

# Cucumis Melon Fibers Seed and Leaf Extracts as Natural Descaler and Corrosion Inhibitor in Acid and Brine Solution

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Received: 8 November 2022 Accepted: 5 January 2023

DOI: 10.30473/IJAC.2023.66039.1250

## Abstract

Effect of fibers of seeds inside Cucumis melon (Persian melon) extract (FE) and Cucumis melon leaves extract (LE) were investigated as descaler and inhibitor. In one part of this research, the effect of LE and FE for the solvent of Ca precipitates was investigated by gravimetric and electrochemical measurements. The decrease in the weight of calcium precipitate in the solution containing the extracts shows that the FE has the best effect as a solvent of Ca precipitate with 62% efficiency due to lower pH. A comparison of the hardness of the solutions after dissolving the calcium precipitate shows an increase of about 3 times the hardness of the solution containing FE compared to the blank. The second part is dedicated to the study of corrosion inhibition behavior of extracts on Cu corrosion in brine and acid solution by Tafel plot and electrochemical impedance spectroscopy (EIS). The solution containing the LE and FE shows higher polarization resistance ( $R_p$ ) (about 3.5 times) and lower corrosion current density than the blank solution. While the LE shows more positive potential than the FE. Based on the results, the FE can be used to formulate a low-cost and environmentally friendly descaling agent, and the LE can be used as a green corrosion inhibitor.

## Keywords

Cucumis melon; Fiber seed extract; Leaf extract; Descaler; Corrosion inhibitor

## 1. INTRODUCTION

Several methods are used to control the corrosion process and scale in the industry. Chemical antiscalants, inhibitors, and chemical descalers are usually used to control scale depositions and corrosion, a major concern in the industry. Antiscalants are a family of chemicals such as polyacrylic acids, carboxylic acids, polymaleic acids, organophosphates, polyphosphates, phosphonates, and anionic polymers, designed to inhibit the formation and precipitation of crystallized mineral salts that form scale [1]. A corrosion inhibitor reduces the corrosion rate by substantial pH variation, or oxygen and hydrogen sulfide scavengers, causing the removal of aggressive species from the solution. Mainly nitrogen, sulfur, oxygen-containing compounds like azoles, amines, thioureas, amino acids, and their derivatives are reported in the literature as corrosion inhibitors [2,3]. Descaling agents are typically acidic compounds such as hydrochloric acid, acetic acid, citric acid, phosphoric acid, sulfamic acid and also chelating agent such as ethylenediaminetetraacetic acid (EDTA) used to remove limescale or calcium precipitate from metal surfaces in contact with hot water [4]. Some of these compounds may be harmful to environment or toxic to humans so the environmentally friendly antiscalants, inhibitors and, descaler are needed.

Have established that the extract of some plants acts as a new type of green inhibitor for acidic corrosion of metal. The plant extracts such as *Musa paradisica* (Banana) peel [5], olive leaf extract [6], and *Nigella Sativa* seeds [7], are employed as a corrosion inhibitors. Polyacrylamide grafted with Okra mucilage was tested as a corrosion inhibitor for mild steel in acid media [8].

The scale inhibition properties of plant extracts have been investigated by different methods [9]. The properties of *Herniaria glabra* (*H. glabra*) towards  $\text{CaCO}_3$  formation were tested by using chronoamperometry and fast controlled precipitation methods [10]. Fig leaf were investigated as an environmentally friendly antiscalant for  $\text{CaCO}_3$  calcareous deposits on steel [11]. Carboxymethyl cellulose was investigated as a green antiscalant in the RO system by flux change measurement and membrane surface observation [12]. The use of natural extracts as descaling agents is less reported. The descaling properties of fermented whey on mild steel coupons were validated using both electrochemical and electron microscopy techniques [13].

The muskmelon (*Cucumis melon*) plant contained 6 saturated fatty acids that mostly include hexadecanoic acid, octadecanoic acid and, tetradecanoic acid [14]. Fatty acids are aliphatic carboxylic acids with varying hydrocarbon lengths at one end of the chain joined to the terminal

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carboxyl (-COOH) group at the other end. To the best of our knowledge, the use of leaf and fiber of seed inside of melon extract as descaler and inhibitor has not been reported. Sweet Melon peel extract as a corrosion inhibitor was evaluated by weight loss and potentiodynamic polarization methods [15]. The effect of cantaloupe juice and seed extracts was investigated on corrosion of Aluminium in acidic and alkaline solution using hydrogen evolution measurement [16].

In this article, the extracts taken from melon leaves (LE) and the fibers of the seeds (FE) inside were investigated as anti-scaling and corrosion inhibitors using gravimetric, hardness measurement and, electrochemical techniques.

## 2. EXPERIMENTAL

### 2.1. Materials

Distilled water and analytical reagent grade were used for preparing solutions. Ethylenediaminetetraacetic acid (EDTA) with indicator eriochrome black T and ammonia buffer (pH = 10) (200 ml water solution containing 11.7 gr  $\text{NH}_4\text{Cl}$  and 20 ml Ammonia) was used for the hardness test.  $\text{CaCl}_2$  brine solution was prepared to a concentration of 0.7 M NaCl, 0.0025 M  $\text{NaHCO}_3$ , 0.028 M  $\text{Na}_2\text{SO}_4$  and 0.01 M  $\text{CaCl}_2$  [6]. All reagents and  $\text{H}_2\text{SO}_4$  were purchased from Merck

### 2.2. Preparation of LE and FE extracts

For the preparation of stock solutions of Cucumis melon leaf (LE) and fibers of the seeds inside melon (FE) extract, a certain amount of leaves and the separated fibers from the seed, separately dried in an oven at 70 °C for 2 h and grinding to powdery form. A 30 g sample of the powder was refluxed in 500 mL ethanol for 2 h by a soxhlet extractor. The refluxed solution was filtered to remove any contamination. The precipitate remaining from the evaporation of the filtered solution (about 55%) was used for further work. The concentration of the stock solutions was expressed in terms of a milligram per liter (ppm).

### 2.3. Electrochemical measurements

Electrochemical corrosion tests and impedance spectroscopy (EIS) analysis were studied with a potentiostat/galvanostat model IVIUM made in Netherland. For corrosion and impedance measurements a three-electrode system was applied with Cu electrode as a working electrode, a platinum wire and saturated calomel electrode were used as counter and reference electrodes. The electrochemical corrosion measurement starts by the preparation of a Cu electrode. For the preparation of the Cu electrode, 1 cm<sup>2</sup> Cu sheets were soldered to copper wire and insulated with epoxy resin.

### 2.3. Scaling test

Scaling tests were performed by preparing very hard water.  $\text{CaCl}_2$  (3 g L<sup>-1</sup>) dissolved in the hard well water;  $\text{Na}_2\text{CO}_3$  was added (3 g L<sup>-1</sup>) and mixed thoroughly. The polished Cu electrodes were fixed to the bottom of the metal pot and 300 mL of prepared very hard water was poured into the pot. The solution was boiled until it evaporated completely, and the next portion of very hard water was added and boiled based on the same procedure. After the boiling procedure, the scaled Cu electrode was left to dry at room temperature for 48 h.

## 3. RESULTS AND DISCUSSION

### 3.1. Application of extracts as a corrosion inhibitor

In order to evaluate the ability of extracts as corrosion inhibitors, electrochemical measurements were performed in acidic ( $\text{H}_2\text{SO}_4$  0.5 M) and brine solutions in the presence and absence of LE and FE extracts. At first, the comparison of anti-corrosion properties of the LE and FE was evaluated by electrochemical impedance spectroscopy (EIS). In this method, alternating current signal with low amplitude. It is applied to an electrochemical cell (corrosion cell) and the response of the system is checked. Since EIS is a non-destructive technique due to the small amplitude of the excitation voltage, this analysis was performed first. The 1 cm<sup>2</sup> Cu electrode was immersed in an acidic solution for 10 min to attain a steady state open circuit potential (OCP) and then the impedance measurements were carried out at the OCP. Fig 1 shows the Nyquist plots of the Cu electrode in an acidic solution containing 50 ppm LE and FE. In the Nyquist plot, the real (Z') and imaginary (Z'') parts were measured in the frequency range of 100 kHz–100 mHz. The obtained curves were fitted with the electrochemical equivalent circuits given in the figure background. In an equivalent circuit,  $Q$  is a constant phase element (CPE) that is related to geometric origins such as surface heterogeneity or surface roughness [6]. The impedance " $Z_{\text{CPE}}$ " is composed of constant phase element  $Q$  and exponent " $n$ " as follows:

$$Z_{\text{CPE}} = 1/(j\omega)^n Q$$

where " $\omega$ " is the angular frequency and " $j$ " is the imaginary number ( $j = \sqrt{-1}$ ). Therefore the " $n$ " is known as the heterogeneity coefficient. In other words,  $Q$  corresponds to double layer capacitance for a non-homogenous system. When the electrode shows the capacitive behavior, the  $n$  takes a value from 0 to 1. Generally, the value of  $n$  decreases with increases in the heterogeneity of electrode surface. The decrease in  $Q$  values with the addition of extracts can be attributed to an increase in the

thickness of the electrical double layer [17]. The Equivalent circuit parameters after being fitted to the impedance curve are summarized in Table 1. The equivalent circuit also contains the elements corresponding to the solution resistance ( $R_1$ ), the resistance of double layer of the electrode surface ( $R_2$ ), and charge transfer resistance ( $R_3$ ). The data show that the value of  $R$  increased with the addition of 50 ppm extracts. LE extract shows a greater increase in  $R_3$ . The increase in  $R_3$  values is attributed to the formation of an insulating protective film at the metal/solution interface. Furthermore, the electrical double layer capacity  $Q$  is reduced due to inhibitor absorption.

**Table 1.** Nyquist data fit and Tafel analysis parameters for Cu electrode in  $H_2SO_4$  0.5 M in absence (blank) and presence of 50 ppm LE and FE

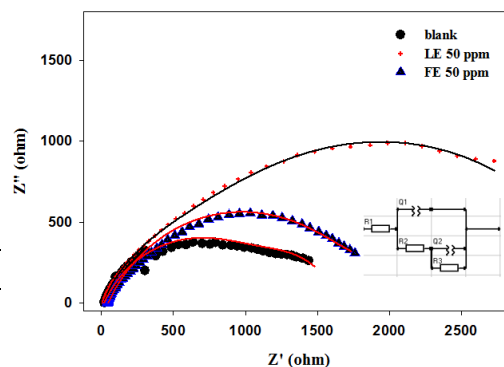
	blank	LE	FE
$R_1$ Ohm	13.7	18.9	45.3
$R_3$ Ohm	347	2276	564
$R_2$ Ohm	1249	1289	1820
$Q_1$ ( $Q_2$ ) $\times 10^{-6}$	91.4 (1836)	74.8 (222)	128 (1110)
$n_1$ ( $n_2$ )	0.7 (0.87)	0.76 (0.7)	0.70 (0.71)
$E_{corr}$ V	-0.0137	0.0097	-0.0264
$I_{corr}$ $\mu A/cm^2$	3.14	1.70	2.06
$R_p$ Ohm	3015	10440	10310
$\beta_a$ V/dec	0.05	0.13	0.16
$\beta_c$ V/dec	0.59	0.35	0.36
C. rate mm/y	0.58	0.31	0.21

The corrosion inhibition of LE and FE on the Cu electrode also was evaluated by Tafel plots in  $H_2SO_4$  0.5 M. A Tafel equation for anodic and cathodic reactions in a corrosion system was expressed with Butler-Wolmer equation [23] as follows:

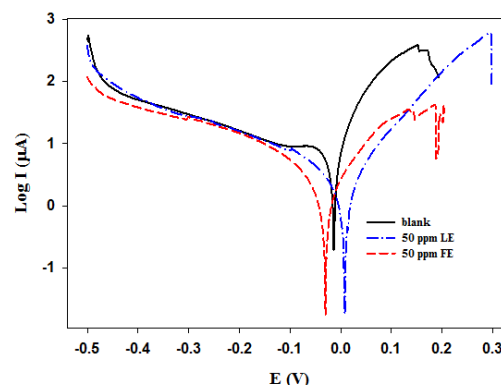
$$I = I_{corr} (e^{2.303(E-E_{corr})/\beta_a} - e^{-2.303(E-E_{corr})/\beta_c})$$

where  $I$  (A) and  $E$  (V) are current and potential of electrode,  $I_{corr}$  and  $E_{corr}$  are corrosion current and potential of electrode and  $\beta_a$  ( $RT/nF(1-\alpha)$ ),  $\beta_c$  ( $-RT/nF\alpha$ ) are anodic, cathodic Tafel constant. The  $\alpha$  is charge transfer coefficient for cathodic half-reaction, and  $(1-\alpha)$  is charge transfer coefficient for anodic half-reaction. The electrodes were then polarized cathodic to an anodic direction from the OCP at a scan rate of 1 mV/s. The potential and corrosion current densities were determined by extrapolating the linear portion of the anodic and cathodic curves. Fig 2 shows the comparison of Tafel curves of the electrode in presence of LE and FE in an acidic solution. It is observed from the figure that the curve in presence of LE is shifted to a more positive potential relative to FE and also their corrosion current ( $I_{corr}$ ) value is decreased compared to the blank solution. This shift of

corrosion potential ( $E_{corr}$ ) and decrease in  $I_{corr}$  indicates the higher corrosion resistance property of the solution as well as the increase in polarization resistance ( $R_p$ ). The increase in  $R_p$  in presence of an inhibitor suggests that a non-conducting physical barrier is formed at the metal electrolyte interface [8]. The kinetic parameters of the Tafel curves are given also in Table 1.



**Fig. 1.** Nyquist plot of the Cu electrode in  $H_2SO_4$  0.5M (blank) and 50 ppm of LE and FE



**Fig. 2.** Tafel plot of the Cu electrode in  $H_2SO_4$  0.5 M (blank) and 50 ppm of LE and FE

Fig 3 a shows the Nyquist diagram drawn for the copper electrode immersed in brine solution at different concentrations of LE extracts. In appearance, the diameter of the curves increases with increasing concentration and, the results of the fit diagrams are given according to the equivalent circuit given in Table 2. Solution resistance  $R_1$  and charge transfer resistance  $R_3$  increased with increasing the LE concentrations from 50 to 200 ppm. The Tafel curves of the Cu electrode in brine solution and different concentration of LE illustrate in Fig 3b. A decrease in current density and corrosion rate (C. rate) with an increase in LE concentration was observed as shown in Table 2.

Gravimetric experiments were carried out by immersing polished and weighted Cu sheets ( $1 \times 2.5$   $cm^2$ ) in plates containing 50 mL of the 1%  $H_2SO_4$  without and with different amounts of extract. The

plates were kept in an air and at room temperature at 24 h. The specimens were then removed from the solution, washed thoroughly with distilled water, dried and, weighed. The calculated weight is less given in Table 2. The inhibition efficiency (IE) was computed using the following formula:

$$IE\% = \frac{w_0 - w}{w_0} \times 100$$

where  $w$  and  $w_0$  are the weight loss with and without the addition of extract, respectively. The maximum inhibition efficiency observed is about 69 % in the concentration of 200 ppm LE, which also indicates that the inhibitor molecules have the capability of forming a compact adsorbed layer over the metal surface.

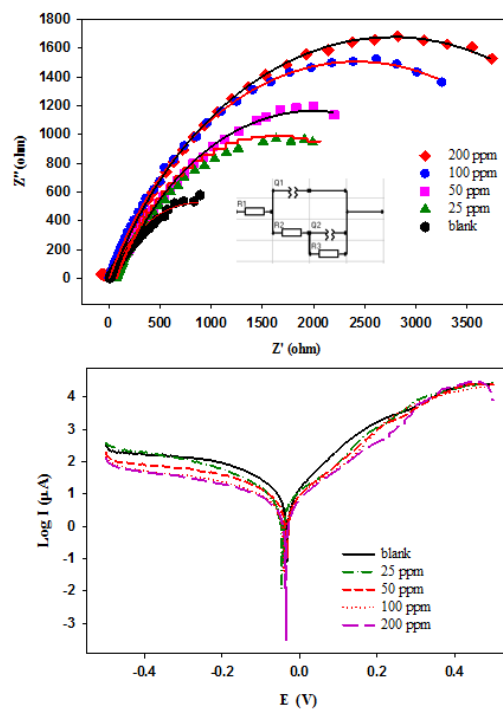
### 3.2. Application of LE and FE extracts as descaler

The effect of extracts as a descaling agent was investigated by three methods. The first method is based on the remaining weight of calcium deposits. The different concentrations of the extract were added to the calcium sediments and after 3 hours of stirring, the weight of the remaining sediments was measured. Based on the results obtained in Table 3, with the increase in the concentration of the extract, the residual weight was lower. A solution containing 1000 ppm of the FE showed about 55% efficiency for the dissolution of calcium deposits. In addition, a comparison was made between FE and LF as anti-scaling agents. Based on the results, the FE has pH = 5 and higher efficiency in dissolving calcium deposits

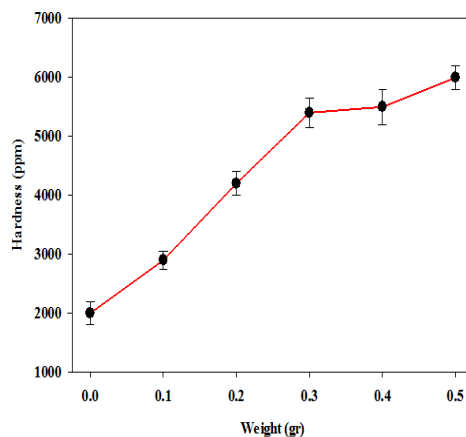
In the second method the soluble calcium was determined by titration with EDTA. Fig 4 shows the curves of the hardness of solutions resulting from the dissolution of different weights of calcium deposits in the same concentration of extract. The result showed that 2000 ppm of FE can dissolve calcium deposits up to 4000 ppm.

The electrochemical investigation of the descaling process of the scaled electrode was performed by immersing the scaled Cu electrode in 50 mL of 2000 ppm of LE, FE, H<sub>2</sub>SO<sub>4</sub> 1%, and distilled water (DW) for 2h. After immersion, electrodes were washed gently with distilled water and dried at room temperature for 24 h. Then electrochemical measurement, Tafel analysis and, EIS were performed for these electrodes in a brine solution. A clean Cu electrode was also tested for comparison. Fig 5 illustrates the OCP for all five electrodes. The OCP values depend on the surface electrode, electrolyte concentration, and other variables of the system. The OCP values descaled in DW have more negative than clean electrode, and other OCP takes place between them. The OCP increases because of the formation of a passivation layer [18]. Scaled electrodes achieve higher OCP as the fouling layer protects partially the surface from anodic corrosion. On the other hand;

immersions in FE nearly restore the original clean electrode, indicating the removal of the scale layer.



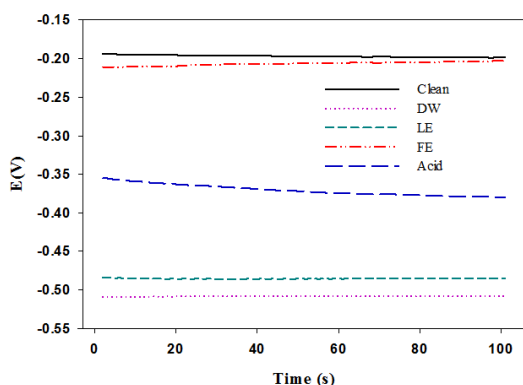
**Fig. 3** Nyquist (up) and Tafel (down) plot of the Cu electrode in H<sub>2</sub>SO<sub>4</sub> 0.5 M (blank) and different concentration of LE.



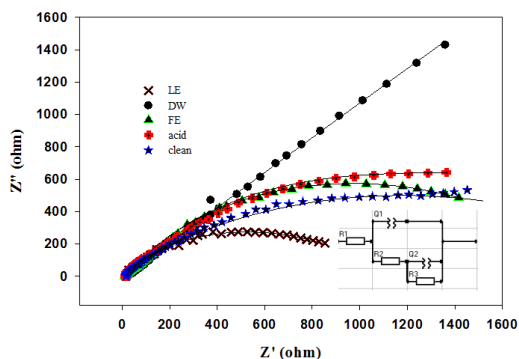
**Fig. 4.** The hardness of solutions resulting from the dissolution of different weights of calcium deposits in 2000 ppm FE

The electrodes were then polarized cathodic to an anodic direction from the OCP at a scan rate of 1 mV/s. Fig 6 show the Nyquist plot for five electrodes, and data fitting of curves base on the given equivalent circle, given in Table 4. The  $R_1$  represents the solution resistance. The  $R_2$  is the resistance associated with the layer of products formed during immersion which has the highest value for the descaled electrode in FE. The  $R_3$  corresponds to the charge transfer resistance, in other words, the same as  $R_p$  at the solution/metal

interface. The  $Q_1$  is associated with the capacitance of the same layer, and  $Q_2$  is associated with the double-layer capacitance. Increasing  $R_1$  and  $R_2$  is accompanied by decreasing in none ideal layer  $Q_1$ , and double layer capacitance  $Q_2$  values for descaled in DW due to the remaining the scale layer. The  $Q_2$  increased after the immersion in FE, due to the partial removal of the scale layer, by an increase in double layer capacitance [19]. The  $Q$  increased following the and the  $R$  decreases again following FE treatment such as electrode descaled in acid, confirming that extract especially FE effectively removes the scale layer.

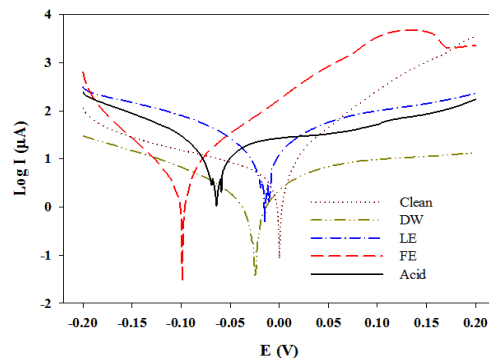


**Fig. 5.** The OCP of Cu electrode ( blank (clean), descaled in distilled water (DW), descaled in LE, descaled in FE and descaled in acid) in brine solution



**Fig. 6.** The Nyquist of Cu electrode (blank (clean), descaled in distilled water (DW), descaled in LE, descaled in FE and descaled in acid) in brine solution

Tafel plots of five electrodes in brine solution showed in Fig 7, and parameters are given in Table 4. The  $I_{\text{corr}}$  for descaled in DW electrode was lower than that of the clean specimen, which might be due to the blocking of the active regions of the Cu surface, hence calcium carbonate scale acting as corrosion inhibitor [20,21]. The  $E_{\text{corr}}$  for descaled in LE is near to DW but  $I_{\text{corr}}$  of its increased. The  $E_{\text{corr}}$  of descaling in FE is more negative and its  $I_{\text{corr}}$  is lower than electrode descaled in acid. Accordingly, FE acts as an anti-corrosion agent while it behaves similarly to acid as a scale remover.



**Fig. 7.** The Tafel plot of Cu electrode ( blank (clean), descaled in distilled water (DW), descaled in LE, descaled in FE and descaled in acid) in brine solution

#### 4. CONCLUSION

In the present work, a comparison was done between LE and FE as descaler and corrosion inhibitor. The ability of extracts as descaler was tested by gravimetric measurement, Hardness test and, electrochemical descaling test. 2000 ppm of FE has the ability to dissolve calcium deposits up to 4000 ppm. The corrosion inhibition of extracts was evaluated by Tafel extrapolation and EIS techniques. The amount of change in the electrochemical parameters, such as the increase in resistance and the decrease in the capacity of the electric double layer, shows that the inhibition process takes place through the absorption of the chemical compounds of the extracts on the metal surface. Overall, the results show that LE and FE can be used as corrosion inhibitors in both acidic and brine environments, moreover, FE can perform better as a low-cost and environmentally friendly scale remover.

#### Acknowledgments

This research was supported by the Nano Structured Coatings Institute of the Payame Noor University of Yazd.

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**Table 2 :** Corrosion and inhibition parameters obtained from Gravimetric (in H<sub>2</sub>SO<sub>4</sub> 0.5 M), Tafel and EIS (in brine solution) analysis for Cu electrode without and with various concentration of LE.

LE (ppm)	Gravimetric				Tafel extrapolation				EIS						
	weight less	IE%	E <sub>corr</sub> (V)	I <sub>corr</sub> $\mu$ A/cm <sup>2</sup>	R <sub>p</sub> Ohm	$\beta_a$ V/dec	$\beta_c$ V/dec	C. rate mm/y	IE%	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	Q <sub>1</sub> (Q <sub>2</sub> ) $\times 10^{-6}$	IE%	
0	0.052	-	-0.01	14.5	1992	0.09	0.23	1.49	-	12.5	982.7	751.2	628(1026)	-	
25	0.022	57.6	-0.04	7.36	3869	0.16	0.25	0.46	49.2	33.1	2211	1995	365(672)	62.3	
50	0.021	59.6	-0.02	6.99	4637	0.11	0.23	0.72	51.8	69.5	3354	2099	285(502)	64.2	
100	0.017	67.3	-0.03	5.39	6668	0.13	0.24	0.55	62.8	128.6	4258	1453	167(156)	48.3	
200	0.016	69.2	-0.02	4.63	7062	0.11	0.27	0.48	68.1	243.5	3053	2172	144(60)	65.4	

IE% = (I<sub>0</sub>-I)/I<sub>0</sub>×100, or = (R<sub>3</sub>-R<sub>3,0</sub>)/R<sub>3</sub>×100, I<sub>0</sub> and R<sub>3,0</sub> are the parameter values in the absence of LE extract

**Table 3: Concentration** compare residual weight of calcium precipitate in presence of LE and FE

Sample	Concentration (ppm)	pH	Residual precipitate (g)	Solubility%
LE	500	7.27	0.202	19.2
	1000	7.24	0.170	32
	2500	7.32	0.153	38.8
	5000	7.35	0.131	47.6
FE	500	5.41	0.141	43.6
	1000	5.29	0.113	54.8
	2500	5.26	0.101	59.6
	5000	5.10	0.095	62

**Table 4.** Electrochemical parameters from the Nyquist and Tafel plots of Cu electrode in brine solution.

Sample	Clean	Descaled in DW	Descaled in LE	Descaled in FE	Descaled in Acid
E <sub>corr</sub> V	-0.0085	-0.0786	-0.0151	-0.1108	-0.0642
I <sub>corr</sub> $\mu$ A/cm <sup>2</sup>	5.58	5.54	19.2	4.53	20.2
R <sub>p</sub> Ohm	3833	10590	1521	2832	2296
C. rate mm/y	0.58	0.57	2.3	0.47	2.1
R <sub>1</sub> Ohm	22.04	50.84	15.25	18.43	11.85
R <sub>3</sub> Ohm	2374	9199	354	505.6	1388
R <sub>2</sub> Ohm	1281	706.4	664.5	1332	780.3
Q <sub>1</sub> $\times 10^{-6}$	149	8.25	225	296	284
Q <sub>2</sub> $\times 10^{-6}$	1832	16.2	1411	199	346

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## عصاره فیبرهای دانه و برگ خربزه به عنوان رسوب زدا طبیعی و بازدارنده خوردگی در محلول اسید و نمک

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تاریخ دریافت: ۱۷ آبان ماه ۱۴۰۱ تاریخ پذیرش: ۱۵ دی ماه ۱۴۰۱

### چکیده

اثر عصاره فیبر دانه های داخل خربزه (FE) و عصاره برگ خربزه (LE) به عنوان رسوب زدا و بازدارنده بررسی شد. در بخشی از این تحقیق، اثر LE و FE به عنوان حلال رسوبات کلسیم با اندازه گیری های وزن سنجی و الکتروشیمیایی مورد بررسی قرار گرفت. کاهش وزن رسوب کلسیم در محلول حاوی عصاره نشان می دهد که FE به عنوان حلال رسوب کلسیم با راندمان ۶۲ درصد به دلیل pH پایین تر بهترین اثر را دارد. مقایسه سختی محلول ها پس از حل شدن رسوب کلسیم، افزایش حدود ۳ برابری سختی محلول حاوی FE را نسبت به بلانک نشان می دهد. بخش دوم به بررسی رفتار بازدارندگی خوردگی عصاره ها بر خوردگی مس در محلول اسید و نمک توسط نمودار تافل و طیف سنجی امپدانس الکتروشیمیایی (EIS) اختصاص دارد. محلول حاوی LE و FE مقاومت قطبش بالاتر (Rp) (حدود ۳٫۵ برابر) و چگالی جریان خوردگی کمتری نسبت به محلول شاهد دارد در حالیکه، LE پتانسیل مثبت تری نسبت به FE نشان می دهد. بر اساس نتایج حاصل، عصاره FE را می توان برای فرموله کردن یک عامل رسوب زدا کم هزینه و سازگار با محیط زیست و عصاره LE را به عنوان بازدارنده خوردگی سبز استفاده کرد.

### کلید واژه ها

خربزه، عصاره فیبر دانه، عصاره برگ، رسوب زدا، بازدارنده خوردگی