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**Chromium(VI) removal from aqueous solutions using powdered coconut shell
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Corrosion Rate of Stainless Steel Tubes Calculated by Electrochemical Frequency Modulation

Đorđe Ž. Petrović, Ksenija R. Kumrić, Marija D. Mirković, Snežana S. Ilić-Stojanović*,
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Introduction

The use of high energy, ionizing radiation in nuclear medicine is now pervasive and routine. The interaction of gamma radiation with aqueous solutions produces different kinds of reducing and oxidizing agents. Reducing agents represent e^-_{aq} , $H\bullet$ and H_2 while oxidizing agents represent H_2O_2 , $\bullet OH$, O_2 , O_2^- and HO_2 [1]. Production of such species under gamma irradiation, there may affect the rates or mechanisms of corrosion attack modes.

In recent times, electrochemical frequency modulation (EFM) has caught the attention of corrosion scientists as a rapid and non-destructive technique for the instantaneous determination of corrosion rate. The advantage of the EFM technique is the fact that the measurement can be completed in a short time period [2]. The EFM technique offers an excellent [alternative](#) for the analysis of electrochemical corrosion behavior of metals compared with contemporary techniques like potentiodynamic polarization, linear polarization, electrochemical impedance spectroscopy and weight loss. This technique provides measurements of corrosion rate directly without knowledge of the Tafel constants. Even though the Tafel constants are not required, it measures them and a change in Tafel constant may indicate a change in corrosion mechanism. The theory behind the technique is the same Butler-Volmer kinetics that underpins all corrosion rate measurements. It is a small signal ac technique where two sine waves (at different frequencies) are applied to the cell simultaneously.

In our work, we used the EFM technique to calculate the corrosion rate on stainless steel samples in saline and under gamma radiation.

Methods

Stainless steel tubes used in this study were AISI 304 purchased from different companies. Their composition was determined by x-ray fluorescence spectrometer for elemental analysis Analyticon Instruments gmbh XL3-101210. Three types of tubes with dimensions of ϕ 0.5 mm and 100 mm length were prepared for the electrochemical tests.

Prior to each measurement, the samples were ground using a carbide emery papers ranging 600 grit size, subsequently rinsed by bidistilled water and acetone, and finally dried at room temperature.

Electrochemical measurements were performed using a Gamry potentiostat/galvanostat model 750 ZRA.

Electrochemical experiments were carried out at room temperature in saline purchased from Hemofarm A.D, in an electrolytic cell with an AgCl reference electrode tip placed

close to the working electrode to minimize ohmic resistance. The working electrode was a stainless steel tube while the platinum plate (20 mm x 80mm) was a counter electrode. After immersion in bidistilled water and saline for ten days samples have been analyzed. The second group of samples was tested after irradiation by gamma rays obtained by ^{60}Co in bidistilled water and saline. The delivered dose of the radioactive source was 25 kGy. Samples immersed in saline for ten days were marked with Roman numerals and with letter S (S I, S II, and S III) (Table 1); while the samples that were aged in saline and additionally irradiated were marked with Roman numerals and the letters FG (S I-FG, S II-FG, and S III-FG).

The EFM data have been analyzed using the Gamry Echem Analyst software v561.

Table 1. Composition (wt.%) of different 304 SS tubes from x-ray fluorescence spectrometer for elemental analysis Analyticon Instruments gmbh XL3-101210

Sample	Element / wt.%								
	Mo	Cu	Ni	Mn	Cr	V	Si	P	Fe
S I	0.11	0.19	8,04	1.10	17.56	0.18	0.40		72.39
S II			7.85	1.28	18.40		0.53	0.053	71.831
S III	0.06		9.12	1.63	17.48		0.58		71.04

Results and discussion

The response to the appropriate excitation of a material with a voltage of the appropriate frequency is a graph of the change in current in a given time interval, as shown in Figure 1. EFM chart in Figure 1 shows the I vs. t chart for all of the data collected. The number of cycles (16) is chosen that it is neither small due to uncertainty nor large so that the measurement does not take too long. The maximum number of cycles with a given device is 255. For the EFM analysis, the data from the first cycle is not used but the device itself adds this number given that it should not be taken into account when setting the parameters. A frequency spectrum of an EFM current response, illustrating the different harmonic and intermodulation frequencies, is depicted in Figure 2.

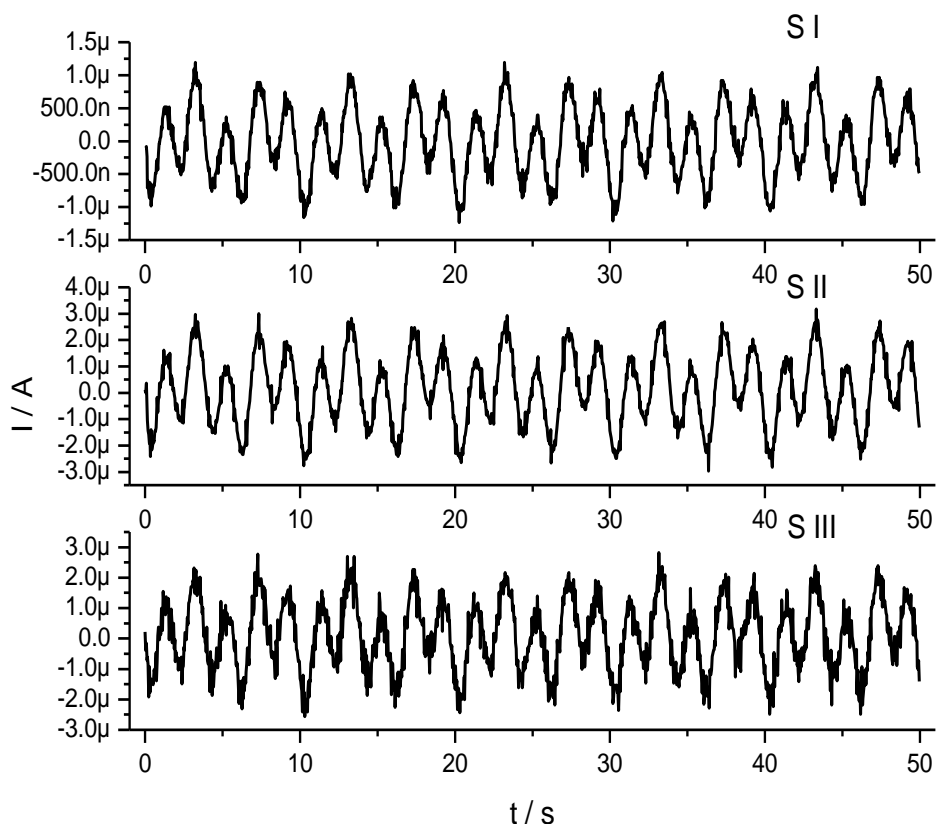


Figure 1. Current I vs. time t for sample I, II, and III

The baseline frequency was 0.1 Hz with four cycles, the multipliers were 2 and 5 and the amplitude was 10 mV. Harmonic current responses at ω_1 , $2\omega_1$, $3\omega_1$ and ω_2 , $2\omega_2$, $3\omega_2$ as well as intermodulation frequencies like $2\omega_1 \pm \omega_2$ and $2\omega_2 \pm \omega_1$ caused that bandwidth Δf of the EFM measurement was $0.1 < \Delta f \text{ (Hz)} < 1.5$.

The currents obtained at certain frequencies such as 0.2 Hz and 0.5 Hz as well as 0.3 Hz and 0.7 Hz have equal values, which agree with the claims Bosch et al. can be seen in Figures 2 and 3 [3].

The chosen frequencies were considered a reasonable compromise of some arguments. The harmonics and intermodulation frequencies should not influence each other and the frequency should be as low as possible to avoid the influence of the capacitive behavior of the electrochemical double layer. Also, the frequency should be as large as possible to reduce the time needed to accomplish a measurement.

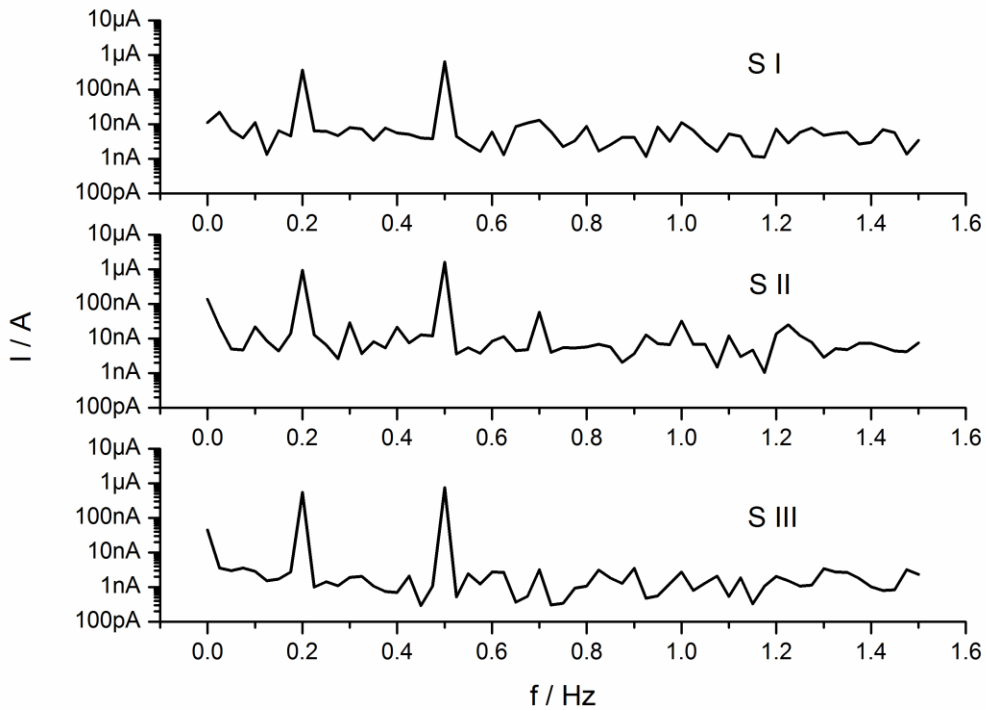


Figure 2. Current I vs. frequency f plot for samples I, II, and III.

Figure 2 shows results obtained from samples that were immersed in saline for ten days and analysed using a perturbation signal with an amplitude of 10 mV for both frequencies. On sample S II two peaks can be seen at 0.3 Hz and 0.7 Hz which are missing in the other two samples. In the S II-FG sample, four additional peaks appear compared to the S I-FG and S III-FG samples. These peaks are positioned at 0.3 Hz, 0.4 Hz, 0.7 Hz and 1 Hz. From the given figures 2 and 3, it can be seen that sample S II is more susceptible to corrosion and much more if it is exposed to chloride ions and gamma radiation.

Assuming that the cathodic reaction is under diffusion control the results of the analysis are given in Figure 4. In Figure 4, the corrosion rate of samples S I, S II, and S III under various influences of the corrosive environment is shown.

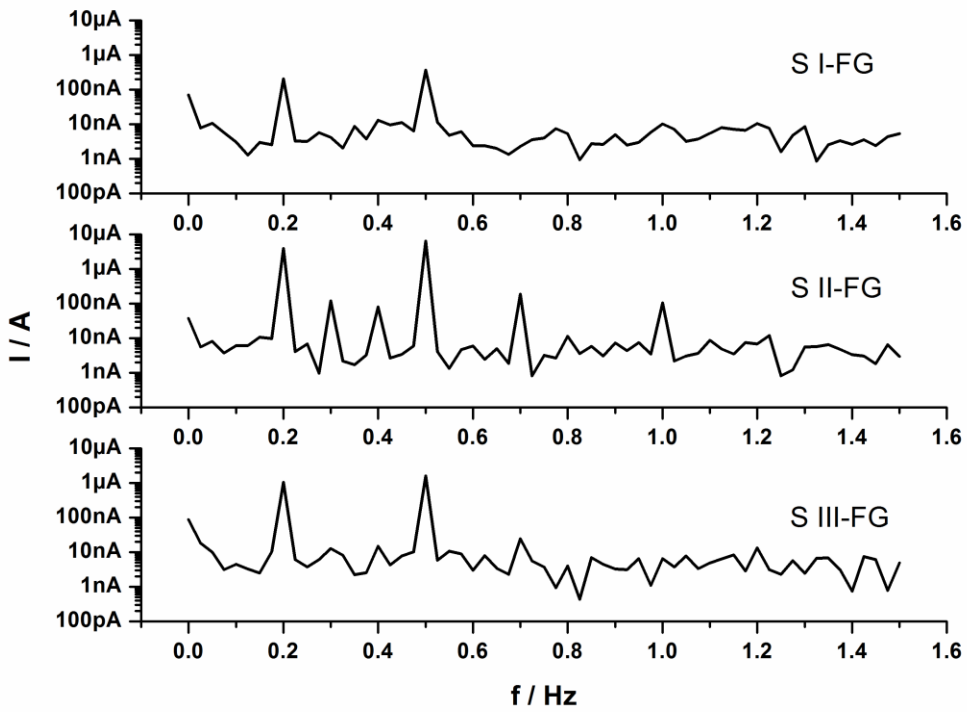


Figure 3. Current I vs. frequency f plot for samples I, II, and III.

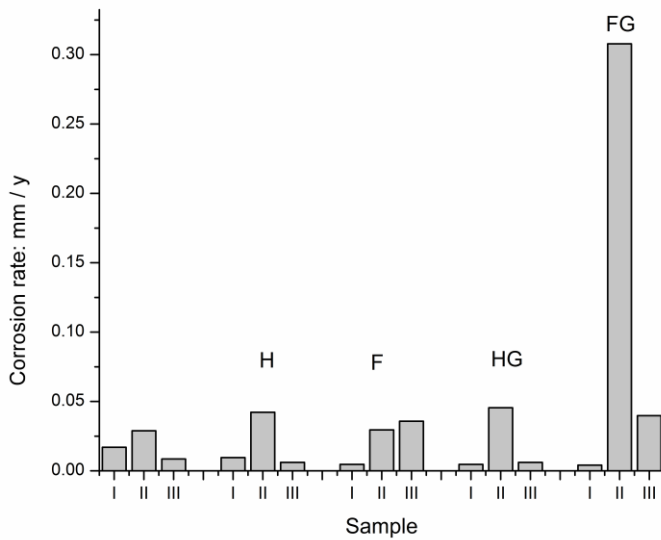


Figure 4. Corrosion rate in mm/yr for samples S I, S II and S III under various influences of the corrosive environment (the S mark has been omitted for clarity).

The sample marked as S II shows a significant influence of chloride ions on corrosion rate but a dramatic increase in the corrosion rate can be seen when sample S II simultaneously was immersed in saline and irradiated with gamma rays.

The composition of tubes determined by x-ray fluorescence spectrometer is presented in table 1. It is obvious that the content of macroelements in all samples is similar, but sample S II shows a lack of some elements, such as Mo, Cu and V. Also, this sample contains a small amount of phosphorus, while the two other samples do not have the same element.

Conclusion

A corrosive environment containing chloride ions is detrimental to materials such as stainless steel. A slight increase in the corrosion rate in material immersed in saline occurs due to a low concentration of NaCl in the solution. Gamma irradiation increases the oxidizing nature of the aqueous solutions used in this study through the production of H_2O_2 , $\bullet OH$, O_2 , O_2^- and HO_2 species which with Cl^- account for the observed positive corrosion potential shift for sample S II. The lack of certain elements like Mo in stainless steel, as in the case with sample S II (Table 1), can result in reduced corrosion resistance of materials.

Acknowledgements

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Brzina korozije cevi od nerđajućih čelika izračunata pomoću elektrohemijske frekvencione modulacije

Nerđajući čelici su omiljeni materijali u nuklearnoj medicini zbog jednostavnog održavanja (brisanje, dekontaminacija, itd.). Otpornost na rđanje ovih materijala je smanjena usled istovremenog delovanja hloridnih jona i jonizujućeg zračenja. Brza i nedestruktivna tehnika merjenja korozije, kao što je elektrohemijska frekvenciona modulacija (EFM), korisna je za brzu procenu materijala koji moraju biti otporni na rđanje. Tri različite cevi od nerđajućeg čelika su analizirane pomoću EFM metode i Gamry potencioštata / galvanostata, i izračunate su njihove brzine korozije. Uzorak označen kao S II, koji ne sadrži Mo u svom sastavu pokazuje veću brzinu korozije u odnosu na uzorke koji sadrže Mo ako je istovremeno izložen hloridnim jonima i gama zračenju. Ovaj rezultat je u sagalsnosti sa sastavom nerđajućeg čelika dobijenim rentgenskim fluorescentnim spektrometrom koji pokazuje nedostatak bakra i vanadijuma u tragovima, uključujući molibden.

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