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# Characterization of corrosion inhibition performance of azole compounds through power spectral density of electrochemical noise



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## ABSTRACT

The power spectral density was focused to analyze the electrochemical noise data of stainless steel samples dipped in 1 M  $H_2SO_4$  solution without and with different concentrations of benzotriazole and benzothiazole corrosion inhibitors. Accordingly, a good correlation was observed between corrosion intensity and PSD (*I*) and PSD (*V*) distributions calculated for each frequency point as a percentage of total energy, over the frequency domain. In addition to indicating the inevitable role of PSD distribution in determining characteristic charge from shot noise theory, an approach was developed to choose minimum frequency range to calculate *q*, in particular for the systems with different types of PSD distributions.

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# 1. Introduction

The corrosion inhibition mechanisms of different compounds have been already studied through taking advantage of several electrochemical techniques. Common electrochemical tools, in particular EIS and potentiodynamic polarization methods, have been widely employed to provide a better understanding of inhibition performance [1–3]. In the recent decade, an increasing interest toward applying brand new techniques such as electrochemical noise method (EN) among corrosion researchers could be observed. Although this technique has been used in corrosion monitoring [4,5], evaluation of localized corrosion [6,7], investigation of organic coatings [8–10], and evaluation of inorganic anticorrosive pigments [11], few publications could be found studying the behavior of organic inhibitors in acid solutions using EN [12,13].

Measurement of spontaneous current and voltage fluctuations occurring on the corroding surfaces which is known as electrochemical noise (EN) [14,15] has several advantages [16,17]: measurements are easy to take and application is non-intrusive, i.e. there is no need to apply external perturbation leading to minimal interference with the system. In general, two approaches namely sequence independent analysis methods and sequence dependent analysis methods, are used to interpret EN data [18]. According to literature, the noise resistance ( $R_n$ ) obtained through dividing the standard deviation of potential by the standard deviation of current ( $\sigma v/\sigma I$ ) as a sequence independent analysis method and power spectral density (PSD) plots as a sequence dependent analysis method are extensively used to interpret the time records and frequency domain parameters, respectively [18]. In order to perform PSD analysis, the time domain signal should be first transferred to the frequency domain using maximum entropy method (MEM) or fast Fourier transform (FFT) [19,20]. Being a measure of energy distribution of the signal under study over frequency domain, the resulted PSD may facilitate calculation of the characteristic charge and frequency from shot noise theory.

The present work intends to study the corrosion behavior of stainless steel samples immersed in 1 M H<sub>2</sub>SO<sub>4</sub> solutions with benzotriazole (BTR) and benzothiazole (BNS) as organic inhibitors through considering the PSD (*I*) and PSD (*V*) distribution over frequency domain. The organic molecules containing  $\pi$  bonds, aromatic rings, and heteroatoms such as oxygen, nitrogen and sulfur are commonly used to protect metals against corrosion in acidic solutions [21–23]. It is believed that the corrosion inhibition may happen due to formation of a protective layer on the metal surface, blocking the active sites on the surface [24–26]. In addition, the impact of PSD distribution on calculation of characteristic charge as a parameter derived from shot noise theory is aimed to be investigated. To top it off, the paper tries to develop an approach to choose minimum frequency domain from which the characteristic charge would be calculated.

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# 2. Experimental

# 2.1. Materials

Fig. 1 shows the chemical structure of inhibitors used in this research. The compounds in analytical grade were purchased from Merck and used without further purification. Prior to perform electrochemical noise measurements, panels of stainless steel 304L supplied by Tianjin TISCO and TPCO stainless steel co. Ltd, (Composition wt.%: C: 0.04, P: 0.08, Si: 0.75, Ni: 8, Cr: 18, Mn: 2, N: 0.1, S: 0.8 and Fe: balance) were abraded with abrasive papers starting from 80 up to 2000 grit size. The samples were then rinsed with distilled water and dried in the air, followed by acetone degreasing. One surface of the samples was connected to a copper wire for electrical connection. To seal the edges and back sides of the steel panels, they were covered with a beeswax-colophony mixture, leaving an area of 1 cm<sup>2</sup> unmasked. Electrochemical noise measurements were carried out on the stainless steel panels exposed to 1 M H<sub>2</sub>SO<sub>4</sub> solutions containing 0, 200, 300 and 400 ppm (weight/volume) of benzotriazole (BTR) and benzothiazole (BNS) as corrosion inhibitors at 25 °C ± 2 °C without deaeration.

# 2.2. Methods

Electrochemical potential and current noise were simultaneously measured in a freely corroding system using two nominally identical working electrodes of the same area and a saturated Ag/AgCl reference electrode which was placed between the two working electrodes (Fig. 2). During the electrochemical measurements, the cell was placed in a Faradic cage to minimize possible external electromagnetic interference. The electrochemical noise data was gathered within a period of 1024 s at 1-s interval, which led to a frequency range from close to 1 mHz to 0.5 Hz determined by the expressions  $f_{\text{max}} = 1/2\Delta t$  and  $f_{\text{min}} = 1/N\Delta t$  where  $\Delta t$  and N are the sample interval and the total number of data recorded, respectively. EN measurements were carried out with an Autolab instrument model PGstat 302N containing a noise module with the input range of ±2.5 V, maximum potential resolution of 760 nV and potential accuracy of 300 µV. All data analysis was performed using Nova 1.8 software.

#### 3. Results and discussion

Since previous publication [27] reported a good correlation between the inhibition efficiency and noise resistance,  $R_n$  was focused to assess function of the corrosion inhibitors used in this work. To calculate  $R_n$  properly, the DC trend should be first removed from the electrochemical noise data in a manner that the transients would neither be over-filtered nor be twisted with artificial drifts [27–29]. One of the effective ways to remove DC trend is to subtract a local average from the raw data through moving average removal (MAR) method [30]. In case of a recorded time-potential series consisting of *K* data points, Eq. (1) could be used to remove the DC trend of any data point *i* [12]:

$$V_{i,real} = V_{i,obs} - V_{i,DC} \tag{1}$$



Fig. 1. Chemical structure of (a) benzotriazole and (b) benzothiazole.



Fig. 2. Electrochemical cell used to perform noise measurements.

where *V* is potential and subscripts *obs*, real and *DC* refer to observed fluctuation, real noise fluctuation and DC trend of data point *i*, respectively. In MAR method the *DC* trend component,  $V_{i,DC}$ , is calculated using Eq. (2), where *p* is a natural number.

$$V_{i,DC} = \left(\frac{\sum_{i-p}^{i+p+1} V_{i,obs}}{2p+2}\right)$$
(2)

When the noise resistance is being calculated, the *p* value should be taken appropriately to assure the accuracy and reliability of the data. Although some researchers arbitrarily took p = 3 to remove the DC trend [12,31], Liu et al. [29] showed that too low *p* values could lead to over-filtering of transients. Accordingly, Eq. (3) was proposed to calculate appropriate *p* value involving in MAR method for DC trend removal, where  $\Delta t$  is the data sampling interval [28].

$$p = \frac{32}{\Delta t} \tag{3}$$



Fig. 3. Typical time records of electrochemical current noise of stainless steel samples immersed in  $1 \text{ M} \text{ H}_2\text{SO}_4$  solution with no inhibitor (a) before and (b) after DC trend removal.

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