Study of the protective effect of green inhibitor extracted from seeds oil of *Cannabis sativa* L. against corrosion of brass 60Cu–40Zn in seawater medium

M. Damej,^{1*} M. Zouarhi,¹ M. Doubi,¹ M. Chellouli,² H. Benassaoui¹ and H. Erramli¹

¹Laboratory of organic chemistry, inorganic, electrochemistry, and environment, Faculty of Science, University Ibn Tofail, B.P 133, 1400, Kenitra, Morocco

²Laboratory of Materials, Catalysis and Valorisation of Natural Resources, URAC 24, Faculty of Sciences and Techniques Mohammeda, University Hassan II, Casablanca, Morocco

*E-mail: Damejmohamed1979@gmail.com

Abstract

Several methods are available to prevent or delay the corrosion of metallic materials by ensuring their protection when they are in contact with aggressive media (3% NaCl solution). The use of extracts becomes one of the best sources of ecological inhibitors because they guarantee high efficiency at low-cost. These types of inhibitors do not contain heavy metals or toxic compounds, and they are also biodegradable. In this work, we developed a corrosion inhibiting effect of formulation based on oil of Hemp (*Cannabis sativa* L.) seeds cultivated in North of Morocco labelled as “FCB”. We also determined the chemical composition of fatty acids and some physicochemical characteristics of *Cannabis sativa* L. seeds oil. In order to study the inhibitory effect of this formulation against corrosion of brass 60Cu–40Zn in 3% NaCl solution, we performed stationary and transitory electrochemical measurements. Indeed, the potentiodynamic measurements have shown that the FCB formulation acts as a mixed type inhibitor. The electrochemical impedance measurements confirm the results obtained by the polarization curves. The good protection effect of the FCB formulation is attributed to the formation of an inhibitor film on the brass metal surface. The Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-ray (EDX) analysis strongly supports the obtained results and confirms the formation of barrier film on the metal surface. The inhibition efficiency seems to be interpreted by training of a protective layer on the brass surface.

Keywords: inhibition effect, brass alloys, cannabis sativa plant, 3% NaCl solution, EIS, polarization measurement.

Received: July 2, 2020. Published: December 10, 2020

doi: [10.17675/2305-6894-2020-9-4-24](https://doi.org/10.17675/2305-6894-2020-9-4-24)

1. Introduction

Metal objects, especially those based on copper and its alloys, react spontaneously with their environment to reach their stable thermodynamic state, which is that of oxides. They are

subject to several transformations that make them vulnerable to degradation [1, 2]. Since a long time, we confronted with problems of corrosion; this makes it possible to look for solutions. Thus, the struggle against corrosion remains a scientific and technical discipline in perpetual evolution. Among the means of protection against corrosion, we quote corrosion inhibitors.

Furthermore, environmental protection and preservation of the health of professionals working in the industrial fields encourages us to develop non-toxic corrosion inhibitors, which respect the ecological spirit by taking care of component environmental. These inhibitors are known as “friendly corrosion inhibitors” [3, 4] or “green inhibitors”. Recently, many research works are moving towards the development of effective inhibitors based on biodegradable plant molecules [5–14].

Nowadays eco-friendly compounds, especially derived from plants extracts, draw the attention as effective inhibitors of corrosion protection of metallic substrates in corrosive solutions due to their biodegradability, non-toxic nature, economical aspects and ecological spirit.

In this study, we investigated the inhibitory effect of a novel formulation developed in our laboratory (LMEE), based on fatty acids extracted from *Cannabis sativa* L. seeds labelled “FCB” on the corrosion inhibition of brass 60Cu–40Zn in a simulated seawater medium.

The protective effect of this formulation was evaluated by cathodic and anodic polarization curves and by electrochemical impedance spectroscopy measurements. The surface analysis was performed by using SEM coupled with the elemental analysis EDX.

2. Material and Methods

2.1. Obtaining seeds and oil

2.1.1. Origin of the seeds

Seeds samples were taken from the dried, mature Hemp (*Cannabis sativa* L.) Plant at the harvest season from the year 2017 in Northern Morocco area.

Seeds are first slightly crushed in a mortar, and then homogeneous samples of 30 grams are then subjected to extractions process.

2.1.2. Extraction of the seeds oil

Extraction of the oil from finely ground seeds is carried out by Soxhlet apparatus. After filtration and evaporation of the cyclohexane solvent using a rotary evaporator, the obtained oil is stored in refrigerator at 4°C.

2.1.3. Physicochemical indices analysis

Refractive index was determined at 20°C with a universal refractometer; the density is determined using a pycnometer. Saponification index, peroxide acid and iodine indices are determined according to the French standard AFNOR collection.

2.2. Working electrode and corrosive medium

The copper alloy used in this work is called brass 60Cu–40Zn; this alloy has the following chemical composition: Cu (60.61%), Zn (39.19%), Al (0.12%) and Si (0.08%). The corrosive medium is a 3% NaCl solution prepared by the dissolution the 30 g of NaCl in 1 Litre of distilled water.

2.3. Corrosion inhibitor FCB

We used a corrosion inhibitor based on *Cannabis sativa* L. seed oil noted FCB; this product is patented in OMPIC Office Morocco [15].

2.4. Electrochemical techniques

The electrochemical measurements were performed in a three electrodes electrochemical cell: Consisting of a platinum electrode as an auxiliary electrode, saturated calomel electrode (SCE) as the reference, and brass alloy as the working electrode. These measurements were recorded using a potentiostat SP-200 controlled by EC-Lab software (data management and processing software developed by the company BioLogic Science Instruments). All of these curves (stationary and transitory) were obtained after 1 hour of immersion of the electrode in the electrolytic solution at the corrosion potential in an open circuit (E_{corr}).

2.5. Surface analysis by SEM coupled by EDX

The surface analysis of the 60Cu–40Zn brass was carried out using a scanning electron microscopy (SEM; JEOL, JSM-IT100) coupled with EDX instruments, after 24 h of immersion in the absence and in the presence of inhibitor.

3. Results and Discussion

3.1. Kinetics of extraction, physicochemical characteristics, and fatty acid profile

Oil yield of a plant depends on several factors, but in our study, a single factor influencing the yield has been studied, it is the duration of solid-liquid extraction using a Soxhlet extractor, the solvent used is cyclohexane. The results obtained by varying the extraction time are presented in Figure 1.

The Figure 1 shows that average oil yield progress a little between 10 and 12 hours of extraction. This result makes it possible to consider that almost of the oil is extracted after 10 hours with a maximum yield of about 40% which is close to that found in literature ranging between 35 and 38% [16, 17].

Figure 1. Evolution of yield as a function of extraction time.

This difference is probably due to the origin of the fruits, the period of harvest, the climatic conditions, and the temperature and to the water stress. All of these factors can affect the quantity of the extracted oil in seeds.

Physicochemical characteristics of the oil studied, in particular, density, and refractive, iodine, acid, peroxide, saponification indices are reported in the Table 1. These indices are directly related to the chemical nature of the extracted oil and also they determine their quality.

Table 1. Physicochemical indices of seed oil Hemp cultivated (*Cannabis sativa* L.).

Parameters	Values
Density at 20°C	0.902
Refractive index at 20°C	1.425
Acid number (mg of KOH/g)	5.3
Peroxide index (m_{eq} O ₂ /kg of oil)	12
Iodine index (g/100 g)	150
Saponification index (mg of KOH/g)	179.8

From the results presented in Table 1, we noted that the density and refractive index values measured for hemp seed oil are very similar to those obtained by Borhade *et al.* [18].

Acid and peroxide indices, which are two important parameters, provide information on the state of degradation of the oil.

Concerning the acidity, the value of acid number obtained in Table 2 remains higher than the value obtained in literature [18], this high acidity can be explained by a poor

conservation of oil, although, the fruit pulp contains a very active lipase which causes rapid acidification of oil during shelling.

About the oxidation, the value of peroxide index obtained (Table 2) is greater than that obtained in literature [18]. This means that the oil analysed is oxidized.

Generally, the saponification index and iodine number are significantly higher values, indicating that the hemp of oil is long-chain oil, suggesting that it contains higher percentage of unsaturated fatty acids.

The fatty acid composition expressed as a percentage by weight of the oil obtained from cannabis seeds has been established by the presence of the main fatty acids: linoleic acid C18:2 (51.3%), oleic acid C18:1 (20.3%), α -linolenic acid C18:3 (15.7%), palmitic acid C16:0 (7.9%), stearic acid C18:0 (2.7%), arachidic acid C20:0 (0.8%), arachidoleic acid C20:1 (0.4%) and palmitoleic acid. The oil contains major fatty acid contents C18:2, C18:1, C18:3, C16:0 and C18:0 in comparison with cannabis seeds oils of other origins described in the literature [17, 19].

Furthermore, the balance sheet of polyunsaturated, monounsaturated and saturated fatty acids for this oil shows values of 67%, 20.9% and 11.5% or a ratio of 67:21:11.

3.3. Potentiodynamic measurements

This method consists in imposing a potential variation between working electrode and reference electrode and recording the variation of intensity of current between working electrode and auxiliary electrode.

Cathodic and anodic polarization curves were plotted after 1 hour of immersion in the corrosive solution at the free corrosion potential with a scanning speed of 1 mV/s (Figure 2).

Figure 2. Cathodic and anodic polarization curves of 60Cu–40Zn brass in 3% NaCl solution in the absence and in the presence of different concentrations of the FCB formulation.

The addition of the inhibitor to 3% NaCl solution shows a significant decrease in cathodic current density value from the lowest FCB concentration. Indeed, the current density value varies between $10.68 \mu\text{A}\cdot\text{cm}^{-2}$ in absence of inhibitor to $0.56 \mu\text{A}\cdot\text{cm}^{-2}$ in

presence of 1 g/L of FCB formulation, Furthermore, cathodic potentials, the variation of current density stabilizes at a lower value than that recorded for 1 mA control, which attributed to the diffusion limit current of the dissolved oxygen, which persists in all concentrations of the inhibitor [20].

In the anodic domain, we also find that the addition of inhibitor accompanied by a decrease in current density values all the stronger as concentration of the FCB increases.

This formulation acts without modification of the electrochemical behaviour of brass in the 3% NaCl solution for a concentration of 0.5 g/L, moreover, from this concentration, there is a change of behaviour in the potential domain of the corrosion potential E_{corr} . In this case, the variation of the current density shows the appearance of a pseudo-plateau appears clearly for a concentration of 1 g/L FCB, then there is a sudden increase in the current density, this can be attributed to desorption inhibitor of the metal surface after the current density stabilizes at a value of $10 \text{ mA} \cdot \text{cm}^{-2}$, this means a repassivation of the metal [20, 21].

These results highlight the mixed type of the used inhibitor. The values of the corrosion current density i_{corr} , the corrosion potential E_{corr} , the cathode Tafel b_c and anodic b_a slopes and inhibitory efficiency ($IE\%$) for different concentrations of the FCB are reported in Table 3.

The inhibitory efficiency $IE\%$ is calculated by the following relationship:

$$IE\% = \frac{I_{\text{corr}}^0 - I_{\text{corr}}}{I_{\text{corr}}^0} \cdot 100 \quad (1)$$

I_{corr}^0 and I_{corr} are the current densities in absence and in presence of the FCB respectively, determined by extrapolation of linear part of the curves $\log(I)=f(E)$ to the corrosion potential E_{corr} .

Table 2. Electrochemical parameters and corrosion inhibitory efficacy of 60Cu–40Zn brass in 3% NaCl solution in absence and in presence of different concentrations of the FCB.

FCB (g/L)	Cathodic domain			Anodic domain			$IE\%$
	E_{corr} (mV/Ag-AgCl)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	b_c (mV/dec)	E_{corr} (mV/Ag-AgCl)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	b_a (mV/dec)	
Blank	–235	10.68	–135	–220	10.35	45	–
0.5	–190	1.58	–154	–198	1.90	24	85.20
0.75	–199	0.96	–153	–210	1.58	59	91.01
1	–211	0.56	–120	–219	0.61	61	94.75

From the results presented in Table 2, we noted that inhibitory efficiency increases with the FCB concentration and reached a maximum value of 94.75% at the concentration of 1 g/L.

3.4. Electrochemical impedance spectroscopy (EIS)

3.4.1. Influence of the concentration of the formulation FCB

The electrochemical impedance diagrams obtained after 1 hour of immersion time at the free potential of corrosion E_{corr} in the absence and in the presence of different concentrations of the FCB formulation in 3% NaCl solution are shown in Figure 3 and the electrochemical parameters from these diagrams are shown in Table 3.

Figure 3. Electrochemical impedance and Bode diagrams of 60Cu–40Zn brass in 3% NaCl in the absence and in the presence of different concentrations of the FCB after 1 h of immersion time.

Table 3. Electrochemical parameters in relation to the diagrams presented in Figure 3.

[FCB] g/L	R_{ct} ($\text{k}\Omega \cdot \text{cm}^2$)	CPE _{dl}		θ	IE%
		C_{dl} ($\mu\text{F} \cdot \text{cm}^{-2}$)	n_{dl}		
Blank	1.41	112	0.65	—	—
0.5	8.34	17.05	0.58	0.8309	83.09
0.75	12.46	11.41	0.57	0.8868	88.68
1	22.71	6.26	0.54	0.9379	93.79

The corrosion inhibiting efficiency of brass is calculated from the charge transfer resistance by the relation:

$$IE\% = \frac{R_{\text{ct}}^{\text{inh}} - R_{\text{ct}}^0}{R_{\text{ct}}^{\text{inh}}} \cdot 100 \quad (2)$$

Where R_{ct}^0 and $R_{\text{ct}}^{\text{inh}}$ are the charge transfer resistance respectively without and with inhibitor.

From the impedance diagrams, we observe that in the absence of the formulation, the electrochemical impedance spectrum consists of a single capacitive loop in the form of a relatively flat semicircle [22, 23]. The charge transfer resistance determined by extrapolation of the impedance diagram to the low frequency limit is about $1.41 \text{ k}\Omega \cdot \text{cm}^2$.

The addition of the FCB formulation at different concentration shows a capacitive behaviour of the brass 60Cu–40Zn in a 3% NaCl solution without changing the reaction mechanism, that is to say, always the presence of a single capacitive loop this is clearly seen in the representation from Bode diagrams. In all of the concentration range, the addition of an FCB formulation results in an increase in the value of charge transfer resistance R_t , which inhibits a significant variation as a function of the inhibitor concentration. It reaches a maximum value of around the $21.22 \text{ k}\Omega \cdot \text{cm}^2$ for a concentration of 1 g/L, the associated double layer capacity decreases to reach a value of around the $6.70 \mu\text{F} \cdot \text{cm}^{-2}$. The n_{ct} value increases after the addition of the formulation and can be explained by a certain reduction in the heterogeneity of the initial surface due to the adsorption of FCB at the most active adsorption sites of the surface of the electrode, which protects the metal surface of brass against corrosion. The highest value of the inhibitory efficiency $IE\%$ is 93% attributed for a concentration of 1 g/L of FCB.

Adjustment of electrochemical parameters by EC-Lab software shown that the equivalent electrical circuit of these diagrams can be in the form of RC (*cf.* Figure 4), whose components are attributed to:

- R_e : electrolyte resistance,
- R_{ct} : charge transfer resistance,
- C_{dl} : double layer capacity,
- n_{ct} : coefficient of flattening of Nyquist diagrams.

Figure 4. Equivalent electrical circuit of the metal/solution interface.

3.4.2. Rotational speed effect

The influence of the hydrodynamic conditions on the process of inhibition of metal corrosion is one of the most important parameters to be considered. Indeed, this parameter affects both cathodic and anodic reactions, and even in transport of inhibitor material. Most of the studies on organic inhibitors have been studied under static conditions, so there is some works in the literature concerning the effect of hydrodynamic conditions on performance of inhibitors under laminar or turbulent flow. To evaluate the influence of hydrodynamic conditions, electrochemical impedance curves and representation of Bode diagrams are plotted at different rotational speeds of the electrode after 1 hour of immersion time in corrosion

potential E_{cor} (cf. Figure 5). The electrochemical parameters taken from these curves are recorded in Table 4.

Figure 5. Impedance and bode Diagrams of brass 60Cu–40Zn at different rotation speeds of electrode in presence of 1 g/L of the FCB formulation.

Table 4. Electrochemical parameters at different rotational speeds in the presence of 1 g/L FCB.

Rotational speeds (rpm)	R_{ct} ($\text{k}\Omega \cdot \text{cm}^2$)	CPE_{dl}	
		C_{dl} ($\mu\text{F} \cdot \text{cm}^{-2}$)	n_{dl}
500	13.90	10.23	0.62
1000	22.71	6.26	0.57
1500	28.58	4.97	0.52

The analysis of the Figure 5 and the Table 4 shows that the protective effect of our formulation increases remarkably with the increase in the speed of rotation of the electrode. This is probably attributed to the increased mass transport of our formulation to the metal surface. In fact, the hydrodynamic effect seems to have favored the formation of a less permeable protective layer and to have facilitated the movement of green inhibitor towards the metallic surface in order to form a layer with a higher resistance. This result argues in favor of the hydrodynamic conditions being beneficial for the formation of a protective film after one hour of immersion [24, 25]. In addition, the representation of Bode diagrams shows that the increase in the speed of rotation does not change the reaction mechanism and that the Nyquist diagrams are adjusted by the same electrical circuit RC.

3.4.3. Immersion time influence

Figure 6 shows the electrochemical impedance spectroscopy in Nyquist diagrams mode at different immersion times in 3% NaCl solution at the rotation speed of 1000 rpm of the electrode.

Figure 6. Nyquist plots of 60Cu–40Zn brass in solution the 3% NaCl in the presence of 1 g/L the FCB at different immersion times.

Table 5. Electrochemical parameters associated with the impedance diagrams of brass in 3% NaCl solution in the presence of 1 g/L FCB at different immersion times.

Immersion times (hours)	R_{ct} ($k\Omega \cdot cm^2$)	CPE_{dl}	
		C_{dl} ($\mu F \cdot cm^{-2}$)	n_{dl}
1	22.71	6.26	0.57
2	37.59	3.78	0.65
10	102.00	1.39	0.78
24	125.72	1.13	0.80

From the Figure 6, we have observed that the increase in the immersion time is accompanied by a large increase in the size of the loop of electrochemical impedance curve in the presence of 1 g/L of the FCB. The electrochemical parameters obtained from these impedance diagrams are presented in Table 5. In this case, the increase in R_t increases sharply with the immersion time to reach a maximum value of 125.72 $k\Omega \cdot cm^2$ after 24 hours of immersion time, consequently the associated capacity decreases significantly to reach a value of 1.13 $\mu F \cdot cm^{-2}$. These effects suggest that the inhibitory nature of the formulation results from the establishment on the brass surface of a very strong protective layer which blocks the corrosion process of brass in 3% NaCl solution and that the protective effect improves with the immersion time [26]. This appears clearly in the values of n_{ct} which increases after the addition of green inhibitor and can be explained by a certain decrease in the heterogeneity of the initial surface due to the adsorption of the inhibitor molecules at the most active adsorption sites the surface of the electrode.

3.5. Adsorption isotherm

The inhibition of corrosion of metals by organic or green inhibitors is explained by their adsorption on the metal surface. The adsorption isotherms are then an important complement capable of determining the mode of adsorption of these compounds on the surface. In order to obtain the isotherm, the recovery rate (θ) of the metal surface as a function of the concentration of inhibitor must be obtained. Several adsorption isotherms were evaluated and the Langmuir adsorption isotherms proved to be the best model to describe the adsorption of the studied compounds.

The Langmuir isotherm equation is given by [27]:

$$\frac{C_{\text{inh}}}{\theta} = \frac{1}{K_{\text{ads}}} + C_{\text{inh}} \quad (3)$$

Where K_{ads} is the equilibrium adsorption constant, the adsorption isotherm obtained for this formulation is plotted in Figure 7.

Figure 7. Langmuir adsorption isotherm obtained for brass in a 3% NaCl solution in the presence of the FCB.

The standard free Gibbs energy of adsorption is linked to the adsorption constant by the relation [28]:

$$\Delta G_{\text{ads}}^0 = -RT \ln(1000 \cdot K_{\text{ads}}) \quad (4)$$

Where R is the perfect gas constant, the value of 1000 represents the mass concentration of water in the solution (g/L). The adsorption parameters of the green inhibitor are collated in Table 6.

Table 6. The constant adsorption values for different concentrations of the FCB in 3% NaCl solution.

Inhibitor	K (L/g)	R^2	ΔG_{ads}^0 (kJ·mol ⁻¹)
FCB	7.08	0.9996	-21.59

The standard free Gibbs energy value is negative, which indicates the spontaneity of adsorption of the inhibitor on the metal surface. This value equal to $-21.59 \text{ kJ}\cdot\text{mol}^{-1}$ between $-20 \text{ kJ}\cdot\text{mol}^{-1}$ and $-40 \text{ kJ}\cdot\text{mol}^{-1}$ shows that the green inhibitor is adsorbed by a mixed mechanism (physics–chemisorption) [29].

3.6. Surface analysis by MEB/EDX

The surface analysis was performed by SEM coupled with EDX spectrum on the brass 60Cu–40Zn, immersed in solution the 3% NaCl in the absence and in the presence of the FCB. The results obtained are shown in Figures 8 and 9.

Figure 8. SEM micrographs of the brass surface after 24 hours immersion in the 3% NaCl solution in the absence and in the presence of 1 g/L of the FCB.

In the absence of inhibitor (3% NaCl), the surface of electrode is covered by a layer of heterogeneous product with presence of corrosion pitting. The literature presents some studies involving corrosion prevention of 60Cu–40Zn show that this alloy undergoes a selective dissolution of zinc for long immersion time. on the other hand, in the presence of the green inhibitor (FCB), the micrograph shows the absence of corrosion and the surface of the brass is well protected by the formation of a layer on the metal surface which makes it does barrier against the corrosion of this latter.

Figure 9. EDX spectrum of the brass surface after 24 hours' immersion in solution the 3% NaCl in the absence and in the presence of 1 g/L of FCB.

EDX spectrum analysis (Figure 9) in the absence and in the presence of formulation reveals the presence of carbon, calcium and a remarkable increase in oxygen levels. The important signal of carbon and oxygen certainly corresponds to the carbon chains of fatty acids of the FCB formulation; in addition the detection of calcium shows the presence of a layer on the metal surface which characterizes the protective effect of the formulation in the presence aggressive ions [30].

4. Conclusion

In this context, the objective of this work is to synthesize a new formulation based on *Cannabis sativa* L. seeds oil subsequently used as a green corrosion inhibitor for brass 60Cu–40Zn in 3% NaCl solution by potentiodynamic polarization and electrochemical impedance spectroscopy measurements (EIS), completed with SEM/EDX surface analysis. The results obtained reveal:

- The potentiodynamic measurements have shown that the FCB formulation acts as a mixed type (cathodic and anodic) inhibitor.

- The inhibitory efficiency increases with increasing concentration of the FCB to reach a maximum value of 94.75% at a concentration of 1 g/L of the FCB.
- The electrochemical impedance measurements confirm the results obtained by the potentiodynamic curves, and that the protective effect of the FCB is maintained over immersion time.
- The adsorption of the components of the formulation on the surface of the brass (60/40) according to a Langmuir adsorption isotherm. The values of the thermodynamic parameters indicate that the adsorption of FCB is a mixed adsorption (physics-chemisorption).
- The surface analysis by the SEM coupled with the EDX shows the presence of the FCB formulation elements on the surface of the 60Cu–40Zn brass.

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