Study ability of protection effect of organic acids for multi-metallic anti-corrosion additive system in ethylene glycol-water coolants

Pham Thi Mai Huong[1], Nguyen Thi Huong[2, *]

[1]*Faculty of Chemical Technology, Hanoi University of Industry, 298 Cau Dien Street, Bac Tu Liem District, Ha Noi, Viet Nam*

[2]*Institute of Chemistry and Material, Academy of Military Science and Technology, 17 Hoang Sam, Cau Giay, Ha Noi, Viet Nam*

[*]Emails: [1]*phamthimaihuong76@yahoo.com.vn,* [2]*nguyenhuong0916@gmail.com*

Abstract. In this work, the electrochemical behavior of carbon steel, brass, bronze and aluminum alloys in the presence of various important organic acids (sebacic acid (SbA), terephthalic (TPA), 2-ethylhexanoic (E2C6A), and octanoic (C8A)) and the effect that these have on the growth and protectiveness of the alloys have been investigated. The results show that the presence of organic acids in the coolant increases the protective effect of the system by forming a film on the surface of the alloys. The EIS impedance results demonstrate the formation of active and passive protective layers on the surface of the alloys. The results of surveying 04 types of organic acids for the protection of different alloy substrates show that TPA and SbA acid additives are more effective than C8A and E2C5A. The additive C8A not only did not inhibit corrosion but also promoted this process strongly, especially with aluminum alloys and the TPA has the best anti-corrosion effect for steel, followed by C1 copper alloy and aluminum.

Keywords: coolant, corrosion inhibitors, organic acid, multimetallic.

Classification numbers: 2.3.1, 2.5.3, 2.9.1.

1. INTRODUCTION

In addition to the traditional inorganic additives commonly used for ethylene glycol-based coolants, in recent years, the addition of organic compounds is aimed at enhancing the anticorrosion effect, extending the service life of coolants [1 - 3]. The anti-corrosion performance of organic additives has been attributed to the formation of protective films on metal surfaces, preventing the attack of corrosive agents [4, 5]. Organic additives that contain atoms or functional groups with high electronegativity are believed to cause the adsorption of these additives on metal surfaces [6, 7]. The formation of an adsorbent layer on the surface can also be attributed to the interaction of the p orbitals of the organic inhibitor with the d orbitals of the metals [2]. For studying the effect of functional groups (amine or carboxylic), position, and a

number of substituents on metal corrosion resistance, Ormelese et al. investigated a series of organic compounds such as amines, primary and tertiary amines, amino acids, mono and polycarboxylic, alkanolamines [8].

Research results have shown that coolants often use additives from the salt of a mono aliphatic acid with a carbon atom number from C8 to C12, a salt of a diacid from C8 to C12 or an aromatic acid, sodium salt sebacate, salt of carboxylic acid ether or salt such as benzoate, alkyl benzoate [9 - 11]. The characteristic of these additives is that it has the ability to form a thin protective film of microscopic size on the surface of the material. In addition, this film is formed at the corroded sites rather than on the entire surface of the material, thereby not significantly affecting the heat exchange process of the system. Therefore, the consumption of this additive is relatively slow, the service life of the coolant is extended, and the time required to add this additive is often longer than that of traditional inorganic additive systems.

However, the research results focus on evaluating the anticorrosion efficiency on an alloy substrate, there are no published evaluations on many alloy substrates, while cooling systems are often fabricated from many different alloys [12, 13]. Therefore, the objective of this study is to investigate the anti-corrosion protection ability of some organic acids additives for steel, aluminum, and copper alloy substrates through an electrochemical research method measuring polarization curve and electrochemical impedance spectroscopy (EIS). From there, we choose an organic additive system with good anticorrosion protection for multi-metallic substrates used for EG-based coolants.

2. MATERIALS AND METHODS

2.1. Materials

NaCl, NaHCO$_3$, Na$_2$SO$_4$, KOH, ethylene glycol (EG) and organic acid: sebacic, terephthalic, octanoic, and 2-ethylhexanoic were provided by Macklin (China) and Merck (Germany). The deionized water (DI) with conductivity is not greater than 1 μS/cm.

- The characteristics of the studied alloys are shown in Table 1.

- Autolab PGSTAT 302N multifunction electrochemical analysis equipment that is drived by Nova 2.1.4 software in Institute of Chemistry and Materials.

Table 1. Materials for the Working Electrode.

Sign	Alloys code	Materials	Density (g/cm^3)	Equivalent (g)
G	G10200	Steel	7.87	27.93
A	A23190	Aluminum alloy	2.79	9.31
C1	C1100	Bronze	8.89	31.7
C2	C26000	Brass	8.53	32.15

2.2. Methods

2.2.1. Preparation of Sample

- *Corrosive water (CW)*: CW was prepared according to ASTM D 1384 standard with a composition including 148 mg Na$_2$SO$_4$, 165 mg NaCl, 138 mg NaHCO$_3$ that dissolved in 1000

mL of DI.

- *Coolants: The* composition of the coolants included 40 % EG, 0.0 to 0.3 % organic additives, the rest was DI. The coolants had a clear, colorless to light yellow appearance, pH varied from 7.2 to 7.6.

- *Electrolyte solution* (for electrochemical analysis): including coolant and corrosive water in a ratio of 1:2 by weight.

- *Working electrodes (WE)*: WEs were made from studied alloys. The metals were machined into circle, 0.5 cm in diameter, welded with copper wire and sealed with epoxy on the back. The work surface was polished with sandpaper with a fine grain size from 1,200 to 2,000. The mass loss test piece had dimensions of 3 × 40 × 80 mm and was polished by the same method as above.

2.2.2. Electrochemical measurement

Electrochemical measurements were performed on the PGSTAT 302N instrument with a set of three electrodes: reference electrode (RE) Ag/AgCl, counter electrode (CE) Pt, working electrode (WE) made of studied alloy.

- *Polarization curve:* measuring range ± 0.15 V vesus to open circuit potential (OCP); scan rate 0.001 V/s. The corrosion inhibition efficiency (IE) was calculated according to the following formula*:*

$$IE = \frac{i_{corr}^0 - i_{corr}}{i_{corr}^0} \times 100 \tag{1}$$

where, i_{corr}^0 and i_{corr} are current density (A/cm^2) in the absence and presence of inhibitors, respectively.

- *Electrochemical impedance spectroscopy (EIS)*: scan range from *10^5-0,1 Hz*, the samples were allowed to stabilize the OCP 20 min before measuring.

3. RESULTS AND DISCUSSIONS

3.1. Effect of content of organic acid additives

Corrosion of aluminum alloy A23190 (A), steel G10200 (G) bronze C11000(C1) and brass C26000 (C2) in EG-water (EG-W) media accelerated by CW without the addition of organic acid additives as the reference result which were studied through polarization curve measurement method. The obtained electrochemical parameters show that, without using additives, the aluminum alloy A and C1 copper samples had a low corrosion rate, while the G steel alloy and C2 brass were relatively strong corroded with corrosion rate of 0.0719; 0.0258; 0.4260 and 0.2666 mm/year. Some additives have been further studied to clarify their effects.

3.3.1. Effect of sebacic acid (SbA) and terephthalic acid (TPA)

The anticorrosion capacity of SbA and TPA on alloys A, G, C1, C2 was investigated at different concentrations. The results are shown in Table 2 and Figure 1.

The results in Table 1 show that SbA effectively inhibits corrosion in all 4 types of alloys studied, in which the highest efficiency is achieved with steel (G) samples up to 99.9 %. The survey results also indicate the protection effect is lowest for brass alloy (C2) samples. In addition, there is a certain optimal content for each material. With the aluminum alloy (A), copper (C1, C2) is 0.2 %; while the steel sample is 0.3 %.

Table 2: Electrochemical analysis results of anticorrosion inhibition property of SbA and TPA

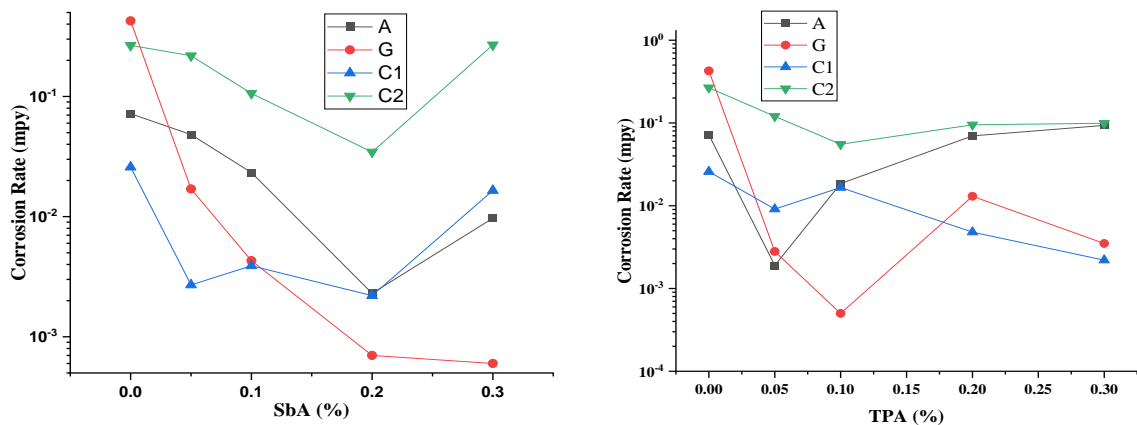| Contents of additives %	Alloys	SbA				TPA			
		E_{cor} (V)	Icor (A/cm^2)	Corrosion Rate mm/year	IE, %	E_{cor} (V)	Icor (A/cm^2)	Corrosion Rate mm/year	IE, %
0.00	A	-0.906	6.59×10^{-6}	0.0719	-	-0.906	6.59×10^{-6}	0.0719	-
0.05		-0.575	4.42×10^{-6}	0.0482	32.9	**-0.514**	**1.73×10^{-7}**	**0.0019**	**97.4**
0.1		-0.768	2.14×10^{-6}	0.0233	67.6	-0.550	1.69×10^{-6}	0.0185	74.3
0.2		**-0.751**	**2.06×10^{-7}**	**0.0023**	**96.9**	-0.600	6.41×10^{-6}	0.0699	7.0
0.3		-0.750	8.91×10^{-7}	0.0097	86.5	-0.991	8.57×10^{-6}	0.0935	-30.0
0.00	G	-0.583	3.67×10^{-5}	0.4260	-	-0.583	3.67×10^{-5}	0.4260	-
0.05		-0.275	1.47×10^{-6}	0.0170	96.0	-0.191	2.40×10^{-7}	0.0028	99.3
0.1		-0.214	3.67×10^{-7}	0.0043	99.0	**-0.281**	**4.44×10^{-8}**	**0.0005**	**99.9**
0.2		-0.213	6.08×10^{-8}	0.0007	99.8	-0.280	1.12×10^{-6}	0.0130	96.9
0.3		**-0.225**	**5.34×10^{-8}**	**0.0006**	**99.9**	-0.265	3.06×10^{-7}	0.0035	99.2
0.00	C1	-0.030	2.21×10^{-6}	0.0258	-	-0.030	2.21×10^{-6}	0.0258	-
0.05		-0.014	2.33×10^{-7}	0.0027	89.4	-0.043	7.81×10^{-7}	0.0091	64.6
0.1		-0.131	3.37×10^{-7}	0.0039	84.7	-0.030	1.42×10^{-6}	0.0166	35.6
0.2		**-0.001**	**1.85×10^{-7}**	**0.0022**	**91.6**	-0.019	4.15×10^{-7}	0.0048	81.2
0.3		-0.004	1.41×10^{-6}	0.0165	36.0	**-0.036**	**1.90×10^{-7}**	**0.0022**	**91.4**
0.00	C2	-0.438	2.16×10^{-5}	0.2666	-	-0.438	2.16×10^{-5}	0.2666	-
0.05		-0.283	1.77×10^{-5}	0.2184	18.1	-0.246	9.76×10^{-6}	0.1203	54.9
0.1		-0.295	8.61×10^{-6}	0.1061	60.2	**-0.169**	**4.48×10^{-6}**	**0.0553**	**79.3**
0.2		**-0.126**	**2.80×10^{-6}**	**0.0345**	**87.1**	-0.221	7.70×10^{-6}	0.0950	64.4
0.3		-0.149	2.18×10^{-5}	0.2692	-1.0	-0.178	8.05×10^{-6}	0.0992	62.8

Figure 1. Effect of concentration of SbA, TPA additives on corrosion rate of alloys.

When the amount of SbA used exceeds this content level, except for steel, but with the remaining 3 types of alloys, the protection effect is reduced, even greatly reduced, and causes a stronger corrosive effect as with the C2 sample. The TPA has the best anti-corrosion effect for steel, followed by C1 copper alloy and aluminum. However, with different alloys, they have different optimal concentration range. With steel, the best additive content is about 0.1%. Meanwhile, with aluminum alloy A showed that TPA only had inhibitory effect in low concentration region. It is best at about 0.05 %, as the concentration increases, the corrosion inhibition efficiency decreases. Even when the content is up to 0.3 %, it also promotes the corrosion process (negative inhibitory effect). In contrast, with the C1 copper alloy, the inhibitory effect increased with the TPA content, the highest value of 91.4 % was obtained when the TPA content was 0.3 %. In the case of C2 brass alloy, although it does not promote corrosion, the inhibitory effect of TPA additive with this alloy is also relatively low. In the studied scope, the optimal content of this additive in C2 brass alloy corrosion inhibition is 0.1 %, the achieved efficiency is 79.3 %. When the additive content, the inhibitory efficiency also ranges from more than 50 to nearly 65 %.

3.3.2. Effect of C8 acids (octanoic and 2-ethyl hexanoic)

Table 3. Electrochemical analysis results of anticorrosion inhibition property of SbA and TPA.

Contents of additives, %	Alloys	C8A				E2C6A			
		E_{cor} (V)	Icor (A/cm^2)	Corrosion Rate mm/year	IE, %	E_{cor} (V)	Icor (A/cm^2)	Corrosion Rate mm/year	IE, %
0,00	A	-0,906	$6,59\times10^{-6}$	0,0719	-	-0,906	$6,59\times10^{-6}$	0,0719	-
0,05		-1,220	$9,48\times10^{-6}$	0,1035	-43,9	**-0,682**	**$2,51\times10^{-7}$**	**0,0027**	**96,2**
0,1		-1,213	$8,75\times10^{-6}$	0,0954	-32,7	-0,692	$3,51\times10^{-6}$	0,0038	94,7
0,2		-1,275	$3,43\times10^{-5}$	0,3747	-264,3	-0,580	$2,49\times10^{-6}$	0,0272	62,2
0,3		-1,296	$4,35\times10^{-6}$	0,4751	-560,5	-0,530	$1,21\times10^{-6}$	0,0132	81,7
0,00	G	-0,583	$3,67\times10^{-5}$	0,4260	-	-0,583	$3,67\times10^{-5}$	0,4260	-
0,05		-0,258	$2,50\times10^{-7}$	0,0029	99,3	-0,923	$4,85\times10^{-5}$	0,5631	-32,2
0,1		-0,274	$6,49\times10^{-7}$	0,0075	98,2	-0,583	$3,12\times10^{-5}$	0,3621	15,0
0,2		-0,266	$3,64\times10^{-7}$	0,0042	99,0	-0,442	$2,57\times10^{-5}$	0,2984	30,0
0,3		**-0,252**	**$1,01\times10^{-7}$**	**0,0012**	**99,7**	**-0,262**	**$1,39\times10^{-6}$**	**0,0161**	**89,6**
0,00	C1	-0,030	$2,21\times10^{-6}$	0,0258	-	-0,030	$2,21\times10^{-6}$	0,0258	-
0,05		-0,044	$1,52\times10^{-6}$	0,0177	31,3	-0,010	$4,06\times10^{-7}$	0,0047	81,6
0,1		**-0,013**	**$2,62\times10^{-7}$**	**0,0031**	**88,2**	-0,023	$2,49\times10^{-7}$	0,0029	88,7
0,2		-0,051	$1,26\times10^{-6}$	0,0147	43,1	-0,009	$8,79\times10^{-7}$	0,0103	60,2
0,3		-0,072	$2,11\times10^{-6}$	0,0246	4,7	-0,012	$2,55\times10^{-6}$	0,0298	-15,3
0,00	C2	-0,438	$2,16\times10^{-5}$	0,2666	-	-0,438	$2,16\times10^{-5}$	0,2666	-
0,05		-0,246	$2,97\times10^{-6}$	0,0366	86,3	-0,314	$1,40\times10^{-5}$	0,1722	35,4
0,1		-0,227	$2,58\times10^{-6}$	0,0318	88,1	-0,221	$1,01\times10^{-5}$	0,1251	53,1
0,2		**-0,258**	**$2,56\times10^{-7}$**	**0,0032**	**98,8**	**-0,164**	**$7,55\times10^{-6}$**	**0,0931**	**65,1**
0,3		-0,255	$5,25\times10^{-7}$	0,0065	97,6	-0,151	$1,32\times10^{-5}$	0,1630	38,9

The anticorrosion effect of two types of C8 acids, including straight chain (Octanoic acid, C8A) and a branched one (2-ethylhexanoic acid, E2C6A) was investigated. The polarization

curve measurement results are shown in Figure 2, and the electrochemical parameters are shown in Table 3.

The results show that the additive C8A not only did not inhibit corrosion but also promoted this process strongly, especially with aluminum alloys. The higher the additive content, the stronger the corrosion. At a concentration of 0.3 % by weight, the corrosion rate of aluminum alloys in the study environment was 0.4751 mm/year, which was more than 6 times higher than in the case of no inhibitor. The results of the polarization curve also show that the Ecor corrosion potential shifts to the negative side in the presence of the C8A additive, which means that the presence of this additive makes the aluminum alloy susceptible to corrosion effects. For the remaining alloys, C8A exhibits very good corrosion inhibition, especially with steel, the anti-corrosion efficiency varies between 98.2 and 99.7 % for the investigated additive contents range. Meanwhile, for additive E2C6A, the corrosion potential changes significantly for aluminum (A), steel (G) and brass (C2) alloys. While with C1 copper, the change in corrosion potential is negligible.
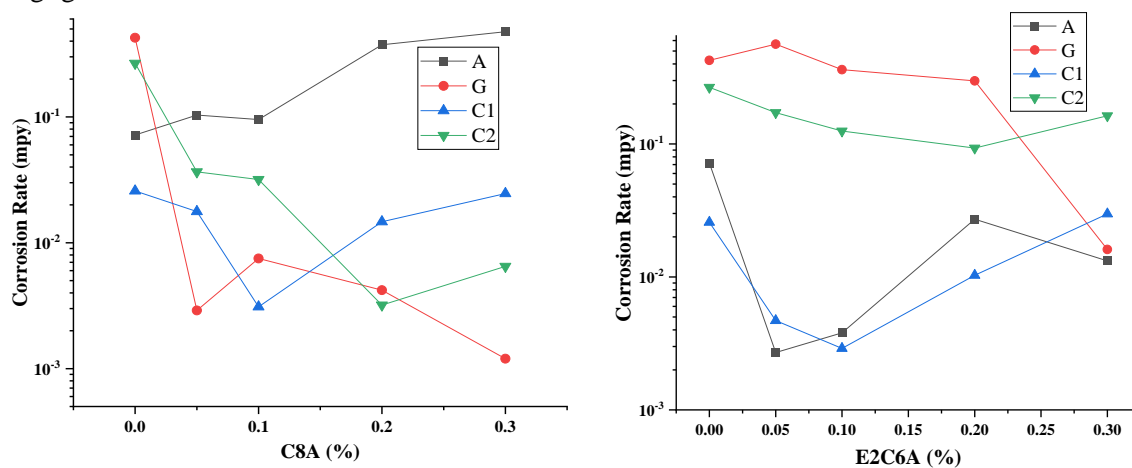

Figure 2. Effect of concentration of SbA, TPA additives on corrosion rate of alloys.

From the measurement results of the polarization curve of the additive E2C6A, it shows that this additive has a good inhibitory effect on aluminum. The best protection effect occurs when the amount of E2C6A used is 0.05 %, and this decreases as the additive content increases. For steel, the inhibitory effect is achieved when the additive content used is large enough. However, the best protection efficiency was only 89.6 %. In small amounts of use they even promote corrosion. With C1 copper alloy, the protection efficiency is in the range of 60 - 80 % when the additive content used is below 0.2 %. With greater use, the protective effect decreases very rapidly, even being negative, that is, they promote corrosion. With the C2 brass alloy, the protective effect of the E2C6A additive is also quite low, the highest value is 65.1 % with the content used of 0.2 %. With higher or lower concentrations, the protective effect is reduced.

The results of surveying 04 types of organic acids for the protection of different alloy substrates show that TPA and SbA acid additives are more effective than C8A and E2C6

A. It shows that the same C8 chain but with different carbon chain structure will create different interactions on the surface of each alloy [14], leading to a difference in the ability to create coatings and anti-corrosion protection. To clarify the protective action mechanism of the

additive, we continue to study the EIS method to build the Nyquist and Bode diagram for the representative additive system, which is TPA acid, the results are given in section 3.2.

3.2. Results of electrochemical impedance spectroscopy

Electrochemical impedance spectroscopy was used to study the electrochemical behavior on the alloy surface in the case of using different additives. The results of EIS spectroscopy of samples with TPA additive are presented in Figures 3 and 4.

The Nyquist and Bode plots show that there is a pairwise similarity in shape between the 0.3 % TPA sample for steel and C1 copper, aluminum and C2 brass. This is consistent with the polarization curve measurement results given in Table 1. Accordingly, with steel and C1 copper, the 0.3% TPA sample has high corrosion inhibition efficiency, the corrosion current density reaches the value of 3.06×10^{-7} and 1.90×10^{-7} A/cm^2, respectively. Meanwhile, for aluminum and C2 brass samples, 0.3 % TPA samples have high corrosion current densities of 8.57×10^{-6} and 8.05×10^{-6} A/cm^2, respectively. At this current temperature, they do not effectively inhibit corrosion but also promote corrosion.

The Nyquist diagram in Figure 3 shows that the difference in impedance is very large between these two groups. With steel and C1 copper, Nyquist graphs are just curves, the impedance value is very large, this is explained by the formation of a protective passive film on the metal surface, the higher the impedance value. The high conductivity indicates that the electrical conductivity, the ability to transfer the charge in these films is very low, so that this film protects the metal well from corrosion. While with aluminum and C2 brass is the opposite, the Nyquist graph has formed semicircles, the impedance value is very low, in which it is easy to see with the aluminum model the Nyquist curve lies the lowest. This shows that the effectiveness of corrosion protection in this case is very poor. When considering the impedance-frequency diagram (Z-f, Bode diagram) in Figure 3, this is even more clearly. Although the impedance Z value in the low frequency region is similar, in the low frequency region the case with steel and copper is much higher than that of aluminum and C2 brass.
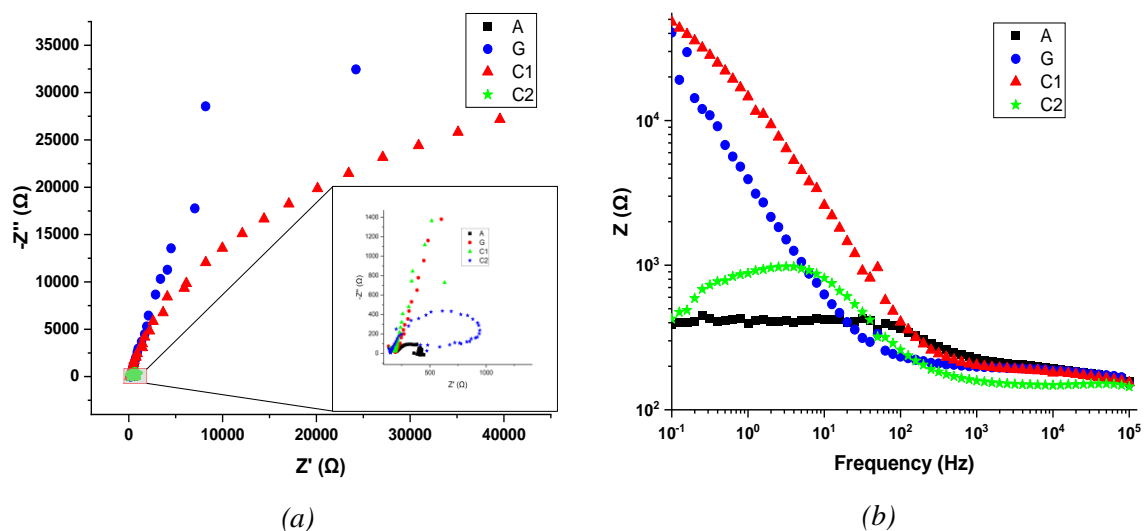

(a)   *(b)*

Figure 3. Nyquist (a) and Bode (b) plot of 0.3 % TPA additive for alloys.

To further clarify the protective action mechanism of the additive, based on the Nyquist and Bode diagrams, the electrochemical behaviour on the surface of the working electrodes was simulated by the equivalent circuit which is shown in Figure 4. The results of the matching circuit show that the built equivalent circuit is quite consistent with the measurement results. That shown in Figure 5.
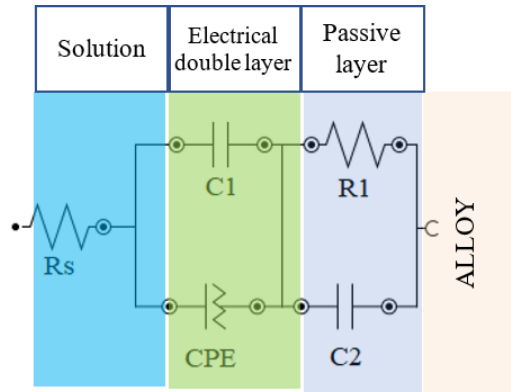

Figure 4. Equivalent circuit diagram simulating the electrochemical behaviour on the alloys surface.

In the equivalent circuit of Figure 4, the formation of film layers on the electrode surface can be assumed. From the metal surface, the first is the passivation layer, which is formed by the active parts of the adsorbent adsorbing on the surface, creating a passive layer covering the metal surface. This passive class is characterized by resistor R1 and capacitor C2. Then, there is the double charge layer, which is formed by the hydrocarbon tail of the additive as the base. Characteristic of the double charge layer is the capacitor C1 and the constant phase element CPE. CPE is an impractical element, built upon the non-ideal capacitor element. It is characterized by a conductance value Y0 (similar to the capacitance C of a capacitor) and a non-ideal exponential factor N. With these films, the higher their impedance, the better the charge-carrying capacity. The less effective the protection, the better the resistance to electrochemical corrosion. Therefore, the lower the C or Y0 values of the capacitance, the better the protection in this case.
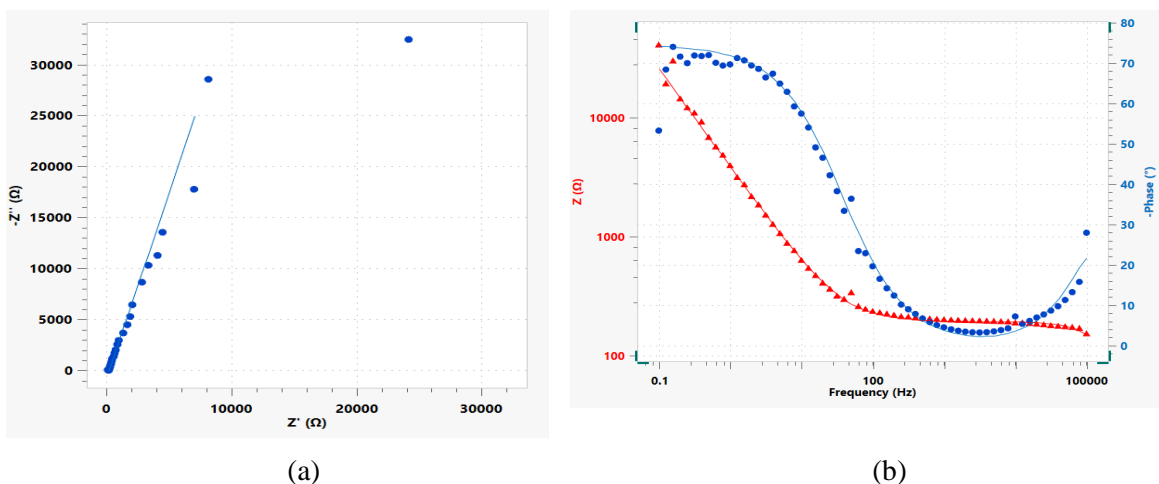

(a)   (b)

Figure 5. Fitting result of G-0,3TPA sample: a-Nyquist plot, b-Bode plot.

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Table 4. Electrochemical characterizatio of equivalent circuit.

Elements	Unit	C1	G	C2	A
Rs	Ω	108.3	99,95	108,18	108,89
C1	F	$4,5 \times 10^{-6}$	$5,3 \times 10^{-7}$	$3,2 \times 10^{-7}$	$3,2 \times 10^{-6}$
Q	Y_0, *Mho*	$2,4 \times 10^{-5}$	$5,7 \times 10^{-5}$	0,02	0,0041
	N	0,41	0,83	0,05	0,15
R2	Ω	85,7	86,4	84,3	80,3
C2	F	$2,9 \times 10^{-8}$	$2,8 \times 10^{-8}$	$1,2 \times 10^{-8}$	$2,0 \times 10^{-8}$

When in contact with the outermost charged double film, solutions are produced, their electrochemical characteristic is the solution resistance Rs. The electrochemical characteristic values of the electrochemical elements are shown in Table 4. From the results in Table 4, the passivation films in the 4 alloys are equivalent. The difference is in the double charge layer. The copper C1 and G steel showed uniformly low values of C1 and Y0, while for the C2 and A alloys, the yield values of Y0 were higher. As explained above, this increases the conductivity of the charge double layer. This explains the poor anticorrosion performance in this case. In addition, from the initial survey results, it shows that, with the coolant on the Glycol - Water system, there is no anti-corrosion additive that can be effective for all cases. Therefore, there is always a combination of different inhibitors to effectively compensate and/or synergize for the best protection.

4. CONCLUSIONS

The survey and assessment of anti-corrosion ability on multi-metallic substrates for additives being organic acids in EG-water cooling system show that sebacic acid (SbA) additive has the best protective effect for steel (99.9 %). The additive terephthalic acid (TPA) has the best protective effect on steel, followed by those of the red copper and aluminum alloys. Octanoic acid (C8A) provides good protection for steel, followed by brass alloys, but C8A promotes aluminum corrosion. The use of organic compounds to inhibit corrosion has been of great significance due to their application in the prevention of corrosion in corrosive environments in general and in EG-water. These compounds have been shown to be effective in inhibiting aqueous corrosion by forming films on metal surfaces. The inhibitory effect of organic compounds is due to the interaction with metal surfaces by adsorption.

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Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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