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Evaluating the recovery of bioactive compounds and antioxidant activity of unripe red grape liquid extracts obtained by maceration

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Abstract

This paper evaluates the results of extracting bioactive compounds using modern maceration technology, which transforms food products into a liquid extract, based on the example of unripe grapes and its by-product called verjuice. During the extraction process, the indicators of niacin and pyridoxine remained stable, while pantothenic acid and choline increased significantly. During the extraction, the extraction system produced 712 kg of liquid extract, which was characterized by a high content of phenolic compounds and water-soluble vitamins, as well as a high antioxidant activity compared to the measurement obtained for green juice from unripe grapes. Since the estimated liquid extract yield from 950 kg of crushed immature grapes is 847 kg, the actual liquid extract yield was about 80%. The advantages of the maceration extraction method included avoiding the use of solvents or preservatives, low temperature treatment and little oxidative damage. Besides, it enabled enhancing the extraction yield due to introduction of certain post-extraction steps.

Keywords: antioxidant activity; extraction; modern maceration technology; biologically active compounds; biochemical characteristics of the food product.

Practical Application: Preparation of extracts for use as functional ingredients or nutritional supplements.

1 Introduction

Food waste is now considered a major social, economic, and environmental problem. Food and Agriculture Organization of the United Nations (FAO) has published data that show that about one-third of the total amount of food produced for global consumption (about 1.3 billion tons of food supplies per year) is lost or wasted (http://www.fao.org/food-loss-and-food-waste) (Nicastro & Carillo, 2021).

Considering fruit processing, about 45% of resources (by weight on a raw basis) are qualified as waste (Kumar & Kalita, 2017). Typically, fruit waste consists of peel, seeds, and pomace. For further use, they must be dried, because due to the presence of a high water content, their susceptibility to microbial destruction increases (Iriondo-DeHond et al., 2018). It is worth noting that factors such as the high cost of technological equipment for drying, storage, and transportation hinder the economic stability of fruit waste processing. Another disadvantage is the low income received from selling dried fruits as animal feed or agricultural fertilizers (Li et al., 2017).

Agro-industrial waste is characterized by great diversity and potential for applying in different fields, such as application as organization and management of fruit and vegetable residues for human and animal consumption (Ramírez et al., 2020), use in the development of alternative culture media (Santos et al., 2022) and a source of minerals, sugars, vitamins, fibers, carotenoids, phenols, and aromatic substances (Costa et al., 2020; Tlais et al., 2020). Thus, the well-known pectin, which is widely used in confectionery, is extracted mainly from the peel of citrus fruits and apple juice, and in order for food products to be stored longer, phenols and carotenoids act as preservatives of natural origin, due to their ability to retain unpleasant odors and bitterness (Vanitha & Khan, 2020). Recently, it has also been proposed as a substitute for fat in meat products (Varga-Visi & Toxanbayeva, 2017). In addition, biologically active compounds are characterized by antioxidant, antitumor, anti-inflammatory, and antiviral properties (Ullah et al., 2020). Also, it is common knowledge that dietary fiber is often used as a food additive because it improves intestinal peristalsis (Yan et al., 2021). Nevertheless, most of fruit waste is currently thrown away. Among other things, this happens due the lack of suitable and commercially available strategies to extract valuable compounds for their further application in global industries (Fierascu et al., 2020).

In the context of new innovative and technological solutions that meet sustainability requirements, good alternatives are technologies using high pressures, high hydrostatic pressures, membrane filtration, pulsed electric fields, enzyme extraction, ultrasound, and microwaves. However, these technologies must preserve the key bioactive compounds found in fruit waste.

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The most innovative and environmentally friendly technologies used for the extraction and recovery purposes must be explored and developed (Del Pilar Sánchez-Camargo et al., 2017).

Fruit waste is a potential source of biologically active compounds, which provide additional health benefits, including phenols (Mukhametov et al., 2021, 2022). Phenols are plant secondary metabolites that contribute to the organoleptic and nutritional qualities of fruits in terms of flavor, aroma, and color (Ullah et al., 2020). Phenolic compounds vary from simple molecules to high molecular weight polymers (polyphenols). The latter comprise more than 8 thousand compounds, and this number is constantly increasing. Based on the number of phenolic rings and structural elements that bind these rings together, it is possible to classify phenolic compounds, according to which their main classes are flavonoids, tannins, phenolic acid, stilbenes, and lignans (Minatel et al., 2017; Behl et al., 2021).

It is important to develop new effective methods for the recovery of biologically active compounds from food products that would replace the outdated methods used in biotechnological production. Therefore, the aim of this work is to assess the recovery of biologically active compounds and antioxidant activity of liquid extracts of unripe red grapes by maceration at different stages of modified extraction (i.e., without the use of solvents). To achieve the above purpose, the researchers are to accomplish several ***objectives***. Firstly, they intend to characterize the samples under investigation (grapes and verjuice as their by-product) from the biochemical point of view. Secondly, the researchers will identify bioactive compounds that are present during the recovery by means of maceration and at the post-extraction stage.

2 Methods and materials

2.1 Characteristics of the plant material and liquid extracts processing

In 2021, red grape samples were handpicked during the growing season for industrial processing of liquid extracts of unripe grapes. Unripe grape samples (about 950 kg) were harvested at the onset of ripening (veraison) on August 13, 2021. The liquid extract was processed according to the established operating procedures. However, two modifications were introduced, which were the addition of larger amount of dry ice to prevent oxidative damage and post-extraction pomace pressing to increase the extraction yield. Using the maceration system, crushed unripe grapes were subjected to extraction, stirred every 5 hours for 20 minutes for a total period of 72 hours at a temperature of +5 °C with the immediate addition of some dry ice (Tartian et al., 2017). Once racked, pomace underwent five pressing cycles in a membrane pneumatic press (Della Toffola, Italy), while the pressure increased from 0.2 to 2.0 bars. During maceration, samples of the liquid extract were obtained in triplicate (12, 24, 48, and 72 hours). All liquid extract samples were stored at –15 °C until their analysis.

2.2 Bioactive compounds extraction and biochemistry

Chemical characteristics and phenolic maturity of grapes were determined according to a predetermined methodology (Rajha et al., 2017). To carry out the process of separating juice and extracts, one hundred and fifty berries were weighed and then pressed. The separated juice was centrifuged at 3,000 g for 12 min and its pH was measured. Before weighing, the squeezes were first washed with water, then dried for 24 hours at +27 °C. The mass of the juice was calculated by subtracting the mass of dry extracts from the mass of berries. By multiplying the mass of dry extracts/juice × 1000, the ratio of extracts/juice was calculated. With the help of spectrophotometric analysis of the juice, the parameters of phenolic maturity were determined. Anthocyanins were measured at 510 nm and expressed in mg equivalents of malvidin-3-O-glucoside/l of extract. All chemicals were purchased from Sigma-Aldrich (Germany). The percentage of skin tannins in phenolic composition (dT of skins) was calculated as pH 3.4 40/1,000. The percentage of seed tannins in phenolic composition (dT of seeds) was calculated as A 270 nm-dT of skins. Total anthocyanin content (TA) was expressed as mg malvidin-3-O-glucoside equivalents/l of liquid extract, and total phenolic content (TP) was expressed as mg catechin equivalents/l of liquid extract. Color intensity (CI) and hue (H) were measured on a ULAB 102 spectrophotometer (China) using a quartz cuvette with a path length of 1 mm with distilled water as a reference. CI was expressed as the sum of optical densities (A) at 410, 510 and 600 nm: $CI = (A410 + A510 + A600) \times 10$. H was expressed as the ratio between optical densities at 410 and 510 nm: $H = A410 / A510$. Vitamin content, phenolic composition, and glutathione content were determined using the method of high-resolution liquid chromatography mass spectrometry according to the method proposed by Xiao et al. (2012). There are two groups of techniques to evaluate the antioxidant capacity in plant extracts. These techniques are classified in tests by electron transfer (ET) and by hydrogen atom transfer (HAT) (León et al., 2022). In this study, antioxidant activity (AA) was measured spectrophotometrically (Santos & Silva, 2020) and expressed as micromoles of Trolox equivalent antioxidant capacity/L of liquid extract.

2.3 Statistical analysis

Using the one-way ANOVA method using Microsoft Excel and the Statistica 10 package, the obtained research results were checked for reliability (Smith, 2018). The experiment was repeated six times. The differences were considered significant at a significance level of $P \leq 0.05$ according to the Student's t-test.

3 Results

3.1 Biochemical characteristics of the grape samples

Table 1 shows data on the biochemical characteristics and phenolic ripeness of the studied grapes, which were collected during the 2021 harvest season.

The pomace to juice ratio was 115.8. This meant that 90.5 kg was the largest amount of liquid extract that could be obtained from 100 kg of unripe grapes, and the dry weight of the pomace was about 9% of the weight of the unripe grapes. The cell maturity index was fairly high – 49.6%. The percentage of skin tannins in phenolic composition (dT = 19.8) was higher than the percentage of seed tannins in phenolic composition

Table 1. Biochemical characteristics and phenolic maturity of the grapes under investigation.

Pomace to juice ratio	pH	Cell maturity index, %	Seed maturity index, %	Percentage of skin tannins in phenolic composition	Percentage of seed tannins in phenolic composition
115.8 ± 5.1	3.4 ± 0.03	49.6 ± 2.2	10.7 ± 0.7	19.8 ± 1.1	1.9 ± 0.02

(dT = 1.9). The seed maturity index was equal to 10.7, and this value was consistent with the behavior described above and indicated seed lignification.

3.2 Recovery of bioactive compounds during maceration extraction

950 kg of crushed unripe grapes after their mechanical destemming (60 kg – 6.3% unripe grape sample) were processed by a maceration extraction system, which can be described as cold maceration extraction.

Figure 1 shows the extraction kinetics of total anthocyanin content, total polyphenolic compounds contents, antioxidant activity, color intensity, and hue of the liquid extracts.

The extraction triggered certain changes in the phenolic profile. To clarify, it significantly increased the contents of multiple phenolic compounds, except for quercetin and myricetin. Of note, the main phenolic compounds found at the initial stage were phenolic acids and flavonols (Figure 2).

At the beginning of extraction, the contents of procyanidin and flavan-3-ol were low, instead, procyanidin B1 and epicatechin could already be detected after 24 hours. After 72 hours, epicatechin-O-gallate was detected, and the content of water-soluble vitamins increased from 895 to 1376 g/L. The initial hue value was high due to the low anthocyanin content, after 24 hours it decreased to less than 0.9 and remained stable.

Peak values were approximately 2650 mg catechin equivalents/l and approximately 135 mg malvidin-3-O-acetylglycoside equivalents/l. The most abundant phenolic compounds were caftaric acid, catechin, and quercetin-3-O-glucuronide, and the most concentrated phenolics found in the liquid extract were phenolic acids and flavan-3-ols.

The total phenol content correlated ($r = 0.59$; $p < 0.003$) with the extract antioxidant activity. The highest positive correlation was confirmed for the antioxidant activity and procyanidin B_1 ($r = 0.982$; $p < 0.005$), procyanidin B_2 ($r = 0.966$; $p < 0.003$), kaempferol-3-O-glucoside ($r = 0.981$; $p < 0.003$) and 3-O-hexoside quercetin ($r = 0.978$; $p < 0.068$).

The enzymatic oxidative form of glutathione disulfide was present, and the amount of 2-S-glutathionylcaftaric acid, which occurs when o-quinones are in the presence of reduced glutathione, was increased. A sign of the effect on leaching is that during the extraction process, the content of niacin and pyridoxine remained stable, while the content of pantothenic acid and choline increased.

It is established that the antioxidant activity of the extract increased linearly, reaching approximately 4,690 µmol of Trolox equivalent antioxidant capacity/L after 72 hours. Extraction led to significant changes in the color intensity. The trend and value of the hue during extraction corresponded to the principle of protecting the liquid extract from oxidation.

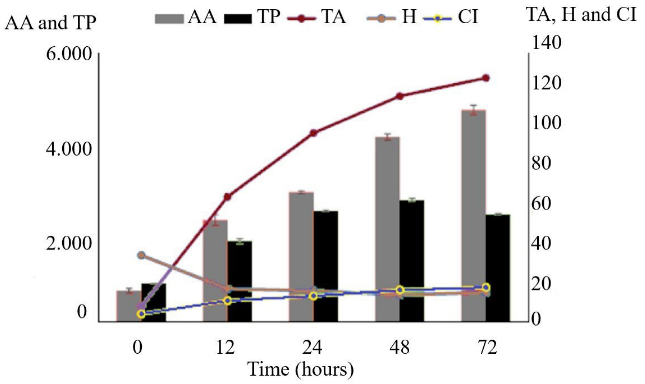

Figure 1. AA – Antioxidant activity, µmol of Trolox equivalent antioxidant capacity/L of liquid extract, TP – total phenol content, mg of catechin equivalents/L of liquid extract, TA – total anthocyanin content, mg of malvidin-3-O-glucoside equivalents/L of liquid extract, H– hue and CI – color intensity, absorption units × 20 of the liquid extracts measured during the extraction phase.

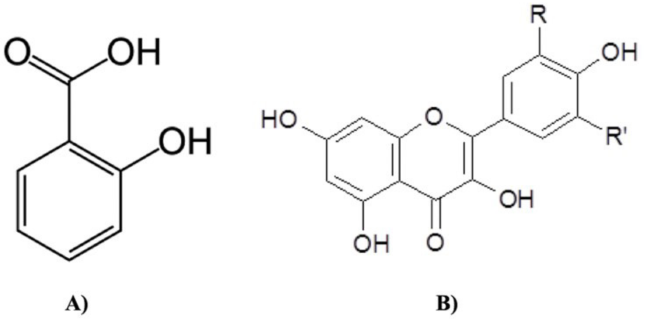

Figure 2. General chemical formulas of phenolic acids (A) and flavonols (B) identified at the initial stages of extraction.

3.3 Recovery of bioactive compounds during post-extraction

The yield of liquid extract (712 kg) was approximately 80%, as the maximum amount potentially obtainable from 950 kg of crushed unripe grapes was estimated at 847 kg (i.e., pomace to juice ratio was 115.8).

At the post-extraction stages, the content of bioactive substances and antioxidant activity were measured. It is worth noting that the total content of polyphenolic compounds and antioxidant activity after pressing increased significantly from 2.650 to 2.811 mg catechin equivalents/l and from 4.690 to 7.995 µmol Trolox equivalent antioxidant capacity/L, respectively.

After decantation, these values remained stable. After filtration, a slight decrease in total polyphenolic compounds was observed, antioxidant activity remained stable, color intensity decreased, and total anthocyanin content was stable. Minor oxidative damage experienced during the post-extraction steps contributed to a slight decrease in hue values.

In addition, after filtration and after 72 hours of extraction, there were some changes in the phenolic profile of liquid extracts. The content of quercetin-3-O-glucuronide and 2-S-glutathionylcaftaric acid in the final extract decreased significantly, while the content of other phenolic compounds remained stable. The contents of water-soluble vitamins were stable, with the only exception of niacin. The concentration of the latter was significantly increased in the final extract. The contents of coutaric acid, caftaric acid, fertaric acid, epicatechin, catechin and procyanidins were higher than those observed after 72 hours of extraction.

4 Discussion

Red and white wine production generates significant amounts of solid organic waste, such as stems, grape skins, pomace, grape seeds, and wine lees (Maicas & Mateo, 2020). To reduce the impact on the environment, these wastes undergo conventional treatment. Common examples include the use of stems, skins, and pomace for the production of compost, the use of stems, pomace and lees for the production of alcohol, organic acids and pigments, or the use of grape seeds for the production of oils (Silva et al., 2021). There are also innovative solutions capable of turning industrial waste into by-products with high added value (Zuin & Ramin, 2018), as evidenced by the renewed interest in the extraction of biologically active compounds from grape by-products, whose functional properties are attracting attention in the food, nutraceutical, and cosmetic industries (Putnik et al., 2018).

Unripe grapes stand out among the potential by-products of wine production. They are derived from thinning operations, which are performed to improve the quality of wine produced. The most common method is crop thinning, which involves the removal of one to two-thirds of the grape bunches to promote ripening of the berries that remain on the vine (O'Brien et al., 2021). The transition from grape growth to berry ripening was chosen as a practice to remove redundant bunches at the verification stage to improve wine quality (Schelezki et al., 2020). Traditionally, unripe grapes are used to produce a semi-finished product called verjus, which is a very acidic juice obtained by pressing unripe grapes. Verjus is used as an ingredient in seasoning mixtures or by boiling with the addition of vinegar and spices to obtain a sour sauce (Öncül & Karabiyikli, 2015). Verjus has some preservative properties due to its acidity and antioxidant activity, which are due to its content of organic acids and phenolic compounds (Togisbayeva et al., 2022). The literature shows that verjus has an acidifying effect in food products on the process of enzymatic darkening of fruits (Zhu et al., 2016), an inhibitory effect (Moon et al., 2020) and protecting effect in the context microbial activity (Silva et al., 2021).

It was found that anthocyanins are not easily extracted from grape cells, because the cell maturity index was quite high (50.4%) (Oliveira et al., 2020). The phenolic profile reflects the data that was previously emphasized by other authors regarding the grape composition (Lorrain et al., 2013; Somkuwar et al., 2018). The appropriate extraction kinetics depends on the localization of phenolic compounds in grape berries. The most abundant compounds found in grape pulp were phenolic acids, which were easily extracted, while flavan-3-ols and procyanidins required longer extraction times due to the thick cell walls of the seeds and skins, where they were concentrated (Zenoni et al., 2016).

To improve the extraction of phenolic compounds in winemaking, the cold maceration technique is widely used, because the protection of phenolic compounds from oxidation can be ensured by the use of dry ice (Lorrain et al., 2013; Grgić et al., 2020). A typical phenomenon of solid-liquid extraction is reflected in the evolution of phenolic compounds and anthocyanins extracted from grapes (Chaves et al., 2020). All liquid extracts in the study showed significantly higher antioxidant activity and phenolic content than grape samples obtained by a number of other researchers and described in scientific sources. For example, other investigators reported the total phenolic levels of about 200–800 mg/L (Lorrain et al., 2013) and 1,150–1,600 mg/L (Moon et al., 2020) and antioxidant activities of about 200–1,000 mol/L (Lorrain et al., 2013) and 2,400–3,900 mol/L (Moon et al., 2020).

A correlation was noted between the total phenolic content of the extract and its antioxidant activity, which is consistent with the results obtained by other scientists (Flieger et al., 2021; Martinez-Gomez et al., 2020). A group of researchers found that antioxidant activity was significantly correlated with levels of proanthocyanins, catechins and procyanidins, but grape variety and extraction method, which affect juice composition, had an effect on antioxidant activity (Wang et al., 2021; Radonjić et al., 2020). In addition, information on the composition of water-soluble vitamins present in unripe grapes has been studied rather superficially. Several authors described the water-soluble vitamin contents in a range of grape varieties. For example, Radulescu et al. (2020) considered the presence of biologically active compounds in different grape varieties; water-soluble vitamins (pantothenic acid, ascorbic acid, vitamin B6, thiamin and riboflavin) have been found in green and red grapes, but there are no data on choline in the literature. Öncül & Karabiyikli (2015), focusing on different stages of berry ripening, identified pantothenic acid, niacin, and pyridoxine in grapes.

5 Conclusions

This paper describes the results of extraction of bioactive compounds by maceration, which transforms food products into a liquid extract, using unripe grapes and verjuice as an example. The extracted liquid had a high extraction yield, a high content of phenolic compounds and water-soluble vitamins, and a high antioxidant activity compared to the measurements obtained for green juice, a traditional semi-finished product from unripe grapes. The results showed that the ratio of pomace to juice is 115.8, which means that the highest amount of liquid extract that can be obtained from 100 kg of unripe grapes was 90.5 kg. Thus, the pomace dry weight represented about 9% of unripe grapes weight. The cell maturity index was fairly high – 49.6%.

The percentage of seed tannins in the phenolic composition (dT = 1.9) was lower than the percentage of skin tannins in the phenolic composition (dT = 19.8). The seed maturity index equaled 10.7. The content of water-soluble vitamins increased from almost 895 to almost 1376 g/L after 72 hours of extraction. During the extraction process, the content of niacin and pyridoxine remained stable, while the content of pantothenic acid and choline increased significantly. As a result of the extraction system, after the last stage of filtration, 712 kg of liquid extract was formed. Given that the highest amount of liquid extract that could potentially be obtained from 950 kg of crushed unripe grapes was estimated to be 847 kg (i.e., pomace to juice ratio of 115.8 as stated above), the yield of liquid extract was approximately 80%. After pressing, the antioxidant activity and total content of polyphenolic compounds increased from 2.650 to 2.811 mg catechin equivalents/l and from 4.690 to 7.995 µmol Trolox equivalent antioxidant capacity/L, respectively.

Advantages of the maceration extraction method included avoiding the use of solvents or preservatives, low temperature processing, and little oxidative damage. In the wine sector, the process can be considered ready for implementation, because the extraction yield has improved thanks to the introduction of post-extraction steps. This implementation will make it possible to obtain extracts that can be used both as functional ingredients that make food products healthier, and as additives of natural origin that protect foods and beverages from the oxidation process.

Conflict of interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Availability of data and material

Data will be available on request.

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