CONVENTIONAL ULTRASOUND AND MICROWAVE-ASSISTED EXTRACTION OF PHENOLIC COMPOUNDS FROM MAVRUD GRAPE, POMACE AND SEEDS

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ABSTRACT

This research is dedicated to the valorization of wine industry residual products as a source of valuable compounds of interest to the food, pharmaceutical and cosmetic industries. Whole grape seeds and grape pomace of organic Mavrud of Bulgarian origin, harvested in 2016 were used. The aqueous ethanolic extracts were prepared by three different techniques: conventional extraction via magnetic stirring, ultrasound-assisted extraction (US) and microwave-assisted extraction (MW). Microwaves seemed to be more efficient in extracting the desired substances through the hard shell of the seeds. The polyphenolic content of the seed extracts, expressed as GAE (gallic acid equivalent) was remarkably higher: 58.57 mg GAE g^{-1} dw (first stage of MW-assisted extraction) compared to the conventional extraction with a magnetic stirrer – 7.03 mg GAE g^{-1} dw. The results obtained for the US extraction were similar - 49.73 mg GAE g^{-1} dw, but the processing time in the microwave field was considerably shorter. The US-assisted extraction was not so convenient for the grape pomace extracts - 20.47 mg GAE g^{-1} dw were obtained for 240 min, compared to 75.02 mg GAE g^{-1} dw for 20 min in the microwave field. Three analyzes were performed to determine the total polyphenolic content, antioxidant activity and anthocyanin content. The MW-assisted extraction was the most suitable for leaching of polyphenolic compounds and the extracts exhibited a higher antioxidant activity. Contrarily it has been observed that the anthocyanin content of the extracts produced via MW-assisted extraction was lower compared to the conventional and US-assisted techniques. Both techniques revealed time and energy-saving and are considered as eco-procedures.

Keywords: valorization, grape by-products, extraction, eco-procedure, polyphenolic content, antioxidant activity, anthocyanin content.

INTRODUCTION

Grape is one of the most valued conventional fruits in the world [1]. Taking into consideration the whole grape production, approximately 75 % is utilized in wine-making [2]. Global wine production is around 27 billion liters per year [3]. The wine health benefits were revealed in 1990s by the theorized “French paradox”. The high consumption of red wine in France reduced the prevalence of coronary diseases even with a traditional consumption of saturated fats and sugar. Studies believed that this paradox was due to the phenolic compounds in wine [4]. The grape pomace is the main solid organic waste from winery industries resulting from the pressing and/or fermentation processes. It is generated in large amounts in many parts of the world [5 - 7]. The main components of grape pomace are seeds and skin. Studies have shown the potential of phenolics and antioxidant fibers recovery from the skin [8, 9], as well as oil recovery from the seeds [10 - 12]. Generally, the grape pomace accounts for 20 - 30 % of the initial weight of the grape [13], and it is characterized by a high residual content of total polyphenols, depending on various factors such as grape variety, winemaking technique, etc. [14].

The most commonly used techniques for the isolation of phenolic compounds from plant material are the solid-liquid extraction [15 - 17] and the supercritical extraction, sometimes the last is modified with co-solvents to enhance the supercritical solvent polarity [18, 19]. Regarding solid-liquid extraction, solvents like methanol, ethanol, diethyl ether, ethyl acetate [20, 21], and their combinations have been widely used for the extraction of phenolics, often as aqueous mixtures with different proportions of water [21].

In the present work we studied the influence of different extraction techniques, particularly conventional solid-liquid extraction (conducted via magnetic stirrer), ultrasound and microwave (US and MW) assisted extraction. The total polyphenolic content (TPC), the antioxidant capacity (AOC) and the anthocyanin content (AC) of whole grape seeds and pomace extracts were examined. The duration of the process and the operating temperature were varied in order to find the optimal parameters.

In red wine processing, grapes are entirely involved in the fermentation and in this case, juice and pomace ferment together. The presence of the skin during this stage provides pigments such as anthocyanins, necessary to create the red color of the wine [22]. During winemaking, grapes are crushed and pressed, which does not alter their chemical composition. It is reported that approximately 70 % of the phenolic content is preserved in the grape pomace after processing [22, 23]. The potential use of grape by-products can be a promising alternative, not only motivated by environmental issues, but also by the possibility of enhancing food quality and developing high added-value ingredients and products [5, 24]. This kind of study could encourage the winemaking companies recycle the grape pomace instead of disposing it, which will prevent environmental problems, and thus - attribute an added value to a material commonly considered as a waste.

EXPERIMENTAL

The pomace and seeds from bio-Mavrud red grape were provided by the wine-making factory *Lozev wine Ltd*. in Trakiets, Bulgaria from the vintage of 2016.

The chemicals used for the experiment were ethanol 96 % v/v (Valerus, Bulgaria), methanol 100 %, HPLC grade sodium carbonate (> 99 %), gallic acid - anhydrous (> 99 %), DPPH (2,2-diphenyl-1-picrylhydrazyl), (Sigma-Aldrich Co, Germany), sodium nitrite, aluminum chloride hexahydrate (Merck, Germany), 2N solution Folin-Ciocalteu reagent, rutin hydrate, ammonium tetrahydrate, quercetin hydrate (≥ 95 %), tannic acid (≥ 91 %), pyrogallic acid (≥ 98 %), (+) – catechin hydrate (≥ 96 %), sodium hydroxide, (Sigma Aldrich, Germany). Deionized water purified by Elix 70 C Gulfstream - Merck was used throughout the experiment.

The same solvent (a 50 % vol. aqueous-ethanolic solution) and solid-liquid ratio (1:20) were used for all the experiments [25]. The samples were taken at different times during the conventional and ultrasound-assisted extractions in order to trace the kinetics of the processes. The measurements were carried out using PG INSTRUMENTS T60 UV-Visible spectrophotometer (United Kingdom).

Conventional stirring extraction

The mixture was agitated for 240 min by a magnetic stirrer MMS-3000 (Boeco, Germany) at ambient temperature and pressure and at constant stirring rate of 800 rpm.

Ultrasound-assisted extraction

This technique was conducted in an Ultrasonic bath "Siel" (Gabrovo, Bulgaria) for 240 min.

Microwave-assisted extraction

The extraction was carried out in a domestic microwave oven "Crown" with the lowest microwave power - 20 % of 1800 W. The extraction was performed in three stages each of them being 20 min, using fresh solvent and the same solid phase.

Total phenolic content (TPC)

The TPC was measured at 765 nm on a PG Instruments T60 UV/VIS spectrophotometer according to the modified Folin-Ciocalteau method [27, 28]. The TPC was presented as milligrams gallic acid equivalents per gram dry weight (mg GAE g^{-1} dw). Gallic acid is pure, stable and less expensive substance that is considered equivalent to most phenolics on a mass basis. The results were presented also as pyrogallic acid, tannic acid, vanillic acid and ferulic acid equivalents per gram dry weight (mg PAE g^{-1} dw; mg TAE g^{-1} dw; mg VAE g^{-1} dw and mg FAE g^{-1} dw).

Antioxidant capacity (AOC)

The reactivity of the extract with DPPH was estimated according to the method described by Brand-Williams et al. [29], and later changed by Sánchez-Moreno et al. [30]. The antioxidant capacity is defined as antiradical activity on a stable form of synthetic product DPPH. The decrease in its dark-purple color allowed us to estimate the effectiveness of the examined antioxidants. The incubation was performed at room temperature, and the absorbance was followed spectrophotometrically at 517 nm. The antioxidant activity was defined by plotting a calibration curve of Trolox, concentration range (2.5 - 175 μmol L^{-1}) vs. inhibition (%), C/I. The results were expressed as μmol Trolox equivalent antioxidant capacity per gram dry weight (μmol TEAC g^{-1} dw).

Total anthocyanin method

The total anthocyanin content of the grape pomace and seeds extracts was determined by the pH differential method according to Lee et al. [31] using a spectrophotometric assay whereby the absorbance of the extracts was measured at pH 1.0 and pH 4.5. The absorbance of the samples was determined at wavelength 510 and 700 nm, and the anthocyanin content was expressed as mg CGE g^{-1} dw (CGE- cyanidin-3-glucoside equivalent) and mg MGE g^{-1} dw (MGE - malvidin-3-glucoside equivalent) according to the equation:

$$CGE(MGE) = \frac{A * Mw * DF * V * 10^3}{\varepsilon * L * M}$$

where

$$A = (A_{510} - A_{700})_{pH1.0} - (A_{510} - A_{700})_{pH4.5}$$

Mw - the molecular weight of cyanidine-3-glucoside (449.2 g.mol^{-1}) or malvidin-3-glucoside (493.4 g mol^{-1}),
DF - the dilution factor,
V - volume of the extracting solution (L),
M - mass of the solid material (g),
ε - molar extinction coefficient (absorptivity) of cyanidine-3-glucoside (26900 L cm^{-1} mol^{-1}) or malvidin-3-glucoside (28000 L cm^{-1} mol^{-1}),
L - path length (1 cm).

RESULTS AND DISCUSSION

It is believed that the reduction of the particle size should increase the superficial area available for mass transfer. Pekić et al. [26] found that the grinding of grape seeds could reduce the extraction time but did not increase the yield of proanthocyanidins, and furthermore, caused a significant increase in the extraction of undesired concomitant components. As consequent they used entire grape seeds. In this study, we also study the entire seeds. It is important to know if the processes of grinding, sieving, collection and separation of the fractions could be avoided. This could save energy and time and the sustainable winemaking production that we were interested in, could become more economically and environmentally friendly. Furthermore, it could appeal to the winemaking factories most of which are currently disposing of the grape by-products to reuse them.

The extraction efficiency can be improved mainly by changes in the solvent type, particle size, temperature, and extraction time, as well as the presence of interfering substances in the matrix [17]. Recently traditional extractions with organic solvents are switched to novel techniques with lower consumption of organic solvents and reduced time. The US-assisted extraction is a non-conventional method that consists in sonication via sound waves above human hearing, but below microwave frequencies. The high temperature and pressure involved in the process destroy the plant cell walls and release its contents into the bulk.

Total Polyphenolic Content

It is well established that mixtures of alcohol and water are more efficient than the corresponding mono-component solvent system for extracting phenolic constituents from grape seeds [33 - 35]. For that reason, the extraction was carried out using 50 vol. % ethanol-in-water solutions [25]. Yilmaz and Toledo [34] found out that the phenolic content (as GAE) of ethanolic extracts from grape seed powder increased when the water in the mixture was increased from 0 to 30 vol. %, being constant for 30; 40 and 50 vol. %, and decreased for a higher percentage.

The MW-assisted extraction was conducted in a domestic microwave oven. During the preliminary test, we determined that the long extraction time in a microwave oven was not appropriate, because the solvent evaporated too fast in a non-modified domestic oven. Therefore, we decided to perform a three-stage extraction as each stage had a duration of 20 min and the solid material was mixed with a new portion of fresh

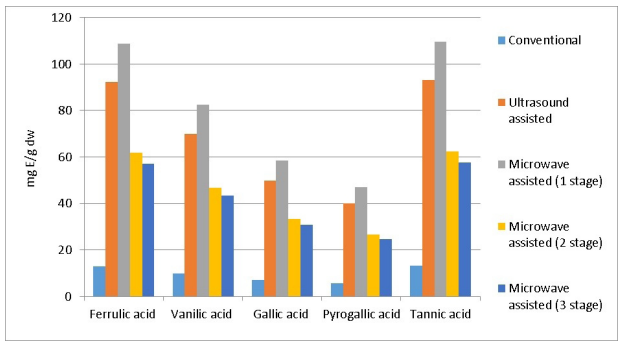

Fig. 1. TPC of grape seeds extracts prepared with different extraction methods expressed as mg equivalents of ferulic acid, vanillic acid, gallic acid, pyrogallic acid and tanic acid per g dry weight.

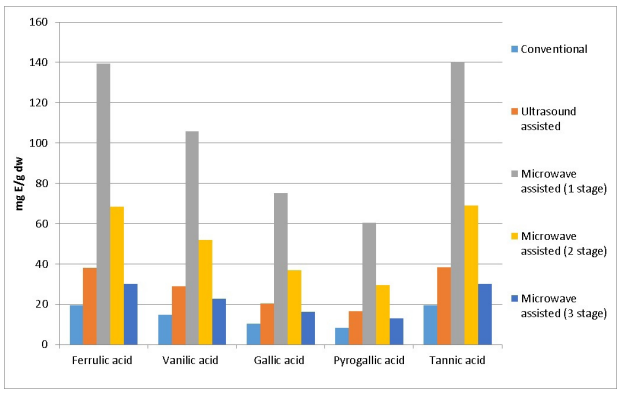

Fig. 2. TPC of grape pomace extracts prepared with different extraction methods expressed as mg equivalents of ferulic acid, vanillic acid, gallic acid, pyrogallic acid and tanic acid per g dry weight.

solvent at each stage. Many phenolic compounds were identified in grape seeds and pomace as reported by other authors [37 - 39]. Our experimental results concerning the TPC of grape seed and pomace extracts, prepared from non-grounded or chopped material extracted with an aqueous ethanolic solution (50 vol. %) and solid to solvent ratio 1:20 are summarized in Fig. 1 and Fig. 2.

As shown in Figs. 1 and 2 the yield obtained using MW-assisted extraction was higher for both seeds and pomace extracts. For the grape seeds, the results from the 1st stage of the MW-assisted extraction were comparable to those obtained from the US-assisted extraction. The situation was not the same for the pomace extracts: the yield obtained in the ultrasound bath was remarkably lower compared to the one from the microwave oven. We can conclude that for the TPC, the MW-assisted extraction was more suitable because it saved time, energy and solid material. The drawback was the loss of solvent due to evaporation. A further investigation in regards to the modification of the equipment with an additional unit for solvent condensation could be performed.

The conventional and the US-assisted extraction were both carried out for 240 minutes in order to trace the kinetics of the process (Figs. 3 - 6).

As can be seen in Fig. 3 the TPC of the extracts expressed as mg GAE g^{-1} dw increased in time, without reaching a plateau. From the beginning of the process until approximately the 80th min we observed no significant changes in the amount of the extracted phenolic compounds. This fact could be explained by the difficult permeability for the solvent at the beginning of the process. Seemingly it was caused by the hard shell of the whole seed that made the closed cells inaccessible. The structure of the plant matrix most likely changed in time and the subsequent rupture of the plant cell allowed access of the solvent to the enclosed phenolic substances. That may explain the following continuous increase of the extracted amounts. Thus, this technique was considered as not suitable for application when the purpose is time and energy saving.

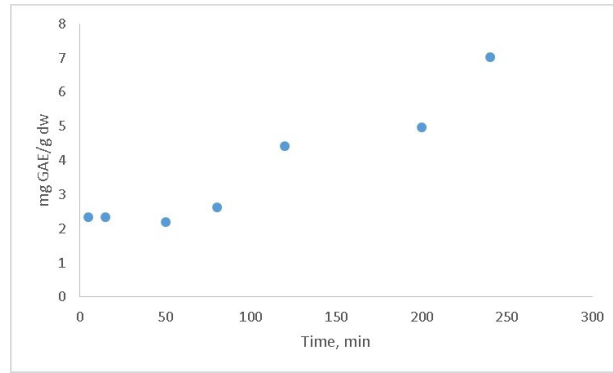

Fig. 3. Kinetics of the TPC of grape seed extracts prepared via conventional extraction.

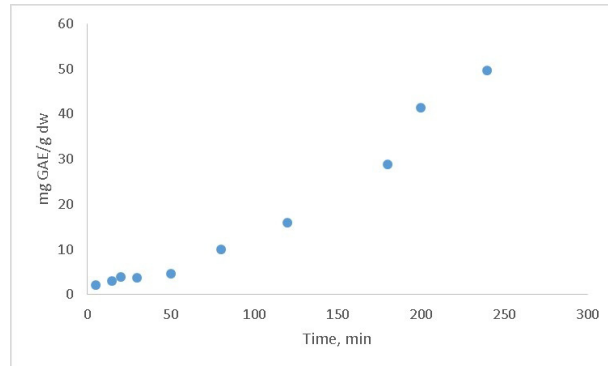

Fig. 4. Kinetics of the TPC of grape seed extracts prepared via US-assisted extraction.

As can be seen in Fig. 4 when the process was carried out in the US field, the overall trend was similar, but the amount of the extracted polyphenolic compounds was about 7 times higher. This fact may be attributed to the cavitation effect caused by the US field, as well as the local temperature increase in the bulk. The plateau was not reached during the time the experiment was carried out. Comparing these results with those obtained for the MW-assisted extraction, we can conclude that a significant amount of polyphenolic compounds was not extracted. During the 1st stage of the process 58.57 mg GAE g^{-1} dw were extracted for 20 min in the MW field and then 33.27 mg GAE g^{-1} dw during the 2nd stage and 30.8 mg GAE g^{-1} dw during the 3rd stage, while 49.73 mg GAE g^{-1} dw were obtained for 240 min in the US field and only 7.03 mg GAE g^{-1} dw via conventional extraction. From the results, we concluded that the external force field was favorable for the extraction through hard-shelled particles. In this case, the microwaves allowed the extraction of a larger amount of polyphenols for significantly shorter time and about nine times lower energy consumption (according to the apparatus specifications). The energy consumption of the US-assisted extraction was about six times lower compared to the conventional technique.

In Fig. 5 are presented the results obtained for TPC of the grape pomace extracts. It can be seen that until the 260th min the plateau was still not reached. From the beginning of the process until approximately the 80th min we observed an increase in the amount of the extracted phenolic compounds. This may be due to the bigger and more easily accessible mass transfer surface of the pomace particles. Then the velocity of the process decreased and the extraction of polyphenols did not

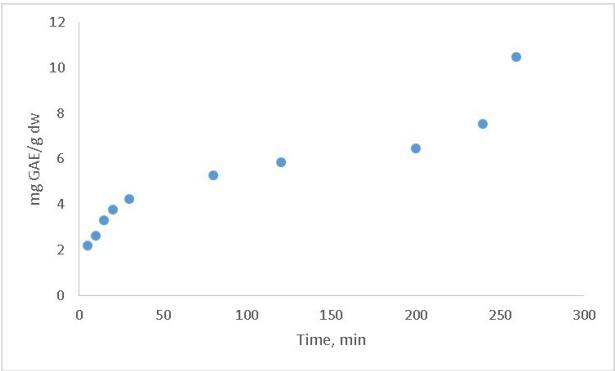

Fig. 5. Kinetics of the TPC of grape pomace extracts prepared via conventional extraction.

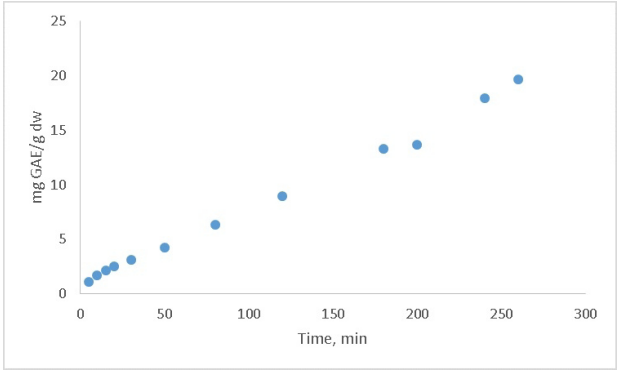

Fig. 6. Kinetics of the TPC of grape pomace extracts prepared via US-assisted extraction.

change significantly until the 200th min. It was assumed that after the 200th min, the cell walls of the plant material were destructed and the enclosed substances became available for the solvent. This could be the reason for the following continuous increase of the TPC leaching.

In Fig. 6 the amount of the extracted polyphenolic compounds in the US field increased constantly in time until 260 min. During the experimental time, a plateau was not reached. This indicated possible remains of hardly extractable valuable compounds in the raw material. A comparison between the results obtained for the different extraction techniques was made. During the 1st stage of the MWA process 75.02 mg GAE g^{-1} dw were extracted for 20 min, 36.78 mg GAE g^{-1}dw during the 2nd stage and 16.12 mg GAE g^{-1} dw during the 3rd stage. The TPC extracted in the MW field was about 3.7 times higher than the TPC from the US-assisted extraction (20.47 mg GAE g^{-1} dw) and 7 times higher than the yield from the conventional extraction (10.47 mg GAE g^{-1} dw). As mentioned above the MW field allowed the extraction of a greater amount of polyphenols for a significantly shorter time and lower energy consumption compared to the US and conventional extraction.

Antioxidant Activity

The research aimed to get better insight into the optimization of antioxidant and polyphenolic extraction from grape pomace and seeds from Mavrud. We investigated some variables selected based on the available literature about the same subject.

An obstacle considered by Brenes et al. [40] for grape pomace utilization is its composition which can be different depending on grape variety, location, fertilization conditions, soil and harvest period. However, these

Table 1. Antioxidant activity of the extracts produced via different extraction methods expressed as μmol Trolox equivalent antioxidant capacity per g dry weight.

Extraction technique	μmol TEAC g^{-1}	
	Seeds	Pomace
Conventional - 240 min	11.06 ± 3.6	21.67 ± 2.86
US-assisted - 240 min	32.34 ± 0.07	31.13 ± 0.21
MW-assisted (1st stage) - 20 min	33.36	23.20 ± 2.81
MW-assisted (2nd stage) - 20 min	32.42	30.13 ± 0.46
MW-assisted (3rd stage) - 20 min	32.15	31.44 ± 0.07

differences should not be a problem, since they represent different application possibilities. A better knowledge of the grape pomace composition could enable the possibility to find an industrial use [41] and to evaluate the importance of the raw material variability [13] on the final application. Despite a substantial number of studies using grape pomace for different applications, in reality they are often ineffective as they are not successfully implemented in larger scales [6].

Alcoholic and aqueous extracts (solvents not selective for phenols) from fruits processing by-products contain inevitably sugars and polysaccharides. From a commercial point of view, a higher purity would give a higher value to the extract. However, purification is an expensive step, inevitably separates free from bound polyphenolics, or different phenols classes (mixing of which may bring to antioxidant synergistic effect [42]). Depending on the extract application (dietary supplies, antioxidants into food systems) sugars removal might not be necessary.

Many works confirmed the correlation between high phenolic content and the antioxidant activity of the extracts [10, 14, 28]. The same was confirmed in our work. All analyses were performed in triplicate. The only discrepancy was related to the high inhibition of the grape pomace extracts obtained through the US-assisted extraction where, as shown in Fig. 2, the total polyphenolic content was lower. This may be due to the anthocyanins content.

The conventional and the ultrasound-assisted extractions were both carried out for 240 minutes in order to trace the kinetics of the processes (Figs. 7 and 8).

As can be seen in Fig. 7 the AOC of the extracts expressed as μmol TEAC g^{-1} dw increased in time and reached a plateau. The experimental data showed a curve with a typical pattern for conventional and ultrasound-assisted extractions. The steep part of the curve (approximately till the 50th min for the conventional and 100th min for the US-assisted extraction) corresponded to the fast kinetics when the leaching of valuable compounds was from the external surface of the seeds. Then the slope decreased which means that the substance was extracted from the interior of the particles where the access of the solvent was more difficult and the transfer to the solid-liquid interface was slower. About 120th min the curve reached a plateau and the extracted substances decreased with the driving force.

When the process was carried out in the US field, the overall trend was similar to the conventional extraction, but the antioxidant capacity of the extract was about 3 times higher. Comparing these results with those obtained for the MW-assisted extraction, we concluded that the antioxidant capacity of the extracts prepared via the US-assisted extraction was similar. During the 1st stage of the process 33.36 μmol TEAC g^{-1} dw were extracted for 20 min in the MW field and then 32.42 μmol TEAC g^{-1} dw during the 2nd stage and 32.15 μmol TEAC g^{-1} dw during the 3rd stage, while 32.34 ± 0.07 μmol TEAC g^{-1} dw were obtained for 240 min in the US field and only 11.06 ± 3.6 μmol TEAC g^{-1} dw via conventional extraction (Table 1). The MW field compared to US allowed the extraction of the same amount of compounds possessing AOC for a significantly shorter time and about nine times lower energy consumption.

In Fig. 8 are presented the results obtained for AOC of the grape pomace extracts and it can be seen that for the conventional extraction the plateau was still not reached until the 260th min. From the beginning of the process until approximately the 80th min an increase in the antioxidant capacity was observed. Then the velocity

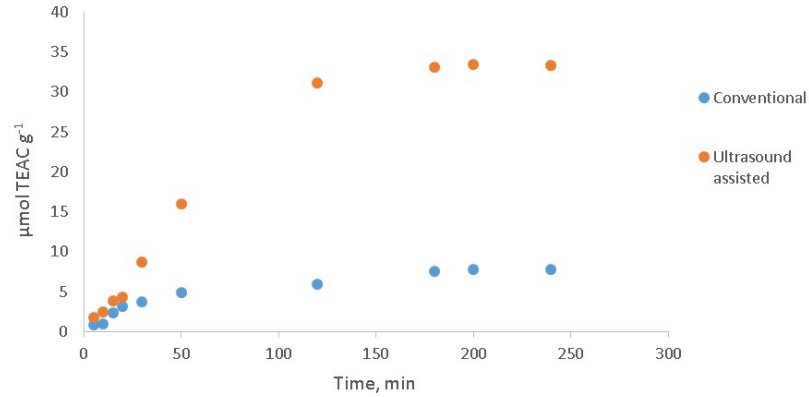

Fig. 7. Kinetics of the conventional extraction and US-assisted extraction from grape seeds.

of the process decreased and the extraction of antioxidant active compounds did not change significantly until the 200th min. Most likely at this time, the cell walls of the plant material were destroyed and the enclosed substances, initiated discharging into the bulk. This could be the reason for the following continuous increase of the AOC. As can be seen the extraction in the US field reached a plateau at about 180th min. A comparison between the results obtained for the different extraction techniques was made. During the 1st stage of the MW-assisted process 23.20 ± 2.81 µmol TEAC g^{-1} dw were extracted for 20 min, 30.13 ± 0.46 µmol TEAC g^{-1} dw during the 2nd stage and 31.44 ± 0.07 µmol TEAC g^{-1} dw during the 3rd stage. The amount of antioxidants extracted in the MW field was similar to the amount of antioxidants extracted via US-assisted extraction (31.13 ± 0.21 µmol TEAC g^{-1} dw) and slightly higher than the yield from the conventional extraction (21.67 ± 2.86 µmol TEAC g^{-1} dw) (Table 1). As mentioned above the MW field allows the extraction of a greater amount of polyphenols for a significantly shorter time and lower energy consumption. The processes were arranged according to the energy consumption from the lowest to the highest as follows: MW-assisted extraction 21.6 kW, US- assisted extraction (28.8 kW) and conventional extraction (132 kW).

Total Anthocyanins

Cacace and Mazza [36] presented results showing that the extraction of anthocyanins from black currants using ethanol-in-water solutions increased with ethanol concentration up to a maximum of about 60 vol. % and then decreased with further increase in the solvent concentration.

The frozen samples have been dried with an air conventional drier at 57 °C for 4 hours. The effect of the

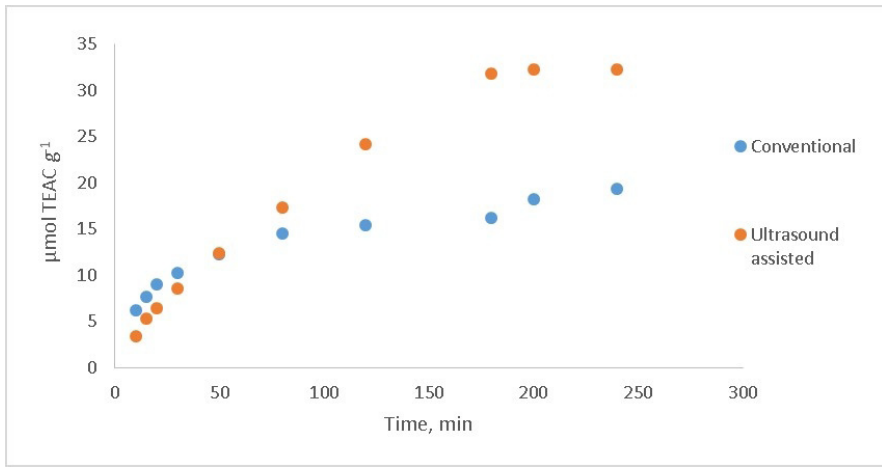

Fig. 8. Kinetics of the conventional extraction and US-assisted extraction from grape pomace.

Table 2. Anthocyanins content of the grape pomace extracts expressed as cyanidin-3-glucoside equivalent (mg CGE g^{-1} dw) and malvidin-3-glucoside equivalent (mg MGE g^{-1} dw).

	Conventional stirring extraction		Ultrasound-assisted extraction	
Time, min	CGE	MGE	CGE	MGE
5	15.17 ± 2.44	15.99 ± 2.57	6.18 ± 1.04	6.51 ± 1.09
10	24.81 ± 2.56	26.16 ± 2.70	15.28 ± 2.96	16.12 ± 3.12
15	35.36 ± 1.36	37.28 ± 1.43	10.05 ± 0.30	10.59 ± 0.31
20	43.10 ± 3.36	45.44 ± 3.54	36.53 ± 2.81	38.52 ± 2.97
30	61.88 ± 6.18	65.25 ± 6.51	35.59 ± 5.33	37.53 ± 5.62
50	78.26 ± 4.22	82.51 ± 4.45	48.15 ± 1.18	50.77 ± 1.25
80	92.12 ± 7.36	97.13 ± 7.76	71.60 ± 1.18	75.50 ± 1.25
120	107.39 ± 7.15	113.23 ± 7.53	88.67 ± 1.92	93.49 ± 2.03
180	116.73 ± 5.58	123.08 ± 5.88	112.33 ± 2.52	118.44 ± 2.65
200	123.16 ± 7.57	129.86 ± 7.98	123.32 ± 3.55	130.02 ± 3.75
240	132.10 ± 5.98	139.29 ± 6.31	136.82 ± 0.15	144.26 ± 0.16
260	154.71 ± 6.48	163.12 ± 6.83	141.01 ± 4.89	148.68 ± 5.15
	Microwave-assisted extraction			
Stage	CGE		MGE	
1 - 20 min	25.52		26.91	
2 - 20 min	15.32		16.15	
3 - 20 min	6.39		6.74	

temperature could not be generalized since it strongly depends on the typology of the compounds. For example, Cacace and Mazza [36] found a maximum of 30 - 35°C for the extraction of anthocyanins from ribes with ethanol 85 %. On the other hand, it was shown by Larrauri et al. [43] that drying red grape pomace peels at 60°C did not significantly affect the stability of polyphenols and antioxidant activity, and, indeed, it was reported an increase in the antioxidant capacity of grape extracts by means of a simple thermal treatment at 60°C, due to phenol polymerization [44, 45].

Anthocyanins are pigments characteristic of the red color and produced during ripening [46, 47]. They are highly susceptible to chemical transformations due to the action of agents such as light, temperature, oxygen, pH, solvents and metallic ions. Due to these characteristics, one of the great focuses of investigations on this compound is its stabilization for use as a natural colorant in the food industry. The main anthocyanins found in grape skin are: 3-O-glycosides of malvidin, petunidin, cyaniding, peonidin and delphinidin, however, factors such as variety, maturity and climate may alter the presence of these compounds [48, 49].

The experiments were carried out in triplicate to ensure better reproducibility and are presented in Table 2.

The trend of anthocyanins extraction from the grape seeds is not shown because the yields were too low. This could be due to the fact that in general, their level is higher in the grapes than in the seeds.

The yield for the extracts produced via MW and US-assisted extraction is lower. This could be due to the low selectivity of these methods as well as to the higher temperature generated in the microwave and ultrasound fields. We can conclude that among the three extraction techniques, conventional extraction is the most suitable for anthocyanin extraction.

CONCLUSIONS

It was determined that the microwave-assisted extraction of grape seeds provides higher polyphenolic content 12 times faster compared to the ultrasound-assisted extraction. The pomace extracts obtained in microwave field are 3.5 times richer in polyphenols than

those produced in an ultrasound bath.

The antioxidant activity for both microwave and ultrasound-assisted extractions was similar, but the time and energy, used for the microwave process were considerably reduced.

The use of microwave-assisted extraction showed an increase in the extraction yield with reduced extraction time and solid-phase quantity compared to conventional solid-liquid extraction (with a magnetic stirrer) and ultrasound-assisted extraction. The disadvantage is the loss of solvent. A modification of the microwave oven could eliminate this issue.

It was determined that the anthocyanins content of the extracts produced via microwave-assisted extraction is lower. This result could be related to the fact that this method has lower selectivity, so it could be used for applications that do not require high purity of the extracts.

Further experiments on the incorporation of the extracts into cosmetic products will be conducted.

An accurate economical evaluation of the incidence of the energy cost of the extraction stage on the overall production cost per unit mass of the final extract will allow confirmation of the choice of extraction method.

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