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Chapter

Electrochemical Impedance Spectroscopy (EIS) Characterization of Kitchen Utensils Used as Materials for Local Cooking in Two Culinary Media

Jacques Sawadogo and Jean Boukari Legma

Abstract

This study is inscribed in the framework of the valorization of traditional kitchen utensils recycled from aluminum waste in Burkina Faso. In fact, these traditional kitchen utensils made of recycled aluminum alloys occupy a very important place in Burkina Faso's kitchen. The effect of foods for consumption on its local utensils was studied using the non-stationary technique and electrochemical impedance spectroscopy. For this purpose, a sample of utensil has been deduced on traditional production site. The corrosion behavior of the recycled aluminum alloy ok know chemical composition was evaluated by analyzing the impedance spectra obtained at the open circuit potential, in the salt media titrated at $3 \text{ g}\cdot\text{L}^{-1}$ and rice. Modeling electrical properties by using of a simple equivalent circuit made it possible to interpret the results obtained by impedance spectroscopy. The results showed a susceptibility to pitting corrosion and were confirmed by the electrochemical impedance spectroscopy method.

Keywords: corrosion, electrochemical impedance spectroscopy, equivalent circuit, foods, recycled aluminum alloy

1. Introduction

The pots made by craftsmen from recycled aluminum alloys play an important role in the cooking process in Burkina Faso. These alloys stand for very reactive materials and react instantly to media containing oxygen. This is why their outside surface is covered with an isolating oxide film. The thickness of this film reached around 10 nm and plays a protective role in those materials to corrosion which was generally observed in some aggressive media. The state in which these materials are located is called passivity state. The passivity condition can be stopped at any time when defects are found in the oxide film (discontinuity and heterogeneity) or the presence of aggressive ions in the electrolytic media (halogen, cyanide, etc.).

This can lead to a release of a localized aggression [1]. Aluminum alloys have a low density ($2.7 \text{ g}\cdot\text{cm}^{-3}$), a good thermal and electrical conductivity, a low melting point, easy to shape, a relatively low price, which is advantageous for local people [2]. Moreover, they are of high mechanical characteristics which make them to be utilized as structural materials. In Africa and particularly in Burkina Faso, craft industry turns to profit these properties in the recycling of aluminum alloys for kitchen utensils manufacturing; the raw material used in this field is made of combined or non-aluminum waste, from old car spare parts, beverage cans and tins [3]. Manufacture techniques remain empirical and recycled aluminum alloys are not homogeneous. Corrosion phenomena is favored when utensils are used for food cooking at high temperature or for long cooking time and when acidic or alkaline food are stocked in these same containers for long time [4, 5]. The humidity, the high temperature, and the cooking times are factors which favor the metallic materials corrosion, from which some of the component elements of the corroded material get through the surrounding aqueous media. Despite the numerous studies related to aluminum and their alloys corrosion, few scientific, strict and comprehensive studies on the behavior to corrosion, recycled allows in the craft industry have been conducted. The objective of this work was to study the corrosion behavior of a recycled aluminum alloy collected in the city of Ouagadougou (Burkina Faso) in various culinary media and to evaluate the anti-corrosion effect in these media. This study was carried out by the use of an electrochemical technique: electrochemical impedance spectroscopy (EIS).

2. Materials and methods

2.1 Sample preparation

The chemical composition of the recycled aluminum alloy is shown in **Table 1** [6]. Before each measurement, the aluminum allow surface preparation of these discs for electrochemical tests was the following: the discs were first ground with 400 through 4000 grit SiC papers and then polished with diamond down to $6 \mu\text{m}$ and followed by $1 \mu\text{m}$ alumina – 30% chrome oxide suspension, and finishing with 5% oxalic acid solution. Later, each polished sample was rinsed with acetone and put in an ultrasonic cleaner for 10 min. Subsequently, it was rinsed with milliQ water (with a conductivity of $0.7 \mu\text{s}/\text{cm}$) and ethyl alcohol and finally dried under a hot air flow. The contact area with the culinary media is 3.46 cm^2 .

2.2 Culinary media preparation

To simulate similar Burkina Faso operation, the testing media was local culinary media whose composition is given below. The media used in this study are: salt water (WS) titrated at $3 \text{ g}\cdot\text{L}^{-1}$ and broken rice (WR) in tap water (5 g of broken rice in 250 ml water) reserve for local consumption. The selection is made based on the fact that rice is the most consumed cereal in Burkina Faso. In this country,

Chemical elements	Al	Si	Fe	Cu	Zn	Mg	Cl	Mn	P	Pb	Remainder
Wt. %	82.8	12.76	0.76	1.21	1.27	0.48	0.19	0.27	0.22	0.01	0.03

Table 1.
Chemical composition of the recycled aluminum alloy determined by X-ray fluorescent technique.

the people consume on the average once daily prepared with vegetables, fish, and meats. These media were chosen to simulate a cooking process similar to that of Burkina Faso. All electrochemical measurements were performed in five replicates for each cooking media and show a reproducibility up to around 3–9%. Before each test, we assure that all the electrodes are submerged in the media, at the same depth in the electrochemical cell. As the cooking is most of the time performed at a hot temperature, the media were tested at boiling temperature (100°C) in order to simulate the real cooking conditions [7, 8].

2.3 Detection of aluminum in different culinary media using Eriochrome black T

2.3.1 Principle

Eriochrome Black T is a (3-hydroxy-4-[(1-hydroxy-2-naphthalenyl)azo]-7-nitro-1-naphthalenesulphonic) acid sodium salt, Mordant Black 11. In the presence of colored indicator [9], diluted aluminum in the buffer solution forms a complex which changes at wine-colored. The formed complex is more stable. Acidity of the obtained solution depended on aluminum content. The various measures of aluminum content in culinary media will be done by colorimetric with a spectrophotometer 7315 of JEWAY mark at 560 nm wavelength [10, 11].

2.3.2 Aluminum quantitative analysis method

Local kitchen utensil samples have been thoroughly washed and rinsed using distilled water then air dried. Each kitchen utensil has been filled out with studied solution WS and WR then heated at boiling temperatures. To compensate evaporation during heating phase, the final volume is adjusted to the end of each operation with distilled water [12].

2.3.3 Colorimetric dosage of aluminum released from local kitchen cooking

The loss of aluminum quantity released from two local kitchen utensils was determined by colorimetric dosage to 5 ml sample for each cooking solution. For that it was placed in the graduated flask of 50.0 ml containing 10.0 ml distilled water: 5.0 ml of EBT solution, 20.0 ml of buffer solution acetyl acetic acid ($C_4H_6O_3$) permit to hold a pH at 6; 1.0 ml ascorbic acid 2% and a volume of solution S_0 specified in **Table 2** then filled up to the line of gauge with distilled water. After stirring and resting during 20 minutes, samples were analyzed with spectrophotometer. The standard was measured with a solution without aluminum and tally with no absorbance. The concentration of aluminum in the different solution was expressed in mg/L.

2.4 Data analysis

Data obtained were analyzed for duration and temperature variations using the Student's t-test and XLSTAT 7.5.2 statistical software. Mean parameter concentrations were compared according to the Ryan-Einot-Gabriel-Welsch (REGWQ) test.

2.5 EIS-electrochemical impedance techniques

Electrochemical impedance spectroscopy (EIS) is a well-established quantitative method for the accelerated evaluation of the anti-corrosion performance of

	Control sample	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11	S12
Volume of S0 (ml)	0	0.08	1.6	3.2	4	5	6.25	6.5	7.5	9	10	12.5
Distilled water	10	10	10	10	10	10	10	10	10	10	10	10
EBT (ml)	5	5	5	5	5	5	5	5	5	5	5	5
Buffer pH = 6 (ml)	15	15	15	15	15	15	15	15	15	15	15	15
Ascorbic acid 2% (ml)	1	1	1	1	1	1	1	1	1	1	1	1
Complete distilled water (ml)	50	50	50	50	50	50	50	50	50	50	50	50
Al concentration (mg/L)	0	0.016	0.032	0.64	0.8	0.1	1.25	1.3	1.5	1.8	2	2.5

Table 2.
Composition of standard scale.

protective coatings. Within short testing times, EIS measurements provide reliable data, allowing for the prediction of the long-term performance of the coatings. The result of EIS is the impedance of the electrochemical system as a function of frequency. EIS is a versatile testing procedure and can be performed under different conditions of stress, depending on the performance of the tested coatings. Electrochemical impedance spectroscopy (EIS) is a powerful technique that utilizes a small amplitude, alternating current (AC) signal to probe the impedance characteristics of a cell. The AC signal is scanned over a wide range of frequencies to generate an impedance spectrum for the electrochemical cell under test. EIS differs from direct current (DC) techniques in that it allows the study of capacitive, inductive, and diffusion processes taking place in the electrochemical cell.

2.6 Electrochemical measurements

The electrochemical measurements were conducted in the Analytical Chemistry and Interfacial Chemistry (CHANI) of the University of Brussels (ULB). The EIS measurements were determined in a three electrodes electrochemical cell containing the culinary media. There are three electrodes – the reference electrode, the auxiliary electrode, and the working electrode. A saturated calomel electrode (SCE) was used as the reference electrode, a platinum metal gate as the auxiliary electrode, and a recycled aluminum alloy as working electrode (WE) made in the laboratory. The EIS measurements were performed with employed Princeton Applied Research potentiostat (model PGSTAT 50). A microcomputer was used for data acquisition. The measurements were carried out after 60 minutes of cooking.

3. Results and discussion

3.1 Open-circuit potential measurements

Open-circuit potential, E_{oc} , changes were measured against a standard saturated calomel electrode placed in the same compartment. The recycled aluminum alloy was immersed in the culinary media exposing a circular area of about 3.46 cm^2 . A copper wire was soldered at the rear of the electrode which was housed in a glass tube to protect it from the test culinary media. Results of the open circuit

potential (E_{oc}) are shown in **Figure 1**. In the curve (**Figure 1**), a rapid increase of the open circuit potential was observed followed by a decrease of the value in the two culinary media. Open-circuit potential was studied for 15 min of cooking in the various culinary media. From the curve, a rapid increase of the open-circuit potentials followed by a decreasing of the value in the two culinary media (salt water at 3 g.L^{-1} and broken rice) were observed. It can be noticed that these curves vary toward higher values during the first 150 seconds of cooking but after that, an almost decrease of the potential is observed. In this case, we can observe the aluminum passivation tendency which could have many forms: passivation caused by hydroxides which are absorbed at the metal surface, that caused by absorption of the existing components of the two cooking media or their combination.

A comparison of the behavior of recycled alloy in the media (broken rice and salt water) indicated that significantly higher corrosion potential was recorded in the salt water compared to broken rice media. This could be explained by their negative effect susceptible of influencing the passivation during the first minutes of cooking. According to literatures, the presence of chloride ions in study media could compete with media hydroxides ions when absorbed at the surface, allowing a localized corrosion and then a deterioration of the passive film [13]. In order to understand more about the existing behavior for metal/media in the cooking media, a series of curves was set out by electrochemical impedance spectroscopy in the context of comparative study in the different media.

3.2 Electrochemical impedance spectroscopy (EIS) measurements

Behavior to corrosion from recycled alloy in the two cooking media simulating a similar process to Burkina Faso cooking habit was studied by electrochemical impedance spectroscopy at 100°C and different cooking times. The frequency ranged from 100 KHz to 100 mHz, and the amplitude was set at 10 mV. Nyquist and Bode plots were used in broken rice media and that of salt water titrated at 3 g.L^{-1} and up to boiling temperature after various cooking times in an open-circuit. Data

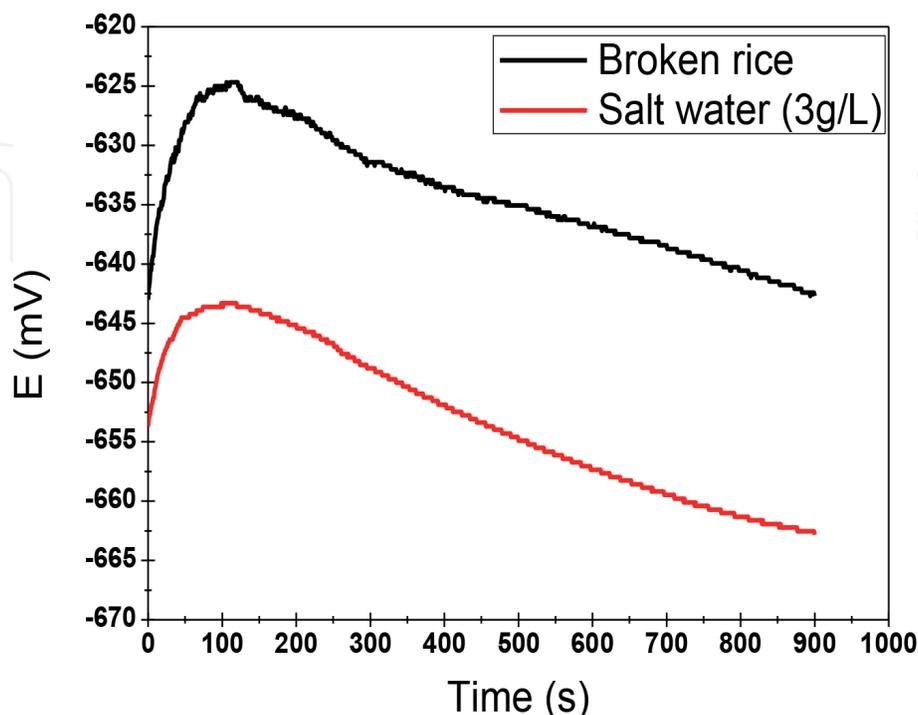


Figure 1.
Open-circuit potential for recycled aluminum alloy in salt water and broken rice.

acquisition and analysis were performed with microcomputer. The spectra were interpreted using the ZSimpWin program. These measures were performed in five replicates to ensure the results reproducibility.

3.3 Effect of cooking times

Measuring electrochemical impedance consists in studying the response of the electrochemical system, due to disturbance which is most often a low amplitude double signal. The strength of this technique is to differentiate the reaction phenomena from their relaxation times. Only quick processes are characterized in high frequencies; when the applied frequency decreases, appears the contribution of slower steps as transport phenomena or solution diffusion. To evaluate the behavior of the passive layer in various culinary media, the sample of aluminum alloy was immersed continuously for 60 minutes (00, 15, 30, and 60 minutes) for broken rice and salt water. During these cooking times, only measurements of impedances have been regularly performed since they do not disturb the system. Nyquist graph (**Figures 2** and **3**) illustrates the experimental impedance diagrams to corrosion potential obtained from the aluminum alloy in the studied culinary media. Indeed, **Figure 3** shows a progressive decrease in the size of the impedance spectrum in a more or less flattened half circle shape, characterizing the formation of the protective layer (alumina Al_2O_3). This leads to a decrease of the total recycled aluminum resistance with regards to the cooking time. In contrast to the salt water media, the broken rice media (**Figure 2**) show an increase in the spectra size, confirming the sample resistance of the media [14]. We find a phase difference with respect to axis of real (**Figures 2** and **3**), which may be explained by the surface none-homogeneity. However, for a better correlation between the experimental data and simulation, we introduced into the procedure for calculating a constant phase element and the surface none-homogeneity is realized through this constant phase element as follows (Eq. 1) [15–18].

Despite a constant phase element being utilized for data fitting instead of an ideal capacitor, since n values obtained from data fitting were in the range from 0.85 to 0.95, the value obtained from data fitting was taken as the capacitance (Eq. (1)).

$$Z_{CPE} = C [(j\omega)]^{-n} \quad (1)$$

Z_{CPE} = the impedance of the CPE;

CPE: constant phase element.

C = the capacitance associated to an ideal capacitor;

j = the complex imaginary number;

ω = the angular frequency and

$$-1 < n < 1$$

When $n = 1$, CPE is an ideal capacitor (Eq. (2)) [19, 20].

$$Z_{dc} = C [(j\omega)]^{-1} \quad (2)$$

Z_{dc} = double layer capacitance.

A true capacitive behavior is rarely obtained. The n values close to 1 represent the deviation from the ideal capacitive behavior [21].

The best simulation is obtained from the use of equivalent circuit proposed for metal/electrolyte interface and illustrated in **Figure 4**. This equivalent circuit was

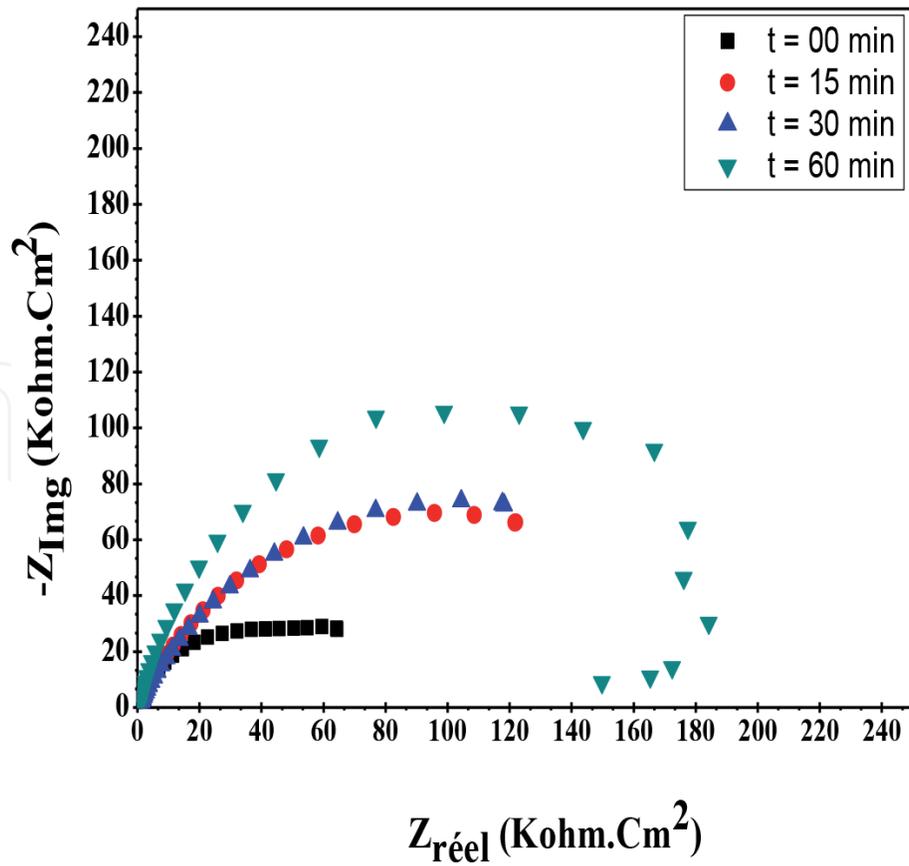


Figure 2.
Nyquist plots for recycled aluminum alloy tested in broken rice media.

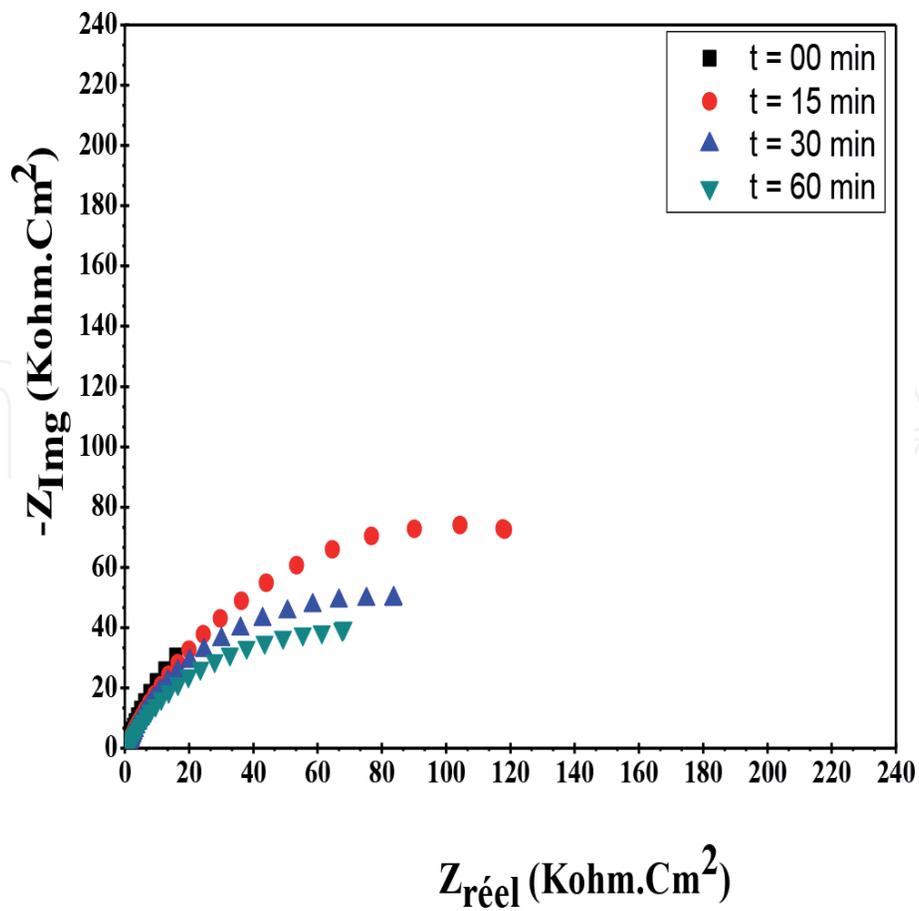


Figure 3.
Nyquist plots for recycled aluminum alloy tested in salt water media.

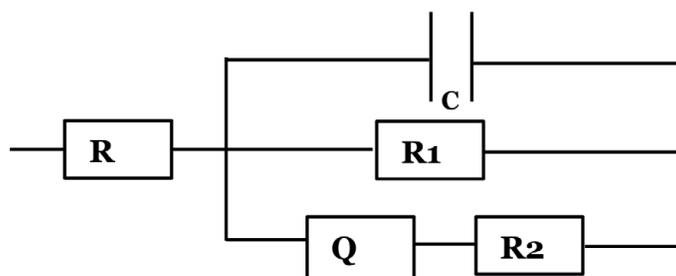


Figure 4.
Equivalent circuit to aluminum alloy in the cooking food of Burkina Faso.

Media	Times	R	C	R1	Q	n	R2
	(min)	$k\Omega\cdot\text{cm}^2$	$\text{F}\cdot\text{cm}^{-2}$	$k\Omega\cdot\text{cm}^2$	$\text{S sn}\cdot\text{cm}^{-2}$		$k\Omega\cdot\text{cm}^2$
Broken rice	0	0.697	0.0017	0.3480	0.0048	0.73	75.4
	15	$1.81\cdot 10^{-8}$	$1.72\cdot 10^{-5}$	0.4830	0.0053	0.78	196.8
	30	$3.21\cdot 10^{-8}$	$1.53\cdot 10^{-5}$	0.5364	0.0060	0.76	223.8
	60	$1.96\cdot 10^{-6}$	$9.14\cdot 10^{-6}$	0.3061	0.0015	0.87	259.2
Salt water	0	$6.70\cdot 10^{-11}$	$1.41\cdot 10^{-5}$	0.4474	0.0426	0.80	330.9
	15	$2.30\cdot 10^{-7}$	$2.04\cdot 10^{-5}$	0.5586	0.0080	0.74	155.9
	30	$2.40\cdot 10^{-8}$	$1.53\cdot 10^{-5}$	0.5366	0.0060	0.76	222.3
	60	$2.89\cdot 10^{-7}$	$1.58\cdot 10^{-5}$	0.6573	0.0105	0.66	152.8

R, solution resistor; R1, polarization resistance; R2, oxide pore resistance; Q, constant phase; C, coating capacitance.

Table 3.
Electrical parameters of equivalent circuit obtained by fitting the experimental results of EIS tests.

proposed by Zhang et al. [9] to describe the bi-layer oxide film formed on aluminum and aluminum corrosion aqueous media.

This circuit is valid for all determinations. In the equivalent circuit, R is the salt water and the broken rice resistance, R_1 is the resistance to polarization, C is the corresponding capacity to the dense oxide layer, R_2 is the resistance in porous oxide position, and Q is the corresponding component of the constant phase to porous oxide positions. The results of the parameters in the equivalent circuit are shown in **Table 3**.

For the recycled aluminum alloy, different resistivity profiles in both media, regardless of the cooking time are observed as the impedance diagrams vary with the immersion time (**Figures 5 and 6**). It shows that parameters in the salt water media decrease in contrast to those in broken rice media for different cooking times up to 60 minutes. This behavior may be associated with physicochemical variations which occurred in the oxide film (alumina) during cooking in the salt water media (penetration of the electrolyte into the oxide layer and hydration of alumina) containing chloride ions. Comparison of the curves (**Figures 5 and 6**) clearly shows that resistivities in the layer alumina developed on the alloy recycled aluminum are higher in the broken rice media than those in the salt water. This could be explained by the presence of a more homogeneous and dense layer for the recycled aluminum in the media and also that of chloride ions in the salt water. Because, the behavior of interface/media is completely different with the latter. The overall behavior is reflected in the impedance diagram by a decrease in

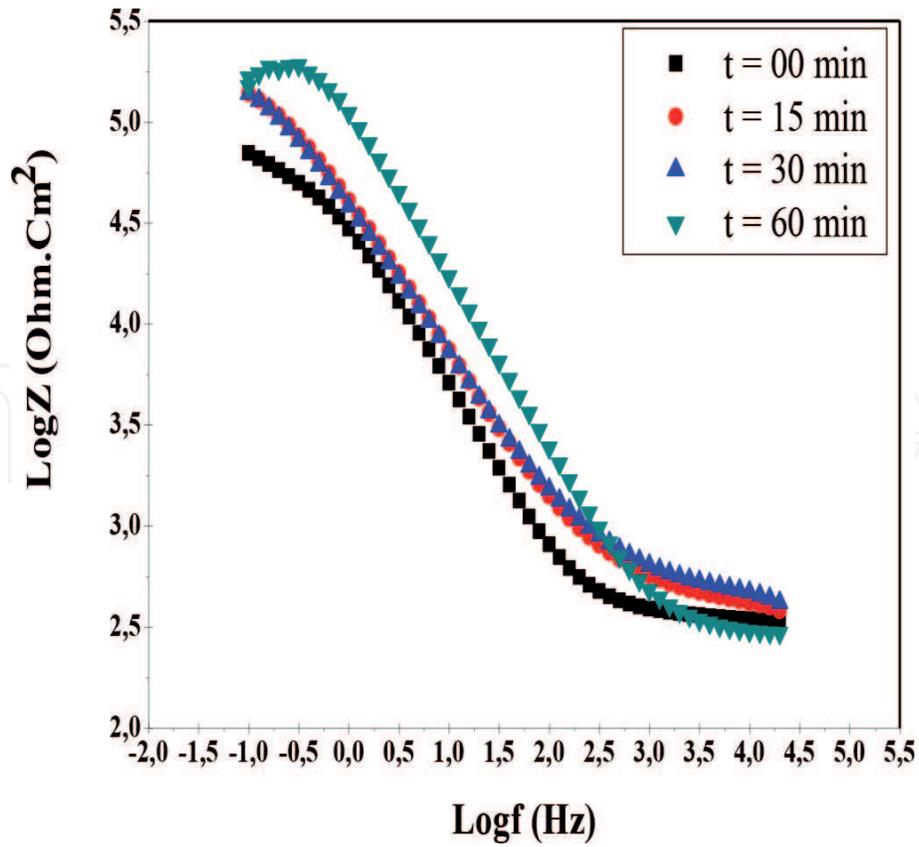


Figure 5.
Bode plots spectra for recycled aluminum alloy tested in broken rice media.

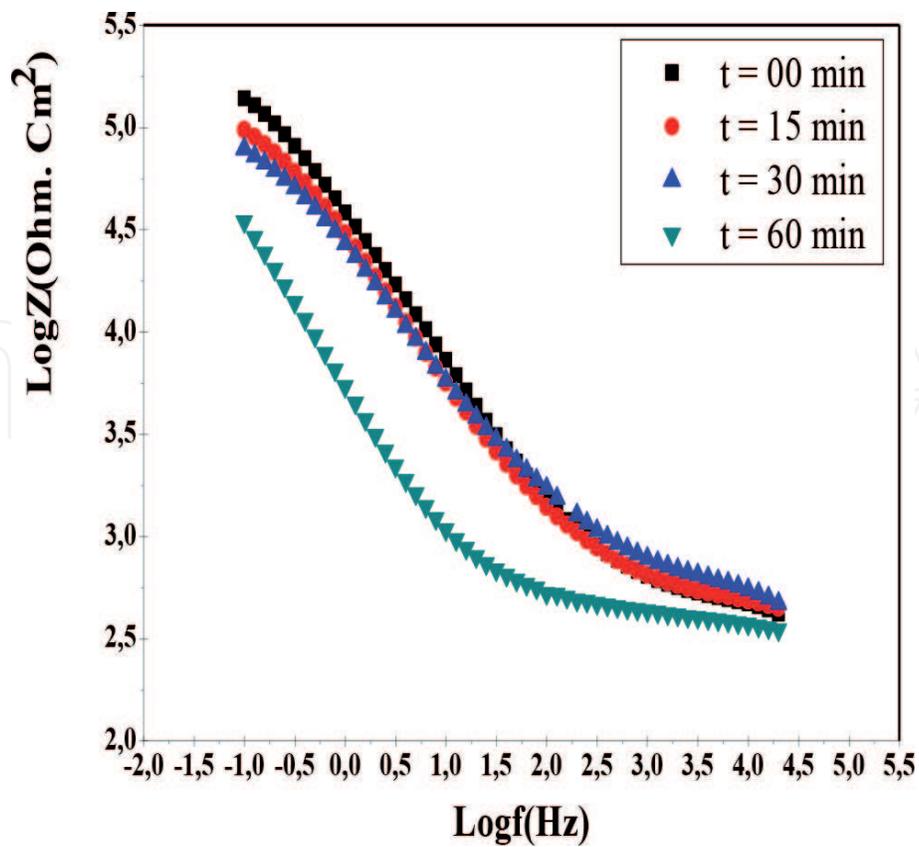


Figure 6.
Bode plots spectra for recycled aluminum alloy tested in salt water media.

size of the capacitive phenomenon. This can be explained by the weakening and destruction of a film which is likely to be developed on the surface of the studied alloy allowing disappearance of the distribution phenomenon and the decrease of the resistance. These differences may be explained by the oxide layer composition developed on the alloy which is influenced by the chemical composition of material solid media and by the chemical composition of the intermetallic particles [22–24].

In conclusion, resistivity profiles obtained for recycled aluminum alloy showed that the oxide layer developed is less protective in the salt water media than the broken rice. This result would be bound to the zinc presence which would return this less resistant system [25–27]. The negative effect of chlorides in the salt water media are presented in **Table 3**. This result was translated by the decrease in the polarization resistance. There also appeared an increase in the capacity associated with the polarization resistance. This increase may reflect the dissolution of the recycled alloy in the salt water media. The polarization resistance stands for the sum of the dense oxide layer resistance and that of the two cooking media (salt water and broken rice) [28]. In this case, R_2 is much larger than R_1 , therefore, it can be considered as the polarization resistance. **Table 3** illustrated the simulation parameters. It shows that the polarization resistance increases gradually with the increased cooking time up to 60 minutes for media broken rice while for the salt water media, a decrease is observed followed by a slight increase. Highest values of the polarization resistance in broken rice media as compared to the salt water can be explained partly by the chemical composition of the recycled alloy capable of modifying the physical and chemical properties of the oxide layer into more or less noble depending on the studied media, and second, by the resistance of the charge transfer (R) which is not identical for both media. **Figure 7** indicated a clear difference between the polarization resistance values from the two cooking media.

Observation **Figure 7** curves show that the sample from the broken rice media is less corroded than that from the salt water media. This confirmed the destructive effect of the salt water media on our sample [29, 30].

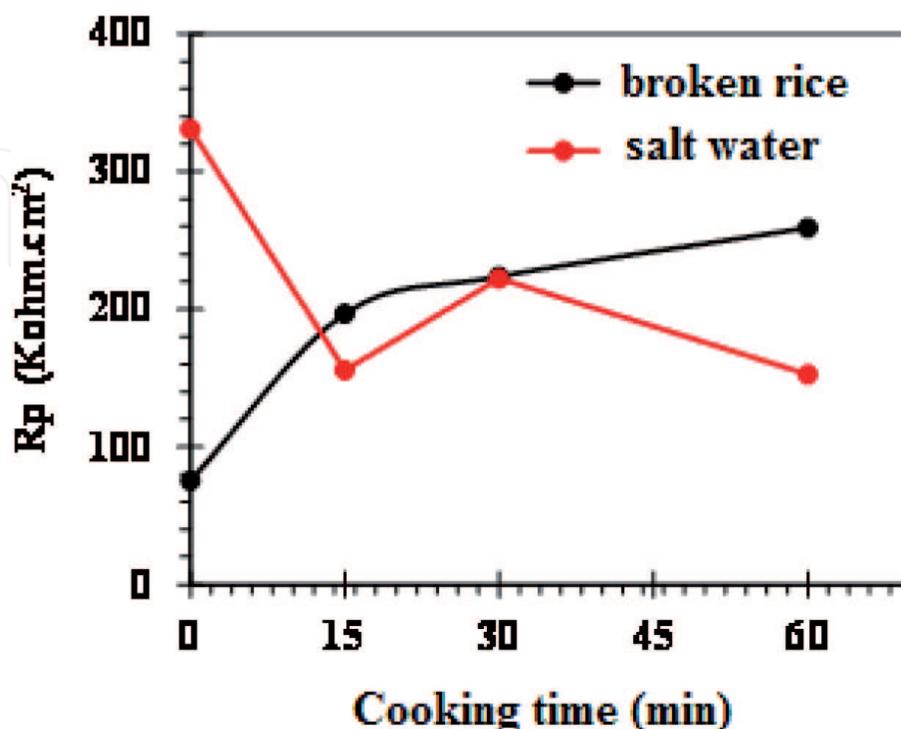


Figure 7.
Polarization resistance according to cooking times.

Sample	Times (min)	WS		WR	
		25°C	100°C	25°C	100°C
D	1440	5,84i		5.24j	
	15		57.85h		52.39g
	30		62.78e		56.82d
	60		70.78a		64.24a
	ddl		3		3
Probability		<0.0001		<0.0001	
Manning		HS		HS	

Results are means of 3 replications; HS = high significant. Test Ryan-Einot-Gabriel-Welsch (REGWQ), the difference is not significant between values added by the same letter in the same line.

Table 4.

Aluminum content measured in various cooking media of sample D after different contact times at room temperature (25°C) and at boiling temperature.

3.4 Influence of media, WS, and WR

Aluminum content released after 30 and 60 minutes in various cooking media is given in **Table 4**.

The same absorbance measured 30 and 60 minutes of cooking duration in the media WS (titrated at 3 g.L⁻¹ of salt) has given more important result in the other media (WR) of study. The high quantity of aluminum in this media has been probably linked to the presence of chloride ion and also to environmental pH. This result is according to the study conducted by Bommersbach and Duggan [31, 32]. Similar increase of aluminum loss with the increase of alcohol-free drinks acidity package in the aluminum bottle. These contents are very comparable with those got using tape water, concentrated tomato, and media WR in the same conditions. Other minerals in the tape water added to chloride ion have a significant influence on aluminum leaching with local kitchen utensils. For media WR important contents of aluminum had been lost in the cooking media after 30–60 minutes in the four local kitchen utensils. These results are similar to those decrypted by some authors [33, 34], in cooking breaking rice (WR) found to be not aggressive operation for sample containing more silicon. Studies showed that concentrated tomatoes caused more effect on cooking utensils [35]. Acidity of this product is so probability equivalent to those of fresh tomatoes, that is surely again a consequence of their origin and mode of production. Contributions of water at room temperature and tomato are so low that aluminum quantities swallowed and are relatively independent form the proportion of rice water. But, toxicity norm by some authors [26, 36] do state of acceptable daily dose to 1 mg by kilogram of body weight for human. This dose is a maximal tolerable quantity by human organism above which aluminum became toxic for him [22, 37]. This simplified outcome showed that we are far from the critical threshold for which human health is in danger. From this study, we can conclude that kitchen cooking utensils in Burkina Faso have not involved in particular toxicological danger.

4. Conclusions

This study contributed to the characterization by electrochemical impedance spectroscopy of the local kitchen utensils used for cooking. From this study, we

conclude that variations of the impedance spectra in Niquist Z diagram based on the cooking time confirms the development of a protective oxide layer (alumina) of this alloys in electrochemical tests, resulting in an increase of the polarization resistance jointly with a decrease in the capacity of the double layer. Electrochemical tests showed a good efficacy of the sample in the broken rice media and having a good resistance to corrosion comparatively to salt water media. The low resistance to corrosion of sample in the salt water media is certainly caused by chloride ions. Susceptibility to corrosion by pitting has been confirmed by the method of electrochemical impedance spectroscopy.

Analysis of two local kitchen utensils with known composition and with various cooking media frequently used in Burkina Faso showed that aluminum content released increases with temperature influence, cooking time, and media. However, insignificant values of aluminum concentration released at room temperature in all solution are may be caused by the short stocking time, may be a decreasing of stocking temperature or another factor not deal with in this study. This study permits to update literature data and must support agribusiness and socio-economic interest of local kitchen utensils made in Burkina Faso according to the area. As precaution to take for limit risk of aluminum migration in foods:

- Avoid using spoil kitchen utensils, aluminum migrate more easily when kitchen utensils are worn;
- Avoid cooking or preserving food in kitchen utensil in aluminum. Food will absorb more aluminum if it is cooked or preserved in kitchen utensil (pan, leaf,...) made in this material;
- Avoid cooking vegetable or acid foods as tomatoes, citrus fruit in aluminum utensil, products absorb more easily this material.

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