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Extraction of biologically active compounds from *Aronia melanocarpa*: Comparison of techniques and multiple response optimization

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Abstract: The presented paper deals with the selection of most efficient extraction method for obtaining biologically active compounds (mainly antioxidants) from the black chokeberry (*Aronia melanocarpa*) fruits. Two conventional methods (maceration and Soxhlet extraction) as well as a more recent accelerated solvent extraction (ASE) were employed. The extracts were evaluated by means of their overall yield, antioxidant activity (TEAC assay), total phenolic content (TPC), ascorbic acid (AA), and malic acid (MA) content. Both Soxhlet extraction and ASE led to the highest overall yields (42 %) of the extract; however, in terms of TEAC and TPC, the extracts obtained by ASE were usually superior. The highest TEAC value (227.7 mg TE/g) as well as the highest TPC (67.9 mg GAE/g) were obtained by ASE using 40 % (v/v) ethanol at 140 °C after 30 min. The ASE method was further analyzed using a 2^3-factorial design where the effect of temperature (40—140 °C), extraction time (5—30 min), and solvent composition (40—96 % ethanol) was investigated. While temperature and solvent composition significantly affected the extract properties, the effect of extraction time was small or even insignificant. The regression model obtained from ANOVA was further used for multiresponse optimization of ASE conditions using the global desirability function as a criterion for overall extract quality.

Keywords: Accelerated solvent extraction, antioxidant activity, desirability function, factorial design

Introduction

Genus *Aronia* consists of three distinct species, namely *Aronia melanocarpa* (black chokeberry), *Aronia arbutifolia* (red chokeberry), and their potential hybrid *Aronia prunifolia* (purple chokeberry) (Jurendić and Ščetar, 2021). Owing to their astringency, black chokeberry fruits are rarely consumed in their raw form; however, there is a substantial market for juices, purées, jams, syrups, and various food supplements derived from black chokeberry fruits (Jia et al., 2022; Çelik et al., 2022). In fact, the astringent nature of these fruits is mainly caused by exceptionally high amounts of polyphenols (Sidor et al., 2019), which are considered beneficial for human health (Cory et al., 2018). Polyphenols are secondary metabolites found abundantly in certain parts of plants, especially in fruits, seeds, and leaves (Szwajkowska-Michałek et al., 2020; Dufour et al., 2018; Gao et al., 2019; Wu and Zhou, 2021; Swallah et al., 2020; Bae et al., 2022). In general, polyphenols can be divided into three classes: phenolic acids, flavonoids, and non-flavonoids (Rudrapal et al., 2022). Oszmiański and Wojdylo (2005) identified procyanidins, oligomeric and polymeric catechins/epicatechins as the major classes of polyphenolic compounds present in chokeberries. Even though chokeberries are usually associated with high amount of polyphenols, like chlorogenic acid, neochlorogenic acid, and anthocyanins, they are also moderately rich in other beneficial plant-derived compounds, such as ascorbic acid, β-carotene, and dietary fiber (about 55 g/kg, (Chiorean et al., 2022)).

Notwithstanding the benefits of consuming native food (Aguilera and Toledo, 2022), there is a growing demand for various food supplements in which beneficial compounds are present in significantly higher concentrations or have better organoleptic properties. Extraction is frequently employed in the manufacturing of such supplements. However, conventional extraction techniques such as maceration and solvent extraction at room temperature are rather time consuming and require large amounts of solvents whose removal (or recovery) is an energy-demanding process. Moreover, these techniques may not yield extracts of the desired quality and/or quantity. Recently,

more efficient and "green" methods of extraction have been employed at industrial scale (Chemat et al., 2012). Among said methods, accelerated solvent extraction (ASE) is especially valued for relatively simple implementation and ease of full automation (Repajić et al., 2020). In ASE, extraction is carried out using a liquid solvent which can be heated above its normal boiling point as the process takes place in a closed system at elevated pressure. The elevated temperature employed in ASE improves both the kinetics and the equilibrium of extraction by increasing the diffusion rate and solubility of analytes while decreasing the solvent viscosity and surface tension (Alhallaf et al., 2022; Khanyile et al., 2022; Efthymiopoulos et al., 2019). Moreover, high pressure improves penetration of the solvent into porous matrices (Efthymiopoulos et al., 2019) thus improving the overall contact surface area. Apart from the operation at high pressures, the closed system also makes ASE suitable for extraction of oxygen-sensitive compounds (Liang et al., 2022).

This work deals with qualitative and quantitative comparison of extracts from *Aronia melanocarpa* fruits obtained by various extraction techniques. The results of ASE were compared with those of conventional methods, maceration and Soxhlet extraction. Since the performance of ASE depends on various parameters (solvent used, extraction time, temperature, pressure), a 2^k factorial design was employed and the effects of solvent, extraction time and temperature on five chosen extract properties were assessed. Finally, the extraction parameters were optimized with respect to various extract properties using the response surface methodology with the global desirability function.

Material and Methods

Plant material and chemicals

Chemicals used in the study were of analytical grade and were purchased from local and international vendors and used without further purification. Food grade ethanol (96 % v/v, Centralchem, Slovakia) and its mixtures with deionized water in various proportions (80, 60, 40 % v/v) was used as extraction solvent.

Fresh *Aronia melanocarpa* fruits originating from Ekofarma Aronia in Central Slovakia were ground by blender ETA 1-012 (ETA a.s., Slovakia) and dried in a thin layer at 60 °C for 24 hours. Dry biomass was further pulverized and extracted by means of maceration, Soxhlet extraction, or ASE. Between the various stages of handling, the plant material was stored in a freezer at –20 °C.

Extraction procedures

ASE was carried out using accelerated solvent extractor Dionex ASE 350 (Thermo Scientific, MO, USA) with 5 g of pulverized biomass. The 2^k factorial design was performed using 96 % and 40 % (v/v) ethanol for 5 min and 30 min at 40 °C and 140 °C. Center point conditions were: 17,5 min, 90 °C, and 68 % (v/v) ethanol. In all ASE runs, the pressure was maintained at 10.3 MPa.

Maceration was done at 22 °C for 2 hours using ethanol—water mixtures (40 %, 60 %, 80 %, and 96 %, v/v) and the biomass to solvent ratio of 1:10 (w/w) with 5 g of pulverized biomass. During maceration, the mixture was stirred in a laboratory stirrer.

Soxhlet extraction was carried out for 2 hours using Soxtherm apparatus (Gerhardt, Bonn, Germany) with an extraction thimble filled with 5 g of pulverized biomass. The biomass to solvent ratio was the same as in case of maceration; extraction temperature (78—83 °C) was determined by the boiling point of the solvent (ethanol 40—96 % v/v).

Finally, the extracts were dried in a rotary evaporator Heidolph Hei-VAP Precision, (Heidolph instruments, Germany) under reduced pressure at 40 °C until a gum-like semisolid matter was obtained. Dried extracts were used for all subsequent analyses.

Antioxidant activity

Trolox equivalent antioxidant capacity (TEAC) assay was carried out using a stock solution of DPPH (350 $\mu mol \cdot L^{-1}$ in ethanol) and extract samples dissolved in ethanol (0.1 $g \cdot L^{-1}$); the Trolox calibration curve was measured in the concentration range of 5—75 $\mu mol \cdot L^{-1}$. The spectroscopic assay was performed using a microplate reader BioTeK EPOCH 2 (Germany) where an extract sample of 200 µL was mixed with 75 µL of DPPH solution. Absorbance was measured every 10 min over a 1-hour interval at λ = 517 nm.

Total phenolic content

Total phenolic content (TPC) was determined in extract samples dissolved in water (10 $g \cdot L^{-1}$) and homogenized in an ultrasound bath; gallic acid in the mass concentration range of 0.001—0.9 $g \cdot L^{-1}$ was used for the calibration curve construction. The assay was carried out using a 100 µL sample of extract mixed with 500 µL of Folin-Ciocalteu reagent. After 3 min, the mixture was alkalized with 1.5 mL of sodium carbonate (20 % solution), filled up with water to 10 mL and left in dark for 2 hours. Subsequently, absorbance of the mixture was measured at the wavelength of 765 nm using a microplate reader BioTeK EPOCH 2 (Germany).

HPLC analysis of organic acids

HPLC analysis was conducted using 1260 Infinity II LC System (Agilent Technologies, USA) with a diode array detector (190—950 nm). The extracts were diluted in water (1 $g \cdot L^{-1}$); solutions of ascorbic acid, malic acid, and fumaric acid were prepared in 0.001—1 $g \cdot L^{-1}$ mass concentration range for calibration. Mobile phase was 0.020 $mol \cdot L^{-1}$ solution of potassium dihydrogen phosphate buffered with phosphoric acid to pH = 2.4; HALO AQ-C18 column was used as the stationary phase. Analysis conditions were as follows: flow rate of 0.6 $mL \cdot min^{-1}$, sample volume of 20 µL, pump pressure of 307 bar.

Statistical analyses

All quantitative analyses were performed in duplicates; the mean values with standard deviation are presented if not stated otherwise. ANOVA was performed using Minitab 17 (Minitab Inc., PA, USA); optimization of the response surface was done using Design-Expert 13 (Stat-Ease, MN, USA). Data plots were created in OriginPro 8.1 (OriginLab, MA, USA).

Results and discussion

Maceration and Soxhlet extraction

Maceration and Soxhlet extraction are considered "classical" methods for extraction of valuable components from biomass. Maceration is especially suitable for thermally sensitive compounds; however, despite the process being usually performed for several days, it may provide low yields. Table 1 lists the overall yields from both maceration and Soxhlet extraction.

In case of maceration, the highest overall yields were obtained using 40 % and 60 % ethanol; higher ethanol fractions led to only slightly lower yields. Almost identical trend with respect to solvent composition (r = 0.96, P < 0.05) was observed for the Soxhlet method, however, it led to yields approximately twofold higher (up to 42.2 %), most probably due to extraction at the boiling point. It is generally recognized that elevated temperatures contribute to extraction efficiency more prominently than prolonged extraction time (Hidalgo and Almajano, 2017).

Regarding the antioxidant activity, both conventional methods led to comparable results except for maceration with 96 % ethanol, which gave inferior product in almost all analyzed properties. Except for this solvent, the TEAC values fell into a narrow range from 158 to 185 mg TE per gram of dry extract. A direct comparison of these results with published ones is difficult not only because of differences in the extraction conditions but also due to the changes in antioxidants content during the ripening of chokeberry fruits (Ünal et al., 2023). Since phenols are probably responsible for a major part of antioxidant activity of chokeberry extracts, it is not surprising that TEAC and TPC values are highly correlated for both methods (r = 0.96—0.97, P < 0.05). Similarly, the AA and MA content did not vary much with the extraction method employed or with extraction conditions. The AA content in extracts was remarkably low (mostly bellow 6 mg/g); however, the *Aronia melanocarpa* fruits are reported as a rather poor source of dietary AA with only 0.13 mg AA per gram of fresh fruit (Benvenuti et al., 2004). It is interesting to note that while maceration led to significantly lower yields, all qualitative parameters of the extracts were similar if not better compared to Soxhlet extraction. However, with respect to the starting weight of the fruit biomass, the Soxhlet method led to approximately twofold increase in the total amount of extractives.

Results summarized in Table 1 show that a binary solvent system containing aqueous mixtures of or-

Tab. 1. Yield and other extract parameters obtained by conventional methods (extraction time 120 min).

	Temperature (°C)	Solvent (EtOH %, v/v)	Yield[a] (%)	TEAC[b] (mg TE/g)	TPC[c] (mg GAE/g)	AA (mg/g)	MA (mg/g)
Maceration	22	96	14.9	86.3 ±0.5	38.2 ±0.9	5.66 ±0.05	32.5 ±0.2
	22	80	16.3	178.3 ±0.1	56.6 ±0.4	5.91 ±0.07	40.2 ±1.1
	22	60	18.0	184.5 ±0.3	57.8 ±0.8	5.97 ±0.02	40.8 ±0.2
	22	40	17.9	157.8 ±0.9	47.6 ±0.4	6.09 ±0.04	32.2 ±0.4
Soxhlet extraction	78	96	22.7	137.1 ±2.0	46.4 ±1.4	5.33 ±0.03	30.1 ±0.2
	79	80	36.9	167.8 ±3.8	60.9 ±2.7	5.47 ±0.02	32.2 ±0.2
	81	60	42.2	166.7 ±0.6	60.1 ±1.4	5.51 ±0.01	33.1 ±0.1
	83	40	42.0	158.7 ±0.2	59.6 ±0.4	5.58 ±0.01	28.8 ±0.2

[a]overall yield of dried extract with respect to the starting biomass (determined from extract mass)

[b]trolox equivalent antioxidant capacity in mg of Trolox equivalents per gram of dry extract

[c]total phenolic content in mg of gallic acid equivalents (GAE) per g of dry extract

ganic solvents is more effective than a single-component solvent system (ethanol) in the extraction of phenolic compounds. While ethanol is responsible for disrupting the bond between the solutes and the plant matrix, water plays an important role in matrix swelling thus improving the mass transport of extractives between the phases.

Comparison of ASE with conventional methods

Due to the influence of multiple factors (solvent, time, temperature) and monitoring of several parameters of the resulting extracts, the comparison of ASE with conventional extraction methods is not straightforward and unambiguous. Thus, the performance of ASE was investigated using a 2^3-factorial design in a wide range of conditions; the investigated factors and their levels are listed in Table 2. For the sake of simple graphical comparison with conventional methods, the results of ASE performed at 140 °C for 30 min (for TPC and TEAC) and at 40 °C for 30 min (for AA and MA) were chosen. Figure 1 compares all three methods with respect to TEAC, TPC, AA and MA content. In all cases, the ASE method provided better extract quality while the overall yield (see Table 3), after 30 min at 140 °C using 96 % and 40 % ethanol, ranged between 23 % and 42 %, respectively. As it is further discussed in section *ANOVA of ASE performance*, extract of similar yield and quality can be obtained even after 5 min of ASE which corresponds to a significant improvement compared to conventional methods.

Tab. 2. Values and labelling of factors employed in 2^3-factorial design of ASE.

Label	Factor	Coded levels	
		–1	+1
A	Temperature (°C)	40	140
B	Time (min)	5	30
C	Solvent composition (% EtOH, v/v)	40	96

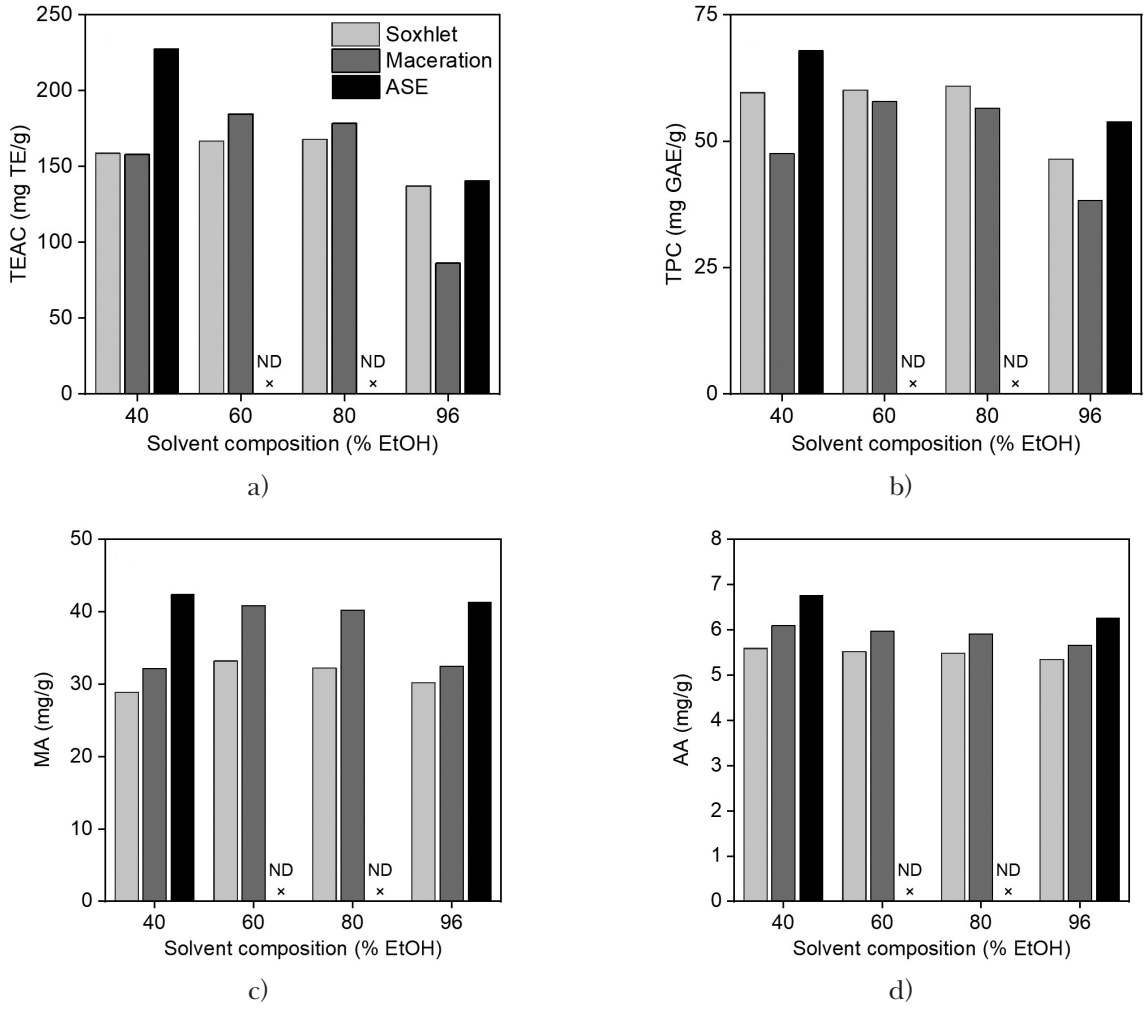


Fig. 1. Antioxidant activity (a), total phenolic content (b), malic acid content (c), and ascorbic acid content (d) of extracts obtained using various extraction methods and solvent composition (ND — value not determined).

Tab. 3. Yield and other extract parameters obtained by ASE.

Run #	Coded levels			Yield	TEAC	TPC	AA	MA
	A	B	C	(%)	(mg TE/g)	(mg GAE/g)	(mg/g)	(mg/g)
1	−1	−1	−1	25.8	125.9 ±0.3	51.7 ±1.6	6.52 ±0.01	42.4 ±0.3
2	+1	−1	−1	37.1	219.7 ±1.4	66.7 ±1.1	6.12 ±0.01	36.4 ±0.1
3	−1	+1	−1	26.9	149.6 ±2.0	53.7 ±0.9	6.76 ±0.03	40.4 ±0.2
4	+1	+1	−1	41.9	227.7 ±1.2	67.9 ±0.5	5.93 ±0.01	31.7 ±0.3
5	−1	−1	+1	17.3	83.5 ±0.9	31.8 ±0.7	6.19 ±0.09	36.5 ±0.2
6	+1	−1	+1	22.1	128.7 ±0.3	48.8 ±0.4	5.28 ±0.01	30.6 ±0.4
7	−1	+1	+1	18.1	91.7 ±0.8	38.8 ±1.5	6.26 ±0.08	41.4 ±0.3
8	+1	+1	+1	23.7	140.7 ±7.0	53.8 ±1.1	5.09 ±0.10	7.0 ±0.2

ANOVA of ASE performance

Additional ASE experiments in a 2^3-factorial design arrangement were conducted to assess the effect of solvent composition, extraction time, and temperature on the overall yield of the extract, amount of malic acid (MA), ascorbic acid (AA), and phenolic compounds (TPC) in the extract from *Aronia melanocarpa L.*, and its antioxidant activity (TEAC). Marginal levels employed for each factor are given in Table 2; extraction pressure was kept at 10.3 MPa in all runs. The analysis was performed with respect to main effects and all possible pairwise interactions; regression model for each response was:

$$y_i = \sum_i \beta_i x_i + \sum_{i<j} \beta_{ij} x_i x_j \qquad (1)$$

In addition to 2^3 corner points a center point with five repetitions was included to assess repeatability of the results and to check for curvature of the response surface. High *P* values corresponding to the quadratic curvature for all responses (see Table 4) suggest that first-order model with pairwise interaction terms is adequate.

As it can be seen from Table 4, all three factors affect the overall yield of the extract with statistical significance ($P \leq 0.001$). Among the factors, the positive main effect of temperature and negative main effect of ethanol content in the solvent dominate over a surprisingly small effect of extraction time. As a matter of fact, in all responses (overall yield, TEAC, TPC, AA, and MA) the extraction time is the weakest factor and even fails to attain significance in case of AA and MA content. Similarly, the interaction terms involving time (*AB* and *BC*) are mostly insignificant or borderline significant, which indicates that relatively rapid extraction equilibrium is achieved during ASE regardless of the temperature and solvent used. In terms of overall yield, TEAC, and TPC, the best results were obtained using 40 % ethanol at 140 °C. On the other hand, AA and MA content was maximized at 40 °C (with only a negligible effect of time). This can be explained by increased solubility of phenolic compounds, as well as structure breakdown following their release from macromolecules due to higher temperature. Chokeberry is a rich source of polyphenols with antioxidant properties which also explains the increase of TEAC with higher temperature. In case of organic acids, thermal degradation could be the reason of their decrease

Tab. 4. Regression coefficients and *P* values from ANOVA of 2^3-factorial design.

	Yield (%)		TEAC (mg TE/g)		TPC (mg GAE/g)		AA (mg/g)		MA (mg/g)	
	Coeff.	***P***	**Coeff.**	***P***	**Coeff.**	***P***	**Coeff.**	***P***	**Coeff.**	***P***
Constant	26.62	< 0.0005	145.6	< 0.0005	51.66	< 0.0005	6.01	< 0.0005	33.27	< 0.0005
A	4.60	< 0.0005	33.26	< 0.0005	7.63	< 0.0005	–0.41	< 0.0005	–6.87	0.002
B	1.02	0.001	6.49	0.001	1.92	< 0.0005	–0.009	0.40	–3.17	0.052
C	–6.30	< 0.0005	–34.78	< 0.0005	–8.35	< 0.0005	–0.31	< 0.0005	–4.42	0.015
AB	0.57	0.012	–1.50	0.2	–0.34	0.009	–0.084	< 0.0005	–3.89	0.025
AC	–1.99	< 0.0005	–9.72	< 0.001	0.36	0.007	–0.11	< 0.0005	–3.19	0.051
BC	–0.43	0.036	–1.42	0.2	1.09	< 0.0005	–0.021	0.09	–1.49	0.30
Curvature	—	0.95	—	0.62	—	1.00	—	0.60	—	1.00
R^2	0.9954		0.9947		0.9993		0.9957		0.8193	

after exposition to high temperature, especially for AA, which is known to have very poor thermal stability (Dönmez and Kadakal, 2023; Tomsone et al., 2020). Such co-occurrence of high yield, TEAC, and TPC on one side and high AA and MA content on the other can also be seen from pairwise correlations among these variables: yield, TEAC, and TPC all exhibit strong positive correlation among themselves ($P < 0.001$); the same applies to the AA and MA content ($r = 0.849$, $P < 0.001$). Thus, high yield is a good predictor of both high TEAC and high TPC values (and *vice versa*).

In general, the AA content exhibited the smallest variability among the evaluated properties of the extracts. Based on the thermolabile nature of AA, a detrimental effect of extraction at 140 °C on its content is not surprising; however, the effect is quite small (6.5 *vs.* 6.1 mg/g of AA after 5 min at 40 °C and 140 °C, respectively). Moreover, contrary to expectation, the effect of elevated temperature is essentially independent from extraction time (6.1 *vs.* 5.9 mg/g of AA after 5 and 30 min using 40 % ethanol, respectively).

Optimization of ASE performance with desirability function

Finding optimum conditions for the extraction of a complex matrix such as *Aronia melanocarpa* fruits is a typical problem of multiple responses: certain set of conditions may lead to optimization of TEAC or TPC while the content of AA and MA is maximized at a different set of conditions. Thus, a trade-off between different desired properties of the extract is usually sought by maximizing appropriately defined global desirability function. In this case, all responses (yield, TEAC, TPC, AA, and MA) can be considered desirable and the corresponding partial desirability functions d_i can be written as

$$d_i = \frac{y_i - y_i^{\min}}{y_i^{\max} - y_i^{\min}}, \quad (2)$$

where y_i is the particular value of the i-th response and $y^{\min}$ and $y^{\max}$ are the extreme values found during factorial design. Global desirability function is usually expressed as a weighed product of partial desirability functions:

$$D = \left(\prod_i d_i^{w_i} \right)^{1/\Sigma w_i}, \quad (3)$$

where w_i is the weighing coefficient associated with the importance of the i-th response variable. It should be noted that, in this case, the choice of weights is rather arbitrary as there is no quantitative measure of "beneficial" effect of extract properties and the weights ultimately may determine the optimum solution. In this study, the overall yield ($w = 4$) was prioritized over both TEAC and TPC ($w = 2$) as both parameters are expressed per gram of extract and such weighing maximizes the absolute amount of desired extractives with respect to the starting biomass. Concentration of ascorbic acid, which is more abundant in other dietary sources, was optimized with $w = 1$; concentration of malic acid was omitted from global desirability as its beneficial properties are not conclusive. Apart from the extract properties, the global desirability function also included partial desirability functions for extraction time and temperature:

$$d_i = \frac{x_i^{\max} - x_i}{x_i^{\max} - x_i^{\min}}, \quad (4)$$

so that short extraction times and low extraction temperatures are favored. Optimization (maximization of D function) was done using Design-Expert 13 (Stat-Ease, Minneapolis, MN, USA) software; results (in uncoded values) are summarized in Table 5. Since the overall yield was given the highest weight in the global desirability, the location of optimum is quite close to the highest yield (see Table 3); however, inclusion of time and temperature into the optimization procedure led to a much more economic set of conditions (119 °C and 6 min).

Tab. 5. Factors maximizing the global desirability function and the corresponding responses.

Factor/response	Value	d_i
Temperature (°C)	119.1	0.210
Time (min)	6.3	0.948
Solvent composition (% EtOH, v/v)	40	–
Yield (%)	35.2	0.727
TEAC (mg TE/g)	199.0	0.801
TPC (mg GAE/g)	63.7	0.883
AA (mg/g)	6.2	0.655
MA (mg/g)	39.1	–

Conclusions

Compared to maceration and Soxhlet extraction, the ASE method has proven to be more efficient for extracting the valuable compounds from *Aronia melanocarpa* fruits. The results of ANOVA show that temperature, extraction time, and ethanol content significantly affect the overall yield, antioxidant activity and total phenolic content of extracts. While elevated extraction temperatures have positive effect on all the aforementioned extract parameters, the content of ascorbic acid and malic acid show an opposite trend. Despite meeting the significance criteria, the effect of time is surprisingly small, and 5 min of ASE can provide extracts of similar quan-

tity and quality as do conventional methods after 120 min. Solvent composition (ethanol percentage) was shown as the most important factor affecting the extract yield and properties regardless of the extraction method; the best results were obtained using water—ethanol mixtures containing 40—60 % (v/v) of ethanol. In addition to the results of factorial design, a different set of optimum conditions for ASE was obtained by optimizing the global desirability function with a goal to maximize extract yield and quality while considering the process economy. Tailoring plant extracts using the desirability function can be valuable especially when various properties of the extract lead to contradictory sets of optimum conditions.

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