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Effect of polka raspberry *(Rubus idaeus* L.*)* extract on corrosion inhibition of bronze

ABSTRACT

Plant extracts contain a large number of organic compounds, and one of the large groups of compounds present are phenolic compounds. Researchers have shown that a certain number of these compounds can be used as effective metal corrosion inhibitors. Plant extracts of raspberries (leaves, flowers, and fruit) were obtained by ultrasonic extraction using 96% ethanol as a solvent. The UV/Vis spectrophotometric method was used to determine the content of total phenols in plant extracts. Phenolic acids and flavonoids in plant extracts were separated and quantified using the HPLC method. Tafel extrapolation was used for electrochemical characteristics. The corrosion characteristics and behavior of bronze in 3% NaCl solution, with and without the presence of plant extracts were investigated. The content of total phenols in leaves was found to be 107.14±3.63 mg/g in flowers 148.99±9.02 mg/g and in fruits was 8.75±0.61 mg/g. Leaf extract in a concentration of 0.04828 g/L according to the Tafel extrapolation method provides the best protection for bronze in a 3% NaCl solution. The same concentration in the case of flower and fruit extracts proved to be the most favorable.

Keywords: *raspberry extract, ultrasound, corrosion inhibition, bronze*

1. INTRODUCTION

There are many effective corrosion inhibitors used in industry, but the use of most of them has been banned today due to their toxicity. The development of new environmentally friendly corrosion inhibitors is focused on finding natural, non-toxic, biodegradable molecules to protect the environment. Therefore, nowdays, intensive work is being done to find such compounds.

Reducing or completely stopping the use of a number of highly effective corrosion inhibitors has arisen from the increasing concern for environmental preservation, which has resulted in stricter regulations regarding the use of chemicals that can have a harmful impact on the environment [1].

Chromates were removed from the list of technically applicable corrosion inhibitors due to their toxicity. The use of polyphosphates was removed because it was found that their release into natural waterways could lead to eutrophication-excessive algae growth [2]. Benzotriazoles and their derivatives were effective corrosion inhibitors for copper. The use of highly effective inhibitors was primarily eliminated because of their toxicity, so even today, these inhibitors are being replaced by new environmentally friendly corrosion inhibitors.

A large number of studies have been focused on natural organic compounds that can be obtained from plant material. By extracting plant material or synthesizing it, natural antioxidants can be obtained that are cheap, available, and renewable. Studies have shown that a certain number of these compounds can be applied as efficient metal corrosion inhibitors [3,4], as well as efficient copper corrosion inhibitors [5-8], known as green corrosion inhibitors [9].

Plant extracts contain a large number of organic compounds, and one of many are phenolic compounds. Phenols are aromatic compounds with one or more hydroxyl groups (-OH) attached to a carbon atom of a benzene ring (C_6H_5OH). They play an important role in the formation of color, bitterness, astringency, and aroma of fruits and fruit products. A large number of studies have shown

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that some of them possess anticancer and antimutagenic properties as well as antioxidant activity. Some phenolic compounds form chelate complexes with metals [10].

Polka is a very important variety of raspberry. It was first introduced in Poland in 2001. It is a everbearing raspberry, created by crossing the *Autumn Bliss*, *Lloyd George*, and *Rubus crataegifolius* varieties. Polka raspberries have small to medium, cone-shaped, light red fruits, very attractive in appearance and of excellent and better quality than most biennial raspberry varieties, with high yields, greater resistance to disease and weather conditions. Due to all these qualities, Polka raspberries quickly spread to production plantations in Poland, Serbia, Bosnia and Herzegovina, Ukraine, and others [11].

Extraction is an important step in isolating bioactive compounds from plant material. Extraction is defined as a mass transfer process, during which solid materials in contact with a solvent undergo a mass transfer, and the soluble components are transferred from the solid matrix to the liquid phase [12].

2. EXPERIMENTAL PART

2.1. Plant extract preparation

The aim of this paper is to investigate the effectiveness of environmentally friendly inhibitors of bronze corrosion. The corrosion resistance of bronze in a 3% NaCl solution was tested with and without plant extracts present.

In this research, also the influence of extract concentration on inhibition efficiency will be investigated.

The cultivated raspberry variety Polka from the Maglaj area was used as the plant material. Extracts were made from raspberry leaves, fruits, and flowers.

Bronze with a chemical composition of Cu 86 %, Zn 0.16 %, Sn 12.2 %, Fe <0.01 %, and Pb 0.49 % was used to test the corrosion properties of the selected plant extracts in a 3 % NaCl solution. [13-15]. The chemical composition of bronze was tested at the "Kemal Kapetanović" Institute in Zenica.

Extractions were carried out in an ultrasonic bath under defined conditions: frequency (20-40 kHz), power (250-500 W), temperature (40 ºC), and extraction time (30 min) [16]. Ethanol (96 %) was used as the solvent. After treatment, the extracts were filtered and evaporated to dryness. The extracts obtained in this way were stored in dark bottles in the refrigerator at a temperature of +4 °C.

2.2. Total phenolic content

Solutions of leaf, flower, and fruit extracts were prepared: 100 mg of dry extract was dissolved in ethanol in a volumetric flask to 10 mL (γ=10 g/L). The Folin-Ciocalteu method was used to spectrophotometrically determine the total phenol content [17].

The reaction mixture was prepared by mixing 40 µL (in some cases 100 µL) of extract (concentration 10 g/L), 500 µL of Folin-Ciocalteu reagent (1:2), 1500 µL of 20% Na_2CO_3 in a 10 mL volumetric flask, and then diluting with distilled water. At the same time, a blank test was prepared with distilled water instead of the extract. After 45 minutes, absorbance is measured at a wavelength of 725 nm.

Based on the measured absorbance, the concentration (mg/mL) of phenolic compounds was calculated, and then the content of total phenolic compounds in the extract was expressed as gallic acid equivalent (mg GAE/g dry extract) which was used as a standard.

2.3. HPLC analysis

In raspberry leaf, flower, and fruit extracts of the Polka variety, phenolic acids (gallic acid) and flavonoids (rutin and quercetin) were identified and quantified using reverse phases on the Shimadzu Prominence (modular high-performance liquid chromatography, HPLC), which was equipped with a UV/Vis detector, mobile phase degasser, pump, autosampler, and column oven.

2.4. Electrochemical investigations

The electrochemical cell for electrochemical tests has three electrodes. A carbon electrode is used as an auxiliary electrode, and a saturated calomel electrode (SCE) with a potential of 0.2415 V is used as a reference electrode. All potentials given in the paper are relative to the SCE electrode. The working electrode is in the form of a disk. Before each measurement, the working surface of the tested material was mechanically processed by sanding with sandpaper of different fineness, degreased in ethanol and washed with distilled water. The tests were performed in a 3% NaCl solution without and with the addition of plant extract.

3. RESULTS AND DISSCUSION

Discussion: The extraction yield Y, expressed as a percentage, was calculated based on the dry weight of the plant material (mS) and the extract (mE) after solvent separation, according to the following formula (Milenković Anđelković, 2016):

$$Y\ (\%) = (mE\ /\ mS) \times 100 \tag{1}$$

Table 1. Extraction Yield of plant material

Tabela 1. Prinos biljnih ekstrakata

Plant extract	Mass of plant (g)	Mass of extract (g)	Yild (%)
ULPM	15.16	1.34	8.84
UCPM	23.95	1.48	6.18
UPPM	99.85	5.48	5.49

The analyzed samples are marked as follows: leaf extract (ULPM), flower extract (UCPM), and fruit extract (UPPM).

Plant extracts were prepared using ultrasonic extraction. The results in Table 1 show that the highest yield was for the leaf (8.84%), and the lowest for the fruit extract (5.49%).

3.1.Total fenolic content

The results obtained in this work, in Table 2, show that extracts from leaves, flowers and fruits raspberries (*Rubus idaeus* L.) of the Polka variety contain significant amounts of total phenols.

Table 2. Total phenolic content

Tabela 2. Sadržaj ukupnih fenola

Plant extract	Total phenolic content (mg GAE/g extract)
ULPM	107.14±3.63
UCPM	148.99±9.02
UPPM	8.75±0.61

According to Pavlović et al. [18], the highest content of total phenols was detected in *R. idaeus* ˝Meeker˝ (144.20±1.58 mg GAE/g plant dry weight) and ˝Willamette˝ (143.38±4.68 mg GAE/g dry weight of the plant), while the lowest content of total phenols was recorded in the variety ˝Tulameen˝ (84.64±2.05 mg GAE/g dry weight of the plant).

The content of total phenols determined by the Folin-Ciocalte method does not provide a true picture of the quantity and quality of phenolic compounds in the investigated plant extracts. Various phenolics compounds have different responses to the Folin-Ciocalteu reagent. According to Vinčić [19], it is possible the presence of interfering compounds (sugars, aromatic amines, sulfur dioxide, vitamin C, organic acids, Fe(**II**) and other substances that are not of polyphenolic origin), affect unrealistic increase in results.

3.2. HPLC analysis

To determine the concentration of rutin, quercetin, and gallic acid in the tested raspberry leaf, flower, and fruit extracts, an HPLC method was used. The results of HPLC analysis of Polka raspberry leaf, flower, and fruit extracts from the Maglaj area are shown in Table 3. All experiments were repeated three times. The values are expressed as mean ± standard deviation.

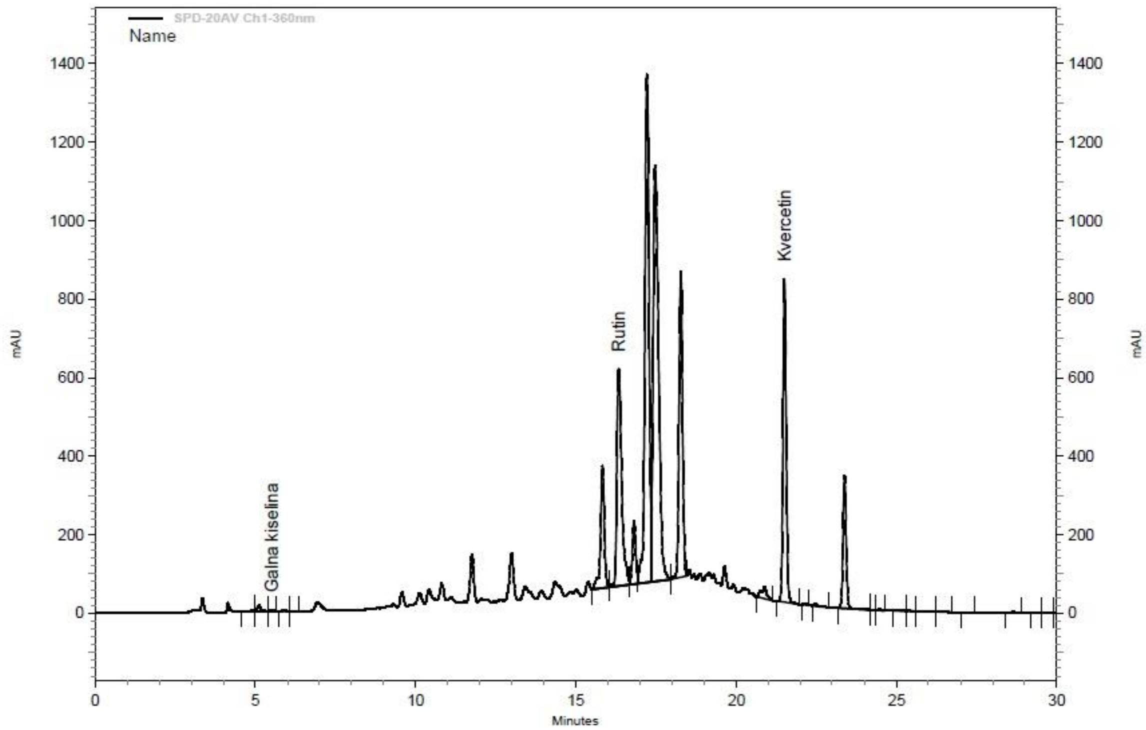

Figure 1. HPLC chromatograms of Polka Maglaj leaf: gallic acid, rutin, quercetin

Slika 1. HPLC hromatogrami lista Polke Maglaj: galna kiselina, rutin, kvercetin

Table 3. Results of HPLC analysis of raspberry Polka Maglaj leaf, flower, and fruit extracts obtained by ultrasound-assisted extraction, for rutin, quercetin, and gallic acid

Tabela 3. Rezultati HPLC analize ekstrakata lista, cvijeta i ploda maline sorte Polka Maglaj dobijenih ultrazvučnom ekstrakcijom, za rutin, kvercetin i galnu kiselinu

Plant extract	Rutin, γ (μg/mL)	Quercetin,γ (μg/mL)	Gallic acid, γ (μg/mL)
ULPM	3.77±0.06	4.83±0.01	124.57±52.92
UCPM	29.88±21.22	5.59±1.29	118.96±26.87
UPPM	0.59±0.07	0.20±0.01	20.78±1.70

Based on the calibration curves of standard solutions of phenolic compounds and the obtained chromatograms, the concentrations of identified phenolic acids (gallic acid) and flavonoids (rutin, and quercetin) were calculated. Results are shown in Table 3.

3.3. Electrochemical investigations

The potentiodynamic polarization method is used to measure the change in potential of the working electrode from the open-circuit potential in the cathodic and anodic directions in a range of ± 250 mV with a scan rate of 0.5 mV/s [18]. The experimentally measured values of current density, *i,* and potential, E, are shown in semi-logarithmic form as E - log(*i*).

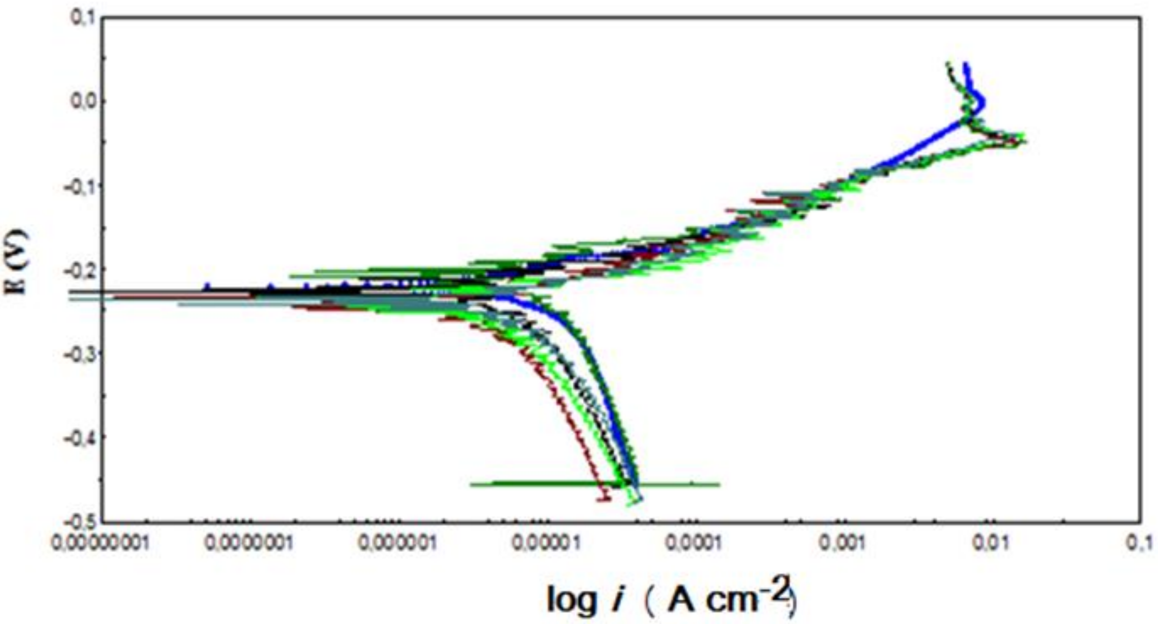

Figure 2. Anodic and cathodic polarization curves of bronze in 3% NaCl with and without the addition of Polka Maglaj leaf extract at different concentrations: ----3% NaCl; ----3% NaCl+0.01612 gdm^{-3}; ----3% NaCl+0.03221gdm^{-3}; ----3% NaCl+0.04828 gdm^{-3}; ---- 3% NaCl+0.06432 gdm^{-3}; ----3% NaCl+0.08033gdm^{-3}

Slika 2. Anodne i katodne krivulje polarizacije bronze u 3% NaCl bez i uz dodatak ekstrakta od lista Polke Maglaj u različitim koncentracijama: ----3% NaCl; ----3% NaCl+0.01612 gdm^{-3}; ----3% NaCl+0.03221gdm^{-3}; ----3% NaCl+0.04828 gdm^{-3}; ---- 3% NaCl+0.06432 gdm^{-3}; ----3% NaCl+0.08033gdm^{-3}

The other Tafel curves were recorded in a same conditions. In Table 4 are given summarized results for corrosion parameters for all extracts.

The effectiveness of the inhibitor was calculated according to the equation:

$$\eta = \frac{i - i_{inh}}{i} \times 100 \quad (2)$$

where *i* is the current density without the inhibitor, and i_{inh} is the current density with the inhibitor. Based on this equation, the inhibitor effectiveness was calculated at a concentration of 0.04828 g/L, resulting in 78.86% effectiveness for the leaf, 95.17% for the flower, and 13.44% for the fruit.

Table 4. Corrosion parameters determined by the Tafel extrapolation method for bronze in 3% NaCl with and without the addition of leaf (ULPM), flower (UCPM), and fruit (UPPM) extracts at different concentrations are presented

Tabela 4. Korozijski parametri određeni metodom Tafelove ekstrapolacije bronze u 3% NaCl bez i uz dodatak ekstrakata lista (ULPM), cvijeta (UCPM) i ploda (UPPM) u različitim koncentracijama

γ (g/L)	ULPM				UCPM				UPPM			
	E(mV)	i(mAcm^{-2})	βc(mV	βa(mV	E(mV)	i(mAcm^{-2})	βc(mV)	βa(mV)	E(mV)	i(mAcm-2)	βc(mV)	βa(mV)
0	-227.3	3.76	-544.6	503.8	-263.3	2.69	-415.9	507.0	-236.8	1.19	-193.8	51.8
0.01612	-246.5	2.45	-396.3	387.2	-267.4	4.32	-626.4	685.0	-244.8	1.15	-151.4	52.9
0.03221	-264.8	3.79	-518.2	534.2	-275.8	0.52	-99.3	102.4	-230.9	1.59	-190.6	50.2
0.04828	-258.0	0.87	-131.2	134.5	-266.8	0.13	-36.0	35.9	-232.2	1.03	-155.4	47.8
0.06432	-247.8	5.17	-568.3	620.9	-267.6	3.48	-452.5	584.4	-220.6	2.25	-168.2	581.5
0.08033	-255.5	3.38	-457.5	477.4	-283.2	0.69	-129.1	119.9	-241.7	2.05	-201.5	55.2

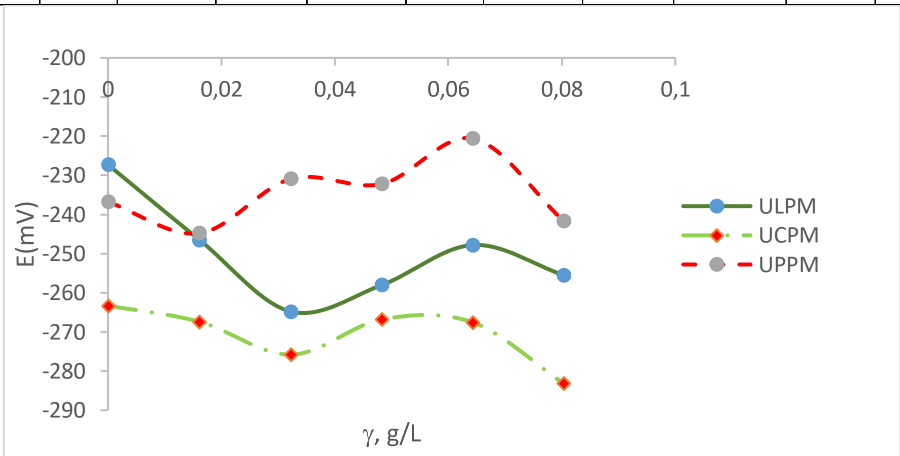

Figure 3. The dependence of the corrosion potential on the concentration of leaf, flower, and fruit extracts obtained by ultrasonic extraction on the corrosion characteristics of bronze

Slika 3. Zavisnost korozionog potencijala od koncentracije ekstrakata lista, cvijeta i ploda, dobivenih metodom ultrazvučne ekstrakcije, na korozione karakteristike bronze

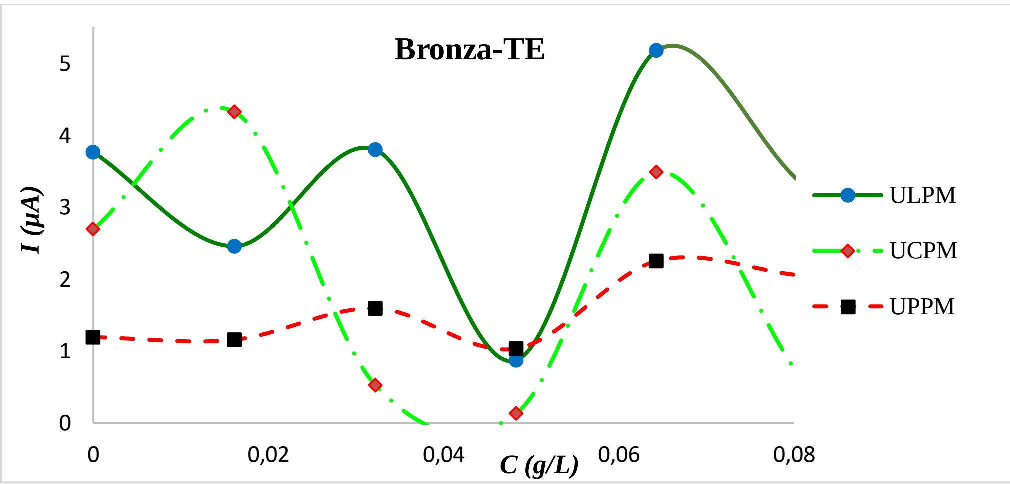

Figure 4. The dependence of the corrosion current on the concentration of leaf, flower, and fruit extracts obtained by ultrasonic extraction on the corrosion characteristics of bronze

Slika 4. Zavisnost struje korozije od koncentracije ekstrakata lista, cvijeta i ploda, dobivenih metodom ultrazvučne ekstrakcije, na korozione karakteristike bronze

Based on the obtained results, it can be observed that a concentration of 0.04828 g/L leads to a shift in corrosion potential towards lower values, indicating the best inhibitory effect. The corrosion current density follows this dependence. Figures 3 and 4 show E_{corr} (corrosion potential) and current densities obtained from Tafel extrapolations, depending on the concentrations of individual extracts, as well as their origins.

The corrosion parameters shown in the Tables 4 and 6 indicate that the concentration of 0.04828 g/L of the extract provides the best protection for bronze in 3% NaCl, as determined by both the Tafel extrapolation and the electrochemical impedance spectroscopy methods.

The contribution of individual components of the extract to the corrosion characteristics of bronze was also examined. The contribution of inhibitory substances whose concentration gave the best inhibition results was taken, which was 0.0322 g/L.

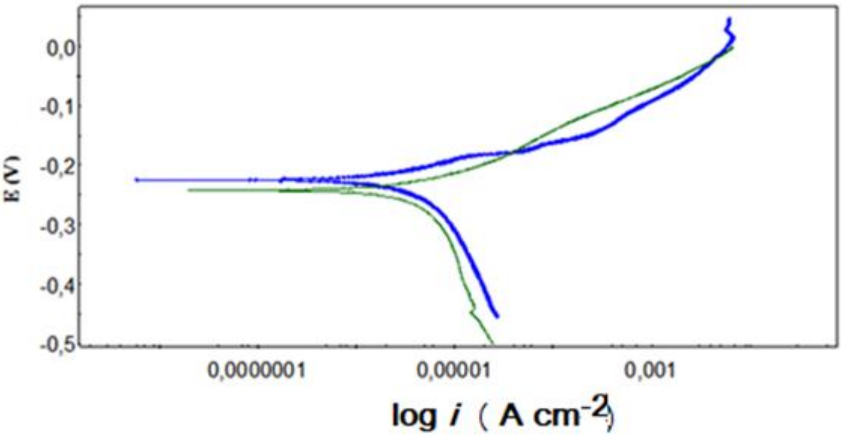

Figure 5. Anodic and cathodic polarization curves of bronze in 3% NaCl with and without the addition of a standard solution of gallic acid: 3% NaCl; 3% NaCl+0.03221g/L

Slika 5. Anodne i katodne krivulje polarizacije bronze u 3% NaCl bez i uz dodatak standardne otopine galne kiseline: 3% NaCl; 3% NaCl+0.03221g/L

Table 5. shows the influence of other substances expressed through corrosion parameters obtained by Tafel extrapolation.

Table 5. Corrosion parameters determined by the Tafel extrapolation method for bronze in 3% NaCl with and without the addition of a standard solution of gallic acid, quercetin, and rutin.

Tabela 5. Korozijski parametri određeni metodom Tafelove ekstrapolacije bronze u 3% NaCl bez i uz dodatak standardne otopine galne kiseline, kvercetina i rutina

Standard, γ (g/L)		**E, (mV)**	**I, (μA)**	**Bc,(mV)**	**Ba, (mV)**
Gallic acid	0	-225.550	6.903	415.781	83.000
	0.03221	-242.444	6.206	480.388	77.904
Quercetin	0	-220.063	11.48	423.491	69.768
	0.03221	-224.405	6.116	465.024	69.847
Rutin	0	-231.203	15.43	522.456	95.650
	0.03221	-238.475	4.871	564.412	83.071

The parameters presented in the Table 5. and the figure 5 indicate that all of these components reduce the corrosion rate of bronze in a 3% NaCl solution.

The electrochemical impedance spectroscopy (EIS) method was used to determine the kinetic parameters of the electrochemical reaction of bronze corrosion in a 3% NaCl solution with and without plant extracts [21].

The electrochemical cell can be represented by an electrical model. The electrochemical reaction that occurs on the electrode surface is analogous to an electrical circuit consisting of a resistor and a capacitor. Figure 6 shows the equivalent circuit diagram of a simple electrochemical cell.

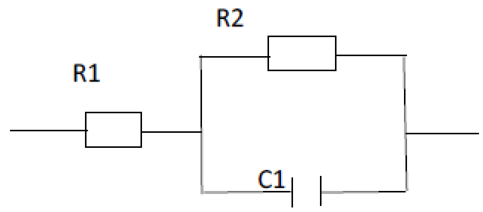

Figure 6. Equivalent circuit diagram of a simple electrochemical cell

Slika 6. Shema ekvivalentnog kruga jednostavne elektrohemijske ćelije

Results obtained by electrochemical impedance spectroscopy (EIS) can be presented using an equivalent electrical circuit (Figure 6) where R_1 represents the electrolyte resistance, R_2 represents the resistance of the oxide film or inhibitor, and C1 represents the capacitance.

Figure 7. shows the Nyquist plots of the effect of different concentrations of raspberry leaf extract on the corrosion resistance of bronze in a 3% NaCl solution.

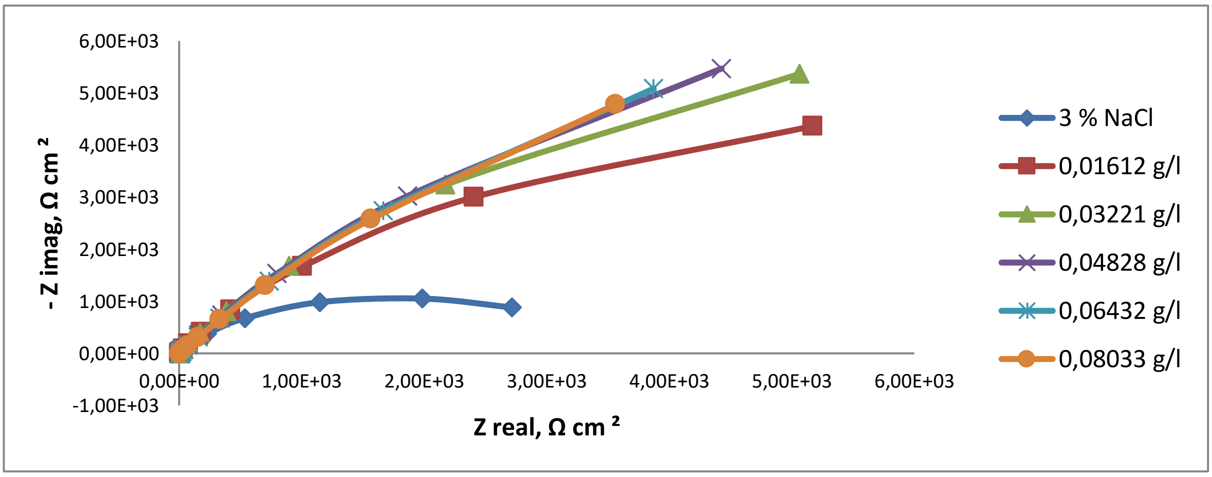

Figure 7. Nyquist plots for bronze in 3% NaCl with the addition of different concentrations of Polka Maglaj leaf extract.

Slika 7. Nyquist-ovi dijagrami za bronzu u 3% rastvoru NaCl i uz dodatak različitih koncentracija ekstrakta lista Polka Maglaj

The parameters obtained by the electrochemical impedance spectroscopy method for bronze in 3% NaCl with the addition of leaf (ULPM), flower (UCPM), and fruit (UPPM) extracts at different concentrations are shown in Table 6.

Table 6. Parameters obtained by the electrochemical impedance spectroscopy method for bronze in 3% NaCl with the addition of leaf, flower, and fruit extracts at different concentrations

Tabela 6. Parametri dobiveni metodom elektrohemijske impedansne spektroskopije bronze u 3% NaCl bez i uz uz dodatak ekstrakata lista, cvijeta i ploda u različitim koncentracijama

γ (g/L)	ULPM			UCPM			UPPM		
	R_1 (Ω)	R_2 (Ω)	C(F)	R_1 (Ω)	R_2 (Ω)	C(F)	R_1 (Ω)	R_2 (Ω)	C(F)
0	78.24	2644	$2.19\cdot10^{-4}$	32.5	778.9	$2.95\cdot10^{-4}$	69.88	2307	$2.82\cdot10^{-4}$
0.01612	138.4	8404	$2.78\cdot10^{-4}$	47.1	1583	$3.51\cdot10^{-4}$	113.3	6100	$3.37\cdot10^{-4}$
0.03221	139.8	10360	$2.96\cdot10^{-4}$	67.7	3197	$4.52\cdot10^{-4}$	127.6	11580	$3.31\cdot10^{-4}$
0.04828	135.2	10850	$3.36\cdot10^{-4}$	76.8	5080	$5.60\cdot10^{-4}$	129.8	12260	$3.22\cdot10^{-4}$
0.06432	129.8	10250	$3.80\cdot10^{-4}$	80.8	6126	$6.27\cdot10^{-4}$	132.3	13840	$3.18\cdot10^{-4}$
0.08033	126.3	9703	$4.09\cdot10^{-4}$	85.3	6306	$6.53\cdot10^{-4}$	136.0	14550	$3.16\cdot10^{-4}$

The summary graph shows the dependence of the corrosion parameters obtained by the electrochemical impedance spectroscopy method on the concentration of leaf, flower, and fruit extracts obtained by ultrasound-assisted extraction.

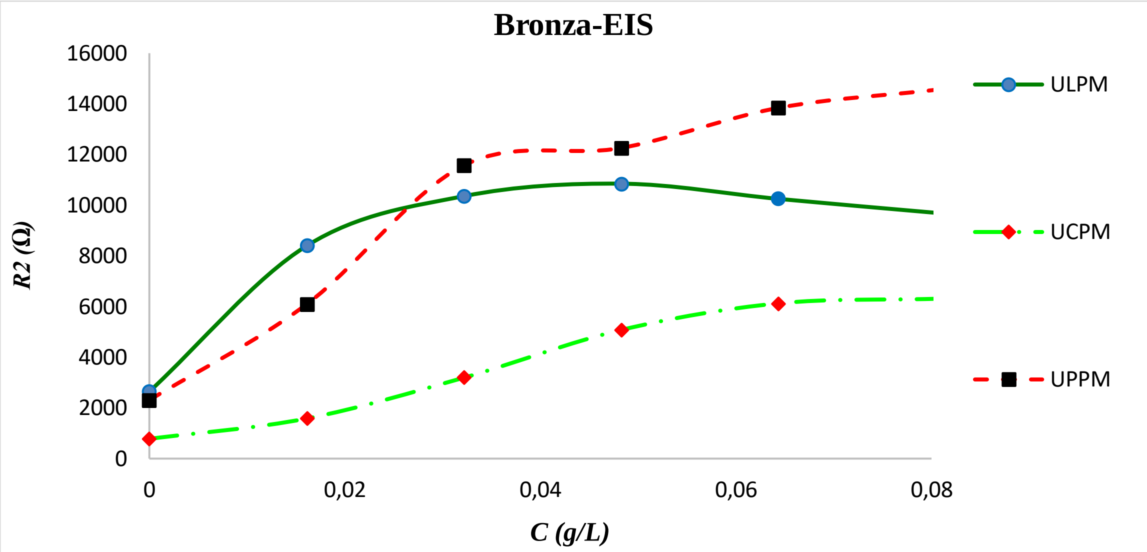

Figure 8. Summary diagram presenting the results of the influence of ultrasound-extracted extracts of leaves, flowers, and fruits on the corrosion characteristics of bronze using the EIS method.

Slika 8. Sumarni dijagram prezentacije rezultata uticaja ekstrakata lista, cvijeta i ploda, dobivenih metodom ultrazvučne ekstrakcije, na korozione karakteristike bronze metodom EIS

4. CONCLUSION

The results undoubtedly show that the content of total phenols can affect the inhibitory action and reduction of the bronze corrosion rate. The content of total phenols in leaves is 107.14±3.63, in flowers 148.99±9.02 and in fruits 8.75±0.61 mg GAE/g. Tafel extrapolation method revealed that the concentration of leaf, flower and fruit extract at 0.04828 g/L provides the best protection for bronze in 3% NaCl. By using EIS method, it can be concluded that raspberry leaf extracts obtained by ultrasonic extraction show inhibitory effect on bronze corrosion. The highest protection for bronze was provided by ULDM extract at a concentration of 0.06432 g/L with a resistance of 15030 Ω. By using the potentiodynamic polarization method with the extension of the anodic polarization curve, it was proven that there is a displacement of the linear Tafel, pseudopassive region and the region in which there is an increase in current density in the areas of lower current densities with the application of raspberry extracts.

These results confirm that in aggressive media, such as a 3% NaCl solution, the examined extracts of raspberry leaves, flowers and fruits of the Polka variety can be used to protect bronze from corrosion. Small differences in the efficiency of inhibition obtained by different methods can be explained by the fact that chemical methods give the average value of corrosion rate, while electrochemical methods give instantaneous values of corrosion rate [6].

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IZVOD

Uticaj ekstrakta polka maline (Rubus idaeus L.) na inhibiciju korozije bronze

Biljni ekstrakti sadrže veliki broj organskih spojeva, a jedni od mnogih su fenolni spojevi. Ispitivanja su pokazala da se određeni broj ovih spojeve može primijeniti kao efikasni inhibitori korozije metala. Biljni ekstrakti maline (list, cvijet i plod) dobijeni su metodom ultrazvučne ekstrakcije uz primjenu 96% etanola kao rastvarača. UV/Vis spektrofotometrijskom metodom je određen sadržaj ukupnih fenola u biljnim ekstraktima. HPLC-metodom su razdvojene i kvantificirane fenolne kiseline i flavonoidi u biljnim ekstraktima. Za elektrohemijske karakteristike korištena je Tafelova ekstrapolacija. Ispitivane su korozione karakteristike i ponašanje bronze u 3% rastvoru NaCl i u prisustvu biljnih ekstrakata. Sadržaj ukupnih fenola kod lista je 107.14±3.63, cvijeta 148.99±9.02 i ploda 8.75±0.61 mgGAE/g ekstrakta. Metodom Tafelove ekstrapolacije je utvrđeno da koncentracija ekstrakta lista, cvijeta i ploda od 0.04828 g/L pruža najbolju zaštitu bronzi u 3% NaCl.

Ključne riječi: *ekstrakt maline, ultrazvuk, inhibicija korozije, bronza*

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