



Microwave-assisted extraction of phenolic compounds from red grape skins (*Babeasca neagra* variety)

Daniela Serea^{*} Nicoleta Stănciuc, Gabriela Bahrim, Oana-Viorela Nistor, Gabriela Răpeanu

"Dunărea de Jos" University of Galati, Faculty of Food Science and Engineering, Domnească Street, 111, RO-800201, Galati, Romania

**Corresponding author: daniela.serea@ugal.ro*

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Abstract: Red grape skins (*Babeasca neagra* variety) represent a valuable byproduct resulted in rose winemaking, due to their content in polyphenolic compounds, such as: flavan-3-ols, anthocyanins, flavonols, phenolic acids, etc.

The aim of this study was to extract phenolic compounds from red grape skins (*Babeasca neagra* variety) using a microwave oven. For microwave-assisted extraction, power (525 and 1050 W), extraction time (5, 10 seconds), solvent concentration (aqueous solutions of 50, 70 and 96% ethanol) was selected as independent variables. The dependent variables were total phenolic content (TPC), total monomeric anthocyanin content (TMA), total flavonoid content (TFC) and antioxidant activity (AA). The highest total monomeric anthocyanin (TMA) content of 7.05 ± 0.79 , mg C3G / g d.w., the total polyphenols (TPC) of 4.99 ± 1.13 mg GAE / g d.w., and the total flavonoid content (TFC) of 0.92 ± 0.16 mg CE/g d.w., was obtained using the following extraction conditions: 10 seconds of treatment at 1050 W, solid solvent ratio of 1:10 and ethanol-water mixture at a ratio of 70:30.

Red grape skin (*Babeasca neagra* variety) seems to be a valuable source that can be used to recover high value-added compounds for the formulation of new products (food supplements, cosmetics, medicines).

Introduction

Recently, economic and environmental problems have raised concerns about the large volume of waste generated in the food industries.

In this sense, one of the current challenges is to reuse such waste as "co-products" that can be reprocessed, in order to reduce their negative impact, as well as to obtain high value-added ingredients.

Wine industry generates a large amount of waste, which is generally used as organic fertilizer or intended for animal feed. The main residue of the wine industry is grape pomace, which is composed mainly of skins and seeds, being known for its high levels of phenolic compounds. The presence of these bioactive compounds, which have antioxidant and antimicrobial activity, adds value to these residues due to their potential for more noble applications, whether in the food, pharmaceutical or cosmetic industries.

Aims

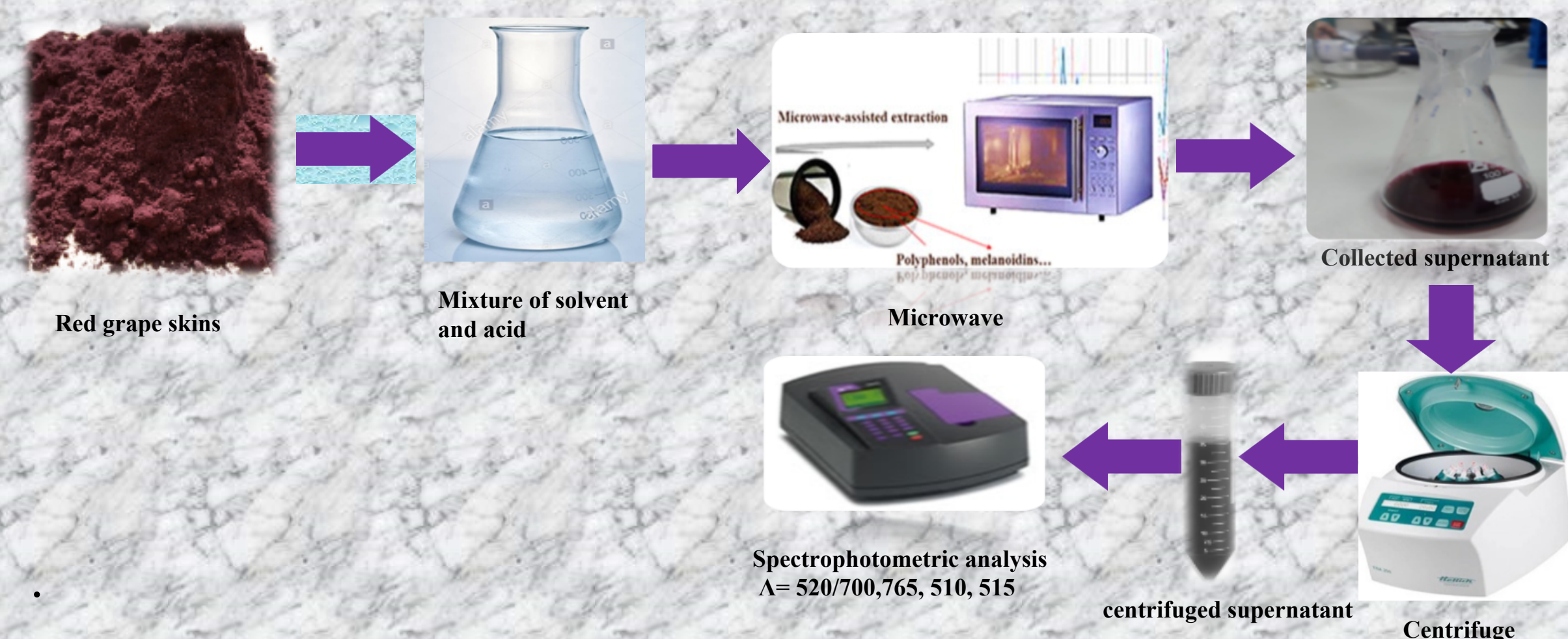
The purpose of this study was to recover polyphenolic compounds from red grape skin of the *Babeasca neagra* variety, using unconventional extraction methods (microwave-assisted extraction).

Materials and methods

The grapes (*Babeasca neagra* variety) were destemmed and then the separated berries were washed with water in a ratio of 1:2 (w/w). After washing, the skins were manually separated, washed with distilled water and wiped on paper towels to remove any residual pulp and were freeze dried.

The method used to extract bioactive compounds from red grape skin is microwave-assisted extraction (MAE). For this method, the variation of solvent, acid, power and extraction time was chosen. Therefore, three different concentrations of solvent (50%, 70%, 96% ethanol), two level of power (525 and 1050 W) and two extraction times (5 and 10 seconds) were used. The solvent used for extraction was acidified with three different acids, two organic (glacial acetic acid, 99.5% citric acid) and one inorganic acid (0.1 N hydrochloric acid).

Solid ratio:liquid of 1:10, 70% ethanol and power 525 W were chosen as the most suitable conditions for the recovery of phenolic compounds. Extracts obtained from red grape skins were characterized in terms of total monomeric anthocyanin content (TMA), flavonoid content (TFC), total polyphenol content (TPC) and antioxidant activity (AA). All determinations were performed in triplicate.



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Results and discussion

Total monomeric antocyanins content, C3G/g DW

Solvent / microwave power		Total anthocyanin content, mg C3G / g d.w.		
		Glacial acetic acid/ 10 sec	Acids/treatment times Citric acid 99.5%/ 10 sec	0.1 N hydrochloric acid/ 10 sec
50%	525W	4.37±0.85 ^d	5.59±0.49 ^{a,c}	6.18±0.65 ^{a,b}
	1050W	5.53±1.48 ^{a,d}	7.05±0.79 ^a	6.32±0.71 ^{a,b}
70%	525W	5.21±0.26 ^{b,d}	5.50±1.20 ^{a,d}	4.15±1.77 ^{a,e}
	1050W	4.66±0.42 ^{c,d}	5.15±0.60 ^{b,d}	4.63±0.16 ^{c,d}
96%	525W	1.89±0.49 ^e	3.86±0.20 ^{d,e}	4.31±0.92 ^{b,d}
	1050W	2.54±0.84 ^{d,e}	3.96±0.66 ^{d,e}	5.29±0.47 ^{a,d}

By using 70% ethanol acidified with citric acid, it was possible to extract the largest amount of anthocyanins of 7.05 ± 0.79 , mg C3G/g d.w. after 10 seconds of extraction at power of 1050 W.

Total flavonoids content, mg CE/g DW

Solvent / microwave power		Total flavonoid content, mg CE / g d.w.		
		Glacial acetic acid/ 10 sec	Acids/treatment times Citric acid 99.5%/ 10 sec	0.1 N hydrochloric acid/ 10 sec
50% ethanol	525W	0.74±0.22 ^{a,c}	0.82±0.10 ^{a,c}	0.92±0.16 ^{a,c}
	1050W	0.68±0.06 ^{c,c}	0.87±0.03 ^{a,b}	0.92±0.09 ^a
70% ethanol	525W	0.72±0.08 ^{a,c}	0.84±0.16 ^{a,c}	0.90±0.07 ^{a,b}
	1050W	0.79±0.13 ^{a,c}	0.80±0.16 ^{a,c}	0.91±0.10 ^{a,b}
96% ethanol	525W	0.53±0.06 ^c	0.54±0.02 ^{b,c}	0.89±0.23 ^{a,c}
	1050W	0.64±0.15 ^{a,c}	0.67±0.12 ^{a,c}	0.73±0.03 ^{a,c}

70% ethanol combined with 0.1N HCl, extracted the highest flavonoid content of 0.92 ± 0.16 mg CE/g d.w., after 525 W microwave treatment.

Total polyphenols, mg GAE/g DW

Solvent /microwave power		Total polyphenol content, mg GAE/g d.w.		
		Glacial acetic acid/ 10 sec	Acids/treatment times Citric acid 99.5%/ 10 sec	0.1 N hydrochloric acid/ 10 sec
50% ethanol	525 W	3.75±0.15 ^{b,f}	4.71±0.83 ^{a,b}	4.35±0.67 ^{a,c}
	1050 W	3.66±0.22 ^{c,f}	4.66±0.29 ^{a,c}	4.58±0.38 ^{a,c}
70% ethanol	525 W	3.20±0.58 ^{d,f}	3.78±0.27 ^{b,e}	4.99±1.13 ^a
	1050 W	3.18±0.53 ^{d,f}	3.90±0.25 ^{b,d}	4.22±0.29 ^{a,c}
96% ethanol	525 W	1.94±0.17 ^h	2.81±0.13 ^{e,h}	2.82±0.32 ^{a,h}
	1050 W	2.04±0.24 ^{g,h}	2.73±0.28 ^{f,h}	3.02±0.25 ^{d,g}

0.1N hydrochloric acid in combination with 70% ethanol extracted the highest polyphenol content of 4.99 ± 1.13 mg GAE/g d.w. after 10 sec of microwave treatment at 525 W.

Antioxidant activity, mMolTrolox/ g DW

Solvent / microwave power		Antioxidant activity, mMol Trolox/g d.w.		
		Glacial acetic acid/ 10 sec	Acids/treatment times Citric acid 99.5%/ 10 sec	0.1 N hydrochloric acid/ 10 sec
50% ethanol	525W	12.69±0.59 ^{c,d}	12.87±0.31 ^{c,d}	16.00±0.44 ^{b,c}
	1050W	5.53±1.93 ^{a,f}	6.80±0.85 ^{a,f}	16.32±0.78 ^b
70% ethanol	525W	16.25±0.77 ^b	5.97±0.63 ^{e,f}	2.67±2.34 ^f
	1050W	10.21±2.03 ^{d,e}	12.73±.58 ^{c,d}	12.05±0.86 ^d
96% ethanol	525W	19.30±0.40 ^a	13.13±0.82 ^{c,d}	16.02±1.53 ^{a,c}
	1050W	18.8±0.46 ^a	11.84±1.12 ^d	10.42±1.57 ^d

Microwave-assisted extraction with pure ethanol acidified with glacial acetic acid at power of 525 W for 10 seconds, allowed the extraction of polyphenolic compounds that possess an antioxidant activity of 19.30 ± 0.40 mMol Trolox/g d.w.

CONCLUSIONS

The processing of red grapes generates large quantities of by-products each year, which are not usually recovered.

According to our results, red grape skin extracts are rich in biologically active compounds that can be used to obtain a functional product with added value.