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Impact of Different Solvents and Temperatures on the Extraction of Bioactive Compounds from Rose Fruits (*Rosa rugosa*) Pomace

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Abstract: The use of waste brings many environmental and economic benefits to the country. One of the by-products of the fruit industry in Poland is rose fruits pomace. Rose fruit has great nutritional value and is a rich source of beneficial bioactive compounds. The aim of this study was to investigate the effects of temperature (25, 45, and 65 °C), time extraction (15, 30, 45, and 60 min), and different solvents on the recovery of total phenolic compounds (measured by Folin–Ciocalteu) and L-ascorbic acid (measured by the HPLC method) of rose fruits (*Rosa rugosa*) pomace. Higher temperatures (45 °C and 65 °C) showed a higher content of L-ascorbic acid but also faster degradation of this acid after 30 min of extraction. The highest content of polyphenolic compounds was obtained using 50% acetone at 65 °C (average 37.28 mg gallic acid equivalents/g of freeze-dried pomace) and the lowest using 100% acetone at 25 °C (average 12.46 mg gallic acid equivalents/g of freeze-dried pomace). The highest yield of L-ascorbic acid from pomace was obtained using water as a solvent (average 33.64 mg L-ascorbic acid/g of freeze-dried pomace). Rose fruits pomace extracts could therefore be used as natural bioactive molecules for many industrial applications.

Keywords: rose fruits; extraction; polyphenols; L-ascorbic acid; pomace

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1. Introduction

Poland is a very important producer of fruit and fruit processing products, such as juices, pulps, and jams, which results in an increase in waste associated with their processing. Currently, manufacturers strive for waste-free production, and waste is increasingly managed. In large amounts, these products are used as compost or added to animal food. Sometimes, for a new participant in the production chain, a by-product may become the main raw material, which is a source of valuable, previously ignored, or not fully obtained ingredients. Some waste may have health-promoting properties; others may be used as functional additives, replacing synthetic compounds, or supporting the functioning of the human body. Reusing waste has many advantages, including reducing the costs of waste disposal, reducing seasonality, and improving the level of hygiene. Such behavior brings not only financial and environmental benefits but is also consistent with the concept of a circular economy [1]. During the production of juices, the waste mass consists of pomace, the amount of which can amount to 25% of the raw material [2,3]. Fruit pomace is still rich in bioactive compounds, primarily polyphenols and dietary fiber, which can be recovered [4,5]. Polysaccharides included in fiber can perform technological functions in food and can be used as fillers, thickeners, and structure stabilizers. In particular, pectins have the ability to bind metals and create stable networks through bonds between Ca^{2+} ions and free carboxyl groups. Fiber from fruit is also used as a functional additive in ice cream, snacks, bakery products, and meat products [6]. Dried and powdered pomace is often used in the food industry, such as the confectionery and bakery industries [7]. According to

Tarko et al. [8], pomace, thanks to its use in ethanol production, is an important source of biofuel, and, in the case of apple pomace, it can be a breeding ground for bacteria producing lactic acid.

One of the by-products of the fruit industry in Poland is rose fruits pomace, which is obtained in the juice production process [9]. *Rosa rugosa* is a representative of the *Rosaceae* family with the most appreciated nutritional value among the representatives of this family found and cultivated in Poland. *Rosa rugosa* can occur in a variety of areas because it is resistant to unfavorable environmental conditions, such as poor soil or water restrictions. In Poland, it occurs both in the natural environment and in cultivation and breeding. The commercial planting of wild rose in Poland consists of 1200 ha of *Rosa rugosa*, with a yield of approximately 3000 tons per year [9]. This plant is widely used in various industries. Due to its inedible interior, the fruit cannot be eaten fresh. The rose is used for medicinal purposes and in food processing to produce juices, purees, jams, preserves, confectionery fillings, nectars, tinctures, wines, fruit, and herbal teas [10–16]. It has been proven that rose fruits extracts not only contain high levels of bioactive substances such as vitamin C or polyphenols but also have antibacterial, antiviral, antihypertensive, anti-inflammatory, and antidiabetic properties [9,17–19]. Preparations of this type may inhibit the growth of bacteria and fungi considered pathogenic and contaminating food. As reported in the literature, rose and rose pomace extracts limit the development of microorganisms such as *Escherichia coli*, *Staphylococcus aureus*, *Staphylococcus epidermidis*, *Klebsiella pneumoniae*, *Enterococcus faecalis*, *Bacillus subtilis*, *Bacillus cereus*, and *Candida albicans* [9,17,20–22]. Marmol et al. [10] showed that *Rosa rugosa* inhibits the growth of pathogenic bacteria without changing the growth of lactic acid bacteria. Kamijo [23] showed that phenolic compounds from the tannin group of *Rose rugosa* seem to be a promising prebiotic because they selectively inhibit the growth of pathogenic bacteria. Thanks to the scientific evidence proven and presented so far, we can confidently classify such plant extracts as natural food preservatives [18].

One of the simplest methods of obtaining bioactive ingredients is the extraction of plant material with a solvent. Post-production waste from plant materials from various industries is also perfect for this purpose. Most often, pomace is used, which may also contain large amounts of valuable bioactive ingredients. The extraction process involves the separation of a specific ingredient from the mixture using a solvent by diffusion. The effectiveness of this process depends primarily on the type of solvent but also on the temperature, extraction time, solid-to-solvent ratio, number of extractions, and partial size of the sample material [24,25]. The most important criterion for effective extraction is the appropriate adjustment of the solvent. The most commonly used solvents for extracting active substances from plant material are water, ethanol, methanol, acetonitrile, acetone, hexane, and chloroform. The appropriate selection of the extractant should be based on knowledge of the chemical nature of the extracted substance and the solvent. The temperature used is also a very important element in the extraction process because increased temperature affects the loss of labile compounds. Polar solvents are most often used to extract polyphenolic compounds and vitamin C from plant material [26–28]. The recovery of phenolic compounds during extraction is closely related to their chemical structure. An example of this is gallic acid, which is the most soluble in water because its molecule has four hydroxyl groups and one carboxyl group. The study by Arize et al. [29] showed that water, due to its higher polarity coefficient, washes out more polyphenols from the solid than ethanol. According to Pompeu [30], increasing the share of ethanol in the solvent mixture results in better extraction due to the reduction of the dielectric constant of the solution, which is directly related to the reduction of the energy needed to separate the solvent molecules. Based on research, it is known that chlorogenic acid and flavonoids are better extracted with ethanol [31,32]. In comparison, aqueous acetone is best for extracting higher molecular weight flavanols, and methanol is best for extracting lower molecular weight polyphenols [33]. Ethanol has less toxicity compared to acetone, methanol, and other organic solvents [34]. The use of extraction solvents in industry requires consideration of, among others, factors such as solvent residue in the product, its

removal, and environmental pollution [35]. The most recommended solvent for extraction is water, due to features such as non-toxicity, ecological nature, low cost of use, high extraction potential, and no restrictions on human consumption [33]. According to the European Pharmacopoeia, only class 3 solvents such as ethanol and acetone can be used in pharmaceutical products because they are less toxic and pose less risk to human health [36]. In contrast, solvents such as acetonitrile, chloroform, hexane, and methanol must be limited in pharmaceutical products due to their inherent toxicity. The concentration limits of these products for acetonitrile, chloroform, hexane, and methanol are 400, 60, 290, and 3000 ppm, respectively [36]. To the best of our knowledge, there is no study investigating the effect of different solvents on the recovery of phenolic compounds and L-ascorbic acid from rose fruits pomace waste.

The aim of this study was to investigate the effects of temperature, time extraction, and different solvents, including water, methanol, ethanol, and acetone, and the combination of these organic solvents with water in a ratio of 50:50 (v/v) on the recovery of total phenolic compounds (measured by Folin–Ciocalteu) and L-ascorbic acid (measured by the HPLC method) of rose fruits (*Rosa rugosa*) pomace.

2. Materials and Methods

2.1. Chemicals and Reagents

Anhydrous sodium carbonate and Folin–Ciocalteu reagent were purchased from Chempur (Piekary Śląskie, Poland). Gallic acid anhydrous (GAE), L-ascorbic acid, oxalic acid, m-phosphoric acid, DL-dithiothreitol (DTT), ethanol, acetone, and methanol were purchased from Sigma-Aldrich (Poznań, Poland). All reagents were of analytical grade.

2.2. Plant Material

Fresh fruits (including seeds) of *Rosa rugosa* were harvested at a plantation of the company "Polska Róża" located in Kotlina Kłodzka in September 2020 in Poland. The cultivation of *Rosa rugosa* was carried out in accordance with the cultivation recommendations for this species, and no events occurred that could have affected this cultivation.

2.3. Preparation of Rose Fruits Juice on a Laboratory Scale

Fruits were homogenized by Thermomix® (Vorwerk & Co. KG, Wuppertal, Germany). The pulp was subsequently macerated for 24 h with 0.2 g/kg macerating enzyme pectinase (Rohapect 10 L, AB Enzymes GmbH, Darmstadt, Germany) at room temperature. After this time, the juice was pressed on an automatic press (Bucher Unipektin, Niederweningen, Switzerland).

2.4. Freeze-Drying of Rose Fruits Pomace

Directly after juice pressing, a portion of the pomace was frozen and freeze-dried (Christ, Alpha 1-2 LD Plus, Osterode am Harz, Germany). Lyophilization lasted 45 h at a temperature of −56 °C and a pressure of 1.03 mbar. The obtained dried material was crushed in a Retsch GM 200 homogenizer. The obtained powder was transferred to vacuum packaging bags, closed tightly, and stored in a shaded place.

2.5. Preparation of Extracts from Rose Fruits Pomace Using Classic Extraction by Shaking with a Solvent

A three-way experiment with a completely randomized design was set up. Solvent combinations were created in a cross-arrangement. The following solvents were used for extraction: water, ethanol, methanol, acetone, and methanol: water (50:50, v/v), ethanol: water (50:50, v/v), and acetone: water (50:50, v/v). Seven types of extracts were prepared by shaking with different solvents. The extraction process was performed with a solvent-to-sample ratio of (100:1 v/w). The solvent-to-sample ratio was selected based on the literature data [26,27]. Approximately 0.5 g of freeze-dried pomace was weighed exactly into Falcon tubes with a screw cap, and 50 mL of an appropriate solvent was added. A single extraction

was carried out on an LLG-uni THERMIX 2 pro thermal shaker at a speed of 500 rpm at three different temperatures of 25, 45, and 65 °C and in four different time ranges: 15, 30, 45, and 60 min. Reference samples were prepared in the same manner as tested samples but were not shaken and heated, and their extraction time was 60 min. After extraction, the individual extracts were centrifuged at 5000 rpm for 5 min, filtered through a 0.45 µm PTFE filter, and then the resulting clear supernatants were evaporated with nitrogen using a nitrogen gas generator (Peak Scientific Instruments Ltd., Scotland, UK). After evaporation, the extracts were dissolved in 50 mL of water, transferred to plastic vials, and stored at −20 °C until analyzed.

2.6. Determination of Total Phenolic Content

The total phenolic content (TPC) of the extract solution was determined by the Folin–Ciocalteu reagent's method using a UV1650PC spectrophotometer (Shimadzu, Kyoto, Japan) [37]. Total phenolic content was expressed as mg gallic acid equivalents (GAE) per g of freeze-dried pomace. The equation obtained from the calibration curve of gallic acid in the range of 5–50 mg/100 mL was y = 0.0372x + 0.0826 (r = 0.9965). The test was performed in triplicate.

2.7. Determinations of L-Ascorbic Acid (AA) Content

L-ascorbic acid (AA) (as the sum of AA and L-dehydroascorbic acid (DHAA) after its reduction to AA) was determined using high-pressure liquid chromatography coupled with a UV-VIS detector (Prominence HPLC system, Shimadzu, Kyoto, Japan) according to the method previously stated in the literature with our own modification [11,38]. One mL of extract was transferred to a volumetric flask and diluted to 10 mL with 2% of oxalic acid. Then, the obtained solution was diluted in a 1:1 ratio with 1% DTT (DL-dithiothreitol). The solution was left in a dark place for 1 h to reduce DHAA (L-dehydroascorbic acid) to AA (L-ascorbic acid). This reaction solution was filtered through a 0.45 µm PTFE syringe filter into a chromatographic vial. The sample volume of 20 µL from each chromatographic vial was injected into the HPLC system. The separation was carried out using an Onyx Monolithic C18, 100 × 4.6 mm column (Phenomenex, Torrance, CA, USA), at 25 °C. 0.1% aqueous solution of m-phosphoric acid was used as the eluent at a flow rate of 1.0 mL/min. The analysis was performed in an isocratic system. L-ascorbic acid was determined at 254 nm.

Blank samples were prepared in the same way as the analytes. All results were expressed in mg/g of freeze-dried pomace. The test was performed in triplicate.

The reliability of the HPLC-method was validated for linearity, sensitivity (limit of detection, LOD, and limit of quantification, LOQ), precision, and recovery. Precision was determined by six measurements of an AA standard. The LOD and LOQ were defined as 3 times and 10 times the signal-noise ratio, respectively. Recovery was determined in seven representative extracts (water, methanol, ethanol, acetone, 50% methanol, 50% ethanol, and 50% acetone) from rose fruits pomace. Two levels of AA were added to the tested rose fruits pomace (5 and 20 mg/g), and the recovery level was determined twice for each prepared extract.

2.8. Statistical Analysis

Statistica 13.3 (TIBCO Software Inc., Carlsbad, CA, USA) was used to perform the statistical analysis. The obtained experimental results were subjected to calculations of mean values, standard deviations, and multivariate ANOVA. The significance of statistical differences was verified using the Tukey test with a significance level of $p \leq 0.05$. We also used principal components analysis (PCA) as a multi-trait method describing the relationship among polyphenols and L-ascorbic acid content at study temperatures across a combination of time and solvents.

3. Results and Discussion

3.1. Impact of Process Variables on the Extraction of Total Phenolic Compounds (TPC) from Rose Fruits (Rosa rugosa) Pomace

Results of studies assessing the influence of various variables of the extraction process, i.e., concentration of solvents (water, ethanol, methanol, acetone, and their water mixtures), temperatures (25 °C, 45 °C, and 65 °C), and extraction time (15, 30, 45, and 60 min), on the extraction efficiency of total phenolic compounds (TPC) of rose fruits pomace are presented in Tables 1 and 2. Analysis of experimental data revealed that all three process variables—solvent; time; and temperature—significantly influenced the extraction of TPC ($p < 0.05$). Table 3 presents the results of the multivariate ANOVA analysis for the considered effects on the content of TPC. We observe a significant impact of all main effects on TPC; all interactions between the factors are significant.

Table 1. Content of total phenolic compounds (TPC) in freeze-dried rose fruits pomace after extraction with various solvents (water, methanol, and ethanol) at 25 °C, 45 °C, and 65 °C for 60 min.

Temp (°C)	Time (min)	Solvents			
		Water	Methanol	Ethanol	Acetone
		TPC (mg GAE/g of Freeze-Dried Rose Fruits Pomace)			
25 °C	15	9.85 ± 0.40 ^{Aa}	16.83 ± 0.18 ^{Bb}	26.98 ± 0.39 ^{Bc}	12.93 ± 0.22 ^{BCb}
25 °C	30	17.08 ± 0.62 ^{Cb}	17.13 ± 0.13 ^{Bb}	30.40 ± 0.41 ^{Bc}	12.70 ± 0.51 ^{BCa}
25 °C	45	11.70 ± 0.72 ^{Ba}	15.35 ± 0.62 ^{Bb}	28.39 ± 0.24 ^{Bc}	12.40 ± 0.33 ^{Ba}
25 °C	60	12.77 ± 0.69 ^{Ba}	17.47 ± 0.75 ^{Bb}	30.78 ± 0.27 ^{Bc}	11.82 ± 0.29 ^{Ba}
45 °C	15	11.95 ± 0.71 ^{Ba}	15.94 ± 0.72 ^{Bb}	31.10 ± 1.12 ^{BCc}	9.88 ± 0.52 ^{Aa}
45 °C	30	14.63 ± 0.08 ^{Bb}	17.92 ± 0.66 ^{Bc}	34.18 ± 0.35 ^{Cd}	10.58 ± 0.42 ^{Ba}
45 °C	45	13.62 ± 0.13 ^{Ca}	15.78 ± 0.58 ^{Bb}	33.49 ± 0.24 ^{Cc}	15.51 ± 0.41 ^{Cb}
45 °C	60	15.75 ± 0.15 ^{Ca}	18.17 ± 0.99 ^{BCa}	35.56 ± 0.48 ^{Db}	15.33 ± 0.30 ^{Ca}
65 °C	15	21.36 ± 0.26 ^{Db}	17.59 ± 0.61 ^{Ba}	36.27 ± 0.26 ^{Dc}	14.75 ± 0.69 ^{Ca}
65 °C	30	20.84 ± 0.26 ^{Db}	17.17 ± 0.59 ^{Ba}	33.55 ± 0.51 ^{Cc}	15.42 ± 0.31 ^{Ca}
65 °C	45	25.94 ± 0.25 ^{Dc}	19.97 ± 0.79 ^{Cb}	33.80 ± 0.37 ^{Cd}	14.86 ± 0.72 ^{Ca}
65 °C	60	23.26 ± 0.55 ^{Db}	18.90 ± 0.47 ^{Ca}	33.09 ± 0.46 ^{Cc}	20.36 ± 0.48 ^{Da}
Reference sample		9.34 ± 0.66 ^{Aa}	7.94 ± 0.19 ^{Aa}	12.09 ± 0.26 ^{Ab}	8.16 ± 0.61 ^{Aa}

Data are expressed as the average value over three replications ± standard deviation. ANOVA to compare data; different lowercase letters in a row indicate a significant difference between solvents at one specific temperature and time, and different capital letters in a column indicate a significant difference between one solvent in a combination of time and temperature (Tukey's test, $p < 0.05$). Abbreviations: TPC, total phenolic compounds; GAE, gallic acid equivalents.

The solvents used for TPC extraction had a significant impact on their recovery. Combined organic solvents with water were characterized by higher TPC recovery compared to absolute solvents, with the exception of ethanol. The TPC values of the extracts (25 °C, after 60 min) range on average from 12.46 mg GAE/g of freeze-dried rose fruits pomace for acetone extract (Table 1) to 35.36 mg GAE/g of freeze-dried rose fruits pomace for 50% acetone (Table 2). The TPC values of the extracts (45 °C, after 60 min) range on average from 12.83 mg GAE/g of freeze-dried rose fruits pomace for acetone extract (Table 1) to 34.10 mg GAE/g of freeze-dried rose fruits pomace for 50% acetone (Table 2). The TPC values of the extracts (65 °C, after 60 min) range on average from 16.34 mg GAE/g of freeze-dried rose fruits pomace for acetone extract (Table 1) to 37.28 mg GAE/g of freeze-dried rose fruits pomace for 50% acetone (Table 2). The decrease in TPC of the extracts (at 25 °C and 45 °C after 60 min) was as follows: 50% acetone > ethanol > 50% methanol > 50% ethanol > methanol > water > acetone. The decrease in TPC of the extracts (65 °C after 60 min) was as follows: 50% acetone > ethanol > 50% methanol > 50% ethanol > water > methanol > acetone. For example, at 25 °C, among the aqueous solvents, 50% acetone had the highest recovery of TPC in freeze-dried rose fruits pomace. 50% acetone was closely followed by ethanol and 50% methanol, with about 82 and 79% of TPC being recovered in comparison with 50% acetone.

Table 2. Content of total phenolic compounds (TPC) in freeze-dried rose fruits pomace after extraction with various solvents (50% methanol, 50% ethanol, and 50% acetone) at 25 °C, 45 °C, and 65 °C for 60 min.

Temp (°C)	Time (min)	Solvents		
		50% Methanol	50% Ethanol	50% Acetone
		TPC (mg GAE/g of Freeze-Dried Rose Fruits Pomace)		
25 °C	15	27.81± 0.34 ^{Ba}	27.26 ± 0.25 ^{Ca}	31.63 ± 0.66 ^{Bb}
25 °C	30	28.90 ± 0.39 ^{Bb}	21.55 ± 0.44 ^{Ba}	36.79 ± 0.60 ^{Cc}
25 °C	45	27.40 ± 0.26 ^{Ba}	22.38 ± 0.24 ^{Ba}	36.21 ± 0.31 ^{Cb}
25 °C	60	27.99 ± 0.17 ^{Ba}	24.56 ± 0.35 ^{Ba}	36.83 ± 0.13 ^{Cb}
45 °C	15	30.70 ± 0.23 ^{Cb}	26.11 ± 0.20 ^{Ba}	31.80 ± 0.58 ^{Bb}
45 °C	30	32.97 ± 0.25 ^{Cb}	26.51 ± 0.42 ^{Ba}	37.35 ± 0.45 ^{CDc}
45 °C	45	31.84 ± 0.15 ^{Cb}	26.40 ± 0.43 ^{Ba}	32.93 ± 0.31 ^{Bb}
45 °C	60	33.89 ± 0.51 ^{Cb}	28.93 ± 0.43 ^{BCa}	34.33 ± 0.93 ^{Bb}
65 °C	15	36.92 ± 0.52 ^{Cb}	29.57 ± 0.19 ^{Ca}	36.05 ± 0.57 ^{Cb}
65 °C	30	29.28 ± 0.34 ^{BCa}	30.92 ± 0.41 ^{Ca}	34.33 ± 0.69 ^{Bb}
65 °C	45	32.02 ± 0.52 ^{Ca}	30.66 ± 0.24 ^{Ca}	39.60 ± 0.35 ^{Db}
65 °C	60	34.65 ± 0.34 ^{Ca}	32.90 ± 0.12 ^{Ca}	39.12 ± 0.62 ^{Da}
Reference sample		7.61 ± 0.16 ^{Aa}	10.25 ± 0.22 ^{Ab}	17.82 ± 0.25 ^{Ac}

Data are expressed as the average value over three replications ± standard deviation. ANOVA to compare data; different lowercase letters in a row indicate a significant difference between solvents at one specific temperature and time, and different capital letters in a column indicate a significant difference between one solvent in a combination of time and temperature (Tukey's test, $p < 0.05$). Abbreviations: TPC, total phenolic compounds; GAE, gallic acid equivalents.

Table 3. Multivariate Anova analysis of variance of TPC content on the type of solvent, temperature, and time in freeze-dried rose fruits pomace.

Source	Degrees of Freedom	Sum of Square	Mean Square	F-Value	*p*-Value
Main effects					
temperature	1	554	554.1	243.024	<0.0001
time	4	7082	1770.5	776.535	<0.0001
solvent	6	10,178	1696.3	744.005	<0.0001
temperature:time	4	200	50.1	21.972	<0.0001
temperature:solvent	6	163	27.2	11.915	<0.0001
time:solvent	24	1477	61.5	26.993	<0.0001
temperature:time:solvet	24	209	8.7	3.817	<0.0001
Residuals	140	4648	33.2		

In the tests conducted, the lowest TPC recovery was achieved when using single solvents—acetone and water. By using organic solvents in combination with water, TPC recovery was significantly improved. For example, at temperatures of 25 °C, 45 °C, and 65 °C, fifty percent aqueous methanol provided the highest recovery, about 150% higher than that of methanol. A rapid increase in polyphenol content was observed in the first 15 min of extraction for each temperature and solvent used. After 30 min of heating, relative stabilization occurred. This proves the most effective extraction in the initial stage of heating and stabilization over time, most likely resulting from the complete dissolution of the extractant and the extraction of phenolic compounds. Dent et al. [39] showed that the extraction time after reaching a relatively constant polyphenol value does not significantly affect the results. Moreover, an increase in the applied temperature (25–45–65 °C) resulted in an increase in TPC concentration for identical solvent concentrations and time conditions.

The use of a higher temperature resulted in an increase in the solubility of the solute and the diffusion coefficient, which resulted in the highest TPC concentration being determined at the highest temperature tested [40]. However, according to the recommendations of other authors, the temperature range was kept low (maximum 65 °C) to avoid possible

degradation of phenolic compounds and denaturation of membranes [41,42]. Finally, the highest values of TPC were reached at the conditions of 50% acetone and 65 °C after 45 or 60 min of extraction.

The obtained polyphenol contents in the tested extracts are reflected in the studies of other authors. The obtained results are consistent with Nayak et al. [43], who found that 51% acetone has the highest recovery efficiency of phenolic compounds (12.20 mg GAE/g DM) from orange peel. The obtained results can also be compared with the research conducted by Sielicka and Pawlak [44], in which one of the most effective solvents used in extraction turned out to be mixtures of water and acetone (84.9 mg GAE/g DM) and water and methanol (85.4 mg GAE/g DM). Um et al. [45] showed that the optimal condition for the ultrasound-assisted extraction (UAE) of TPC from rose fruits (*Rosa rugosa*) was 50% ethanol for 30 min. at 50 °C. The content of TPC in the extract (at 50 °C after 30 min) was 95.69 mg TPC/g dry weight. Klewicka et al. [9] reported that four extracts from the pseudofruit pomace of *Rosa rugosa* Thunb. differed in their total content of polyphenols. The content of polyphenols in 60% acetone extracts was higher than in 60% ethanol extracts and amounted to 14.9 g/100 g DM for water-acetone extract crude, 33.2 g/100 g DM for water-acetone extract purified, 8.8 g/100 g DM for water-ethanol extract crude, and 29.2 g/100 g DM for water-acetone extract purified. Dróżdż et al. [46] determined TPC in 80% metanol extract as 29.20 mg GAE/g of dried rosehip pomace. Similarly, according to Ilbay et al. [47], the best TPC (59.69 mg GAE/g DM) in three different brands of rose hip tea (*Rosa canina* L.) was obtained using a 50% MeOH mixture. However, according to Papoutsis et al. [48], the most effective solvent for recovering TPC from lemon pomace was absolute methanol and 50% acetone. Methanol was also the most effective solvent for the extraction of hesperidin, which is a flavonoid compound (flavanone) [49]. According to the report by Lou et al. [50], the most effective solvent for the extraction of TPC from calamondin (*Citrus mitis* Blanco) was hot water compared to absolute methanol, ethanol, or their combination with water. Singh et al. [51] found that ethanol had the highest efficiency of TPC extraction from pomegranate. Research conducted by Cendrowski et al. [52] on rose petals (*Rosa rugosa*) showed that one of the best solvents for the extraction of polyphenolic compounds is a 40% ethanol solution. Olecha et al. [15] reported that the total phenolic content in *Rosa rugosa* samples ranged from 0.14 to 13.9 mg GAE/mL of rose extract (infusion or tincture). Infusions were obtained by pouring 2 g of plant material with 100 mL of boiling water, and tinctures were obtained by extracting 10 g of powdered plant material with 50 g of 70° ethanol. Researchers determined the highest phenol content in tinctures of *Rosa rugosa* flowers, leaves, and roots (12.75–13.9 mg/mL) and the lowest in the infusion of *Rosa rugosa* nuts (0.14 mg/mL). The total phenolic content in *Rosa rugosa* pseudofruit tinctures was 11.88 mg GAE/mL. Similar results were obtained by other researchers, who showed that the content of polyphenols in methanolic rose (*Rosa nutkana* and *Rosa woodsii*) extracts depends on the part of the plant used [21]. The concentration of polyphenols in methanolic extracts from rose fruits, *Rosa nutkana*, and *Rosa woodsii*, was higher than in nut extracts.

The literature results are similar to our research, which showed that extraction solvents significantly affect the recovery efficiency of TPC from plant materials. The data showed that the TPC recovery for individual solvents was different and resulted from different solvent polarities, different polyphenol structures, and the type of plant raw material. According to Marston and Hostettmann [53], for example, flavonoid glycosides and more polar aglycones can be extracted with alcohols or mixtures of alcohols and water, while low-polarity solvents such as acetone are suitable for the extraction of less polar flavonoids such as isoflavones, flavanones, and methylated flavones. The highest extraction efficiency obtained using 50% acetone and ethanol can be associated with the reduced activity of the polyphenol oxidase responsible for the oxidation of phenolic compounds [54].

3.2. Impact of Process Variables on the Extraction of L-Ascorbic Acid (AA) from Rose Fruits (Rosa rugosa) Pomace

Following the extraction procedure, the extract was separated in an Onyx Monolithic C18 column with a UV detector for the analysis of AA. Figure 1 shows an example chromatogram of the aqueous extract of rose fruits pomace.

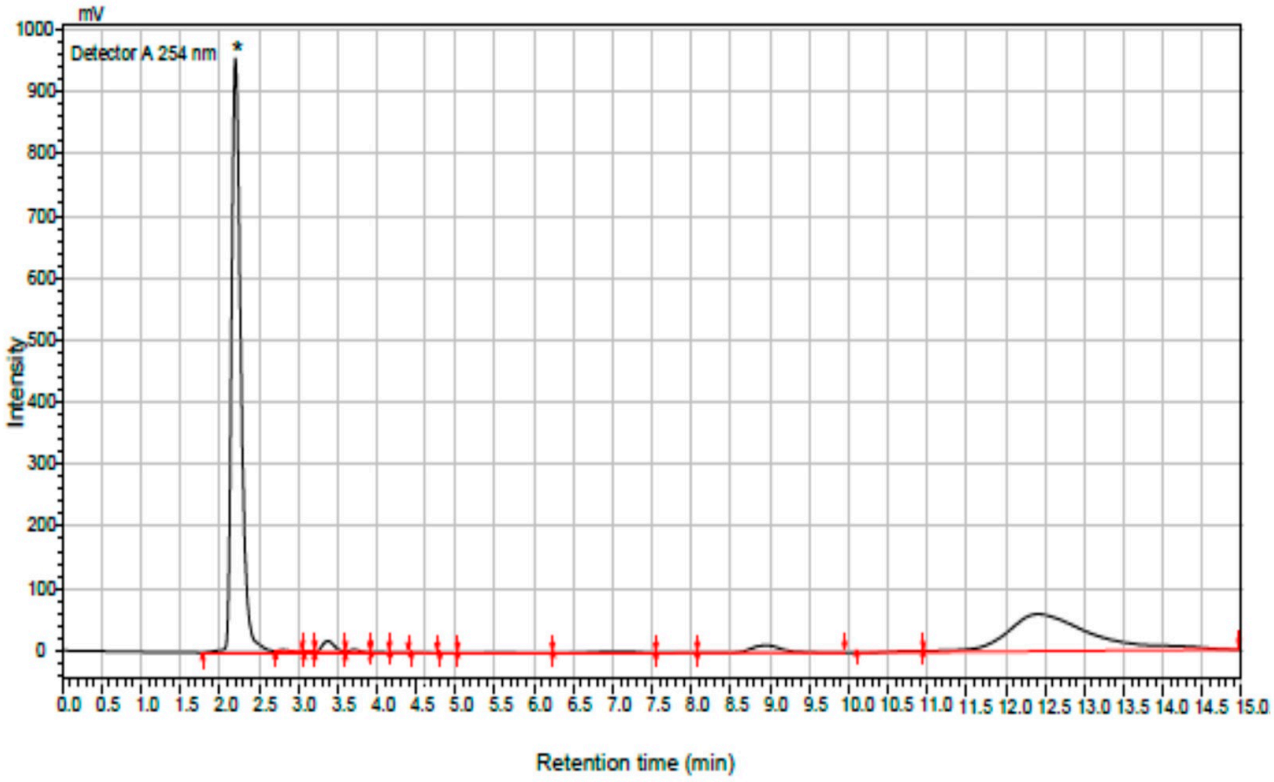

Figure 1. Example chromatogram of the aqueous extract of rose fruits pomace. Asterisks indicate the peak of ascorbic acid.

The quantification of AA was performed by external calibration. The equation obtained from the calibration curve of AA in the range of 0.5–40 mg/100 mL was y = 443,770x − 87,769 (r = 0.9999). LOD and LOQ were 0.14 mg/100 mL and 0.45 mg/100 mL, respectively. Recovery was tested at two fortification levels. The mean recovery percentages were in the range of 94.82%, 92.98%, 93.45%, 91.54%, 93.23%, 92.23%, and 91.77% for water, methanol, ethanol, acetone, 50% methanol, 50% ethanol, and 50% acetone extract from rose fruits pomace, respectively. The precision from six consecutive injections was satisfactory, with an RSD of 1.8%. All recovery and precision values were in the acceptable range. The validation of our HPLC method for the determination of L-ascorbic acid in various extracts prepared from rose fruits pomace is consistent with the validation of the HPLC method for the determination of vitamin C in fruits reported by Odriozola-Serrano et al. [38].

The type of solvent (water, ethanol, methanol, acetone, and their water mixtures) used, different temperatures (25 °C, 45 °C, and 65 °C), and extraction times (15, 30, 45, and 60 min) had a significant impact on the extraction efficiency of AA from rose frutis pomace (Tables 4 and 5). In the case of AA, temperature and solvent turned out to be insignificant among the interactive interactions (Table 6).

Table 4. Content of L-ascorbic acid (AA) in freeze-dried rose fruits pomace after extraction with various solvents (water, methanol, and ethanol) at 25 °C, 45 °C, and 65 °C for 60 min.

Temp (°C)	Time (min)	Solvents			
		Water	Methanol	Ethanol	Acetone
		AA (mg/g Rose Fruit Pomace)			
25 °C	15	31.76 ± 1.89 ^{Bd}	26.43 ±1.02 ^{Ac}	10.67 ± 0.18 ^{Bb}	3.01 ± 0.11 ^{ABa}
25 °C	30	33.75 ± 0.64 ^{Bc}	28.33 ± 0.38 ^{ABc}	11.39 ± 0.75 ^{Ab}	3.32 ± 0.19 ^{Ba}
25 °C	45	35.54 ± 0.12 ^{BCd}	30.54 ± 1.15 ^{Bc}	12.26 ± 0.24 ^{Bb}	3.55 ± 0.21 ^{Ba}
25 °C	60	36.65 ± 0.85 ^{Cd}	31.65 ± 0.68 ^{Bc}	13.08 ± 0.11 ^{Bb}	4.00 ± 0.05 ^{Ca}
45 °C	15	37.80 ± 0.33 ^{Cd}	32.50 ± 1.09 ^{Bc}	13.65 ± 0.41 ^{BCb}	3.96 ± 0.38 ^{Ca}
45 °C	30	38.90 ± 0.09 ^{Cd}	33.76 ± 0.82 ^{Bc}	13.88 ± 0.01 ^{Cb}	4.21 ± 0.17 ^{Ca}
45 °C	45	33.50 ± 0.35 ^{Bd}	28.44 ± 0.78 ^{ABc}	11.44 ± 1.05 ^{Ab}	3.34 ± 0.02 ^{Ba}
45 °C	60	30.54 ± 0.70 ^{Bd}	26.08 ± 0.36 ^{Ac}	10.56 ± 0.07 ^{Ab}	3.06 ± 0.00 ^{Ba}
65 °C	15	38.90 ± 0.27 ^{Cd}	33.32 ± 0.00 ^{Bc}	13.92 ± 0.91 ^{Cb}	4.04 ± 0.33 ^{Ca}
65 °C	30	33.21 ± 1.10 ^{Bc}	33.21 ± 0.19 ^{Bc}	14.05 ± 0.10 ^{Cb}	3.89 ± 0.08 ^{Ca}
65 °C	45	28.21 ± 0.40 ^{Ad}	24.26 ± 0.34 ^{Ac}	9.68 ± 0.75 ^{Ab}	2.80 ± 0.00 ^{Aa}
65 °C	60	25.01 ± 0.00 ^{Ad}	21.65 ± 0.66 ^{Ac}	8.41 ± 0.88 ^{Ab}	2.45 ± 0.07 ^{Aa}
Reference sample		24.83 ± 1.40 ^{Ad}	20.89 ± 0.97 ^{Ac}	8.39 ± 0.73 ^{Ab}	2.43 ± 0.04 ^{Aa}

Data are expressed as the average value over three replications ± standard deviation. ANOVA to compare data; different lowercase letters in a row indicate a significant difference between solvents at one specific temperature and time, and different capital letters in a column indicate a significant difference between one solvent in a combination of time and temperature (Tukey's test, $p < 0.05$). Abbreviations: AA, L-ascorbic acid.

Table 5. Content of L-ascorbic acid (AA) in freeze-dried rose fruits pomace after extraction with various solvents (50% methanol, 50% ethanol, and 50% acetone) at 25 °C, 45 °C, and 65 °C for 60 min.

Temp (°C)	Time (min)	Solvents		
		50% Methanol	50% Ethanol	50% Acetone
		AA (mg/g Rose Fruit Pomace)		
25 °C	15	18.53± 0.66 ^{Ba}	21.43 ± 1.01 ^{Bb}	25.19 ± 0.85 ^{Ac}
25 °C	30	19.83 ± 0.36 ^{Ba}	23.00 ± 0.90 ^{Bb}	26.87 ± 0.03 ^{Bc}
25 °C	45	21.45 ± 0.57 ^{Ca}	24.45 ± 1.14 ^{Bb}	28.17 ± 0.82 ^{Cc}
25 °C	60	23.78 ± 0.43 ^{Ca}	26.06 ± 0.25 ^{BCb}	28.97 ± 0.74 ^{Cc}
45 °C	15	22.75 ± 0.17 ^{Ca}	27.43 ± 0.05 ^{Cb}	29.88 ± 0.44 ^{Cc}
45 °C	30	23.61 ± 0.37 ^{Ca}	27.85 ± 0.51 ^{Cb}	30.76 ± 0.61 ^{Cc}
45 °C	45	19.98 ± 0.56 ^{BCa}	23.43 ± 0.87 ^{Bb}	26.54 ± 0.12 ^{Bc}
45 °C	60	18.23 ± 0.71 ^{Bb}	12.82 ± 1.30 ^{Aa}	24.66 ± 0.87 ^{Bc}
65 °C	15	23.24 ± 0.20 ^{Ca}	27.81 ± 1.12 ^{Cb}	30.75 ± 0.40 ^{Cb}
65 °C	30	23.47 ± 0.25 ^{Ca}	28.25 ± 0.08 ^{Cb}	31.48 ± 0.08 ^{Cc}
65 °C	45	16.82 ± 0.85 ^{Ba}	17.81 ± 0.91 ^{Aa}	22.39 ± 0.45 ^{Bb}
65 °C	60	13.19 ± 0.09 ^{Aa}	14.82 ± 0.63 ^{Aa}	18.91 ± 0.12 ^{Ab}
Reference sample		14.66 ± 1.82 ^{Aa}	16.98 ± 0.05 ^{Ab}	19.63 ± 2.09 ^{Ac}

Data are expressed as the average value over three replications ± standard deviation. ANOVA to compare data; different lowercase letters in a row indicate a significant difference between solvents at one specific temperature and time, and different capital letters in a column indicate a significant difference between one solvent in a combination of time and temperature (Tukey's test, $p < 0.05$). Abbreviations: AA, L-ascorbic acid.

This extraction efficiency, depending on the solvent used, was influenced by the structure of vitamin C, which is a polar molecule, and its solubility increased with the polarity of the solvent [28]. In general, Vitamin C is a natural antioxidant that primarily exists in and is available in its reduced form as L-ascorbic acid and its oxidized form as L-dehydroascorbic acid [55].

Our research showed that the highest content of AA was in the water extract, followed by methanol, 50% acetone, 50% methanol, 50% ethanol, and acetone. These results are consistent with those reported by Shalmashi and Eliassi [28], who found that the solubility of

vitamin C decreases in the following order: water, methanol, ethanol, acetone, acetonitrile, and ethyl acetate.

Table 6. Multivariate Anova analysis of variance of L-ascorbic acid content on the type of solvent, temperatures, and time in freeze-dried rose fruits pomace.

Source	Degrees of Freedom	Sum of Square	Mean Square	F-Value	*p*-Value
Main effects					
temperature	1	41	40.6	17.008	<0.0001
time	4	1688	421.9	176.634	<0.0001
solvent	6	17,693	2948.8	1234.435	<0.0001
temperature:time	4	872	218.1	91.306	<0.0001
temperature:solvent	6	18	3	1.268	0.276
time:solvent	24	372	15.5	6.489	<0.0001
temperature:time:solvet	24	165	6.9	2.876	<0.0001
Residuals	140	334	2.4		

The content of AA in the extracts (25 °C, after 60 min) ranges from 3.47 mg AA/g of freeze-dried rose fruits pomace for acetone extract to 34.42 mg AA/g of freeze-dried rose fruits pomace for water (Table 4). The content of AA in the extracts (45 °C, after 60 min) ranges from 3.64 mg AA/g of freeze-dried rose fruits pomace for acetone extract to 35.18 mg AA/g of freeze-dried rose fruits pomace for water (Table 4). The content of AA in the extracts (65 °C after 60 min) ranges from 3.29 mg AA/g of freeze-dried rose fruit pomace for acetone extract to 31.33 mg AA/g of freeze-dried rose fruit pomace for water (Table 4). The use of 50% acetone or 50% ethanol (Table 5) as a solvent showed higher recoveries of L-ascorbic acid in rose fruits pomace compared to acetone and ethanol (Table 4). These recoveries were higher on average for the three temperatures (25 °C, 45 °C, and 65 °C) by approximately 87% and 48% for 50% acetone and 50% ethanol, respectively. The higher extraction efficiencies obtained during extraction using 50% acetone and 50% ethanol can be attributed to the presence of water, which can increase the polarity of the solvents.

These results can be compared with the research conducted by Milal et al. [56], who reported an average vitamin C content of 3500 mg/100 g dry weight (DW). Um et al. [45] showed that the optimal condition for the ultrasound-assisted extraction (UAE) of AA from rose fruits (*Rosa rugosa*) was 50% ethanol for 30 min. at 30 °C. The content of AA in the extract (30 °C, after 30 min) was 6.38 mg AA/g dry weight (DW). Differences in the content of L-ascorbic acid depend on many factors, such as species, degree of maturity, and type of climate. Adamczak et al. [57] conducted research comparing different species of roses in terms of the content of L-ascorbic acid. The results ranged from 510 to 2250 mg/100 g dry weight (DW). *Rosa villosa* had the highest content, while *Rosa canina* L. had the lowest content. Other researchers reported significant differences in the vitamin C content of *Rosa corymbifera* (760.52 mg/100 g), *Rosa rugosa* Thunb. (577.13 mg/100 g), *Rosa alba* L. (733.86 mg/100 g), and *Rosa canina* L. (628.11 mg/100 g) cultivated in the same growing conditions [58].

Paunovic et al. [59] showed significant differences in the content of vitamin C in fresh (429.55 mg/100 g dry matter) and dried (187.67 mg/100 g dry matter) rose hips (*Rosa canina*). The vitamin C content in dried rose hips was significantly reduced by 56.3% compared to the initial value in the fresh sample. As the extraction temperature was lowered (25 °C), the concentration of L-ascorbic acid remained at a similar level in the tested pomace over time. At higher temperatures (45 °C and 65 °C), a higher content of L-ascorbic acid was demonstrated, but also faster degradation after 30 min of extraction. The decrease in vitamin C content after exceeding a certain temperature may be due to the fact that it is a thermolabile vitamin. Testing the vitamin C content in pomegranate juice at different temperatures showed a constant concentration of vitamin C at 90 °C for a period of 15 to 75 min [60]. Therefore, it is safe to assume that an increase in temperature will further increase the extraction efficiency but may degrade the antioxidant more quickly. Higher

temperatures may promote the solubility of extracts in the solvent and initiate a sudden increase in the diffusion rate, followed by faster mass transfer.

Figure 2 shows the results of Prinicpal Component Analysis (PCA). Our PCA analysis data shows a positive correlation both between the L-ascorbic acid content and the tested extraction temperature, as well as between the polyphenol content and the tested extraction temperature. We observed a very strong correlation between the extraction temperatures used and the content of L-ascorbic acid as well as polyphenols. In general, a higher content of L-ascorbic acid and polyphenols was observed for extraction with 50% acetone, while low contents were observed for extraction with acetone alone at all times.

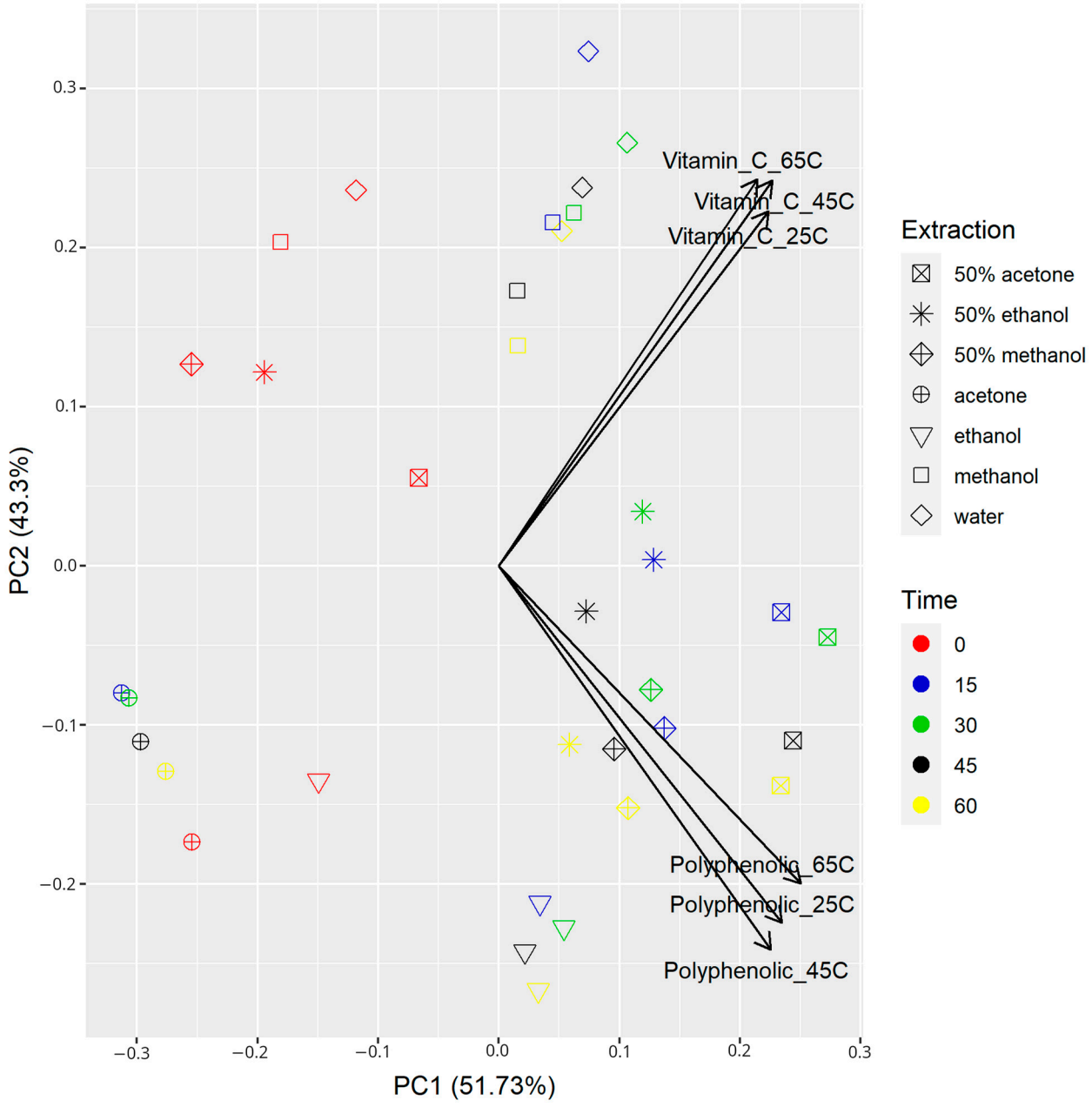

Figure 2. The biplot of principial componets analysis for extraction methods across time points.

4. Conclusions

One of the by-products of the fruit industry in Poland is rose fruits pomace, which is obtained in the juice production process. On the basis of this study, it was shown that rose fruits (*Rosa rugosa*) pomace, rich in polyphenols and ascorbic acid, can be an excellent addition to other products produced in the food industry. Analysis of experimental data

revealed that all three process variables—solvent; time; and temperature—significantly influenced the extraction of total phenolic compounds and L-ascorbic acid. A rapid increase in polyphenol content was observed in the first 15 min of extraction for each temperature used. After 30 min of heating, relative stabilization occurred. The use of a higher temperature resulted in an increase in the solubility of the solute and the diffusion coefficient, which resulted in the highest TPC concentration being determined at the highest temperature tested. Finally, the highest values of TPC were reached at the conditions of 50% acetone and 65 °C after 45 or 60 min of extraction. Higher temperatures (45 °C and 65 °C) showed a higher content of L-ascorbic acid but also faster degradation of this acid after 30 min of extraction. Water turned out to be the best solvent for the extraction of L-ascorbic acid from pomace (average 33.64 mg AA/g of freeze-dried pomace). This extraction efficiency, depending on the solvent used, was influenced by the structure of L-ascorbic acid, which is a polar molecule, and its solubility increased with the polarity of the solvent.

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