



DEVELOPMENT AND EVALUATION OF RESPIROMETRIC IN SITU CORROSION MONITORING SYSTEM

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ABSTRACT: *There is a growing need for a system to be used for real-time in-situ monitoring of corrosion rates. This system/method allows the determination of real-time corrosion rates under realistic exposure conditions and is capable of following changing exposure conditions in situ. This is realized by a combination of optical Oxygen sensor measurements with either gravimetric volume sensitive techniques or pressure sensor based techniques in a closed chamber. This study was therefore aimed at developing and evaluating a low-cost, real-time corrosion monitoring system, using copper (Cu) as a test sample. Materials used were sourced locally, the circuitry was designed and used to develop the system with incorporation of sensors that can monitor temperature, humidity and pressure within an airtight glass bottle and placed in a housing which was fabricated. The developed system was then evaluated using a piece of Cu exposed to 5% Sodium Chloride (NaCl). Hydrogen evolution reaction (HER) and Oxygen Reduction Reaction (ORR) within the closed chamber were monitored; and thus Ideal gas and Henry laws were adopted to calculate the amount of gas molecules, and convert them to cathodic reactions. The methods were carried out in accordance with existing literature and standard procedure. Results of evaluation of the system showed that, the more Oxygen is being consumed, the higher the corrosion. Mass loss validation measurements carried out at the end of exposure showed a good correlation with the total recorded cathodic charge. Immersion corrosion kinetics can be monitored non-destructively and in real-time. Manometric approach showed that HER leads to a pressure increase while ORR leads to a decrease in pressure. ORR monitoring is possible based on the amount of consumed O₂ by manometric and sensor-based approaches; sensitive, non-destructive corrosion rate measurements are possible on Cu and could be monitored remotely.*

KEYWORDS: Respirometric, Corrosion, Kinetic, Monitoring, Static Manometric.



INTRODUCTION

Corrosion is the destructive attack of a metal by chemical or electrochemical reaction with its environment. Deterioration by physical causes is not called corrosion, but is described as erosion, galling, or wear. For many years, huge attention has been paid to the problem of corrosion damage and destruction of metallic materials. Experience shows that failures due to corrosion problems are very important, and statistics at the world level shows that the damage resulting from the effects of various forms of corrosion is substantial and that, for example, in industrialized countries it reaches 4-5% of national incomes. Significant funds are determined annually for the prevention and control of corrosion. In the case of ignoring the problem of corrosion, in some cases the penalty is financial, whereas in other cases it is the loss of human lives. For metal infrastructure elements, corrosion negatively affects their durability and operability. It is reported that corrosion is a dominant form of defects with 42% of frequency of failure mechanisms in engineering structures (Petrovic, 2016).

Monitoring and detection of corrosion is useful for plant engineers and maintenance personnel to replace the corroded parts in petrochemical industries only after damage has occurred. On the basis of recorded data, observers can predict at how much rate the corrosion should occur. Common methods used for corrosion monitoring and detection are destructive and non-destructive. In non-destructive methods, ultrasonic guided wave, radiographic and electromagnetic and in destructive methods corrosion coupons, electrical resistance and linear polarization resistance are included (Kansara *et al.*, 2018).

Corrosion also refers to the loss of mass of metallic material as a function of time through interaction with the environment. The loss of mass may be uniform over the material surface or highly localized as in crevice and pitting corrosion (Buchanan & Stansbury, 2012). Corrosion can be viewed as a universal phenomenon which is bound to occur everywhere in our environment. It is an irreversible interfacial reaction of a material (metal, ceramic, and polymer) with its environment which results in its consumption or dissolution into the material of a component of the environment.

Presently, there are several methods for determining corrosion rate of metals, such as gravitational-base mass loss (weight-loss measurement), quartz crystal microbalance-based mass loss, electrochemical, electric resistance and solution analysis (Badea *et al.*, 2010). Zou *et al.* (2011) stated in their paper that the most direct and reliable method of measuring corrosion rate is the weight-loss measurement but it is time consuming and does not give enough information i.e its information limited. According to Shaw and Kelly as stated by Mastoor and Authority (2015), electrochemical measurement happens to be fast and sensitive and can give corrosion measurement in minutes or hours. There are various electrochemical methods used to determine corrosion rate. Among them are polarization curves, polarization resistance transient and electrochemical impedance techniques (Mastoor & Authority, 2015). These methods make use of a device called potentiostat for the purpose of current input, voltage and current measurement required to generate data. The polarization measurement methods such as in-situ respirometric, based on electrochemical concepts, enable determination of instantaneous reaction rates at electrode/solution interface, such as the exchange current density in case of electrode equilibrium and the corrosion current density in case of corrosion system, from a single experiment (Kansara *et al.*, 2018). All other methods require multiple measurements over time to provide information on the corrosion rate.



The key advantage to implementing corrosion monitoring as applied by this study is to detect early warning signs of corrosion and to determine trends and processing parameters that may induce a corrosion environment. This study therefore engages different types of sensors to measure and monitor different conditions (environmental conditions) that affect and cause corrosion such as temperature, pressure and relative humidity. It was noted that when the potential of the metal surface is polarized due to the effect of the environment, the application of the current in a positive direction is said to be anodically polarized, a negative direction signifies it is cathodically polarized. The degree of polarization is a measure of how the rates of the anodic and cathodic reactions are retarded by various environmental such as concentration of metal ions, dissolved oxygen in solution, and/or surface process (absorption, film formation, ease of release electrons, e.t.c) factors. The former is called concentration polarization and the latter is called activation polarization. The variation of potential as a function of current (a polarization curve) allows one to study the effect of concentration and activation processes on the rate at which anodic or cathodic reactions can give up or accept electrons.

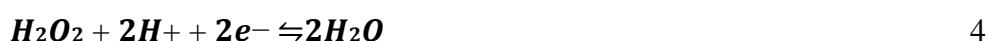
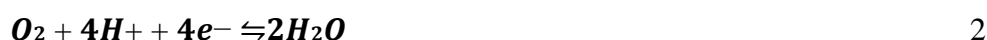
The respirometric method used in this study presented electrochemical techniques by immersion and is applicable to any metallic sample. In the field of atmospheric corrosion, Stratmann *et al.* (1990) used a differential pressure sensor to monitor the pressure decrease due to oxygen consumption of a corroding iron sample. Matthiesen (2007) measured the consumption of O₂ due to the degradation of cultural heritage materials with an optical O₂ sensor in closed vials, flexible gas-tight bags and in situ on larger artifacts. Hydrogen evolution measurements are more common in corrosion studies, especially for immersion conditions (Curioni, 2014).

According to Strebl *et al.* (2020), atmospheric corrosion under thin electrolyte films is one of the most important but also most complex corrosion scenarios. The factors to be considered are e.g. contamination by hygroscopic particles, gas composition of the atmosphere, electrolyte film thickness, surface conditions, temperature and the influence of wet-dry cycles. As a result, lab tests or accelerated corrosion tests often fail to predict the corrosion behavior found in the real world. The determination of reliable corrosion rates under different exposure conditions in laboratory experiments is a key prerequisite for gaining insight into the corrosion mechanisms and for the development of better accelerated tests. However, the existing methods to measure corrosion rates during atmospheric exposure come with several limitations.

The idea of the respirometric methods is to monitor both reactions (HER and ORR) through the changes noticed in the number of gas molecules within the system. In ORR, O₂ gas is consumed, while HER is accompanied by the generation of gaseous H₂ molecules. An analogous approach is widely used in the studies of metabolic processes like respiration of animals, plants, cells, or microorganisms. The term respirometry describes techniques to assess the rate of a metabolic process in terms of the uptake rate of an electron acceptor (e.g. O₂, nitrate) or in terms of the production rate of its reduced form (e.g. methane in the case of anaerobic respiration). In a corrosion process, the electron acceptor is usually either O₂ or H₂O and it is more convenient to monitor the reduced form of water, namely H₂ (Hölzl, 2018). Although it is obvious that monitoring the cathodic reaction rate provides a direct, non-destructive measurement of the corrosion rate, there are only few examples where this approach was used in corrosion science (Cáceres, 2007).



Fajardo and Frankel (2015) showed that measuring H₂ evolution rates can be used to monitor atmospheric corrosion rates of Mg alloys in real-time. Electrical resistance sensors have been developed to monitor the rate of reduction of the cross section of a metal track by corrosion. The ability to monitor real-time corrosion rates in the field and during accelerated testing (Prosek, 2014) makes this approach very promising. Possible drawbacks of the resistance method lie in the conductive electrolyte film or inhomogeneous corrosion of the sensitive track that could influence the measured resistance (Steen, 2017). The corrosion behavior of the narrow metal track could differ from a bulk sample and could also depend on the microstructure resulting from the deposition method of the track. The sensors are commercially available only for a limited range of substrates. To monitor the rate of the cathodic reactions, according to mixed-potential theory, the rate of the oxidation reactions and hence the corrosion rate is directly related with the rate of the cathodic reduction reactions. The main cathodic reactions in aqueous corrosion are the O₂ reduction reaction (ORR, 1) and the H₂ evolution reaction (HER, 2).



MATERIALS AND METHODS

Materials and Equipment

Table 1 presents a list of the elements, components, items, various tools, equipment and machines used for this study. The materials were sourced locally and procured from different dealers in Ibadan, Oyo State, Nigeria. All components, element and items were coupled together at the Fabrication Workshop (Metal Shop) of the Department of Physics, University of Ibadan, Ibadan while all the tools and equipments were also available at the said workshop.

Table 1: List of materials and equipment

I	Purpose
Arduino microcontroller	Interfacing between sensors and computer
ESP32	This collects data and send to cloud
Transparent glass tight chamber (Aspirator bottle)	For electrochemical cell (storing electrolyte)
Pressure gauge	To monitor increase and decrease in pressure
Digital weighing scale	To measure the weight of samples
Oxygen Sensor	Measuring Oxygen reduction within the system
Pressure sensor	Partial pressure measurement
Temperature sensor	Temperature measurement



Epoxy syringe	To protect all the sensor from possible condensation
Teflon Nylon	To check the linkages in the aspirator bottle
Relative humidity sensor	To protect sensors from possible condensation
USB cable	To measure RH/water vapor
Saturated salt solution	To collect the equipment with the computer
16 bit ADC (ADS1116)	To control relative humidity
	ADS1115 is a precision analog-to-digital converter [ADC] with 16 bits of resolution offered in an ultra-small, leadless QFN-10 package.
Bread board	Wire joint
Bolt and nut	Fastening
Electrolyte	For hydration, conducting electricity
Metal alloys (Mill steel)	To use for testing corrosion rate
Perspex	To house all the apparatus
Screw	For connect of joint
Gum	To join and smooth finished all Perspex angle
Drilling machine	Drilling of holes
Screw driver	Fastening
Hark saw	For cutting of Perspex
Brush	Removal of dust and diet
Grinding machine	To redress to surface
	All the materials listed above was sourced for locally

Design, development and setup of the system

The respirometric in-situ corrosion monitoring system was designed and constructed as a simple laboratory instrument for real-time corrosion monitoring. The design, construction and evaluation was done in accordance with International Electrotechnical Commission (IEC) and Strebl (2019, 2020 and 2021). Proteus software was used for circuitry design and simulation (Figure 1). The voltages in the system were between 3.5 to 5 volts. Two different microcontrollers were used. The first one is known as ESP32. This is a low-cost, low-power system on a chip microcontrollers with integrated Wi-Fi and dual-mode Bluetooth. The second microcontroller consists of a physical programmable circuit board and a piece of software or Integrated Development Environment that runs on the computer, to write and upload computer code to the physical board.

To measure the pressure, temperature and relative humidity within the system, several sensors were used in conjunction with the microcontroller. The 16 bit ADC assists in converting analog signal from the circuit to digital signal, which is easily processed by the microcontroller. On the other hand, digital signal from the microcontroller is converted to analog signal by the 12 bit DAC which is then transferred to the circuit. To assess the environmental conditions, a BME 280 temperature, relative sensor combined and pressure sensor were used. These sensors monitored the activities regarding the rise and fall of temperature, partial pressure and RH within this closed established system.

Zirconia O₂ sensor was used for the measurement of partial oxygen pressure at liquid phase. The sensor was fixed to the base of the aspirator bottle. Cathode and an anode were submerged in an electrolyte. Once oxygen enters the device, it is reduced at the cathode, and

an electric current is generated. The Arduino microcontroller was used to collect information from the BME280 sensor, pressure sensor and the zirconia O₂ sensor, and the results were returned in a comma separated format. The microcontroller was connected to a computer and the Cool Term terminal software (telemetry) was used to convert the data to a text file. In addition, a live plotter was developed that enables one to analyze and visualize the data in a real-time graph both on the computer and anywhere through ThingSpeak (MathWorks) over the internet. To avoid data loss, an SD card was also used with the system to capture the data, record them even when ESP32 has no internet to send information to the cloud.

The housing was constructed with Perspex material, and screwed together (Figure 2). The construction was done in the Metal Workshop of the Department of Physics, University of Ibadan.

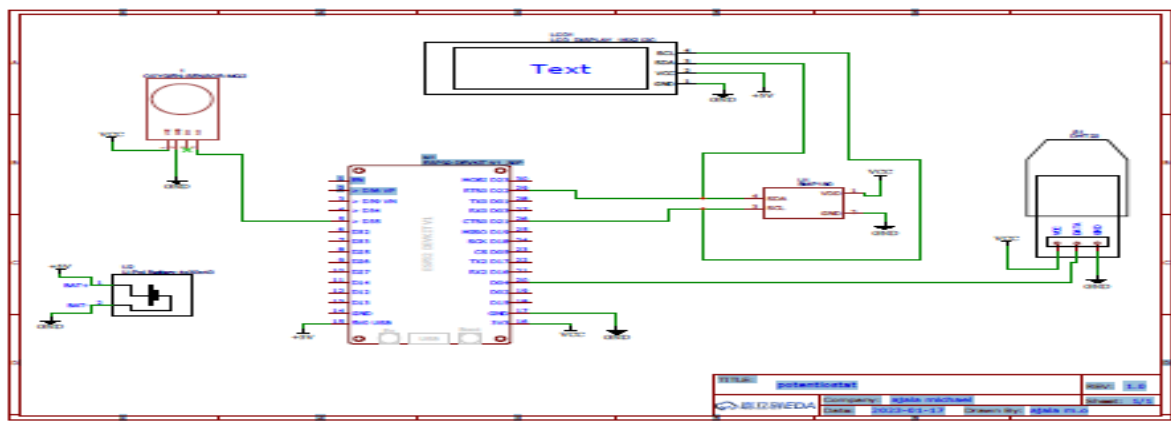


Figure 1: Circuit diagram for corrosion monitoring system

Evaluation of the System

The final assemblage and evaluation of the developed system were done at the Metal Workshop of the Department of Physics, University of Ibadan. Copper was used as a test sample with which the performance of the developed system was evaluated. The metal (Cu) was cut to 100mm by 15mm with 2mm thickness. The pure metal was then exposed to 5% NaCl, exposed and dipped into electrolyte that was kept in an airtight glass bottle (Plate 1). Observation of the system/setup was subsequently made.



Figure 2: Members housing

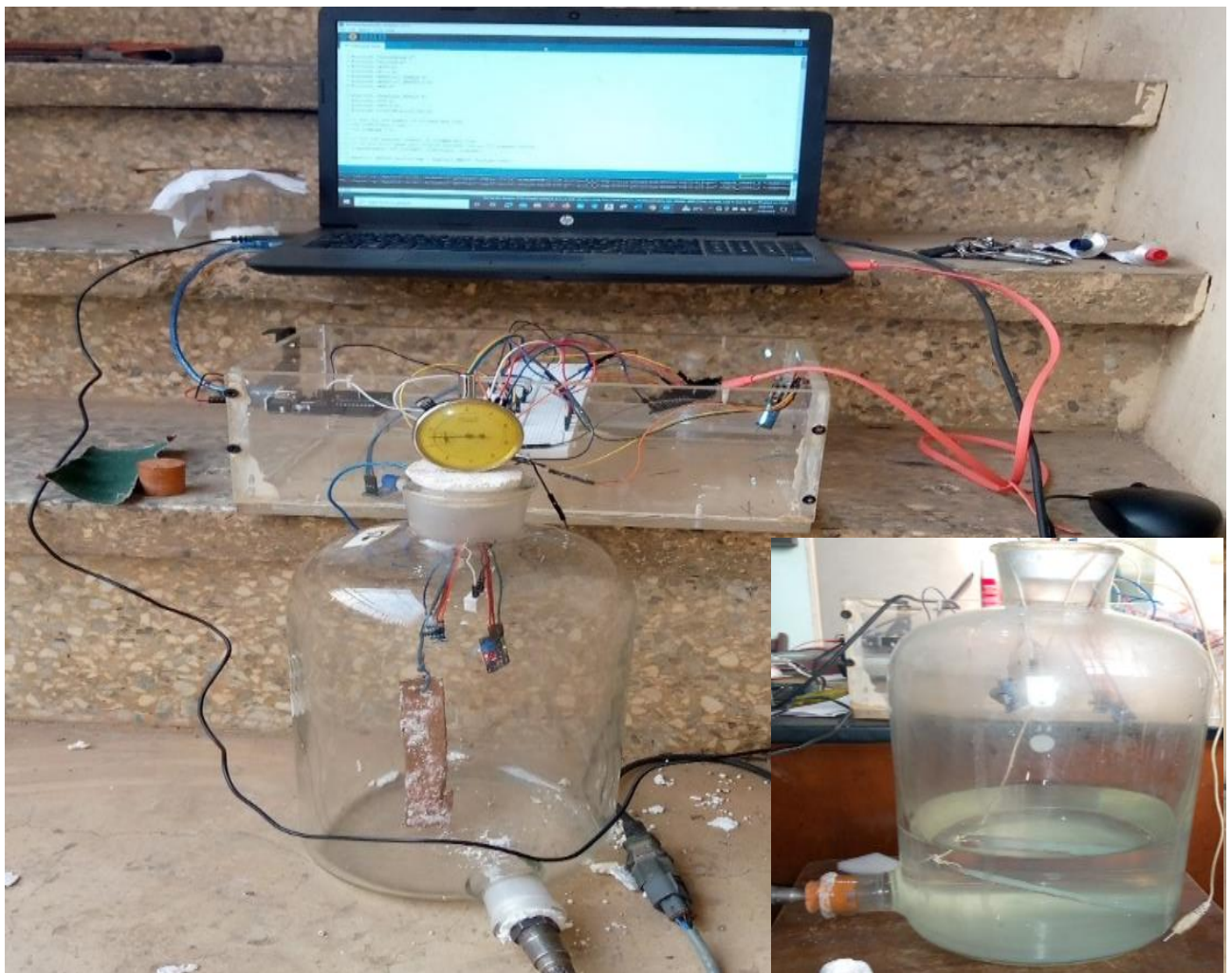


Plate 1: Complete static manometric setup



In order to evaluate corrosion, Hydrogen Evolution Reaction (HER) and Oxygen Reduction Reaction (ORR) is the main reaction that causes corrosion. Therefore, for this study, Ideal gas law (Equation 1) was applied to estimate the amount of ORR and HER in the designed system;

$$PV = nRT \quad (1)$$

$$n = \frac{PV}{RT} \quad (2)$$

Where, n is the total number of molecules.

Henry Law states that the amount of gas that dissolved in a liquid is directly proportional to the partial pressure of that gas above the liquid when the temperature is kept constant. Therefore for the evaluation of the evolved gasses in this study, Henry Law (Equation 3) was applied.

$$C = KP \quad (3)$$

The Ideal gas equation (1) was also used to calculate the amount of substance associated with the volume changes or the pressure changes due to HER and ORR. In a first step, the change in the total number of gas molecules was calculated from the change in total pressure $\Delta p(t)$ for manometric setup:

$$\Delta n_{tot}(t) = \frac{\Delta p(t) \cdot \Delta V(t)}{R \cdot T(t)} \quad (4)$$

Where, R is the Ideal gas constant and $T(t)$ is the temperature of the gas.

Changes in the number of O_2 molecules were also obtained by the changes in the O_2 partial pressure Δp_{O_2} from the O_2 sensor, accordingly (Equation 5).

$$\Delta n_{O_2}^{optical} = \frac{\Delta p_{O_2} \cdot V(t)}{RT(t)} \quad (5)$$

Manometric measurement of temperature was carried out by the BME280 Sensor inside the chamber and humidity will be measured in situ by the RH sensor. This allows calculation of the water vapor pressure $p_{H_2O}(t)$ from the saturation vapor pressure $p_{H_2O}^{sat}$ (Equation 1.6), and compensation for the changes in the number of H_2O molecules in the gas phase Δn_{H_2O} (Strebl *et al.*, 2021).

$$p_{H_2O}(t) = RH(t)p_{H_2O}^{sat} \quad (6)$$



For corrosion occasioned by only either HER or ORR, the number of H₂ generated or O₂ molecules consumed was directly determined from the change in total number of gas molecules by subtracting Δn_{H_2O} (Equation 7).

$$\Delta n_{H_2/O_2} = \Delta n_{tot} - \Delta n_{H_2O} \quad (7)$$

For corrosion occasioned by a combination of HER and ORR contributions, the number of H₂ molecules evolved was determined by subtracting also the changes due to O₂ consumption, as determined by the Zirconia O₂ sensor (Equation 1.8). The H₂/O₂ amount of substance will be converted to a cathodic charge using Faraday's law and the derivative of the charge vs time curve results will be presented which will result in current.

$$\Delta n_{H_2} = \Delta n_{tot} - \Delta n_{O_2}^{optical} - \Delta n_{H_2O} \quad (8)$$

RESULTS AND DISCUSSION

Developed in situ respirometric corrosion monitoring system

The Static Manometric setup was developed to monitor hydrogen evolution reaction (HER) and Oxygen reduction reaction (ORR) during an atmospheric corrosion/immersion with the respirometric approach (Plate 1).

Results of evaluation of the developed system

Figures 3, 4 and 5 show static exposure of a pure Cu sample exposed to NaCl in the manometric setup at elevated RH taking into account pressure, temperature and water vapor pressure, respectively.

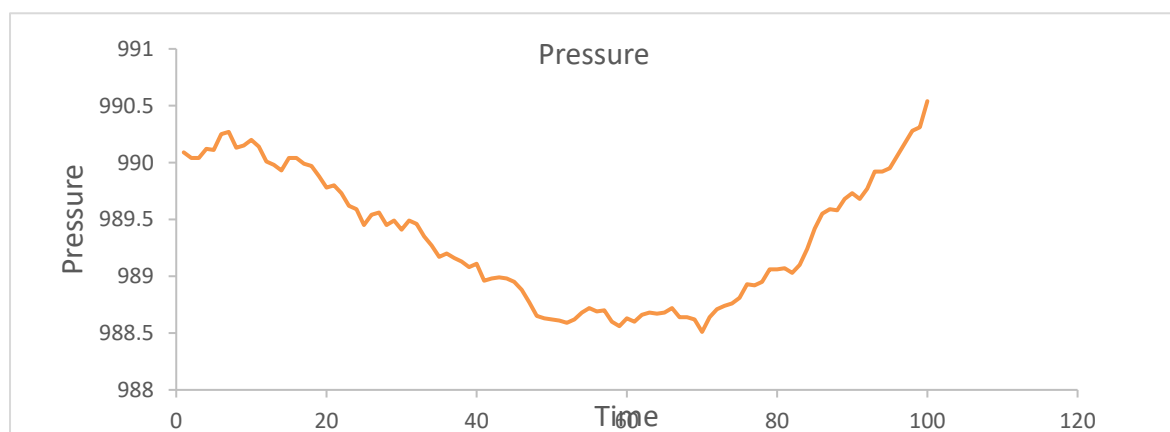


Figure 3: Static exposure of a pure Cu sample exposed NaCl in the manometric setup taking pressure into account

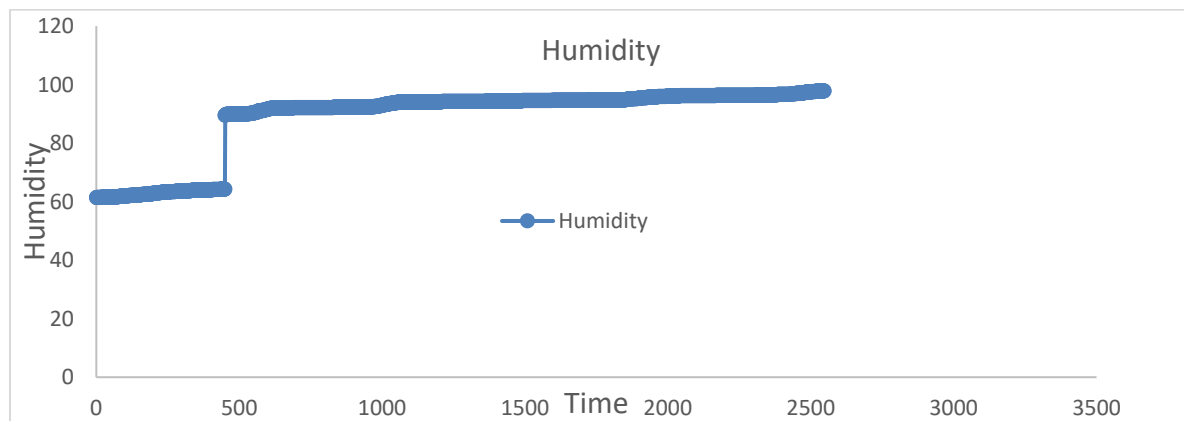


Figure 4: Static exposure of a pure Cu sample exposed to NaCl in the manometric setup taking humidity into account

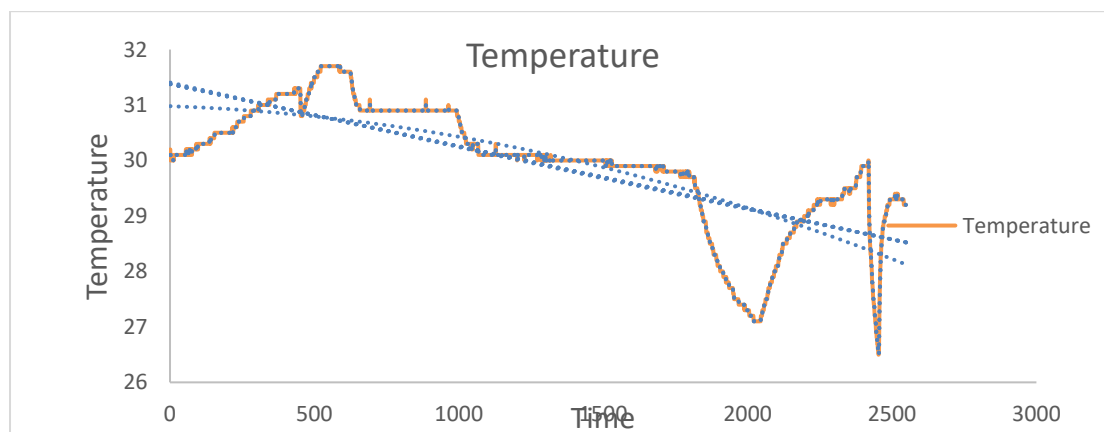


Figure 5: Static exposure of a pure Cu sample contaminated with NaCl in the manometric

Setup taking temperature into account.

It was observed that the signal of the O₂ sensor at liquid phase matches with the signal of the pressure sensor at the gas phase as shown in Figure 6; both registered the decrease in O₂ partial pressure. It can be seen that the charge curves of both sensors overlap after the influence of temperature and water vapor pressure. This leads to the conclusion that reliable measurements are possible both with the O₂ sensor and pressure sensor if the actual temperature and vapor pressure are considered. A direct validation of the Cu experiment with mass loss was also neglected, because the exact oxidation state of Cu in the form of corrosion products is unknown. Therefore, the oxidation state is expected to be somewhere in-between one and two layers of O₂ and pressure curve (figure 6).

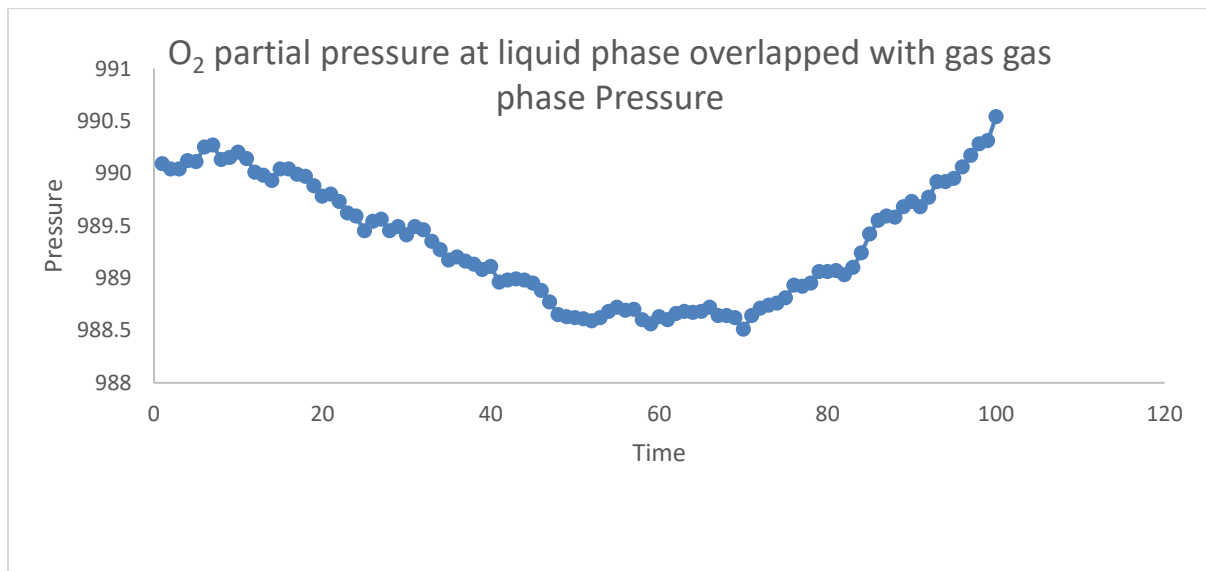


Figure 6: Signal of the Zirconia O₂ sensor when matched with the signal of the pressure sensor

Assuming that the recorded cathodic charge (figure 7) is a good measure of the amount of corrosion that has occurred, the average oxidation state of the corrosion products was calculated using Faraday’s law. In this case z was assumed to be 4 electrons that were consumed to produce one O₂ molecule.

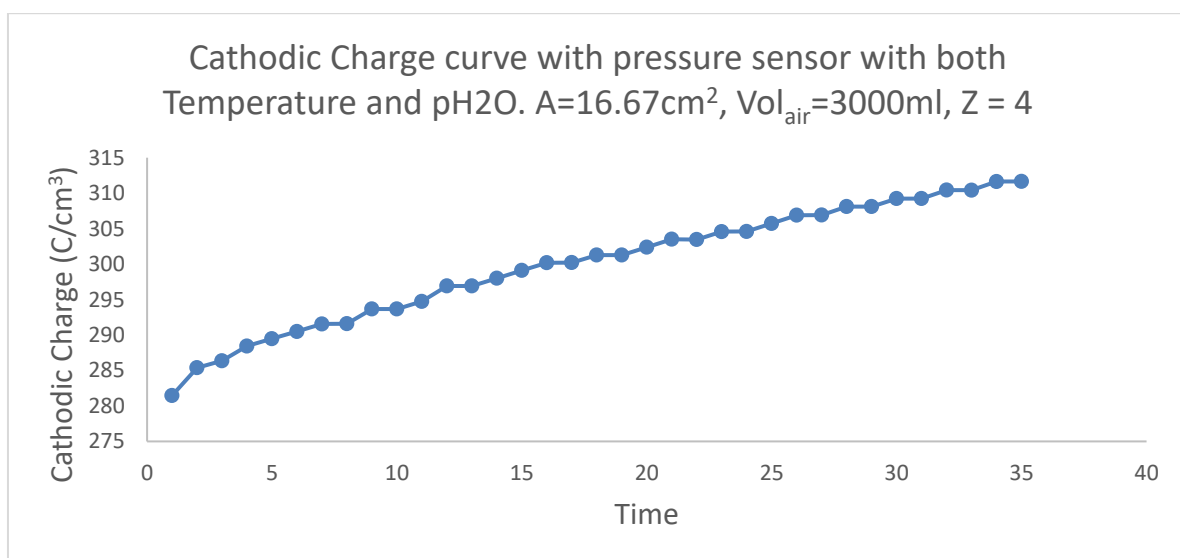


Figure 7: Cathodic Charge curves of Cu samples contaminated with 5% of NaCl at assumed Z = 4

Based on this idea that after having corrected humidity, temperature and pressure variations, and the amount of substance of air is constant. Therefore, $nO_2(t)$ (Figure 8) can be transformed to the O_2 charge density $QO_2(t)$ (Equation 4) via Faraday's law.

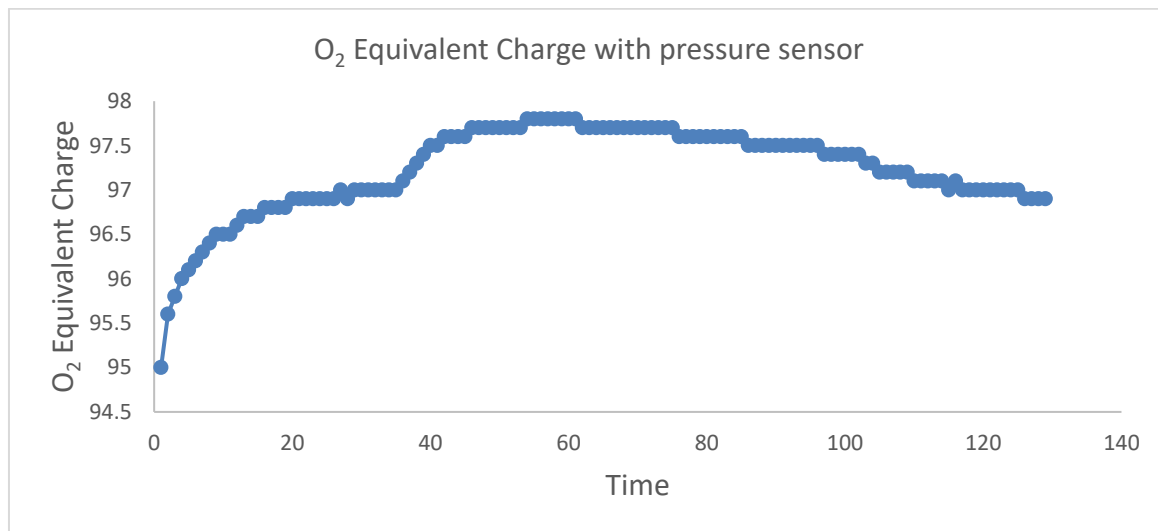


Figure 8: O₂ charge density curve of Cu

CONCLUSION

This study has undertaken the development and performance evaluation of a respirometric in-situ corrosion monitoring system with a graphical user-interface for laboratory use. This instrument is offered to understand the design, functions and optimization of real time corrosion monitoring for electrochemical analysis of polarization measurement in corrosion monitoring. The components materials were sourced locally and the developed system was evaluated. It could be concluded that immersion corrosion kinetics can be monitored non-destructively and in real-time; and in the manometric approach, HER leads to a pressure increase while ORR leads to a decrease in pressure.

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