



A comparative analysis of sensory and chemical properties of grape rakija from different Macedonian producers

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Abstract

Rakija is a strong alcoholic beverage, deeply rooted in Macedonian cultural heritage, where quality varies significantly between industrial, small-scale, and traditional home producers. This study evaluated sensory and chemical differences among three types of producers using 30 samples of rakija from grapes (10 per group). The chemical analysis included gas chromatography (GC) for volatile compounds and analytical methods for alcohol content, specific gravity, and extract. The sensory analysis was performed by a trained panel (6 assessors) following OIV 100-point scoring scale. Spearman’s rank correlation was used for statistical data analysis. Sensory results revealed greater variability in odour and taste (CV 14–29%). Isobutanol showed a significant negative correlation with quality ($\rho = -0.43$ to -0.46 , $p < 0.017$), while extreme variability in esters (CV $>100\%$, ethyl acetate up to 5,654 mg/L) and furfural (CV 383%) was detected. In contrast, the extract content positively influenced the balance ($\rho = 0.43$ – 0.50 , $p < 0.017$) in all of the samples. Between the producer’s groups, the traditional samples had more unpleasant aromas due to improper distillation and fermentation, as confirmed by the chemical analysis results where ethyl acetate reached up to 5,654 mg/L. Routine sensory-chemical correlations can aid quality assessment, linking traditional heritage with modern distillation methods in Macedonian rakija production.

Key words: Rakija, Spearman method, OIV sensory panel, GC – gas chromatography, Distillation.

Introduction

With Ottoman expansion, Middle Eastern distillation techniques and terms like alcohol, arak, raki, and alembic spread to the Balkans, including Macedonia, becoming integral to regional rakija production culture (Mrgić, 2017). Rakija represents a strong alcoholic beverage obtained through distillation in a copper distillation apparatus from previously fermented grapes or other types of fruit that contain sugar (Lučić, 1987). According to Nikićević (2010, 2021), to produce top-quality rakija, a fundamental requirement is the selection of a variety, which yields rakija of a certain quality with specific flavours and aromatic characteristics. He also notes that the temperature for performing distillation and the type of distillation apparatus to be used will have a significant influence on the overall aroma and chemical composition of rakija, all forming the basis of its quality. In addition to its traditional and artisanal roots, rakija production has evolved into a regulated industry with legal rules and regulations that define its composition and sensory profile (Adžić et al., 2023). According to Milanov et al. (2015) and based on the technology of rakija production from grapes in Macedonia, rakija is divided into three types: Lozova, Komova, and Vinovica (Vinjak, Grape Brandy). He also describes Lozova rakija as a national beverage, while Komova rakija is compared to the Italian alcoholic drink "Grappa". This classification is important for understanding the different types of rakija and their characteristics. Regarding the colouring of rakija on the Macedonian market, there is yellow (golden) coloured and transparent rakija. The yellow colour in rakija comes from the oak barrels in which it is stored and aged (Stamenković & Stojanović, 2020), oak chips or boards are the other alternatives for maturation of the young distillate. Different grape varieties also have an impact on chemical compounds that significantly influence the character of the final product. Research on different grape varieties has revealed that the variety has a significant impact on the volatile aromatic compounds and sensory properties of rakija, especially in Muscat varieties (Raičević et al., 2022).

The influence of oenological agents used in the production of wine intended for distillation into strong alcoholic beverages is crucial for shaping the aromatic complexity and structural balance of rakija. Rakija obtained by distilling yeast lees has increased content of ethyl esters of fatty acids, which enhance the complexity of such strong alcoholic beverages (Van Jaarsveld et al., 2005; Chursina et al., 2021). Volatile compounds produced by yeasts as secondary metabolites, including ethyl esters, acetate esters, carbonyls, and fatty acids, together create a broad spectrum of aromatic compounds (Cordente et al., 2012). Unlike *Saccharomyces cerevisiae*, non-*Saccharomyces* yeasts produce increased levels of esters, higher alcohols, and fatty acids, which are key to the aroma of the wine used for distillation (Cordero-Bueso et al., 2013). Scientific

studies indicate that the sensory attributes of rakija are inherently linked to production techniques, raw materials, and aging processes (Stamenković & Stojanović, 2020). Recent studies on fruit spirits show volatile compound patterns, particularly esters and higher alcohols are formed during alcoholic fermentation, and are closely associated with both sensory attributes of technological practices, supporting the idea that controlled yeast metabolism and precise distillation are key for optimising the rakija's aroma (Stanzer et al., 2023; Ferreira et al., 2024).

According to Nikićević (2014), the strengths and flaws of rakija can be analysed visually and by smell and taste. Furthermore, the flaws in smell and taste are divided into two subgroups based on their origin, namely from microbiological spoilage of the distillation raw material and from non-microbiological factors. For high-quality rakija, clarity is required after dilution with water, and precise separation of fractions during distillation (Nikićević, 2014). Using gas chromatography, all desired and undesired aromas can be determined, having their characteristic compounds which influence the overall quality of the final product (rakija). Their concentration in rakija can exceed a certain threshold and lead to unwanted odours in rakija (Cantagrel, 1988).

The aim of this study was to compare the chemical composition and sensory profiles of rakija made from grape, produced by industrial, small family-owned, and traditional home producers, and to establish quantitative relationships between key volatile compounds and sensory quality attributes. The specific aim was to: (1) characterise differences in esters, higher alcohols, aldehydes, and other major volatiles across the three producer categories; (2) evaluate the sensory quality of rakija using a structured descriptive type of sensory scorecard; and (3) identify statistically significant correlations between chemical parameters and sensory domains in order to support technological optimisation of rakija production practices.

Material and Methods

Material

In this study, rakija from three groups of producers was collected and analysed through chemical and sensory analysis. The total number of samples from the targeted group was 30 samples as follows:

- Large-capacity distilleries – distilleries employing a technologist with higher education responsible for controlling the process and quality of the final product in accordance with all legal regulations and standards for producing high-quality strong alcoholic beverages. The equipment owned by these distilleries is modern and with high capacity in line with the latest trends. The

processes are fully or partially automated, and all production processes are monitored by internal laboratories within the distillery. In the production process, these distilleries use all approved oenological agents (yeasts, nutrients enzymes, fining agents etc.).

- Small family distilleries – for this type of rakija producers, production is exclusively from their own vineyards. These distilleries possess professional equipment, but there is no automation of the rakija production processes. In these distilleries, production is limited; the main producer, usually the owner, has at least basic knowledge in the relevant field, understands, and knows how to manage the technological process. These producers mostly use traditional rakija distillation apparatuses, and some of them use column distillation equipment without continuous distillation.

- Traditional home producers – this group includes traditional home producers who make rakija usually for their own needs. The most common group for these producers are micro vineyard owners, people with a long family tradition of producing rakija passed down through generations. Most often, the production technology for these producers is a “family recipe” passed from generation to generation as a legacy for continuing Macedonian culture and tradition. This traditional production means using traditional distillation alembic apparatus, locally produced. Traditional production by these producers means not using oenological agents or minimal use of yeasts, this being an exception, limited knowledge of technological processes based on long-term family experience. The equipment they use almost meets minimum standards, and part of it is passed down from generation to generation.

The sampling was targeted, by following the selection criteria: (1) producers had to be located in the main grape growing regions of Macedonia and registered as rakija producers; (2) each producer had to produce rakija from grapes within the same year; (3) samples represented three clearly defined producer categories (industrial, small family distilleries, and traditional home producers), with 10 samples collected from each category to ensure balanced representation.

For chemical analysis, each rakija sample was analysed in triplicate; the final reported value for each parameter represented the mean of the three samples. No analytical replication was performed for the sensory evaluation, because the sensory scores were already averaged over six trained panelists per sample.

Methods for chemical analysis

The provided samples were subjected to chemical analysis of some parameters at the Laboratory for wine and alcoholic beverages at the Institute of Agriculture – Skopje, according to methods approved by the International

Organisation of Vine and Wine (OIV). The parameters that were examined included the following:

- Classical analytical methods for determination of alcohol, specific gravity, and extract, and
- Gas chromatography analysis of some volatile components

For examination of specific gravity, extract, and alcohol in the samples, a pycnometer method was used (OIV-MA-AS2-01; MA-AS312-01; OIV-MA-AS2-03B; Taskeska – Gjorgjievski & Nedelkovski, 2024).

To determine volatile components in rakija, a gas chromatography analysis was performed by direct injection with prior sample preparation by distilling. The apparatus used to determine the quantity of volatile components was by the Agilent manufacturer, model 8860, which has an autosampler for easier sample handling. The gas chromatograph system was equipped with a flame ionization detector (FID) and appropriate column (30 m × 0.25 mm, 0.25 µm film thickness). The autosampler took 1 µL of the sample in split-splitless mode. The oven temperature was programmed as follows: 40°C for 4 min, then increased at 8.8°C/min to 100°C and at 17°C/min to 200°C, held for 2.3 min. Carrier gases in the method was compressed air at 400 mL/min, N₂ at 30 mL/min, N₂ at 25 mL/min. Data acquisition and analysis were performed using the Agilent software.

Sensory evaluation

Sensory evaluation was carried out by a trained expert panel using a structured scoring system. All panelists had academic background in oenology and high-alcohol spirit production with a minimum of five years of professional experience in the field, to ensure their suitability for sensory evaluation. The procedure followed the general principles of sensory analysis and the recommendations of the International Organization of Vine and Wine (OIV) (Annex 3.3; OIV/CONCOURS 332A/2009). All samples were evaluated individually under controlled conditions.

Each sample was assessed for visual, odour, and taste attributes using predefined ordinal scales with attribute-specific minimum and maximum values, as presented in the sensory evaluation form and scoring scale 100 points. The sensory evaluation included 30 different rakija samples, comprising 10 samples from industrial producers, 10 samples from small distilleries, and 10 samples from traditional household producers. Each sample was independently evaluated by a panel of six trained assessors, three female and three male.

Visual quality was evaluated based on clarity and colour. Odour attributes included typicity, quality, and positive odour intensity, while taste evaluation comprised typicity, quality, and harmony. In addition, an overall harmony score was assigned to each sample to reflect the integrated perception of balance and

coherence. For each sensory attribute, the final score per sample was calculated as the mean value of the scores assigned by the six assessors, and these averaged scores were used in subsequent statistical analyses.

The total sensory score was calculated as the sum of all individual attribute scores, with a theoretical range from 32 to 100 points, and was used as an indicator of overall sensory quality.

Statistical analysis

Descriptive statistics (mean, minimum, maximum, Standard Deviation (SD), and Coefficient of Variation (CV)) were computed for all sensory attributes and chemical parameters. Reliability analysis was performed to assess the internal consistency of the sensory evaluation scale using Cronbach's alpha (α) (Cronbach, 1951) in the Statistical Package for Social Sciences (SPSS). Cronbach's alpha was calculated using all individual assessor scores (30 samples \times 6 assessors = 180 observations). According to commonly accepted guidelines, α values ≥ 0.70 indicate acceptable reliability, ≥ 0.80 good reliability, and ≥ 0.90 excellent reliability (Tavakol & Dennick, 2011).

Bivariate relationships between sensory attributes and chemical parameters were analysed using the Spearman's rank correlation coefficient (two-tailed tests) (Conover, 1980). This non-parametric approach was selected due to the non-normal distribution and high variability observed for several chemical parameters.

Results and Discussion

Descriptive statistics of sensory attributes

As shown in Table 1, the visual attributes, clarity (mean = 4.94) and colour (mean = 4.81), received high average scores with very low variability (CV = 2.29% and 3.90%, respectively), indicating that most samples were visually similar.

In contrast, odour and taste attributes showed wider score ranges and higher variability. Positive odour intensity varied from 2 to 8 (CV = 29.06%), while taste quality ranged from 6.67 to 18.67 (CV = 21.52%), reflecting substantial sensory differences among samples. The overall sensory score ranged from 44.33 to 91.50, with a mean value of 75.24 and moderate variability (CV = 17.42%), indicating that this composite score effectively captures overall sensory differences and is suitable for comparative quality assessment.

Tab. 1 - Descriptive statistics of sensory attributes

Sensory attribute	N	Min.	Max.	Mean	SD	CV (%)
Clarity	30	4.67	5.00	4.94	0.11	2.29
Colour	30	4.33	5.00	4.81	0.19	3.90
Odour typicity	30	2.17	5.67	4.41	0.91	20.60
Odour quality	30	7.33	14.00	11.48	1.75	15.23
Positive odour intensity	30	2.00	8.00	5.53	1.61	29.06
Taste typicity	30	4.00	7.67	6.39	0.90	14.05
Taste quality	30	6.67	18.67	14.59	3.14	21.52
Taste harmony	30	4.33	11.33	8.41	1.81	21.48
Overall harmony	30	6.67	18.33	14.73	3.24	22.00
Overall sensory score	30	44.33	91.50	75.24	13.10	17.42

Reliability analysis of the sensory evaluation scale

It can be seen from Table 2 the sensory evaluation scale demonstrated excellent internal consistency, with a Cronbach’s alpha coefficient of 0.902 for the nine attributes. Corrected item–total correlations were high for odour and taste-related attributes (approx. 0.84–0.92), indicating strong alignment with the overall sensory construct. The corrected item–total correlations revealed that clarity ($r = 0.24$) and colour ($r = 0.25$) showed weak associations with the overall sensory scale, indicating a limited contribution of visual attributes to the internal consistency of the instrument. This result suggests that visual characteristics vary independently from odour- and taste-related attributes, which dominate the perception of overall sensory quality in rakija.

Removing either of these attributes slightly increased Cronbach’s alpha ($\alpha = 0.914$), suggesting that visual attributes contributed less to the internal coherence of the scale. This pattern reflects the nature of rakija evaluation, where sensory quality is primarily driven by aroma and flavour characteristics, while visual attributes tend to show limited variability.

Tab. 2 - Summary of reliability analysis of the sensory evaluation scale

Parameter	Value
Number of items (attributes)	9
Valid cases (N)	180
Cronbach’s alpha	0.902
Range of corrected item–total correlations	0.24 – 0.92
Items with low item–total correlation	clarity = 0.24 colour = 0.25
Alpha if clarity deleted	0.914
Alpha if colour deleted	0.914

Aggregated sensory domain scores

When sensory attributes were aggregated into domains as shown in Table 3, visual attributes showed very high and uniform scores (mean = 4.87; CV = 2.45%), confirming minimal variation in appearance. Odour and taste domains exhibited moderate variability (approximately CV = 19%), indicating stronger discriminatory capacity. Overall harmony displayed the highest variability (CV = 21.60%), suggesting differences in perceived balance among samples. The overall sensory score showed moderate dispersion (CV = 17.42%), supporting its use for ranking samples.

Tab. 3 - Descriptive statistics of aggregated sensory domain scores

Sensory attribute	N	Min.	Max.	Mean	SD	CV (%)
Visual attributes	30	4.58	5.00	4.87	0.12	2.45
Odour attributes	30	4.44	9.00	7.13	1.36	19.13
Taste attributes	30	5.00	12.39	9.77	1.90	19.41
Overall attribute (overall harmony)	30	6.67	18.33	14.71	3.18	21.60
Overall sensory score	30	44.33	91.50	75.24	13.10	17.17

Chemical composition

The chemical composition of the samples showed substantial variability (Table 4). Ethyl acetate and total esters exhibited very high dispersion (CV > 100%), indicating pronounced differences in ester formation, which are associated with yeast strain variability during fermentation, inconsistent distillation cut points (heads/ hearts/ tails separation) (Spaho et al., 2013). Furfural showed the highest relative variability (CV = 382.68%), reflecting that this compound was absent or very low in many samples but high in a few others. In contrast, the ethanol content (mean = 42.94; CV = 6.87%) and specific gravity (mean = 0.94; CV = 1.54%) were highly stable, indicating consistent alcohol strength across samples. Higher alcohols, including isobutanol and isoamyl alcohol, showed moderate variability, while aldehydes exhibited moderate-to-high dispersion.

Ethyl acetate, the dominant ester contributing to fruity flavours in spirits at normal levels ($\leq 150\text{-}200$ mg/L) (Apostolopoulou et al., 2005), was detected in all analysed rakija samples. However, our maximum concentration of 5,654 mg/L far exceeded this threshold, with aromas like nail polish, solvent-like, detected by the sensory panellists. High concentrations of ethyl acetate forms as a negative sensory component during primary fermentation as a result of poorly controlled alcoholic fermentation. This is why the yeast acetyl-CoA synthetase activity on excess acetic acid amplified when non-Saccharomyces strains or

stressed *Saccharomyces* (e.g., high sugar >24°Brix in grape mash) overproduced acetate esters. These high levels could be reduced by the master distiller by removing bigger quantity of heads (first 5-10% distillate fraction). Isoamyl alcohol maxima (4,207 mg/L) similarly arise from leucine catabolism under anaerobic conditions, surpassing harmonious levels (>2,500 mg/L) (Darici et al., 2021; Deng et al., 2020; Spaho et al., 2013; Dimitrov & Ivanova, 2017). These high concentration levels can be traced to the usage of red varieties for distillation while white varieties have a lower concentration (Lukić et al., 2011). The methanol content was detected in all rakija samples, but in low concentrations with maximum of 0.26vol% a.a, which is harmless for humans (Dimitrov et al., 2016).

Tab. 4 - Descriptive statistics of chemical composition parameters

Chemical parameter	N	Min.	Max.	Mean	SD	CV (%)
Ethyl acetate (mg/L % a.a.)	30	163.00	5,654.00	1,003.93	1,116.03	111.17
Ethyl lactate (mg/L % a.a.)	30	-	270.00	68.53	79.19	115.54
Isobutanol (mg/L % a.a.)	30	342.00	1,304.00	688.27	260.76	37.89
Isoamyl alcohol (mg/L % a.a.)	30	960.00	4,207.00	2,363.13	810.70	34.31
2-Phenylethanol (mg/L % a.a.)	30	-	93.00	30.42	28.64	94.15
Furfural (mg/L % a.a.)	30	-	81.00	4.90	18.75	382.68
Total higher alcohols (mg/L % a.a.)	30	1,321.00	5,380.00	3,110.73	988.19	31.77
Total esters (mg/L % a.a.)	30	163.00	5,924.00	1,075.80	1,145.30	106.46
Methanol (VOL% a.a)	30	0.04	0.26	0.15	0.06	37.97
Ethanol (alcohol content) (VOL %)	30	39.60	53.31	42.94	2.95	6.87
Extract (g/L)	30	0.30	15.30	1.75	2.62	149.99
Specific gravity	30	0.87	0.95	0.94	0.01	1.54
Aldehydes (mg/L % a.a.)	30	85.00	554.00	215.97	121.12	56.08

Relationships between sensory and chemical parameters

The correlation analysis revealed that overall sensory performance was most consistently associated with isobutanol (negative correlation) and extract (positive correlation) across odour, taste, overall harmony, and the overall sensory score (Table 5). These findings indicate that excessive levels of certain higher alcohols may negatively affect sensory perception, while higher extract content contributes positively to body and flavour balance.

Tab. 5 - Statistically significant ($p < 0.05$) or marginal ($p < 0.10$) Spearman correlations between sensory domains and chemical parameters

Sensory domain	Chemical parameter	Spearman's ρ	p-value
Visual attributes	Methanol	-0.438	0.015
	Ethanol	-0.438	0.015
	Extract	0.345	0.062
	Specific gravity	0.500	0.005
	Total esters	-0.345	0.062
Odour attributes	Isobutanol	-0.451	0.012
	Extract	0.525	0.003
	Aldehydes	-0.328	0.076
	Total esters	-0.315	0.090
Taste attributes	Isobutanol	-0.432	0.017
	Extract	0.431	0.017
Overall harmony	Isobutanol	-0.448	0.013
	Extract	0.497	0.005
Overall sensory score	Isobutanol	-0.462	0.010
	Extract	0.497	0.005

Visual attributes showed a positive association with specific gravity and a negative association with alcohol content, suggesting that slight differences in physical properties may influence visual perception.

Conclusion

This study highlights significant differences in the sensory and chemical profiles of grape rakija produced by industrial, small, and traditional home producers in Macedonia, demonstrating the relation between production practices and product quality. The sensory analysis revealed high consistency in visual attributes ($CV < 10\%$) but strongly noticeable variability in odour and taste ($CV 19\text{--}26\%$), with traditional home-produced samples exhibiting unpleasant aromas linked to fermentation and distillation. The results can confirm extreme heterogeneity in esters ($CV > 100\%$, ethyl acetate up to 5,654 mg/L) and furfural ($CV 383\%$), alongside negative correlations between isobutanol and sensory quality ($\rho = -0.43$ to -0.46 , $p < 0.017$) and positive associations with extract content ($\rho = 0.43\text{--}0.50$, $p < 0.017$). These patterns highlight how industrial and small distilleries achieve superior balance through modern equipment, oenological agents, and precise fraction separation, while traditional methods that are rooted in cultural heritage often yield off-flavours from excessive heads retention and non-Saccharomyces yeast activity.

Overall, the combined sensory and chemical approach can provide a robust framework for quality evaluation and supports the use of sensory profiling alongside chemical analysis in rakija research.

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Компаративна анализа сензорних и хемијских особина лозове ракије од различитих македонских произвођача

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Сажетак

Ракија је јако алкохолно пиће дубоко укоријењено у македонској културној традицији, при чему квалитет знатно варира између индустријских, малих и традиционалних произвођача за своје потребе. У овом истраживању извршена је оцјена сензорних и хемијских разлика између произвођача ракије употребом 30 узорака лозове ракије (10 по групи). Хемијска анализа укључивала је гасну хроматографију за испарљива једињења и аналитичке методе за садржај алкохола, специфичну тежину и екстракт. Сензорна анализа спроведена је од стране обученог панела (6 стручњака) на основу скале од 100 бодова OIV. Спирманова корелација ранга је кориштена за статистичку анализу података. Сензорни резултати открили су већу варијабилност у мирису и укусу (CV 14–29%). Изобутанол је показао значајну негативну корелацију са квалитетом ($\rho = -0,43$ to $-0,46$, $p < 0,017$), док је откривена екстремна варијабилност у естерима (CV >100%, етил ацетат до 5,654 mg/L) и фурфуралу (CV 383%). Насупрот томе, садржај екстракта позитивно је утицао на равнотежу ($\rho = 0,43$ – $0,50$, $p < 0,017$) у свим узорцима. Између произвођача ракије, традиционални узорци имали су најмање пријатне ароме због неправилне дестилације и ферментације, што је потврђено резултатима хемијске анализе према којој је етил ацетат достигао 5,654 mg/L. Рутинске сензорно-хемијске корелације могу помоћи у процјени квалитета, повезујући традиционалну баштину са модерним методама дестилације приликом производње ракије у Македонији.

Кључне ријечи: Ракија, Спирманова метода, Сензорни панел OIV, гасна хроматографија, Дестилација.

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