohols, as well as acetaldehyde and acetic acid, the selected ion monitoring (SIM) mode was used. The monitored



Oven ramp	Rate	Temp	Hold
Initial	0.0	35	5.00
1	10.0	85.	0.00
2	25.0	200	5.00

Fig. 1. Gas chromatography oven gradient temperature regime

ions and retention time windows were as follows: acetaldehyde (1.20–1.40 min, m/z – 44), methanol (3.85–4.30 min, m/z – 31), propanol (8.00–8.30 min, m/z – 42 and m/z – 59), isobutanol (9.10–9.55 min, m/z – 43 and m/z – 74), butanol (10.10–10.30 min, m/z – 41 and m/z – 56), isopentanol (11.00–11.20 min, m/z – 55 and m/z – 70) and acetic acid (13.00–13.20 min, m/z – 43 and m/z – 60).

Sample preparation for analysis

For quantitative analysis, standard solutions were prepared using ethanol as the solvent. The concentration ranges were as follows: acetaldehyde (0.035–3.5 mg/ml), acetic acid (0.005–1.0 mg/ml), and various alcohols (0.01–2.0 mg/ml).

RESULTS AND DISCUSSION

The chromatograms of the standard solutions and investigated samples are shown in Fig. 2. Under the optimised conditions, the complete separation of all analytes in the standard mixture was achieved. These chromatograms also highlight the significance of using the SIM mode. Chromatogram A, recorded

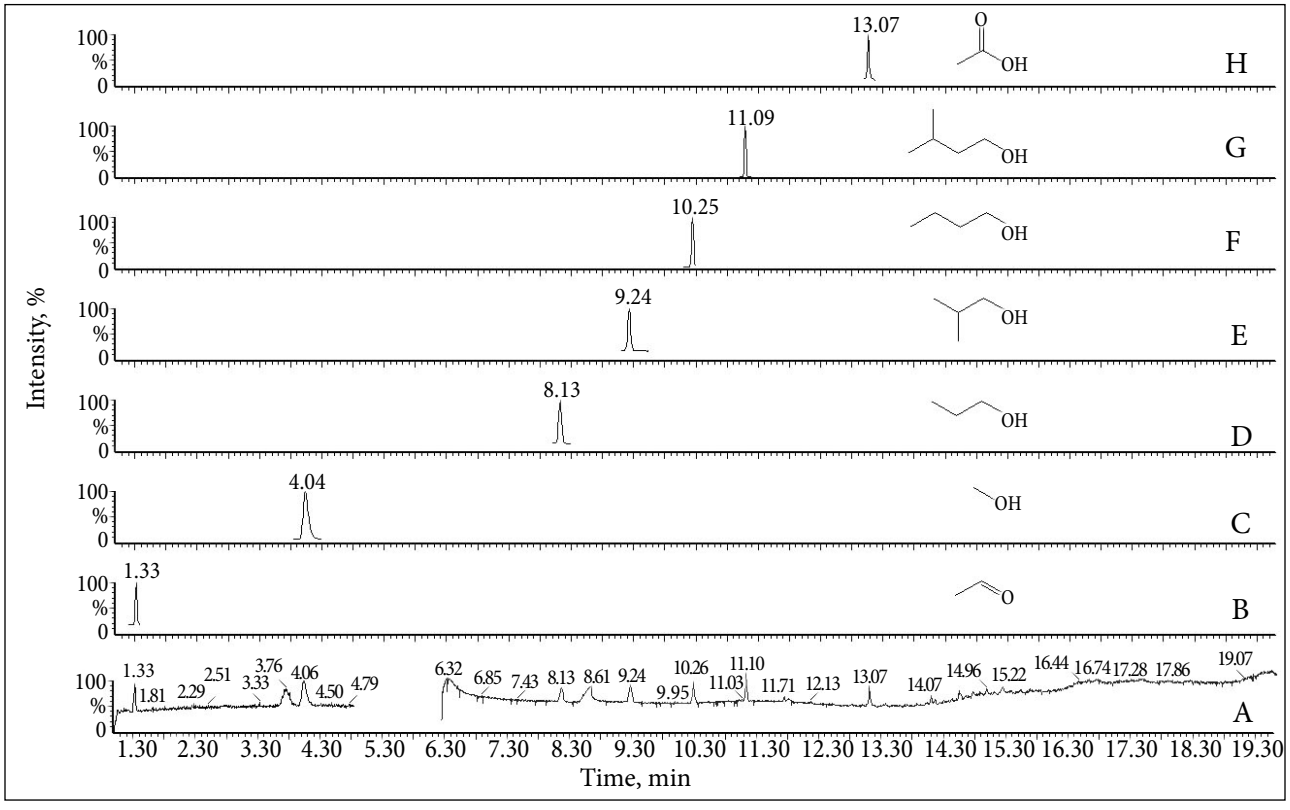


Fig. 2. Chromatograms of standard solutions at the lowest calibration concentrations: recorded in the full scan mode (A) and in the SIM mode for the following analytes – (B) acetaldehyde, (C) methanol, (D) 1-propanol, (E) isobutanol, (F) 1-butanol, (G) isopentanol and (H) acetic acid

in the full scan mode (m/z 30.00–400.00), demonstrates lower sensitivity for analytes present at low concentrations. Consequently, the SIM mode was employed to improve detection sensitivity. Specific *m/z* values were chosen based on the mass spectra of the target analytes. As illustrated in Fig. 3,

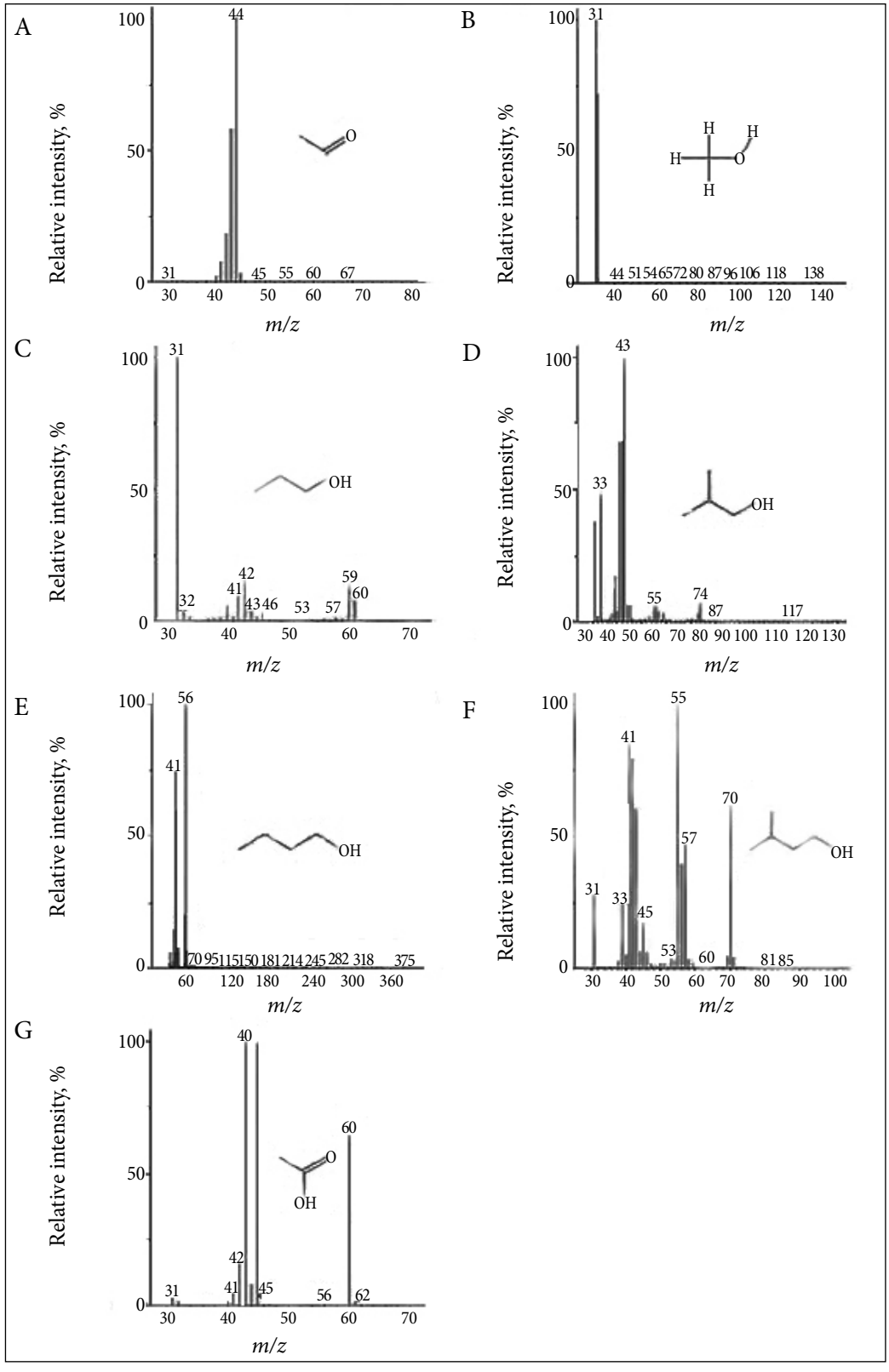


Fig. 3. Mass spectra of the studied compounds: (A) acetaldehyde, (B) methanol, (C) 1-propanol, (D) isobutanol, (E) 1-butanol, (F) izopentanol and (G) acetic acid

the selected ions correspond to the most intense and characteristic fragments of each compound, while avoiding interference from background signals, such as those originating from vacuum pump oil, septa, or the stationary phase (e.g. siloxanes).

A gas chromatography–mass spectrometry system was used for the qualitative analysis of fermented jam distillates. Seven distillation fractions, collected at different time intervals, were analysed throughout the study. The samples were introduced directly into the system without the need for additional preparation. Figure 4 presents the chromatograms of the first (collected after 10 h and 30 min; chromatogram A) and the last (collected after 19 h and 15 min; chromatogram B) distillation fractions. The results clearly demonstrate that the composition of impurity compounds changes significantly over the course of the distillation process.

The changes in the peak areas of the detected compounds across the distillation fractions were analysed. The volatile compounds identified in the different fractions of 'Berrovka' are summarised in Table 1. The variation in

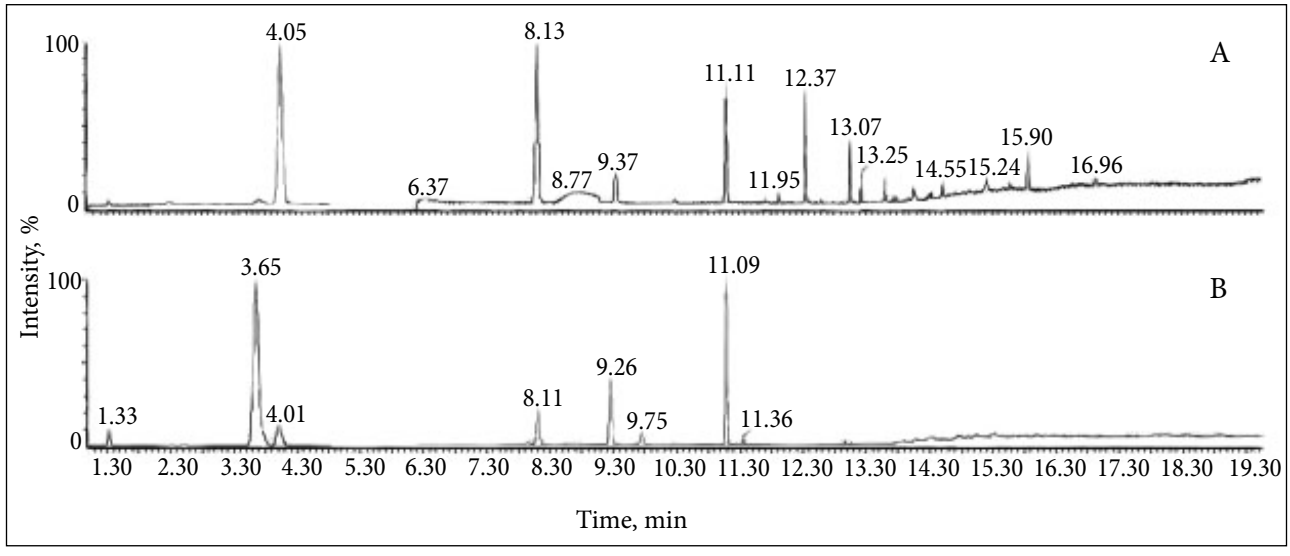


Fig. 4. The chromatograms of two distillation fractions A (taken after 10 h 30 min) and B (taken after 19 h 15 min) of berry vodka

Table 1. **Peak areas of volatile compounds determined in the distillation fractions of berry vodka**

Compound (R.T. min)	Fraction time						
	1 h 30 min (84% ethanol)	11 h 23 min (79% ethanol)	14 h 30 min (70% ethanol)	15 h 50 min (59% ethanol)	16 h 55 min (56% ethanol)	18 h 55 min (40% ethanol)	19 h 15 min (30% ethanol)
Acetaldehyde 1.33	8225618	3741988	1838301	1758777	1451937	154874	164939
Ethyl acetate 3.65	150909200	36110684	4459856	3622137	1955190	495303	469799
Methanol 4.01	18008820	12778510	15450046	12705048	11123627	8879411	10056702
1-Propanol 8.11	39436048	34777660	28652626	18119492	13276659	5926871	6378920
Isobutanol 9.26	63892632	46232236	21122614	8607618	4798785	1043172	1083311
Isoamyl acetate 9.75	14747019	–	–	–	–	–	–
Isoamyl alcohol 11.09	1041004000	89464168	47799956	21479698	11557583	2565361	2663956
Ethyl hexanoate 11.36	3807133	–	–	–	–	–	–
2-Hydroxypropil ethanoate12.36	–	–	1029500	1292480	1388587	1646826	1669696
Acetic acid 13.07	–	–	–	572476	490659	906605	966541
Furfural 13.25	–	–	–	–	233305	353039	361618
1,2-Butanediol 13.62	–	–	–	–	–	340065	316895
Benzyl alcohol 15.88	–	–	–	–	–	974166	1078822

compound profiles is due to the fractions being collected at different stages of the distillation process. The chromatograms of the first and second fractions exhibit similar compound compositions, but with differing peak intensities. Notably, the initial fraction contains additional volatile compounds, such as isobutyl acetate, ethyl butyrate, ethyl hexanoate and ethyl octanoate, which are absent in the intermediate fractions. Among all the samples, the third distillation fraction appears to be the purest product. Most previously detected compounds are no longer present, and only seven volatile compounds remain, with significantly reduced peak intensities in the chromatogram. This indicates a marked decrease in impurities during this stage of the distillation.

As shown in Table 1, the highest concentrations of ethyl acetate and isoamyl alcohol are observed in the early stages of distillation. As the process progresses, the amounts of these compounds decrease significantly. Small quantities of other compounds – such as hydroxypropyl ethanoate, acetic acid and furfural – also begin to appear in the distillates. Toward the end of the distillation process, additional compounds like 1,2-butanediol and benzyl alcohol are detected in the collected fractions. Interestingly, the ethanol concentration in the berry vodka distillates decreases from 84 to 30% as the distillation time increases from 10 h 30 min to 19 h 15 min. A noticeable decline in the peak areas of acetaldehyde, 1-propanol, isobutanol, isoamyl acetate and ethyl hexanoate is observed in the later fractions compared to the first. Although the peak area of methanol shows only a slight variation across fractions, it is markedly lower in the final fraction than in the initial one. In summary, the data presented in Table 1 clearly demonstrate that the peak areas of most compounds decrease substantially, often by several times, as distillation proceeds.

Quantitative analysis of key volatile compounds was also performed. The concentrations of selected distillation products – namely acetaldehyde, methanol, ethanol, 1-propanol, isobutanol, 1-butanol, isoamyl alcohol and acetic acid – were determined. A stock standard solution containing 2.0 mg/ml of each compound was prepared for calibration purposes. For the preliminary quantification of analytes, the GC–MS system was calibrated over a concentration range of 5 μg/ml to 2 mg/ml for all compounds except ethanol, which was quantified separately due to its significantly higher concentration. Ethanol calibration was carried out over a narrower range of 0.1 to 2 mg/ml [26].

Because the concentrations of certain analytes varied widely across distillation fractions, additional calibration curves were constructed at different intervals for compounds such as acetaldehyde, isobutanol and isoamyl alcohol to improve measurement accuracy. These calibration curves are presented in Fig. 5. As shown, the curves for most compounds exhibit a strong linearity, with correlation coefficients greater than 0.99. Concentrations in the samples were calculated using these calibration curves. To enhance precision, the calibration ranges were further refined based on the expected concentration levels. The quantitative results for each compound across the distillation fractions are summarised in Table 2. Notably, performing the analysis in the selected ion monitoring (SIM) mode allowed for the detection of significantly lower concentrations, improving overall sensitivity and accuracy.

Analysis of the quantitative results across all distillation fractions shows a clear trend: the concentration of acetaldehyde decreases steadily from 1594 to 46 mg/mL as the distillation time progresses up to 19 h and 15 min. Methanol concentrations remain relatively consistent across most fractions but drop significantly during the final stage of distillation. The concentrations of 1-propanol, isobutanol and isoamyl alcohol decline sharply after approximately 14–15 h of distillation. In the final fractions, these compounds fall below the detection limit. In contrast, 1-butanol was already undetectable in the fraction collected after 14 h and 30 min. Interestingly, the concentration of acetic acid increases in the later fractions. This may be attributed to the lower selectivity of its detection when other volatile compounds are present at high concentrations. Ethanol concentration decreases steadily in the later fractions compared to the initial and intermediate stages. In summary, the quantitative analysis confirms that extended distillation is effective in reducing the concentration of volatile compounds in homemade berry vodka. The reliability and suitability of the proposed method for determining volatile analytes were also evaluated, and the results are presented in Table 3.

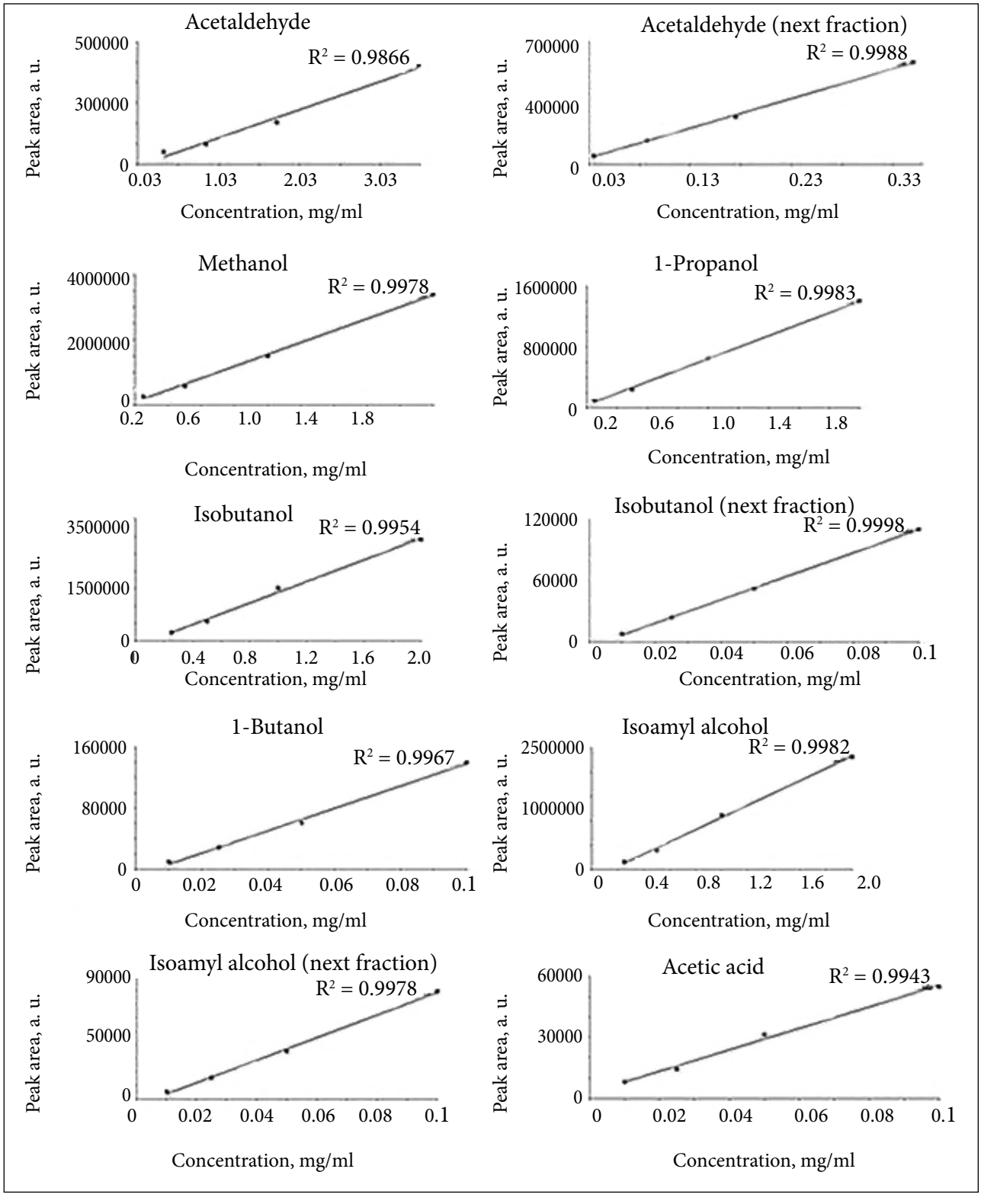


Fig. 5. The calibration curves used for the determination of different volatile compounds in the distillation fractions of 'Berrovka'

These results confirm that the relative standard deviation values were acceptably low, ranging from 4 to 11%. This indicates a good repeatability of the method. Notably, the final distillation fractions of homemade berry vodka show a chemical composition very similar to that of comparable commercial products, suggesting that they could be used in the food industry without restrictions. In contrast, the first two distillation fractions contain higher concentrations of volatile impurities.

Table 2. **The results of the determination of analytes in different distillation fractions of 'Berrovka'**

Analyte	Concentration, mg/l, at different fractions						
	10 h 30 min	11 h 23 min	14 h 30 min	15 h 50 min	16 h 55 min	18 h 50 min	19 h 15 min
Acetaldehyde	1594 ± 335	245 ± 52	63 ± 13	61 ± 13	53 ± 11	50 ± 11	46 ± 10
Methanol	733 ± 59	606 ± 48	623 ± 50	664 ± 53	850 ± 68	742 ± 59	542 ± 44
1-Propanol	765 ± 84	727 ± 80	624 ± 69	485 ± 53	571 ± 63	384 ± 42	276 ± 30
Isobutanol	926 ± 111	840 ± 101	418 ± 50	250 ± 30	230 ± 28	60 ± 7	35 ± 4
1-Butanol	24 ± 3	23 ± 3	–	–	–	–	–
Isoamyl alcohol	1411 ± 183	1667 ± 217	918 ± 119	485 ± 63	468 ± 61	147 ± 19	79 ± 10
Acetic acid	36 ± 4	26 ± 3	31 ± 3	36 ± 4	88 ± 10	98 ± 11	64 ± 7
Ethanol	756000	711000	630000	531000	504000	360000	270000

Table 3. **Analytical characteristics of the method**

Analyte	Characteristics				
	Standard deviation (SD)	Mean, mg/l ($N = 5$)	Relative standard deviation (RSD), %	Accuracy, %	Coefficient of variation, %
Acetaldehyde	144	1349	11	23	21
Methanol	22	516	4	3	8
1-Propanol	25	448	6	10	11
Isobutanol	27	442	6	12	12
1-Butanol	28	446	6	11	12
Isoamyl alcohol	27	429	6	14	13
Acetic acid	25	441	6	12	11

Therefore, the re-distillation of these initial fractions is recommended to improve the overall quality of the final product [2]. The distillation process significantly influences both the presence and concentration of volatile flavour compounds in the final distillate. In the production of strong spirits, it is a common practice to enhance the flavour by selectively removing low-boiling and high-boiling compounds [27]. On the other hand, a well-balanced profile of volatile compounds can contribute positively to the aroma and taste of the beverage [28]. For instance, regulations state that wine spirits and brandy should contain at least 1.25 g/l of volatile substances (expressed per 100% vol. alcohol) and no more than 2.0 g/l of methanol [29]. In the case of all 'Berrovka' distillation fractions, the methanol content was well below this regulatory threshold. The highest methanol levels were found in the final two fractions, yet they did not exceed 1.63 g/l of 100% vol. alcohol. In all fractions collected up to 15 h and 50 min, the methanol content was even lower – less than 1 g/L. These results indicate that all fractions of homemade 'Berrovka' meet safety requirements and can be considered suitable for use in the food industry, provided the sensory properties are acceptable.

CONCLUSIONS

Berries such as strawberries, raspberries, cherries, currants and gooseberries, grown and harvested in a simple household setting and processed into jam, were used to produce high-quality berry vodka 'Berrovka'. In this study, distillates obtained from fermented mixed-berry jam waste were analysed using gas chromatography–mass spectrometry under optimised conditions for both the qualitative and quantitative assessment of distillation products. The qualitative analysis of distillation fractions revealed clear differences in the composition of volatile compounds across various stages of the distillation process. Distillation fractions, collected between 10 h 30 min and 19 h 15 min, were analysed in detail. It was observed that the peak areas of key volatile compounds, such as acetaldehyde, methanol, 1-propanol, isobutanol, isoamyl acetate,

isoamyl alcohol, ethyl hexanoate, 2-hydroxypropyl ethanoate, acetic acid, furfural, 1,2-butanediol and benzyl alcohol, decreased significantly as distillation progressed. The final distillation fraction was found to be the purest, with the lowest concentration of volatile compounds. A quantitative analysis method was developed using GC–MS with the selected ion monitoring mode, enabling the accurate determination of the concentrations of the main volatile impurities. The results confirmed that the main fraction of the homemade berry vodka meets safety and quality standards and can be used in the food and fuel industry without restrictions.

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AUKŠTOS KOKYBĖS UOGŲ DEGTINĖ „BERROVKA“: GAMYBA IR TYRIMAS DUJŲ CHROMATOGRAFIJOS IR MASIŲ SPEKTROMETRIJOS METODU

S a n t r a u k a

Braškių, aviečių, vyšnių, serbentų ir agrastų uogos, užaugintos paprastomis namų sąlygomis ir perdirbtos į uogienes, buvo naudojamos aukštos kokybės uogų degtinei „Berrovka“ gaminti. Šiame tyrime distiliatai, gauti iš fermentuotų mišrių uogienių atliekų, buvo analizuojami dujų chromatografijos ir masių spektrometrijos (GC–MS) metodu optimizuotomis sąlygomis, siekiant įvertinti tiek kokybinę, tiek kiekybinę distiliavimo produktų sudėtį. Kokybinė distiliavimo frakcijų analizė atskleidė ryškius lakiųjų junginių sudėties skirtumus įvairiuose distiliavimo proceso etapuose. Distiliavimo frakcijos, surinktos nuo 10.30 val. iki 19.15 val., buvo išsamiai ištirtos. Nustatyta, kad pagrindinių lakiųjų junginių, tokių kaip acetaldehidas, metanolis, 1-propanolis, izobutanolis, izoamilacetatas, izoamilo alkoholis, etilo heksanoatas, 2-hidroksipropiletanoatas, acto rūgštis, furfurolas, 1,2-butandiolis ir benzilo alkoholis, smailių plotai žymiai mažėjo distiliacijos proceso metu. Galutinė distiliavimo frakcija buvo gryniausia, joje buvo mažiausia lakiųjų junginių koncentracija. Sukurtas kiekybinės analizės metodas, naudojant GC-MS su pasirinktu jonų stebėjimo režimu, leidžiančiu tiksliai nustatyti pagrindinių lakiųjų priemaišų koncentracijas. Tyrimo rezultatai patvirtino, kad pagrindinė naminės uogų degtinės frakcija atitinka saugos ir kokybės standartus ir gali būti naudojama maisto ir degalų pramonėje be apribojimų.