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Polyphenols from Sage Leaves (*Salvia officinalis* L.): Environmentally Friendly Extraction under High Hydrostatic Pressure and Application as a Corrosion Inhibitor for Tinplate

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Abstract: Due to the diversity of organic molecular structures present in sage extract, sage extract is a promising potential source of a cheap and effective biodegradable green corrosion inhibitor for tinplate in 3% NaCl solution, which was evaluated in this study. HHP proved to be a new and emerging technology for the useful extraction of polyphenols from sage as a functional ingredient from natural sources. Analysis of variance among all tested independent factors (ethanol concentration, HHP parameters and temperature) revealed significant differences ($p < 0.05$) in total polyphenol content as well as for rosmarinic acid as the major phenolic compound in sage extract, while extraction time had no effect ($p > 0.05$). The optimum HHP conditions (600 MPa, 30% ethanol, 60 °C and 5 min) gave a maximum extraction yield of total polyphenols of 3811.84 mg/100 g. Sage-leaf extracts were found to be a mixture of phenolic acids, namely rosmarinic and salvianolic acid K, epicatechin and luteolin-7-O-glucuronide glycoside. The corrosion results show that the sage extract at a concentration of 0.6 g/L in 3% NaCl is an effective corrosion inhibitor (93%), forming a passivation layer of sage extract consisting of organic compounds such as polyphenols on the surface of tinplate.

Keywords: high hydrostatic pressure extraction; corrosion inhibitor; sage; rosmarinic acid; polyphenols

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1. Introduction

Sage (*Salvia officinalis* L.) is widely used in Croatia and other Mediterranean countries, and previous studies have shown that it contains various polyphenolic compounds, including diterpenoids, flavonoids, and phenolic acids and their derivatives. The most abundant phenolic acid derivatives in sage are carnosic acid and its derivatives, rosmarinic acid, methyl rosmarinate, caffeic acid, salvianolic acid K, syringic acid, and vanillic acid [1–5]. In addition to phenolic acids, sage contains significant amounts of flavonoids, which occur mainly as flavones, luteolin and apigenin glycosides (luteolin-3-O-glycosides and luteolin-7-O-glycosides) [2,5], and catechins [3,5]. To maximize the extraction of polyphenols from plant material in a shorter time, new and promising extraction methods have recently been introduced, i.e., ultrasonic extraction [6,7], microwave-assisted extraction [4,8], high hydrostatic pressure [6,9–16], and a combination of enzyme-assisted extraction and high hydrostatic pressure [17,18]. In this sense, high hydrostatic pressure (HHP) extraction is an alternative to the extraction techniques mentioned above, as this novel method can be used for the extraction of many polyphenols from sage with less time, lower solvent consumption, and high extraction efficiency. HHP enhances mass transport phenomena where the plant samples are subjected to pressures ranging from 100 to 800 MPa, or in

some cases even up to 1000 MPa [15]. One of the advantages of this extraction technique is that it can be performed at room temperature and low temperatures down to 60 °C, avoiding the structural alteration and degradation of heat-sensitive bioactive compounds. Thus, HHP could be an effective and recommendable non-thermal technique for the extraction of polyphenols from sage. To our knowledge, there is no information on the use of high hydrostatic pressure extraction for the extraction of polyphenolic compounds from sage leaves.

In the field of corrosion protection and public health problems, researchers are focusing on environmental protection and the search for environmentally friendly corrosion inhibitors. Natural antioxidants with multiple polar atoms and electron-rich bonds are potential anticorrosive agents and green alternatives to inorganic anticorrosive agents [19]. Considering the diverse structure of polyphenols present in sage, it is a promising potential source of so-called green inhibitors. There are now several natural plant extracts that have been considered as environmentally friendly potential corrosion inhibitors for steel in acidic media. Research studies in the literature report that plant extracts have a corrosion inhibiting effect and usually act as mixed types of inhibitors. For example, Ficus tikoua leaf extract [20] or ginger extract [21] can be used as natural inhibitors for carbon steel, while Thymus vulgaris for 3014 stainless steel [22], mint leaves [23] and green eucalyptus leaf extract [24] are used for mild steel under acidic conditions. In addition, natural organic antioxidants from green tea [19] act as mixed types of inhibitors: pomegranate extract [25], peach pomace extract [26] for steel, lychee extract for aluminium [27], orange peel extract for magnesium [28], terebinth extract for iron [29], turnip peel extract for copper [30] and olive leaf extract in a sodium chloride solution [31]. In the literature search, we found that sage leaf extract [32] and sage essential oil [33] act as green corrosion inhibitors for steel in acidic media. Few studies have been conducted to investigate corrosion inhibition by natural products on tinplate or pure tin in alkaline media used in the canning industry. For example, pectin isolated from tomato peel waste acts as a cathodic corrosion inhibitor for tin in 2% NaCl, 1% acetic acid, and 0.5% citric acid [34]. In addition, onion essential oil can be used as a natural inhibitor of tinplate corrosion in the production of canned tomato puree [35]. In our previous study, we demonstrated that sage leaf extract obtained after Soxhlet extraction acts as a mixed inhibitor of tinplate in 3% sodium chloride solution [36].

We hypothesized that sage extracts obtained after HHP extraction are rich in polyphenols and represent a potential so-called green inhibitor of tinplate in alkaline media. The aim of this study was to investigate the parameters of HHP extraction as an environmentally friendly strategy for the extraction of polyphenolic compounds from wild sage (*Salvia officinalis* L.) leaves. In selecting the process parameters for HHP extraction, we were guided by the results of previous studies [6,9–14,18], focusing on the use of lower temperatures, lower pressures and shorter extraction times using solvents with a high proportion of the aqueous phase in the organic phase. First, the process parameters for the extraction of total polyphenols and rosmarinic acid as the main phenolic compound in sage were optimised using analysis of variance, response surface methodology (RSM) and partial least squares regression (PLS-R). The polyphenolic composition of the sage extracts was determined by high-performance liquid chromatography (HPLC-DAD). After preparation under the optimum HHP parameters, the sage extract was analysed for its corrosion inhibitory effect on tinplate in 3% sodium chloride solution by potentiodynamic and electrochemical impedance spectroscopy and attenuated total reflectance Fourier infrared spectroscopy (ATR-FTIR).

2. Materials and Methods

2.1. Plant Material and Chemicals

Fresh leaves were collected from wild sage plants (*Salvia officinalis* L.) in the southern Mediterranean region of Croatia. The leaves were air-dried at room temperature (20 ± 2 °C) for one week. The dry leaves were packed in polyethylene bags and stored in a dark, dry

and cool place. Before use, the plant material was crushed using a household blender (Tefal GT1108, Bratislava, Slovakia) and then used for extraction.

Folin–Ciocalteu reagent, anhydrous sodium carbonate, sodium chloride, (+)-catechin, epicatechin, apigenin, luteolin, rosmarinic acid, gallic acid, and chlorogenic acid were used. All chemicals were purchased from Sigma Aldrich (Buchs, Switzerland).

2.2. Extraction Procedure with High Hydrostatic Pressure

A sample (3 g) of sage leaf powder was mixed with a suitable solvent (50 mL) and placed in a polyethylene bottle. The bottle was capped after removing the air from inside and placed in a hydrostatic pressure container; (high pressure: 300, 450, 600 MPa; extraction time: 5, 10, 15 min). At room temperature (25 °C) and at 60 °C, the mixture was filtered through filter paper. The isostatic apparatus with high hydrostatic pressure was purchased from Stansted Fluid Power LTD (Stanford, UK). Effective volume of the vessel: 2 L; maximum working pressure: 1250 MPa; inner diameter: 100 mm; pressure transmission medium: water.

2.3. Total Polyphenols

The determination of total polyphenols in the sage extracts was carried out according to the method described previously [37]. In brief, 250 µL of sage extract was added, followed by the addition of 15 mL of distilled water and 1250 µL of Folin–Ciocalteu's phenolic reagent diluted with distilled water (1:2, v/v). After 3 min, 3750 µL of saturated (20%, w/v) sodium carbonate solution was added to the mixture. The mixture was made up to 25 mL in the volumetric flask with distilled water. After 30 min in a water bath at 50 °C, the mixture was measured against the reagent blank at 765 nm using a Lambda 1 UV-VIS spectrophotometer (Lambda 1, Perkin-Elmer, Waltham, MA, USA). Total polyphenols were calculated using the standard calibration curve for rosmarinic acid ($y = 0.0034x$; $R^2 = 0.9999$) and expressed as milligrams of rosmarinic acid equivalent (RAE)/100 g dry sage. Values were expressed as means ($n = 3$) ± standard deviations (*S.D.*).

2.4. HPLC-DAD Analysis of the Polyphenolic Compounds

An Agilent 1260 LC Infinity quaternary HPLC system (Agilent Technologies, Santa Clara, CA, USA) equipped with UV/Vis and DAD, an automatic injector, and 4.03 Chem Station software on a Luna C18 column, particle size 5 µm (250 × 4.6 mm i.d.) (Phenomenex, Torrance, CA, USA) was used for the analysis. The solvent composition and gradient conditions used have been described previously [2]. The polyphenolic compounds were identified by comparing the retention times and characteristic UV/Vis spectra with those of the authentic standards, with phenolic acids and catechins identified at 280 nm and flavones at 340 nm. The quantitative determination was carried out using the calibration curves of the standards. Calibration curves of different standard compounds were generated for the quantitative analysis: (+)-catechin ($y = 13.241x$; $R^2 = 0.9949$); epicatechin ($y = 10.929x$, $R^2 = 0.9932$); apigenin ($y = 107.04x$, $R^2 = 0.9969$); luteolin ($y = 119.39x$; $R^2 = 0.9978$); rosmarinic acid ($y = 31.594x$; $R^2 = 0.9965$); gallic acid ($y = 33.028x$; $R^2 = 0.9905$); and chlorogenic acid ($y = 69.893x$; $R^2 = 0.9874$). The results were expressed in a mg per 100 g of dry sage. Values were expressed as means ($n = 3$) ± standard deviations (*S.D.*).

2.5. Electrochemical Measurements

Electrochemical potentiodynamic polarisation and impedance spectroscopy measurements were performed with a potentiostat/galvanostat ZRA (Gamry Instruments Inc., Warminster, PA, USA). The electrochemical experiments were performed with three electrodes: a platinum electrode as a counter electrode, a saturated calomel electrode as a reference electrode, and a working electrode in an electrolytic cell containing 3% sodium chloride solution in the absence and presence of different concentrations (0.2, 0.4, 0.6, 0.8, 1, and 2 g/L) of sage extract. The working electrode was cut from tinplate and the tinplate sample was embedded in the polytetrafluoroethylene holder. The tinplate used

for the experiments was tinned carbon steel with a tin coating thickness of about 2.8 g/m, supplied by Lim Samoborka (Samobor, Croatia). The working electrode was washed with distilled water and degassed with ethanol before each test run. To stabilise the steady-state potential, the working electrode was immersed in the solution at 25 °C for 60 min. The potentiodynamic current-potential curves were recorded by automatically changing the electrode potential from −0.6 to a maximum of −0.1 V at a sampling rate of 1 mV/s. The electrochemical impedance spectroscopy measurements were performed in the frequency range of 100kHZ $^{-1}$ mHz at a constant open-circuit potential with an amplitude of 10 mV. Each experiment was repeated three times, and Nyquist and polarisation plots were generated from the individual measurements. The corrosion parameters were determined using the Tafel method of extrapolation and Faraday's laws via software (ZView 3.4 software from Scribner Associates), while the impedance parameters were determined using the appropriate electrical circuit models. The kinetic parameters measured during the corrosion processes were used to calculate the surface coverage θ, the polarisation resistance R_p and the corrosion current density j_{corr}.

2.6. Analysis of Attenuated Total Internal Reflection and Fourier Transform in the Infrared (ATR-FTIR)

The protective adsorption film formed on the tinplate after immersion in the 3% sodium chloride solution with the addition of 0.6 g/L sage extract for 12 h at room temperature was characterized by attenuated total internal reflection and Fourier-transform infrared analysis (ATR-FTIR) (IRAffinity-1; SHIMADZU, Kyoto, Japan). A wavenumber range of 400–4000 cm^{-1} was used to record the IR spectra and identify the protective film formation by comparison with the standard peak positions of the groups, and the spectral resolution was 4 cm^{-1}. Attenuated total internal reflection and Fourier-transform infrared (ATR–FTIR) analysis was carried out.

2.7. Statistical Analysis

The partial least squares regression (PLS-R) approach was used to analyse the effects of linear combinations of extraction variables—temperature, ethanol, pressure, and time (predictors—*X*)—on total polyphenol production (response—*Y*). The analysis was performed using the package "plsdepot" in the statistical software R v.3.2.0. The PLS scores associated with the first two PLS components generated in the model are new variables that summarize the X variables. The scores contain the information about the objects and their similarity and were therefore used to interpret the PLS-R model. We performed a linear model analysis (lm) fitted to the PLS scores using the function aov to test for the significance between phenol production and the extraction variables in a "response–predictor" relationship. The results were determined using R v. 3.2.0 and presented graphically. A threshold value of $p \leq 0.05$ is considered significant in the linear model analysis. The analysis of variance (ANOVA) was performed using Statistica 10.0 software. Three-dimensional categorized plots showed the influence of all observed process parameters on the mass fraction of total polyphenols based on the analysis of variance categorized by solvent, temperature, time, and high hydrostatic pressure. To analyse the possible effects of the extraction parameters at high hydrostatic pressure on the chemical composition of the polyphenols (determined by HPLC-DAD), the non-parametric Spearman rank correlation measure was used. All analyses were performed with R v. 3.2.0 [38]. The significance level was set at $p \leq 0.05$. To visualize the polyphenolic composition of the sage extract, heatmaps were created with the heatmaply function (R package heatmaply v. 0.15.12 https://cran.r-project.org/web/packages/heatmaply/, accessed on 5 September 2023) using standard methods for calculating the distance matrix ('Euclidean').

3. Results and Discussion

3.1. Effect of Extraction with High Hydrostatic Pressure on Total Polyphenols

The parameters of HHP extraction as an environmentally friendly strategy for the extraction of polyphenolic compounds from wild sage (*Salvia officinalis* L.) leaves were investigated. Ethanol concentration, HHP, extraction temperature and time are key factors in the HHP extraction processes, as they both influence the kinetics of total polyphenolic compounds release from sage leaves. The total amount of polyphenolic compounds in sage extracts was determined using a spectrophotometric method (Table 1) and the process parameters for HHP extraction of total polyphenols were optimized using analysis of variance (Table 2), response surface methodology (RSM) (Figure 1), and partial least squares regression (PLS-R) (Figures 2 and 3). Based on the evaluation of the optimal conditions for HHP extraction, solvent selection, temperature, pressure and extraction time, the optimal conditions for HHP extraction were determined. Our results have shown that the sage extracts are rich in polyphenols, which confirms the results of some other available studies [3,5,39,40]. The mass fraction of total polyphenols in sage extracts was determined in the range from 30% ethanolic extract at 60 °C (3811.84 mg/100 g dry matter) to pure ethanol at 25 °C (100.25 mg/100 g dry matter) (Table 1). Some variability was found in the total polyphenolic compounds, which is consistent with the results of another study on the optimization of the HHP process parameters of tomato peels [12]. Some variability may occur in the content of sage samples.

Table 1. The results of mass fraction of total polyphenols of sage extract obtained using extraction under high hydrostatic pressure at 300, 450 and 600 MPa and extraction temperature at 60 °C for 5, 10 and 15 min, using 30% and 96% ethanol. Results of mass fraction of total polyphenols are expressed as mg RAE per 100 g dry matter.

***w*(Total Polyphenols)/(mg GAE/100 g dm)**					
***T*(Extraction Temperature)/(°C)**		**25**		**60**	
***p*(Hydrostatic Pressure)/(MPa)**	***t*(Extraction Time)/(min)**	**30% Ethanol**	**96% Ethanol**	**30% Ethanol**	**96% Ethanol**
300	**5**	108.50 ± 0.90	100.25 ± 0.99	2616.71 ± 9.62	1255.22 ± 6.54
	10	113.14 ± 1.21	102.39 ± 0.82	2602.90 ± 8.51	1676.75 ± 7.31
	15	122.55 ± 0.98	218.32 ± 1.01	2703.79 ± 6.45	1903.35 ± 5.96
450	**5**	104.82 ± 0.52	251.38 ± 1.25	2949.22 ± 10.51	1461.60 ± 5.98
	10	230.22 ± 0.90	262.40 ± 0.98	2846.79 ± 7.97	1641.50 ± 7.41
	15	236.97 ± 1.31	296.57 ± 0.99	2901.91 ± 8.98	1788.43 ± 8.82
600	**5**	112.66 ± 0.78	294.02 ± 1.32	3811.84 ± 4.98	3528.31 ± 6.11
	10	135.63 ± 1.23	302.80 ± 1.25	3491.41 ± 8.54	2288.97 ± 9.21
	15	121.05 ± 1.28	309.63 ± 0.87	2887.13 ± 8.53	1644.05 ± 8.13

Table 2. Linear model fitted with analysis of variance on PLS scores between total polyphenols and rosmarinic acid as the main phenolic compound in sage extracts, and variables associated with extraction conditions, temperature, ethanol, pressure, and time.

Extraction Conditions	**Df**	**Sum Sq**	**Mean Sq**	**F Value**	***p* Value**	**Pr (>F)**
			Total polyphenols			
Temperature	1	45,734,968	45,734,968	209.7043	2.42×10^{-15} ***	<0.001
Ethanol	1	2,137,103	2,137,103	9.7991	0.00379 ***	<0.001
Pressure	1	1,216,634	1,216,634	5.5785	0.02464 ***	<0.001
Time	1	88,912	88,912	0.4077	0.52784	
Residuals	31	6,760,871	218,093			
			Rosmarinic acid			
Temperature	1	545,171	545,171	70.2266	2.36×10^{-9} ***	<0.001
Ethanol	1	137,041	137,041	17.6530	0.0002188 ***	<0.001
Pressure	1	4679	4679	0.6028	0.4435977	
Time	1	6833	3417	0.4401	0.6480596	
Residuals	30	232,890	7763			

Signif. codes: 0 '***'. Df = degrees of freedom, Sum Sq = sum of squares, Mean Sq = mean squares. Means of tested comparisons are significantly different when $p < 0.05$.

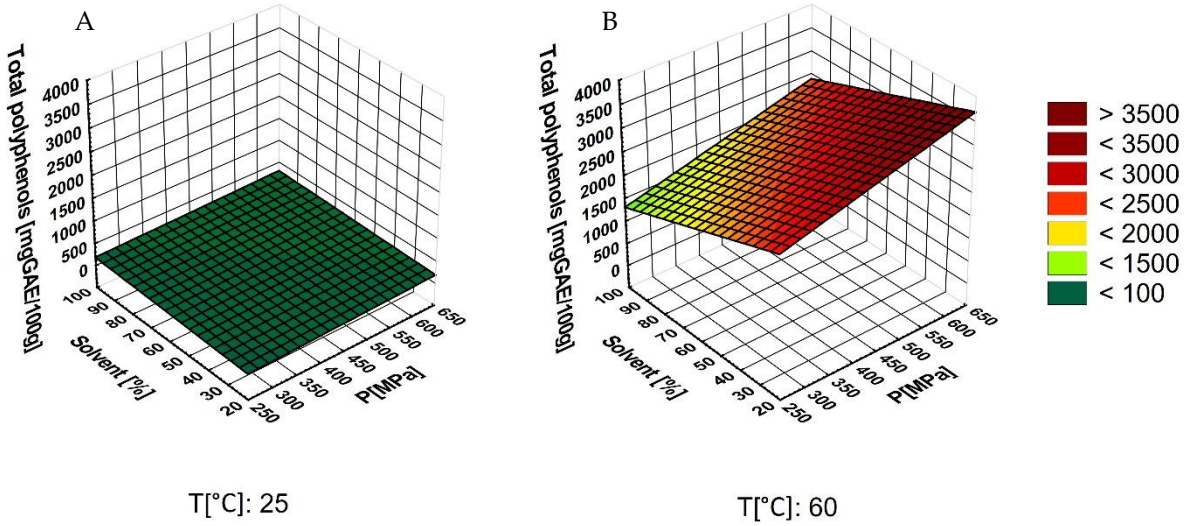

Figure 1. Response surface methodology. Change in the mass fraction of total phenols as a function of the (**A**) solvent/pressure at 25 °C, and (**B**) solvent/pressure at 60 °C.

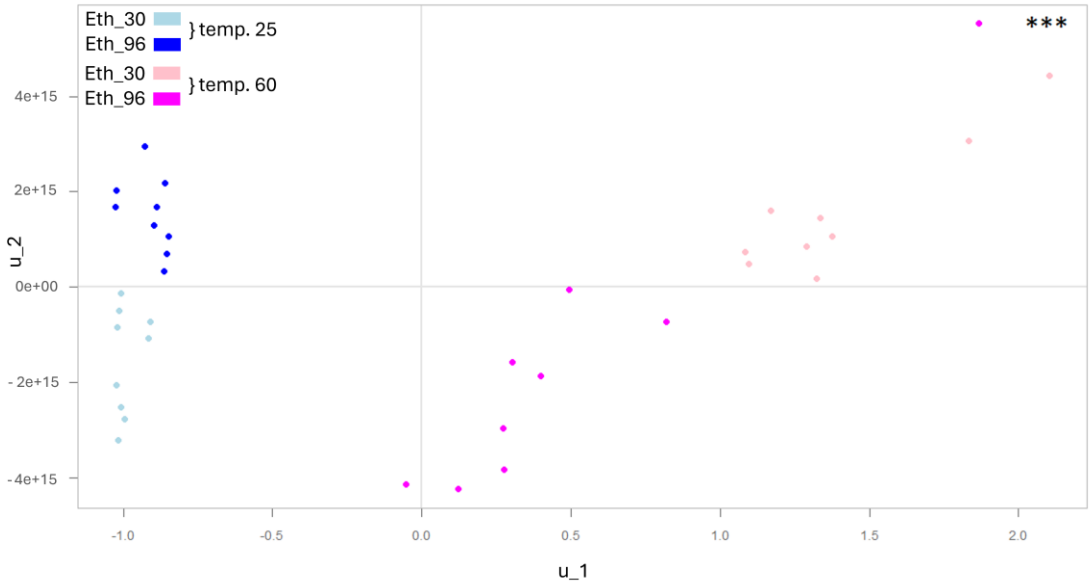

Figure 2. Partial least squares regression (PLS-R) score plot of the total polyphenols, based on y components (u1 and u2). Plots represent the relationship between response variable (total polyphenols) and predictors (temperature and ethanol). Significant effect ($p < 0.01$) in 'response–predictor' relation (performed by using the linear model on PLS-R scores) is indicated by ***.

Increasing the temperature from 25 to 60 °C improved the solubility of polyphenols in the solvent, and the higher mass fraction of total polyphenols (3811.84 mg/100 g) was obtained at 60 °C when 30% ethanol was used, but pure ethanol resulted in the loss of target compounds (3528.31 mg/100 g) at the same extraction temperature and time (Table 1). Therefore, temperature plays an important role in determining the extraction efficiency of polyphenolic compounds, and in the HHP process the temperature is usually between 20 and 60 °C, which is required for the extraction of the bioactive compounds [12,13,41], as degradation may occur at higher temperatures. Increasing the extraction temperature from 25 to 60 °C leads to a higher yield of the target compounds as the diffusivity of the solvent towards the internal components of the matrix increases.

From the results presented, it can be concluded that ethanol concentration and extraction temperature had a significant effect ($p < 0.05$, Table 2) on the change of total polyphenols in sage extract. PLS-R analysis confirmed that the total polyphenolic extrac-

tion was significantly dependent on the extraction temperature and ethanol concentration (Figure 2). A lower ethanol concentration and an increase in temperature and pressure improved the extraction results (Figure 1), although the use of 30% ethanol as the extraction solvent gave a better yield of total polyphenolic compounds than the use of pure ethanol at 60 °C. Almost all extracts extracted with pure ethanol had significantly lower amounts of total phenolic compounds compared to 30% ethanol.

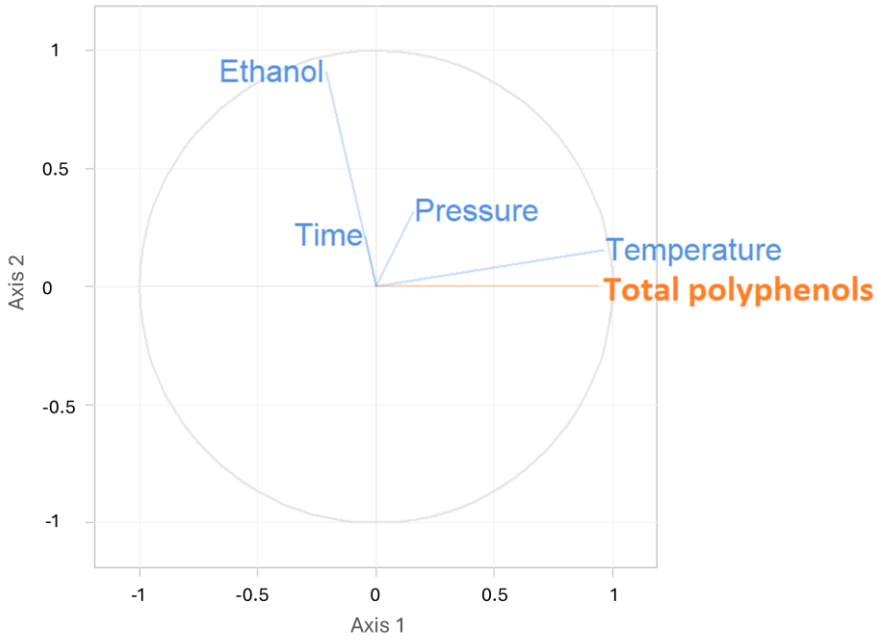

Figure 3. Radar of the partial least squares regression (PLS-R) of the correlation. The orange line represents the mass fraction of total polyphenols (reaction variable), while the blue lines represent the extraction conditions: temperature, pressure, ethanol and time (predictors). Variables that are on the same side of the square within the circle are positively correlated, while those on the opposite side are negatively correlated. Longer lines represent higher correlations, while shorter lines represent lower correlations.

The results showed that increasing the extraction pressure from 450 to 600 MPa had a positive effect on the mass fraction of the total-extracted total polyphenols (Table 1). Statistical analysis of these data revealed a positive effect of HHP on the mass fraction of total polyphenols, while analysis of variance confirmed that the extraction of total polyphenols differed significantly as a function of HHP ($p < 0.05$, Table 2, Figures 1–3). The enhanced effects were pressure dependent, with the extract at HHP 600 MPa showing the highest increase. With increasing pressure, the inner membranes are destroyed, allowing increased solvent influx and increased contact with the solvent. This mechanism was confirmed by previous studies [12–18], which showed that the pressure required to extract the bioactive compound from the biomaterials is generally in the range of 100–600 MPa in a shorter time compared to other extraction methods. Briones-Labarca et al. [6] reported that the application of HHP at 450 MPa destroys the cell wall and increases the permeability of the cells to the solvent, resulting in a high release of polyphenols. Torres-Ossandón et al. [14] showed an increase in total phenolics from Cape gooseberry pulp treated at 400 MPa/3 min compared to 300 MPa. Moreover, pressures below 400 MPa are not strong enough to inactivate polyphenol-degrading enzymes (such as polyphenol oxidases). In addition, 600 MPa HHP at 60 °C for only 5 min was able to extract significantly more total phenols than 300 or 450 MPa pressure. Compared to our results, Shinwari and Rao [13] showed the optimization of the extraction of nutraceuticals from saffron under HHP (580 MPa, 50 °C, 5 min). Statistical analysis of these data revealed a significant influence ($p < 0.001$, Table 2) of almost all variables studied on the extraction results of total polyphe-

nols. Increasing the extraction time from 5 to 15 min had no significant effect on the recovery of total polyphenols ($p > 0.001$, Table 2). Therefore, an optimal pressure treatment time of 5 min was found to be the most suitable for the isolation of total polyphenols, which is related to the main advantages of HHP extraction, i.e., shorter extraction time, which [6,9,12,14] suggests that an extraction time of 1 to 10 min is sufficient for the extraction of phenolic compounds. The extraction time of 5 min is therefore sufficient to extract an optimal mass fraction of polyphenolic compounds from sage. Regardless of the variability of the total polyphenolic compounds, a strong interaction between these process parameters is clearly recognisable, as shown by the analysis of variance (Table 2), the response surface methodology (RSM) (Figure 1) and the partial least squares regression (PLS-R) (Figures 2 and 3). From this, we can conclude that the HHP process is an emerging technology for the extraction of polyphenols from sage, and the best results were obtained under the test conditions with ethanol at a concentration of 30% at an extraction temperature of 60 °C and an extraction HHP of 600 MPa for 5 min.

3.2. *Effect of Extraction with High Hydrostatic Pressure on Individual Polyphenols*

The individual phenolic compounds were determined by HPLC-DAD analysis (Tables 3 and 4, Figure 4). In addition, it should be checked whether the process parameters for the isolation of the total phenolic compounds by HHP extraction correspond to the process parameters of certain individual compounds—for example, the process parameters for the HHP extraction of rosmarinic acid as the most important polyphenolic compound in sage extract. Therefore, the process parameters for the isolation of the individual polyphenolic compounds by HHP extraction were optimized using analysis of variance (Table 2), Spearman's rank correlations (Figure 5) and response surface methodology (RSM) (Figure 6).

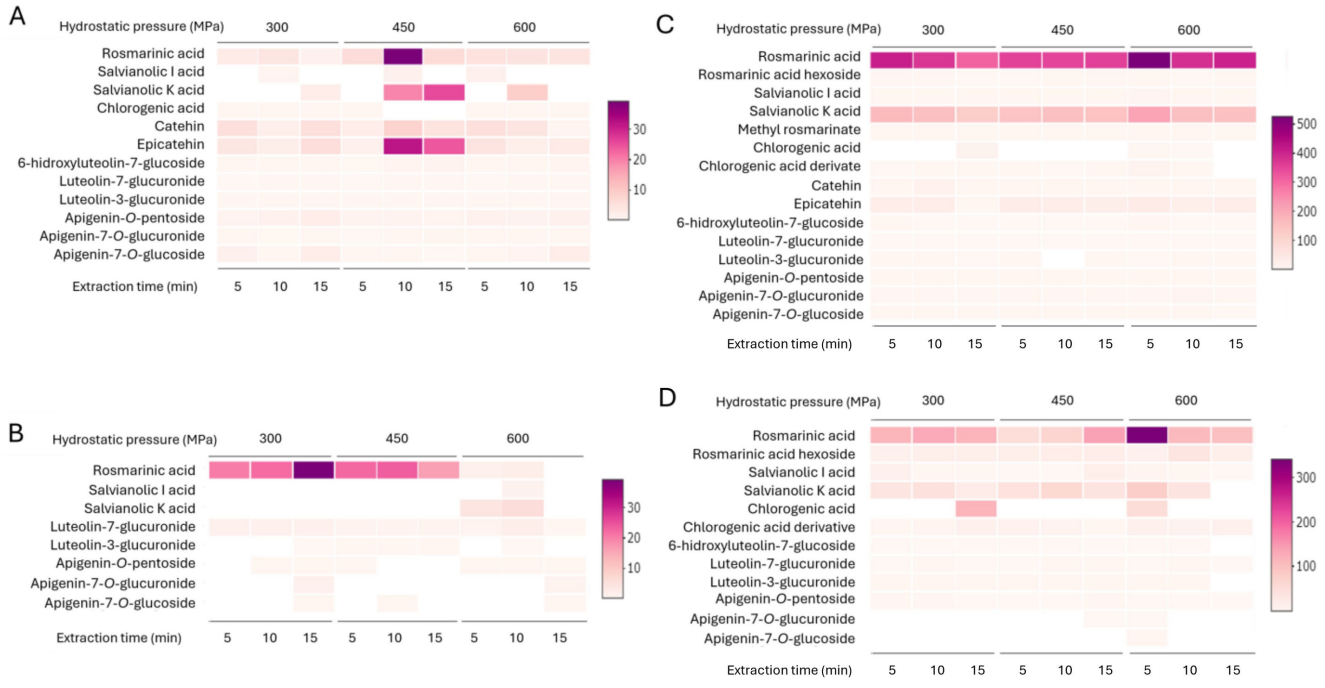

Figure 4. Heatmaps of the polyphenol composition of sage extract, as determined by HPLC. The sage extracts were obtained by extraction under high hydrostatic pressure without different extraction conditions: (**A**) 30% ethanol and (**B**) 96% ethanol at a temperature of 25 °C, (**C**) 30% ethanol and (**D**) 96% ethanol at a temperature of 60 °C The colours correspond to the percentage of a specific compound in the total peak area (as shown in detail in Tables 2 and 3).

Table 3. The results of HPLC UV-DAD determination of sage polyphenols obtained using high hydrostatic pressure at 300, 450 and 600 MPa, and extraction temperature at 25 °C for 5, 10 and 15 min using 30% and 96% ethanol.

***p*(Hydrostatic Pressure)/(MPa)**	**300**			**450**			**600**		
***t*(Extraction Time)/(min)**	**5**	**10**	**15**	**5**	**10**	**15**	**5**	**10**	**15**
					***w*(Polyphenols)/(mg/100 g dm)**				
					30% ethanol				
Rosmarinic acid	2.87 ± 0.13	3.83 ± 0.13	1.48 ± 0.11	5.59 ± 0.23	35.90 ± 0.13	5.85 ± 0.13	4.69 ± 0.28	4.43 ± 0.65	4.21 ± 0.13
Salvianolic I acid	nd	0.85 ± 0.02	nd	nd	1.35 ± 0.08		1.35 ± 0.10	nd	nd
Salvianolic K acid	nd	nd	2.24 ± 0.22	nd	17.84 ± 0.05	23.60 ± 0.56	nd	8.32 ± 0.23	nd
Chlorogenic acid	0.46 ± 0.02	0.25 ± 0.01	0.38 ± 0.15	0.34 ± 0.05	nd	nd	0.50 ± 0.02	0.37 ± 0.17	0.33 ± 0.02
Catehine	5.07 ± 0.18	1.91 ± 0.12	5.18 ± 0.23	2.25 ± 0.19	7.61 ± 0.22	4.35 ± 0.22	5.01 ± 0.11	4.10 ± 0.25	0.70 ± 0.03
Epicatehine	3.57 ± 0.25	2.15 ± 0.14	5.36 ± 0.17	1.61 ± 0.25	29.16 ± 0.65	22.33 ± 0.48	4.26 ± 0.12	1.90 ± 0.18	2.78 ± 0.28
6-hidroxyluteolin-7-glucoside	0.28 ± 0.01	0.36 ± 0.01	0.48 ± 0.09	0.19 ± 0.03	0.68 ± 0.12	0.95 ± 0.05	0.30 ± 0.01	0.59 ± 0.09	0.74 ± 0.08
Luteolin-7-glucuronide	0.55 ± 0.05	13.39 ± 0.31	1.00 ± 0.03	0.79 ± 0.15	4.13 ± 0.15	3.68 ± 0.10	0.65 ± 0.05	1.23 ± 0.05	0.86 ± 0.09
Luteolin-3-glucuronide	0.27 ± 0.01	0.27 ± 0.02	0.27 ± 0.02	0.39 ± 0.08	0.40 ± 0.08	0.29 ± 0.02	0.31 ± 0.02	0.39 ± 0.02	0.48 ± 0.05
Apigenin-*O*-pentoside	1.03 ± 0.11	1.09 ± 0.21	2.36 ± 0.03	0.93 ± 0.25	1.04 ± 0.02	0.72 ± 0.03	1.17 ± 0.05	1.27 ± 0.01	1.48 ± 0.10
Apigenin-7-*O*-glucuronide	0.32 ± 0.01	0.19 ± 0.01	0.46 ± 0.05	0.17 ± 0.04	0.55 ± 0.01	0.6 ± 0.09	0.42 ± 0.02	0.36 ± 0.02	0.36 ± 0.05
Apigenin-7-*O*-glucoside	1.66 ± 0.22	0.37 ± 0.03	2.24 ± 0.08	0.41 ± 0.03	0.44 ± 0.02	0.08 ± 0.01	0.48 ± 0.03	0.67 ± 0.07	2.34 ± 0.02
					96% ethanol				
Rosmarinic acid	20.54 ± 0.28	22.18 ± 0.28	39.27 ± 0.15	22.51 ± 0.23	23.66 ± 0.31	16.43 ± 0.12	1.53 ± 0.20	2.33 ± 0.11	nd
Luteolin-7-glucuronide	1.70 ± 0.06	1.62 ± 0.05	1.79 ± 0.02	1.03 ± 0.05	1.18 ± 0.04	1.17 ± 0.02	1.17 ± 0.03	2.23 ± 0.31	0.39 ± 0.04
Luteolin-3-glucuronide	nd	nd	0.12 ± 0.01	0.52 ± 0.02	0.60 ± 0.02	0.42 ± 0.01	nd	0.20 ± 0.02	nd
Apigenin-*O*-pentoside	4.28 ± 0.08	4.53 ± 0.08	4.88 ± 0.18	3.91 ± 0.11	4.22 ± 0.12	2.14 ± 0.06	2.96 ± 0.07	5.59 ± 0.12	1.12 ± 0.12

Nd—not detected.

Table 4. The results of HPLC UV-DAD determination of sage polyphenols obtained using extraction under high hydrostatic pressure at 300, 450 and 600 MPa, and extraction temperature at 60 °C for 5, 10 and 15 min using 30% and 96% ethanol. Results of mass fraction of individual polyphenols are expressed as mg per 100 g dry matter.

***p*(Hydrostatic Pressure)/(MPa)**	**300**			**450**			**600**		
***t*(Extraction Time)/(min)**	**5**	**10**	**15**	**5**	**10**	**15**	**5**	**10**	**15**
	***w*(Polyphenols)/(mg/100 g dm)**								
					30% ethanol				
Rosmarinic acid	408.70 ± 1.19	374.65 ± 1.39	310.66 ± 1.45	355.84 ± 1.15	352.31 ± 2.19	358.49 ± 1.99	526.14 ± 1.23	384.30 ± 1.49	403.01 ± 1.69
Rosmarinic acid hexoside	4.15 ± 0.50	5.10 ± 0.30	1.75 ± 0.19	1.13 ± 0.02	7.52 ± 0.09	3.31 ± 0.11	5.14 ± 0.37	1.49 ± 0.89	2.86 ± 0.61
Salvianolic I acid	6.52 ± 0.21	6.04 ± 0.91	5.29 ± 0.99	6.57 ± 0.32	6.51 ± 0.82	6.18 ± 0.71	8.84 ± 0.93	5.95 ± 0.29	6.59 ± 0.88
Salvianolic K acid	169.68 ± 1.90	156.78 ± 1.19	123.23 ± 0.97	144.23 ± 0.87	156.34 ± 0.69	148.97 ± 0.80	212.63 ± 0.38	156.02 ± 0.77	155.32 ± 0.89
Methyl rosmarinate	3.78 ± 0.28	5.10 ± 0.11	2.00 ± 0.01	3.44 ± 0.39	3.37 ± 0.51	3.81 ± 0.61	4.52 ± 0.22	4.11 ± 0.25	4.63 ± 0.15
Chlorogenic acid	nd	nd	17.37 ± 0.72	nd	nd	nd	1.96 ± 0.22	0.39 ± 0.05	nd
Chlorogenic acid derivate	1.74 ± 0.54	3.39 ± 0.28	2.47 ± 0.17	4.22 ± 0.68	1.52 ± 0.21	3.46 ± 0.31	12.99 ± 0.42	4.51 ± 0.22	nd
6-hidroxyluteolin-7-glucoside	2.33 ± 0.11	1.35 ± 0.09	1.52 ± 0.05	1.32 ± 0.01	1.32 ± 0.22	1.37 ± 0.31	2.21 ± 0.35	1.42 ± 0.38	1.52 ± 0.41
Luteolin-7-glucuronide	21.00 ± 0.34	22.34 ± 0.87	18.18 ± 0.61	13.64 ± 0.34	21.25 ± 0.29	24.46 ± 0.88	27.18 ± 0.71	16.97 ± 0.55	16.50 ± 0.29
Luteolin-3-glucuronide	3.23 ± 0.28	2.93 ± 0.42	1.23 ± 0.35	2.82 ± 0.54	nd	3.09 ± 0.36	0.54 ± 0.08	2.49 ± 0.15	2.91 ± 0.39
Apigenin-*O*-pentoside	3.79 ± 0.19	4.13 ± 0.64	3.21 ± 0.58	2.68 ± 0.62	4.08 ± 0.39	4.33 ± 0.71	4.70 ± 0.60	3.28 ± 0.17	3.05 ± 0.09
Apigenin-7-*O*-glucuronide	3.52 ± 0.14	3.28 ± 0.39	3.04 ± 0.25	2.65 ± 0.08	3.14 ± 0.07	3.17 ± 0.27	4.42 ± 0.24	9.88 ± 0.09	2.29 ± 0.30
Apigenin-7-*O*-glucoside	3.52 ± 0.54	2.77 ± 0.08	2.64 ± 0.04	2.91 ± 0.15	3.42 ± 0.28	3.02 ± 0.19	4.42 ± 0.41	2.96 ± 0.22	1.16 ± 0.07
Catehine	6.86 ± 0.41	17.14 ± 0.88	7.78 ± 0.61	4.25 ± 0.29	7.26 ± 0.81	4.07 ± 0.61	6.69 ± 0.63	3.64 ± 0.71	4.41 ± 0.26
Epicatehine	33.44 ± 0.90	31.18 ± 1.21	6.83 ± 0.91	37.35 ± 2.20	33.36 ± 1.11	29.17 ± 0.89	37.65 ± 0.71	26.34 ± 0.48	32.32 ± 0.81
					96% ethanol				
Rosmarinic acid	115.41 ± 0.28	130.28 ± 0.78	116.73 ± 0.21	51.42 ± 0.15	69.02 ± 0.12	139.42 ± 0.51	340.01 ± 0.28	108.91 ± 0.28	102.15 ± 0.98
Rosmarinic acid hexoside	12.13 ± 0.25	15.96 ± 0.13	16.03 ± 0.13	17.42 ± 0.03	20.20 ± 0.51	21.99 ± 0.72	11.88 ± 0.23	37.58 ± 0.14	17.47 ± 0.55
Salvianolic I acid	1.51 ± 0.48	2.03 ± 0.12	1.31 ± 0.21	1.08 ± 0.04	1.21 ± 0.09	17.30 ± 0.96	6.87 ± 0.13	2.68 ± 0.09	0.96 ± 0.02
Chlorogenic acid derivative	4.90 ± 0.13	7.52 ± 0.11	13.76 ± 0.14	11.02 ± 0.17	7.84 ± 0.14	1.49 ± 0.22	14.06 ± 0.12	11.44 ± 0.18	12.47 ± 0.44

Nd—not detected.

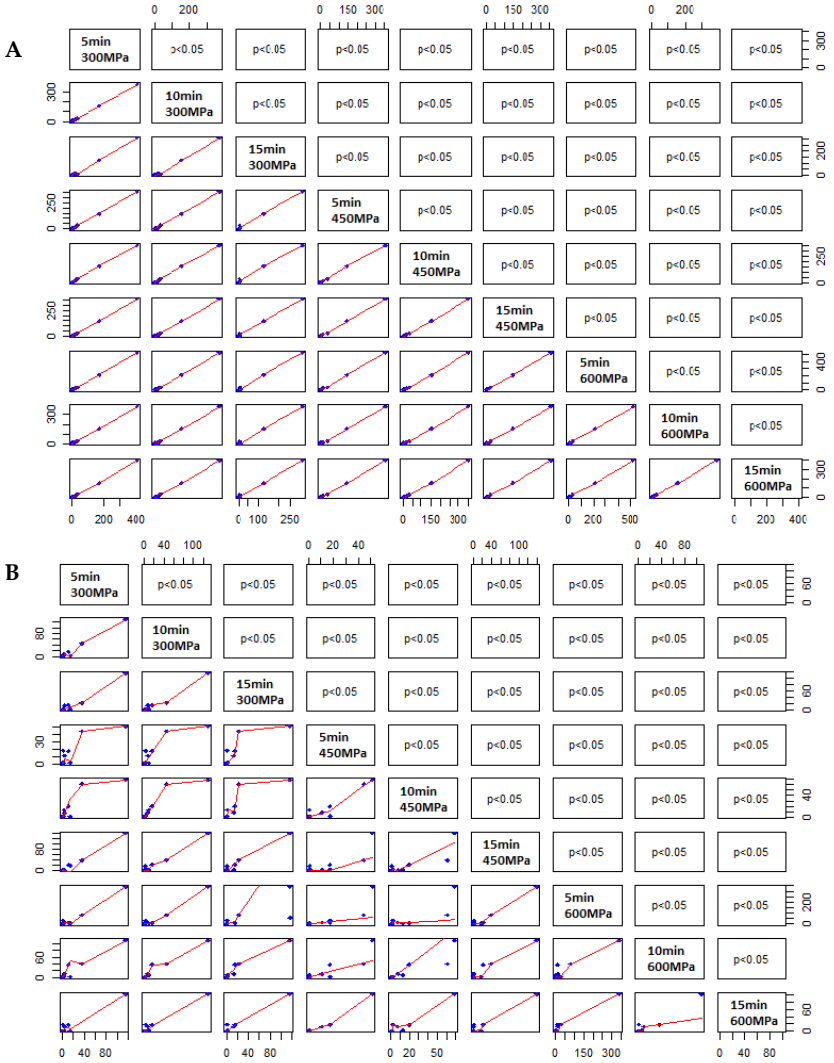

Figure 5. Scatter plot showing correlations between different extraction conditions under high hydrostatic pressure. (**A**) 30% and (**B**) 96% ethanol, pressure 300, 450 and 600 MPa, and extraction time 5, 10 and 15 min (on the diagonal), for the chemical composition of sage extract. Significant *p*-values based on Spearman's rank test are shown above the diagonal, while bivariate scatter plots are shown below the diagonal.

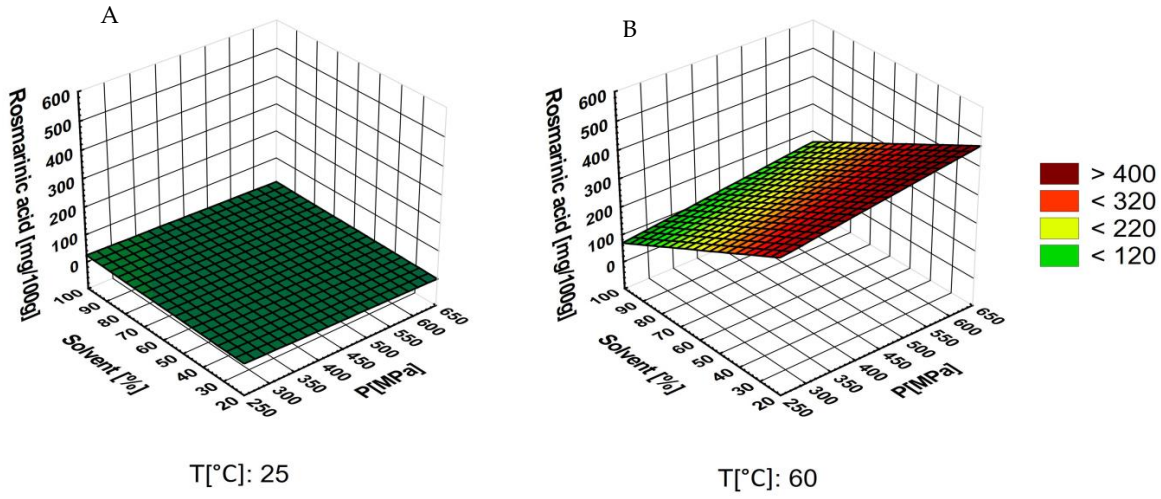

Figure 6. *Cont.*

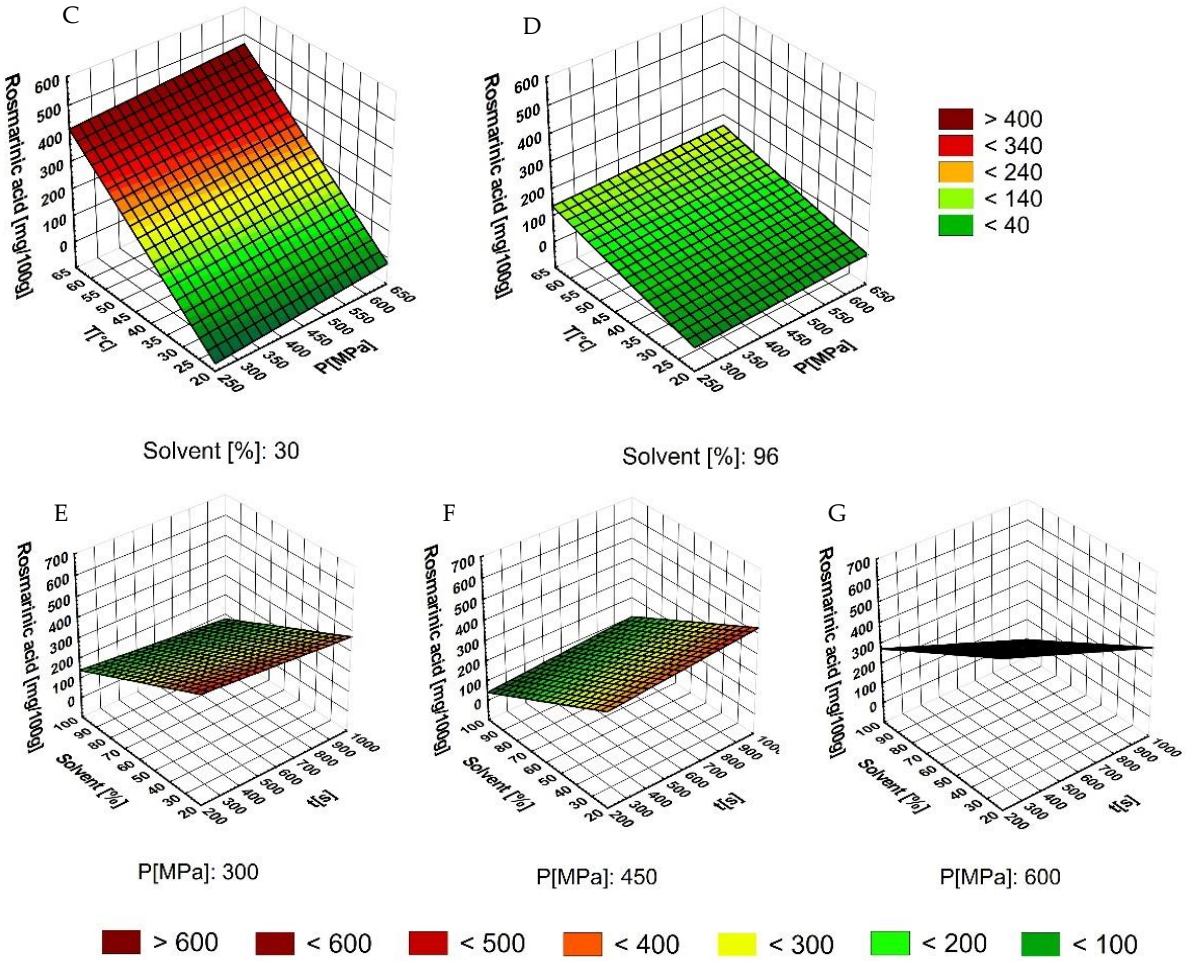

Figure 6. Response surface methodology. Change in the mass fraction of rosmarinic acid as a function of the following: (**A**) temperature (25 °C), solvent (30, 96%), pressure (300, 450, 600 MPa); (**B**) temperature (60 °C), solvent (30, 96%), pressure (300, 450, 600 MPa); (**C**) solvent (30%), temperature (25, 60 °C), pressure (300, 450, 600 MPa); (**D**) solvent (96%), temperature (25, 60 °C), pressure (300, 450, 600 MPa); (**E**) pressure (300 MPa), solvent (30, 96%), time (5, 10, 15 min); (**F**) pressure (450 MPa), solvent (30, 96%), time (5, 10, 15 min); (**G**) pressure (600 MPa), solvent (30, 96%), time (5, 10, 15 min).

Sage extract was shown to be rich in phenolic acids (e.g., rosmarinic acid, rosmarinic acid hexoside, salvianolic acid K, salvianolic acid I, methyl rosmarinate, chlorogenic acid and its derivatives), luteolin glycosides (e.g., 6-hydroxyluteolin-7-glucoside, luteolin-7-glucuronide, luteolin-7-O-glucuronide, luteolin-3-glucuronide), apigenin glycosides (e.g., apigenin-O-pentoside, apigenin-7-glucuronide, apigenin-7-glucoside) and catechins (e.g., catechin, epicatechin) (Tables 3 and 4, Figure 4). Attention was drawn to rosmarinic acid as it is one of the most abundant caffeic acid esters in all sage extracts. The results of our study showed that among all phenolic acids and flavone glycosides, rosmarinic acid was the predominant phenolic acid in all sage extracts, with a maximum mass fraction in 30% ethanolic extract at 60 °C (526.14 mg/100 g dry matter) and minimum at 25 °C (1.48 mg/100 g dry matter) (Tables 3 and 4). Similar results were previously reported for rosmarinic acid in sage extracts at concentrations of 6.3 mg/g prepared by conventional solvent extraction with methanol for 2 h [40] and 5.39 mg/g in sage water residues [5]. Slightly higher results (8.5 mg/g) were previously reported for rosmarinic acid in sage extracts prepared by ultrasonic extraction with two extraction cycles using ethanol [39] or 11.39 mg/g in sage solid residue using a mixture of water and ethanol [5]. Other important compounds detected in sage extracts were salvianolic K-acid (up to 212.63 mg/100 g dry matter), epicatechin (up to 37.65 mg/100 g dry matter), and luteolin-7-O-glucuronide (up to

24.90 mg/100 g dry matter). Our results are consistent with previous literature reports that rosmarinic acid is the most abundant phenolic compound detected in sage. In contrast, the flavone glycosides, catechins, and salvianolic K-acid were the less-concentrated compounds in sage, and have already been detected in sage by other authors [3,5,39].

As mentioned above, sage extracts are rich in polyphenolic compounds, especially rosmarinic acid, which is the predominant polyphenolic compound, so the influence of HHP parameters on the isolation of polyphenolic compounds as well as rosmarinic acid will be discussed in the next section. Regardless of the variability of the polyphenolic compounds, a strong interaction between these process parameters is clearly recognisable, as shown by Spearman's correlation tests (Figure 5). The results of the total polyphenols at both temperatures studied (25 and 60 °C) were in agreement with the results of individual polyphenolic compounds, and the use of 30% ethanol as the solvent resulted in higher extraction yields. Increasing the extraction temperature led to a significant increase in the mass fraction of rosmarinic acid. It is evident from the results that temperature plays an important role in determining the extraction efficiency of polyphenolic compounds, i.e., rosmarinic acid. The effects of ethanol concentration and extraction temperature at HHP on the mass fraction of rosmarinic acid at the extraction temperature (25 and 60 °C) are shown in Figure 6A,B. For example, some phenolic acids (rosmarinic acid hexoside, salvianolic acid K, salvianolic acid I, methyl rosmarinate, chlorogenic acid, and their derivatives) could not be extracted from sage leaves when HHP was performed with pure ethanol at room temperature. The reason for this is probably that the phenolic acids mentioned above are less-polar phenols, which are less soluble in less-polar solvents such as pure ethanol than in 30% ethanol with added water, which increases the polarity of the solvent (Figure 6C,D). The phenolic compounds (catechin and epicatechin) were not extracted from sage with pure ethanol at both temperatures tested (25 and 60 °C). So, based on the results of individual polyphenols (Tables 3 and 4, Figure 4), 30% ethanol at 60 °C was considered the most suitable solvent for the extraction of polyphenols from sage, as the values obtained with this solvent were higher and statistically different ($p < 0.001$, Table 2) from all other values obtained with extraction at room temperature. The change in ethanol concentration had a significant effect on the mass fraction of rosmarinic acid at higher extraction temperature. A positive effect of extraction temperature on the mass fraction of rosmarinic acid in the extract is observed at an ethanol concentration of 30%, while there is almost no effect with pure ethanol. These results suggest that the extractability of polyphenolic compounds from sage, i.e., rosmarinic acid, is significantly affected by the polarity of the solvent used, which could be related to the fact that polyphenolic compounds are often more soluble in polar organic solvents such as aqueous ethanol solution [4]. These results are consistent with previous studies showing that the type of solvent has a major influence on polyphenol extraction capacity in many species, with maximum polyphenol yields determined at an ethanol concentration of 30–50% [39]. So, we can conclude that binary-solvent systems were more efficient than mono-solvent systems, and the highest extraction capacity was obtained by extraction with 30% ethanol, which is in agreement with the results of other authors [11]. According to the mentioned studies, a water–ethanol mixture is an effective solvent for the extraction of polyphenols, in contrast to pure ethanol, which showed the lowest extraction performance. The interaction between the extraction temperature and the high HHP at the ethanol concentration (pure and 30%) for the proposed model is shown in Figure 6C,D. The results showed that increasing the extraction pressure from 450 to 600 MPa had a positive effect on the mass fraction of rosmarinic acid (Figure 6E–G). Statistical analysis of these data revealed no significant effect on the mass fraction of rosmarinic acid (Table 2). Increasing the extraction time from 5 to 15 min had no significant effect on the recovery of rosmarinic acid ($p > 0.001$, Table 2). Therefore, an optimal pressure treatment time of 5 min was found to be the most suitable for the isolation of rosmarinic acid, which is related to the main advantages of HHP extraction, i.e., shorter extraction time, while [6,9,12,14] suggest that an extraction time of 1 to 10 min is sufficient for the extraction of phenolic compounds.

From this, we can conclude that the HHP process is an emerging technology for the extraction of polyphenols from sage and the best results were obtained under the test conditions with ethanol at a concentration of 30% at an extraction temperature of 60 °C and an extraction HHP of 600 MPa for 5 min. Sage extracts obtained by HHP extraction at optimal process parameters are an important source of polyphenolic compounds such as rosmarinic acid, but also of other important polyphenolic compounds such as salvianolic K-acid, epicatechin, and luteolin-7-O-glucuronide. Due to the high content of polyphenolic compounds, sage extracts obtained in this way have the potential to be natural anti-corrosion agents.

3.3. Evaluation of Sage Inhibition Efficiency on Tinplate in a 3% Sodium Chloride Solution

The sage extract obtained by HHP extraction at 600 MPa for 5 min with 30% ethanol concentration at 60 °C was used for further electrochemical analyses. The electrochemical behaviour of tinplate in 3% sodium chloride solution (blank solution) with the addition of different concentrations of sage extract (0.2–2 g/L) at 25 °C was investigated by measuring the potentiodynamic polarisation curve and by using electrochemical impedance spectroscopy (EIS). Figure 7 shows the Tafel diagram for the corrosion of tinplate in a 3%sodium chloride solution in the presence of different concentrations of sage extract (0.2–2 g/L), indicating that the sage concentrations affect the corrosion process. Table 5 and Figure 7 show the calculated values of corrosion current and corrosion potential from the plotted Tafel curves for tinplate in 3% sodium chloride solution with and without sage extract. The addition of sage extract to the blank solution up to concentrations of 0.4 and 0.6 g/L shifted the corrosion potential of the solution to a more positive potential. At concentrations of more than 0.6 g/L, the corrosion potential values shifted to a more negative solution potential (Table 5). Table 5 shows that the OCP values shifted to a more positive potential when sage extract was added to the blank solution (3% NaCl), from −502.9 to −501.0 mV for 0.6 g/L and −453 mV for 0.4 g/L sage-extract concentration. The shift in corrosion potential in the presence of sage extracts (0.4 and 0.6 g/L) ranged from 1.9 to 49.9 mV, indicating that both sage extracts act as mixed-type corrosion inhibitors, since the difference in corrosion potential values does not exceed 85 mV [20,24,36,42–45]. In addition, the addition of sage extract in 3% NaCl shifts both the anodic and cathodic curves to lower current densities, which also proves that the sage extract acts as a mixed-type corrosion inhibitor [21,22,46]. The curves show the dependence of the current density on the sage extract, i.e., the current density decreases with increasing concentration of the sage extract. The value of i_{corr} decreased from 2.880 for the blank value to 0.202 μA/cm^2 in the presence of sage extract (0.6 g/L), which corresponds to a moderately low corrosion level (0.1–0.5 μA/cm^2), and we can conclude that the corrosion process was suppressed. The increase in sage-extract concentration shows the changes in Tafel slopes (βa and βc), which means that sage extract acts as a good protective film on the tinplate surface [47,48]. Moreover, the highest inhibitory effect on tinplate was obtained at a concentration of 0.6 g/L in 3% NaCl (93%).

Table 5. Kinetic parameters for corrosion of tinplate obtained from potentiodynamic polarisation curves with and without the addition of sage extract to 3% NaCl at 25 °C.

γ(Sage Extract)/(g/L)	E_{corr}/(mV)	i_{corr}/(μA/cm^2)	r_{corr}/(mm/year)	βc/(mV/dec)	βa/(mV/dec)	θ	η/(%)
Blank	−502.9	2.880	1.330	−55.24	55.24		
0.2	−522	2.249	1.039	−118.6	66.74	0.219	22.0
0.4	−453	0.768	0.355	−326.1	25.09	0.733	73.3
0.6	−501.0	0.202	0.934	−113.1	55.14	0.929	93.0
0.8	−523.4	2.079	0.960	−71.68	24.45	0.278	27.8
1.0	−552.3	0.826	0.381	−355.0	54.73	0.713	71.3
2.0	−546.2	1.421	0.656	−229.4	32.60	0.507	50.7

i_{corr} = corrosion current density, βc and βa = anodic and cathodic Tafel slope, θ = surface coverage, η = inhibition efficiency.

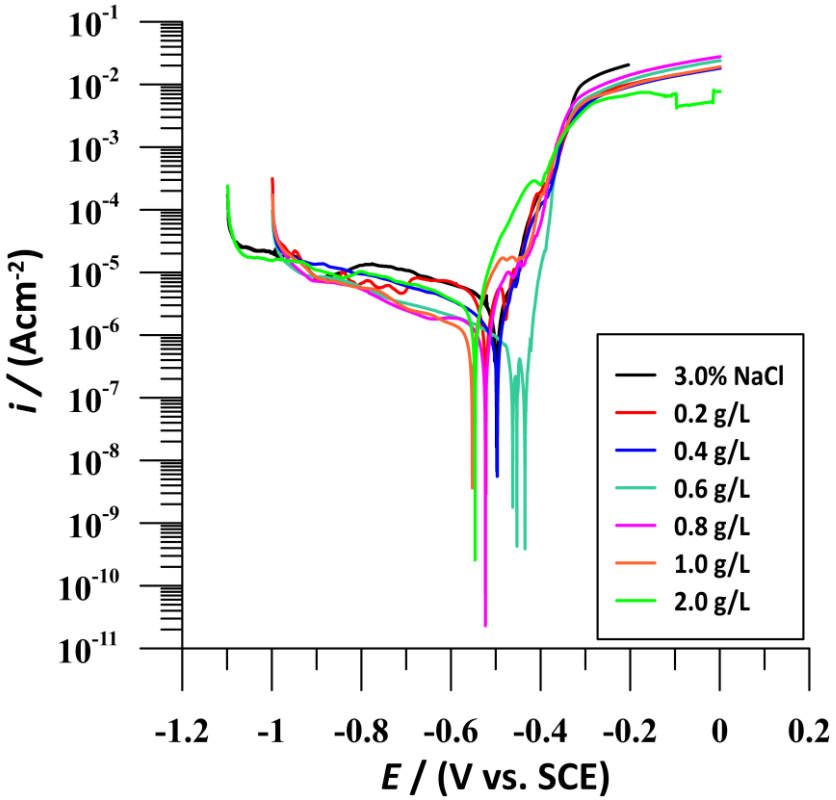

Figure 7. Potentiodynamic polarisation curves (1 mV/s) for tinplate in 3% NaCl solution at 25 °C with the addition of different concentrations of sage extract.

Figure 8 shows the Nyquist plots comparing the corrosion behaviour of tinplate in 3%-sodium chloride solution in the presence of different concentrations of sage extract (0.2–2 g/L) as inhibitor, and it is obvious that the corrosion resistance increases with the increase in sage concentration in 3% NaCl. In the concentration range from 0.2 to 0.6 g/L, the radius of the semicircles increased sharply, which was accompanied by a significant increase in polarisation resistance compared to the unloaded surface. The largest semicircle diameter was obtained with the addition of 0.6 g/L sage inhibitor, with a value of 164.3 kΩ·cm^2 after 2.5 h of testing (Table 6), which was several times higher than that in the non-inhibited solution, consistent with the literature [21,24,29,36,44]. Thus, we confirmed the presence of an adsorbed protective film, which led to an increase in the inhibitory effect. Such an increase in the semicircle is the result of covering the surface of the tinplate with an inhibitor while reducing the corrosion rate [21,31], which is consistent with the results of the potentiodynamic measurements. However, a further increase in the concentration > 0.6 g/L of the added inhibitor leads to a significant decrease in the semicircle radius in the Nyquist diagrams, which is reflected in a decrease in the inhibitory effect. The reason for this inhibitory behaviour could be that the movement of the inhibitor molecules is hindered due to the excessive concentration of inhibitor [49,50], which can lead to the formation of larger structures or complexes with dissolved metal ions [26]. In addition, above a certain inhibitor concentration, active sites are no longer available for adsorption, so that the inhibitor molecules can no longer adsorb to the metal substrate. However, corrosion continues to take place (i.e., the inhibition layer has not changed, but the corrosion activity increases with the immersion time). Table 5 shows that the inhibition effect decreases with increasing exposure time of the corrosive medium to the tinplate. The results show that after the addition of sage extract in 3% NaCl, the impedance response of the tinplate changed and the sage extract at a concentration of 0.6 g/L showed good inhibition properties. Obviously, the dynamics of the anodic process could have a destructive effect on the self-organised adsorption layer, reducing the possibility of a more

successful adsorption of inhibitor molecules. The formation of a protective layer exhibiting protective properties against the corrosion of tinplate in a 3% sodium chloride solution can be attributed to the high content of polyphenolic compounds in the sage extract formed on the tinplate. This was confirmed by ATR-FTIR analysis (Figure S1), which in turn shows that sage extract at a concentration of 0.6 g/L has good inhibition properties. The polyphenolic compounds contained in sage extract, such as rosmarinic acid, salvianolic K-acid, epicatechin and luteolin-7-*O*-glucuronide, could also play an important role in suppressing the corrosion process. Fang et al. [44] concluded that seven aromatic compounds derived from plants, including caffeic acid and gallic acid, act as effective corrosion inhibitors in electrolytes with different pH values. Catechins and flavan-3-ols in peach pomace extract and catechins in green tea have protective effects on steel [19,26] in sodium chloride solution. For example, polyphenolic compounds in (*Pistacia terebinthus* L.) [29], *Malva sylvestris* [47], ginger [21] and olive leaf extracts [31,44] act as effective corrosion inhibitors in a neutral medium, inhibiting the anodic reaction of the corrosion process to a greater extent and forming a protective film on the metal surface [26]. Fekri et al. [30] reported a turnip peel extract to be an effective corrosion inhibitor for copper in 3.5% NaCl. Today's global experience shows that there are several main areas for the development of innovative technologies to improve the corrosion resistance of metal surfaces using corrosion inhibitors based on polyphenolic compounds [50]. One of the major challenges in using organic extracts as corrosion inhibitors is their limited solubility in polar solvents, which enables the application of innovative extraction methods such as high hydrostatic pressure extraction. This innovative HHP technique with high efficiency and low operating temperature is suitable for the extraction of polyphenols with the main target compounds such as rosmarinic acid, catechins and glycosides from sage, making sage extract suitable as a corrosion inhibitor. The electrochemical behaviour of tinplate in 3% sodium chloride solution with the addition of a concentration of sage extract (0.6 g/L) at 25 °C after 2.5 h showed the high inhibition efficiency (86.3%) of tinplate, which was consistent with the results of potentiodynamic measurements (93%).

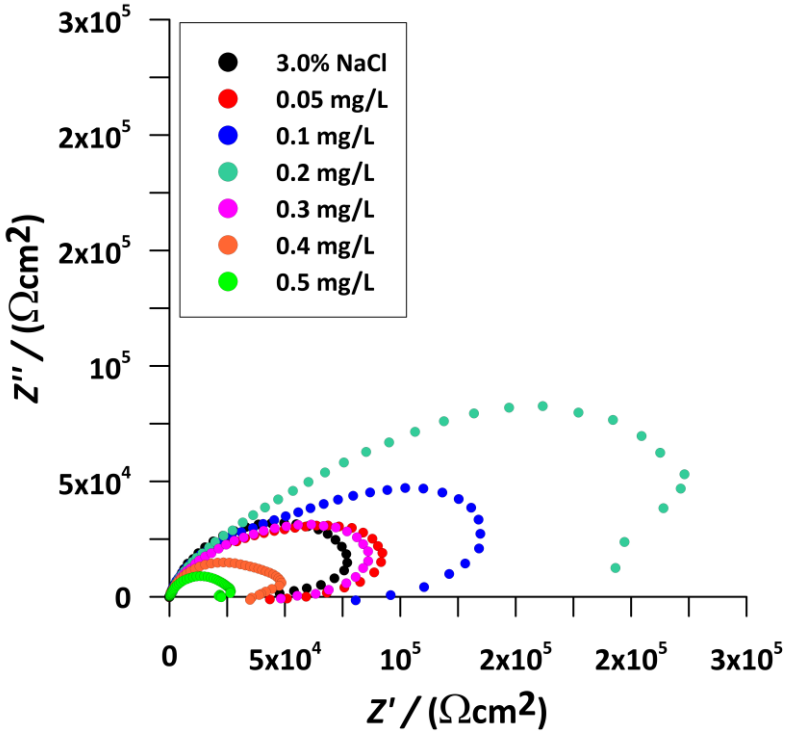

Figure 8. Nyquist plots for tinplate in 3%-NaCl solution at 25 °C with the addition of different concentrations of sage extract.

Table 6. Polarisation resistance and inhibition efficiency for tinplate in 3% NaCl with and without inhibitor addition after different immersion time.

γ(Sage Extract)/(g/L)	R_p/(kΩcm^2)					η/(%)				
t(Immersion Time)/(h)	2.5	5	8	10	12	2.5	5	8	10	12
Blank	25.5	11.6	7.4	5.9	4.7	-	-	-	-	-
0.2	40.1	13.4	8.2	6.2	5.4	36.3	15.2	10.2	3.5	12.5
0.4	73.6	23.3	9.3	12.0	10.3	65.3	51.2	20.8	50.6	53.8
0.6	164.3	87.2	43.8	32.2	26.5	86.3	86.9	83.1	90.3	82.0
0.8	75.4	12.5	12.1	12.3	12.8	66.1	8.8	39.0	51.8	62.7
1.0	53.3	13.1	12.0	10.0	11.5	52.1	12.9	38.3	40.4	58.6
2.0	74.2	17.6	11.7	7.8	5.8	65.6	35.2	36.8	23.9	17.3

R_p = polarisation resistance, η = inhibition efficiency.

4. Conclusions

The results showed that HHP is a fast non-thermal extraction technique suitable for polyphenol extraction from sage, offering high reproducibility in a short time with simplified handling, lower solvent consumption and lower energy consumption. The best results for the polyphenols were obtained under the HHP conditions of the test using ethanol at a concentration of 30% at an extraction temperature of 60 °C and an extraction pressure of 600 MPa, for 5 min. In all sage extracts tested, rosmarinic acid was the predominant phenolic acid, followed by salvianolic K-acid, epicatechin and luteolin-7-O-glucuronide. The results of this investigation can help to find possible ecological alternatives to the extraction of polyphenols from sage, improve the use of environmentally friendly solvents and energy-efficient technologies, and promote the benefits of HHP for extraction purposes and the further application of sage extract as an effective corrosion inhibitor for tinplate. The electrochemical behaviour of tinplate in 3%-sodium chloride solution with the addition of a concentration of sage extract (0.6 g/L) at 25 °C after 2.5 h showed that the inhibition effect increased to 86.3%, which is consistent with the results of potentiodynamic measurements (93%), while the sage extract showed a mixed inhibition effect. Electrochemical impedance spectroscopy and FTIR analysis demonstrate that the inhibitory effect is due to the formation of a protective layer at the interface between the tinplate and the electrolyte. The inhibitory effect of the sage extract was attributed to the adsorption of organic compounds, such as polyphenolic compounds, on the tinplate surface, blocking the active corrosion sites. Sage extract in sodium chloride solution can be used in the food packaging industry as an effective mixed corrosion inhibitor.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/separations11050158/s1, Figure S1: FTIR spectra of the tinplate surface after immersion for 12 h at 25 °C in 3% NaCl with 0.6 g/L sage extract inhibitor.

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