

Utilizing Grape Pomace as a Functional Ingredient: A Case Study in Biscuit Production

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Abstract

The aim of the work was to prepare biscuits with 5, 10, 15 and 20% addition of grape pomace of the Merlot variety obtained from a Slovak wine company. For comparison, a control sample without the addition was also prepared. The amount of dry matter determined ranged in all samples at the level of ~ 94%; the total fat content slightly increased due to the addition, with the lowest value in the control sample (22.51%) and the highest in biscuits with 20% addition of pomace (24.07%). The total ash content ranged from 0.23% (control) to 0.71% (sample with 20 % addition of pomace). The crude protein content increased proportionally with the addition – the control sample showed a content of 5.97%, the sample with a 5% addition had a content of 6.17%, the sample with a 10% addition had a content of 6.29%, the sample with a 15% addition had a content of 6.35% and the sample with a 20% addition had the highest content - 6.41%. The addition of pomace caused an extension of the oxidative stability, which is a positive indicator in terms of the shelf life of the biscuits. The control sample showed a stability of 18.35 hours, while the sample with a 20% addition had up to 35.46 hours. Significantly higher amounts of mineral compounds were found in the biscuits with the addition, especially copper, zinc, manganese, magnesium and iron. The content of the hazardous elements cadmium and lead in the control sample and in the sample with the addition of 5 and 10% was not recorded. In the sample with the 15 and 20% addition, the amounts were found at the level of 0.6 and 0.8 mg/kg, respectively, but these values are in accordance with the legislation. From the sensory point of view, the biscuits were evaluated very well, and the 15% addition appeared to be the best.

Key words: Merlot, ash, fat, crude protein, cereal products, sensory acceptance.

Introduction

The main by-products of the wine industry include grape pomace, grape seeds, grape stems, yeast, and tartrate sediments. Grape pomace is the residual matter left after pressing fresh grapes, whether they undergo fermentation or not. It is one of the most significant by-products in the wine industry and consists primarily of the grape's weight, including the skins, seeds, and other solid components. This by-product is a complex material made up of 30% neutral polysaccharides, 20% pectic substances, 15% insoluble proanthocyanidins, as well as structural proteins and phenolic compounds.¹ Over the years, the utilization of grape pomace has been largely inefficient. In recent years, it is estimated that only around 3% of the grape pomace produced is repurposed for animal feed, while other uses include waste-based compost and potential applications in improving thermal insulation in building construction. Grape pomace is produced in large quantities during the short grape harvest period, resulting in a high concentration of by-products per unit of area.² Disposal methods like incineration or landfilling can be harmful to the environment due to the phenolic compounds in pomace, which lower its pH and increase

its resistance to biological degradation. Other environmental concerns include pollution of surface and groundwater, unpleasant odors, the attraction of flies and pests that can spread diseases, and oxygen depletion in soil and groundwater caused by tannins and other compounds.

With the growing consumer demand for natural over synthetic products and increased focus on sustainable agricultural practices, there are a wide variety of potential applications for grape pomace. These include functional foods (rich in dietary fiber and polyphenols), food processing (biosurfactants), cosmetics (grape seed oil and antioxidants), as well as pharmaceuticals and dietary supplements (grape pomace powder). According to Dwyer et al.³ the market potential for grape pomace in Canada suggests that if every gram of red pomace skin were sold, the potential profit could reach approximately 448 million euros. Additionally, if all grape seeds were used to produce oil, with each 750 mL bottle priced at 4.7 euros, the final profit could exceed 4 million euros. However, the market potential decreases when a significant portion of the grape pomace is used to create compost, which is then recycled back into the vineyard. Several food categories have successfully incorporated wine pomace products to enhance their phenolic content. Cereal products, particularly bread and cookies, represent the category with the highest number of applications, primarily using wine-pomace flours. Cookies made with seedless and wine pomace flours received higher acceptability ratings compared to those made with seed flour, while cereal bars were found to be an excellent option for incorporating grape seed flour. Other food products, such as marmalade, candies, salad dressings, and tomato puree, have also been fortified with wine-pomace ingredients. Additionally, a new functional seafood sausage was developed using meagre (*Argyrosomus regius*), and red skin extracts were incorporated into tea infusions at varying concentrations, ranging from 50% to 100%, to boost the phenolic profile and antioxidant activity of the infusions.⁴

Despite these advancements, a critical knowledge gap persists in understanding the full spectrum of grape pomace's applications and its impact on food quality and safety. While several studies have reported on the benefits of grape pomace in various food categories, comprehensive evaluations of its bioactive compounds and their synergistic effects in food matrices remain limited. Moreover, the economic viability of utilizing grape pomace across different sectors is not thoroughly explored, especially in the context of sustainable practices within the wine industry.

In Slovakian market missing products with grape pomace addition, so the main aim of this study was to prepare model biscuits with 5, 10, 15 and 20% grape pomace incorporation and subsequently determined nutritional and sensory quality of these biscuits.

Material and Methods

Grape pomace from Merlot variety was obtained from private Slovakian wine company from area Tajná (174 m.a.s.l.). Fresh pomace was dried in oven (Binder ED 56, Germany) 5 hours using temperature 50 °C. Dry pomace was homogenized (IKA RCP-24, Germany) for final particle size 0.1 mm.

The biscuits were prepared in accordance with an old family recipe provided by one of the authors (Ivanišová). The ingredients were purchased from local market and included white wheat flour, 00 extra, beet sugar, butter, and vanilla. Each type of biscuits was made and baked separately. Altogether five variants of biscuits were prepared: control sample, samples with 5%, 10%, 15% and 20%. After kneading, the dough was allowed to rest for 60 min at +4 °C. The dough was rolled with a roller to obtain a thickness about 6 – 7 mm. The desired shapes of biscuits were cut out of the dough and formed by hand. The biscuits were baked at 150 °C for 20 minutes in oven (Miwe condo, Germany). After cooling for 30 min, the biscuits were packed in polyethylene zipper resealable food bags and stored at +21 °C and 50% relative humidity prior to the analyses of their nutritional and sensory characteristics.

All the chemicals used were of analytical grade and were purchased from Sigma-Aldrich (St. Louis, MO, USA) and CentralChem (Bratislava, Slovak republic).

Dry matter, ash and protein were determined following the standard AACC method 08 – 01.⁵ Nitrogen content was measured by the semi micro-Kjeldahl method. Nitrogen was converted to protein using the conventional factor of 5.7.

Fat content was determined with Ancom XT15 Fat Extractor (USA) in line with producer instructions – the sample (1.5 g, W1) was weighted to special filter bag (XT4, Ancom, USA) and dried for 3 hours in an oven (WTB, Binder, Germany) at 105 °C to remove moisture prior to the extraction. Samples were placed in a desiccant pouch for 15 minutes, re-weighted (W2) afterwards, and extracted for 60 minutes at 90 °C with petroleum ether. After the process, the samples were removed, dried in an oven at 105 °C for 30 minutes, placed in a desiccant pouch and re-weighted (W3). Fat content (%) was calculated using the following formula: $[(W2-W3)/W1] \times 100$.

The oxidative stability was determined in 892 Rancimat apparatus from Metrohm (Switzerland) according to ISO 6886:1997⁶ utilizing a sample of 0.5 ± 0.01 g. All samples were studied in temperature 120 °C, under a constant air flow (20 L/h). The induction times were printed automatically by apparatus software with the accuracy of 0.005.

The analysis of mineral compounds was performed with Varian model AA 240 FS equipped with a D2 lamp background correction system using an air-acetylene flame (air 13.5 L/min., acetylene 2.0 L/min, Varian, Ltd., Mulgrave, Australia). The results were compared with multielemental standard for GF AAS (CertiPUR®, Merck, Germany). A 1 g of sample was digested with mixture of HNO₃: redistilled water (1:1). Samples were digested in a closed vessel high-pressure microwave digester (MARS X-press, USA) for 55 min. After cooling to room temperature, the suspension was filtered through Munktell filter paper (grade 390.84 g/m², Germany) and diluted to 50 ml with distilled water. Then, the samples extracts were subsequently analysed for Cd, Pb, Cu, Zn, Co, Cr, Ni, Mn, and Fe. The wavelengths at which the heavy metals were analysed following the calibration process were as follows: Cd – 228.8 nm, Pb – 217.0 nm, Cu – 324.8 nm, Zn – 213.9 nm, Co – 240.7 nm, Cr – 357.9 nm, Ni – 232.0 nm, Mn – 279.5 nm, Fe – 241.8 nm.

The sensory properties of the prepared biscuits were determined by a taste panel consisting of 25 evaluators – certified trained panellists (in age from 25 to 65; 15 women and 10 men; STN EN ISO 8586:2023)⁷. The panellists were asked to evaluate general appearance, flavour, taste, overall acceptability, and aftertaste. A 9 – point hedonic scale was used to rate the samples, with scores ranging from 9 (like extremely) to 1 (dislike extremely) for each characteristic. The sensory evaluation was realized in professional sensory laboratory (AgroBioTech Research Centre, Slovak University of Agriculture in Nitra) with accordance to ISO 11132:2021⁸.

All experiments were carried out in triplicate and the results reported are the results of those replicate determinations with standard deviations. The experimental data were subjected to analysis of variance (Duncan's test), at the confidence level of 0.05, using software SAS.⁹

Results and discussion

The results of dry matter content in the tested samples (Table 1) ranged from 93.30% (samples with a 10% addition) to 95.75% (samples with a 15% addition). All values were within the maximum limit for biscuits, which is 8–10%. Biscuits belong to long shelf-life products; under good storage conditions, such as in a dark, cold, and dry place, they can be stored for up to 6 months. In the study by Giosué et al.⁸ the dry matter content in biscuits enriched with grape pomace was like that in our study, at 94%. The ash content (Table 1) was lowest in the control sample and increased linearly, with the highest value found in biscuits containing 20% grape pomace. In the study by Giosué et al.¹⁰ sweet biscuits with a 20% addition contained a higher level of ash compared to our study, at 2%. This may be attributed to the grape pomace, which varied in grape origin and recipe; they used soy milk as a different ingredient, which influenced the ash content. The crude protein content (Table 1) across all tested samples was comparable, with marginally increased levels noted at the 15% and 20% addition rates. These results align with those reported by Giosué et al.¹⁰ who observed crude protein levels of 4.8% at the 20% addition rate and 5.8% at the 30% addition rate in sweet biscuits. In contrast, Troilo et al.¹¹ reported

a protein content of 9% in muffins enriched with 15% grape pomace, which is higher than our findings. However, it is important to note that Troilo et al.¹¹ included milk in their recipe, which contributed to the elevated protein values. Indeed, grape pomace, which is the solid remains of grapes after juice extraction (including skins, seeds, and stems), has been identified as a valuable by-product in the food industry. According to Kumar and Kumar¹² its richness in essential amino acids like lysine and methionine makes it an attractive ingredient, particularly since these amino acids are often limiting in various plant-based and animal-based foods. By incorporating grape pomace, food products can potentially enhance their amino acid profiles, making them more nutritious without relying on traditional sources like soy, which is expensive and can trigger allergies in some individuals¹³.

Table 1: Results of total dry matter, ash and crude protein in analyzed biscuits

Sample	Dry matter [%]	Ash [%]	Crude protein [%]
Control biscuit	94.43 ±0.11 ^{ab}	0.25 ±0.03 ^e	5.97 ±0.33 ^b
B 5 % addition	95.39 ±0.09 ^a	0.42 ±0.05 ^d	6.17 ±0.17 ^b
B 10 % addition	93.72 ±0.12 ^c	0.56 ±0.01 ^c	6.29 ±0.24 ^a
B 15 % addition	95.75 ±0.13 ^a	0.66 ±0.05 ^{ab}	6.35 ±0.12 ^a
B 20 % addition	93.96 ±0.07 ^c	0.71 ±0.02 ^a	6.41 ±0.09 ^a

B – biscuit; mean ±standard deviation; different letters in rows denote mean values that statistically differ one from another

The total fat content in the samples varied between 22.51% in the control samples and 24.07% in the samples with a 20% addition of grape pomace. This difference is not so statistically significant and can be attributed to the main fat source in the biscuits, which is derived from the butter used in the formulation. While grape pomace is generally known to contain between 5% and 10% fat, Pop et al.¹⁴ reported values exceeding 20%. This variability in fat content highlights that the fat percentage in grape pomace can be greatly influenced by the specific variety used. The oxidative stability of fatty products is crucial, as highlighted in Table 2. Grape pomace has been shown to significantly enhance this stability. For instance, the control sample exhibited an induction time of only 18.35 hours. However, as the percentage of grape pomace added increased, there was a linear increase in oxidative stability, with the highest stability observed in samples containing 15% and 20% grape pomace. This improvement is largely attributed to the bioactive compounds present in the pomace, particularly those from the polyphenol family. According to Antonić et al.¹⁵ many polyphenols remain in grape pomace following the processing of grapes due to incomplete extraction. The primary polyphenolic compounds found in this by-product include anthocyanins (which are present only in red grape pomace), catechins, flavonol glycosides, phenolic acids, and alcohols. Together with dietary fibers, these phenolic compounds are among the most valuable constituents of grape pomace, known for their health benefits, including the promotion of intestinal health and the prevention of chronic diseases and cancer. Numerous studies have demonstrated the strong antioxidant potential of polyphenols, making them effective agents for food preservation through the inhibition of lipid oxidation and their antibacterial properties. Enhancing oxidative stability is a critical technological parameter, and grape pomace can serve as a functional ingredient not only in biscuits but also in fatty animal products, which are particularly susceptible to oxidation.¹⁶

Table 2: Results of total fat content and oxidative stability in analyzed biscuits

Sample	Fat [%]	Oxidative stability [h]
Control biscuit	22.51 ±0.22 ^b	18.35 ±0.34 ^d
B 5 % addition	22.61 ±0.23 ^b	18.74 ±0.32 ^d
B 10 % addition	23.69 ±0.12 ^a	21.25 ±0.15 ^c
B 15 % addition	23.79 ±0.32 ^a	32.37 ±0.23 ^b
B 20 % addition	24.07 ±0.12 ^a	35.46 ±0.12 ^a

B – biscuit; mean ±standard deviation; h – hours; different letters in rows denote mean values that statistically differ one from another

Table 3: Mineral compound composition in analyzed biscuits

Sample	mg/kg								
	Cu	Zn	Mn	Fe	Cr	Ni	Co	Pb	Cd
Control biscuit	0.63 ±0.02 ^c	2.40 ±0.12 ^b	3.10 ±0.11 ^d	15.45 ±0.22 ^c	0.10 ±0.02 ^d	ND	ND	ND	ND
B 5 % addition	0.89 ±0.01 ^{ab}	2.58 ±0.11 ^a	3.28 ±0.09 ^c	16.13 ±0.23 ^b	0.27 ±0.01 ^c	ND	ND	ND	ND
B 10 % addition	0.91 ±0.08 ^a	2.61 ±0.09 ^a	3.68 ±0.07 ^b	17.05 ±1.22 ^b	0.30 ±0.01 ^{bc}	ND	ND	ND	ND
B 15 % addition	0.92 ±0.09 ^a	2.65 ±0.08 ^a	3.73 ±0.02 ^b	19.30 ±0.76 ^a	0.35 ±0.02 ^b	0.05 ±0.01 ^a	0.08 ±0.22 ^a	0.28 ±0.05 ^a	0.20 ±0.02 ^b
B 20 % addition	1.07 ±0.02 ^a	2.66 ±0.12 ^a	3.84 ±0.02 ^a	19.20 ±1.33 ^a	0.41 ±0.03 ^a	0.06 ±0.01 ^a	0.07 ±0.22 ^a	0.27 ±0.07 ^a	0.70 ±0.04 ^a

B – biscuit; mean ± standard deviation; ND – not detected; different letters in rows denote mean values that statistically differ one from another

The addition of grape pomace to biscuits significantly enhances their mineral content, particularly increasing levels of essential nutrients such as copper, manganese, iron, and chromium. The most prominent improvements in mineral content were observed at 15% and 20% inclusion rates of grape pomace. This is particularly beneficial for vegan consumers, who are often at risk of anaemia due to insufficient iron intake. Importantly, while these higher percentages also introduced trace amounts of nickel, cobalt, lead, and cadmium, all detected levels remained within the legal limits as stipulated by Slovak food regulations¹⁷, underscoring the safety of consuming these enriched biscuits. Heavy metals like lead, mercury, arsenic, and cadmium are typically recognized as systemic toxicants, posing health risks both in acute and chronic scenarios. Consequently, food contamination with such harmful metals is a significant concern that necessitates regulatory oversight. Research by AntoniĆ et al.¹⁵ highlights the variability in essential mineral compounds found in grape pomace, with iron levels reported to range from 5 mg to over 5468 mg per 100 g, and zinc levels spanning from 2 mg to 2254 mg per 100 g. These minerals contribute not only to dietary nutrition but also play a vital role in enhancing antioxidant potential, which is beneficial for overall health. Additionally, grape pomace serves as an excellent source of potassium, with concentrations reaching up to 3157 mg per 100 g. Potassium is crucial for cardiovascular health, as it helps lower blood pressure and mitigates the risk of osteoporosis by reducing urinary calcium excretion. Thus, incorporating grape pomace into food products like biscuits may represent a strategic approach to improving dietary mineral intake while ensuring safety and regulatory compliance.¹⁸

The sample with 15% grape pomace addition received the best score compared to other samples containing grape pomace. When evaluating the aroma, the evaluators were asked to assign a score from a range of 1 to 9, where a maximum of 9 points indicated an excellent, balanced, and pleasant smell. The best aromas were found in the samples with 15% and 20% grape pomace. Regarding taste, the biscuits with 15% grape pomace were rated as the best, followed by the samples with 5% and 10%. The sample with 15% grape pomace also achieved the highest score for overall acceptability, receiving 8 points (Figure 1). Generally, the samples (Figure 2) containing grape pomace were considered acceptable, though some evaluators detected foreign tastes and aromas reminiscent of herbs or bitterness. Conversely, some evaluators appreciated the nice fruity smell and taste of the samples with grape pomace. Overall, it can be concluded that the biscuits, particularly those with 15% grape pomace, were evaluated very positively, indicating that Slovakian consumers accept grape pomace in innovative food products. In a study by GiosuĆ et al.¹⁰ biscuits with 20% and 30% grape pomace, as well as a control sample without any addition, were evaluated. The grape pomace biscuits were characterized by more pronounced winy and red fruit aromatic and olfactory notes, while the control sample displayed stronger oily notes. The control sample also exhibited a more pronounced biscuit flavor and sweetness, whereas the grape pomace biscuits were notable for their bitterness and acidity. In terms of mouthfeel sensations, the control sample was higher in greasiness, as well as crunchiness and friability; the 20% biscuit also shared these characteristics. The 30% sample was the chewiest, firm, and astringent, and

was accompanied by a seedy texture, like the 20% biscuits. These authors confirmed that smaller particle sizes of grape pomace negatively affected the hardness and color in terms of lightness, as well as the homogeneity of the pores. Muffins with 15% grape pomace in a study by Triolo et al.¹¹ were also rated as very good, possessing a pleasant fruity taste and aroma. Acun and Gül¹⁹ observed that adding more than 5 % red grape pomace to cookies led to an increase in hardness.

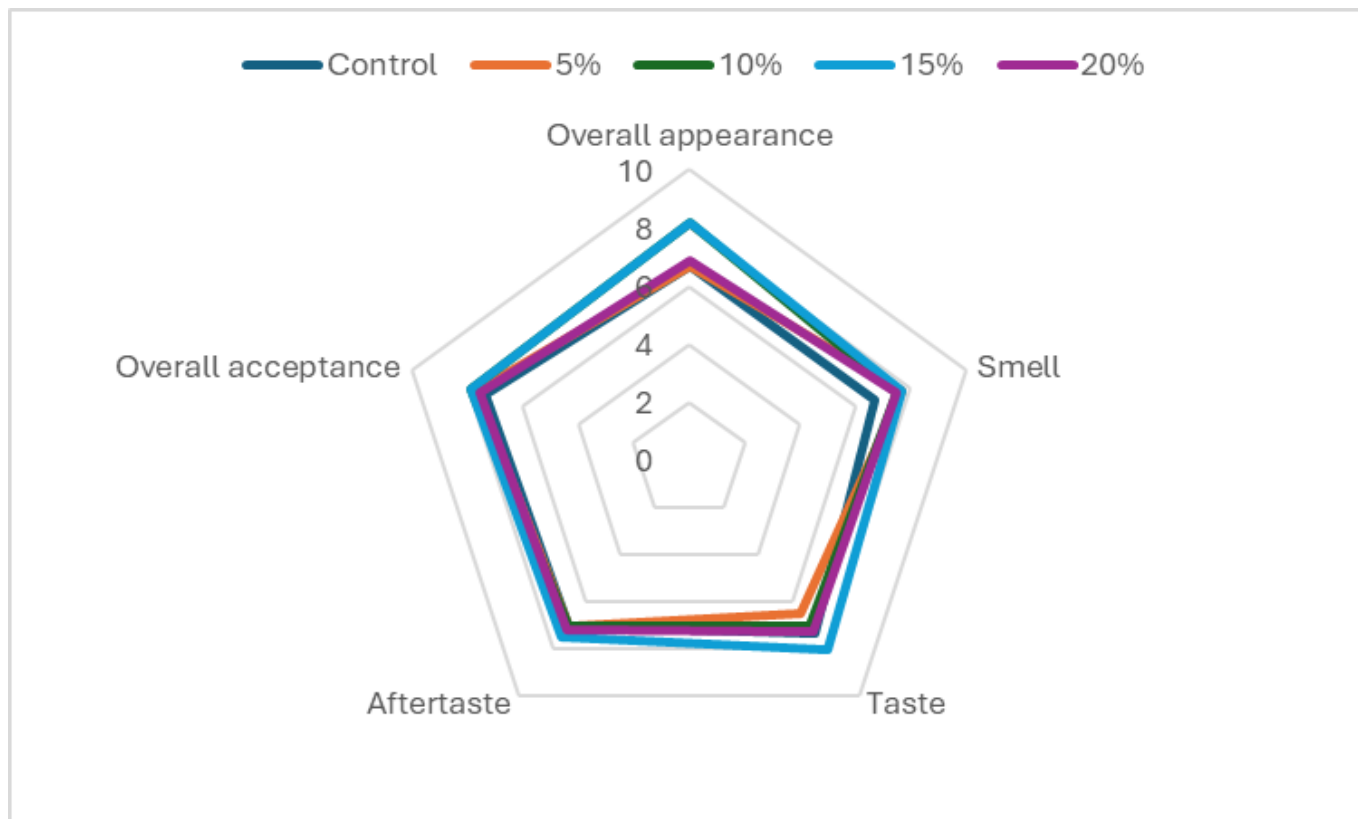


Figure 1: Sensory analysis of prepared biscuits (sum of all evaluators)



Figure 2: Biscuits with grape pomace (from left to right: control samples, sample with 5%, sample with 10%, sample with 15% and samples with 20%) (photo: Eva Ivanišová)

Conclusion

Grape pomace is a promising raw material for food enrichment, particularly in bakery products, which serve as suitable matrices for its incorporation. The benefits of using grape pomace extend beyond nutritional enhancement; it also offers technological advantages in terms of shelf life, as its biologically valuable substances can act as natural preservatives. Based on our results, we recommend incorporating grape pomace at levels of 15% in food applications, particularly in biscuit formulations. While our study evaluated additive levels up to 20%, preliminary findings suggest that these products may tolerate higher concentrations, potentially up to 30%. Although higher additions positively influenced nutritional parameters, sensory attributes remain crucial for consumer acceptance. Therefore, it is

essential to note that exceeding 40% addition may negatively impact taste, particularly through increased bitterness, which could affect overall product acceptance. However, this study has some limitations that should be acknowledged. The sensory evaluation was conducted with a relatively small sample size, which may affect the generalizability of the findings. Additionally, the long-term effects of incorporating grape pomace on the shelf life and quality of baked goods require further investigation. Future research should explore consumer preferences in greater depth, as well as the impact of grape pomace on various types of baked products with differing formulations. Moreover, studies could investigate the sensory attributes and acceptability of formulations with higher pomace levels, alongside a broader assessment of the technological implications in different baking environments. This roadmap for subsequent studies will help to solidify the role of grape pomace in food enrichment and improve the understanding of its full potential in baked goods.

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Využitie hroznových výliskov ako funkčnej zložky: Prípadová štúdia pri výrobe sušienok

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Abstrakt

Cieľom práce bolo pripraviť sušienky s 5, 10, 15 a 20% prídavkom hroznových výliskov odrody Merlot získaných od slovenskej vinárskej spoločnosti. Na porovnanie bola pripravená aj kontrolná vzorka bez prídavku. Stanovené množstvo sušiny sa vo všetkých vzorkách pohybovalo na úrovni ~ 94%; celkový obsah tuku sa pridaním výliskov mierne zvýšil, s najnižšou hodnotou v kontrolnej vzorke (22,51 %) a najvyššou v sušienkach s 20% prídavkom výliskov (24,07 %). Celkový obsah popolovín sa pohyboval od 0,23 % (kontrola) do 0,71 % (vzorka s 20% prídavkom výliskov). Obsah hrubého proteínu sa s prídavkom úmerne zvýšil – kontrolná vzorka vykazovala obsah 5,97 %, vzorka s prídavkom 5 % mala obsah 6,17 %, vzorka s prídavkom 10 % mala obsah 6,29 %, vzorka s prídavkom 15 % mala obsah 6,35 % a vzorka s prídavkom 20 % mala najvyšší obsah – 6,41 %.

Pridanie výliskov spôsobilo predĺženie oxidačnej stability, čo je pozitívny ukazovateľ z hľadiska trvanlivosti sušienok. Kontrolná vzorka vykazovala stabilitu 18,35 hodiny, zatiaľ čo vzorka s prídavkom 20 % mala až 35,46 hodiny. Výrazne vyššie množstvo minerálnych zlúčenín bolo nájdených v sušienkach s prídavkom výliskov, hlavne medi, zinku, mangánu, horčíka a železa. Obsah rizikových prvkov kadmia a olova v kontrolnej vzorke a vo vzorke s prídavkom 5 a 10 % nebol zaznamenaný. Vo vzorke s prídavkom 15 a 20 % boli zistené množstvá na úrovni 0,6 a 0,8 mg/kg, tieto hodnoty sú však v súlade s legislatívou. Po senzorickej stránke boli sušienky hodnotené veľmi dobre a ako najlepšie sa javil prídavok 15 %.

Kľúčové slová: Merlot, popoloviny, tuk, dusíkaté látky, cereálne produkty, senzorickej akceptácia.