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Advances in Experimental Techniques for Corrosion Inhibition Studies: Insights and Applications

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ABSTRACT: Corrosion, a pervasive challenge in materials science and engineering, necessitates continuous efforts to develop effective corrosion inhibition strategies. The article explores recent advances in experimental techniques employed for corrosion inhibition studies, shedding light on insightful methodologies and their diverse applications. The pursuit of novel corrosion inhibitors involves a nuanced understanding of both the corrosive environment and the protective mechanisms at play. The application of these experimental techniques extends beyond fundamental corrosion studies, encompassing practical implications in industries such as oil and gas, aerospace, and infrastructure. Insights gained from advanced experimental methods aid in the design and optimization of corrosion inhibitors, ultimately contributing to the development of more efficient and sustainable corrosion protection strategies. Due to their invaluable insights into corrosion processes, the efficiency of corrosion inhibitors, and the creation of corrosion control schemes, experimental methodologies are vital in studies examining corrosion inhibition weight loss measurements offer a simple and economical method to evaluate corrosion. It is possible to create efficient corrosion control plans and choose the best corrosion inhibitors thanks to these experimental procedures, which offer useful information regarding corrosion behavior, inhibition processes, and material performance.

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The degradation of materials as a result of chemical interactions with their surroundings is known as corrosion. It presents a considerable challenge to numerous sectors of the economy and infrastructure systems. Corrosion has a significant financial impact since it raises maintenance costs, causes equipment to malfunction, and has an adverse effect on the environment. Extensive research has been done to create corrosion inhibition measures in order to lessen the harmful consequences of corrosion (Samuel *et al;* 2023). The efficiency of corrosion inhibitors is studied and evaluated using a variety of ways in these strategies. When introduced to a corrosive environment, corrosion inhibitors greatly slow down the rate at which metals corrode (Ahmad, 2006). Studies on corrosion inhibition are essential for the creation and assessment of practical methods for reducing corrosion's damaging effects. Researchers can examine corrosion processes, judge the effectiveness of inhibitors, and get a thorough grasp of the underlying mechanisms by using a wide variety of experimental techniques. Anodic, cathodic, and mixed corrosion inhibitors are the three primary categories. Anodic inhibitors function by creating a barrier of protective oxide on the metal surface to stop further oxidation. By slowing down the pace of cathodic reactions that take place at the metal surface, cathodic inhibitors function. Anodic and cathodic inhibition

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processes are combined in mixed inhibitors to provide their desired effects (Ma *et al;* 2022). One of the most common experimental techniques in studies of corrosion inhibition is electrochemical measurement. These techniques include precisely electrically controlling the metal sample and monitoring the electrochemical process. For instance, electrochemical impedance spectroscopy (EIS) is used in potentiodynamic polarization to determine variables like double-layer capacitance and charge transfer resistance by analyzing the frequency-dependent response of the metal-electrolyte interface (Burstein, 2006). Analyzing electrochemical noise (ENA) can reveal the rate of corrosion and the efficiency of inhibitors by tracking changes in current or potential over time. Studies on corrosion inhibition also heavily rely on surface analysis techniques (Onen *et al;* 2004). High-resolution imaging of the metal surface made possible by scanning electron microscopy (SEM) makes it possible to examine surface morphology, corrosion products, and inhibitor adsorption. The negative impacts of corrosion in numerous sectors and infrastructure systems must be minimized, and this is where corrosion inhibitors come in. The aim of this paper is to give a brief summary of the experimental methods frequently used in corrosion inhibition investigations. Investigating the mechanisms by which these inhibitors function and evaluating their effectiveness in stopping or slowing down corrosion processes are part of the research of corrosion inhibition. Researchers use a variety of approaches and experimental procedures to achieve this, each of which offers insightful information about the performance of the inhibitor The review is structured as follows: surface analysis methods like scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), fourier transform infrared spectroscopy (FTIR), and energy-dispersive X-ray spectroscopy; corrosion measurement methods like electrochemical, weight loss measurement, corrosion rate calculation; and surface analysis techniques such as scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), fourier transform infrared spectroscopy (FTIR), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD).

MATERIALS AND METHODS

Corrosion Measurement Techniques

Electrochemical techniques: Electrochemical techniques are commonly utilized in research on corrosion inhibition because they can provide useful information about the corrosion behavior of metals and the effectiveness of corrosion inhibitors (Papavinasam, 2008). With these techniques, the electrical conditions applied to the metal surface are precisely controlled, and the electrochemical reactions that result are then monitored. Here are some examples of these techniques in action:

Potentiodynamic polarization: A typical method for determining corrosion rates and gauging the efficacy of corrosion inhibitors is potentiodynamic polarization. The metal surface is subjected to a potential sweep, and the current that results is measured. The potential is swept in a specific range, typically from a more negative potential (cathodic region) to a more positive potential (anodic region), or vice versa. The resulting current-voltage curve, known as the polarization curve, provides information about the corrosion potential, corrosion current density, and the presence of Electrochemical techniques are commonly utilized in research on corrosion inhibition because they can provide useful information about the corrosion behavior of metals and the effectiveness of corrosion inhibitors (Papavinasam, 2008). With these techniques, the electrical conditions applied to the metal surface are precisely controlled, and the electrochemical reactions that result are then monitored. Here are some examples of these techniques in action:passivity or pitting behavior (Al-Amiery *et al;* 2022).

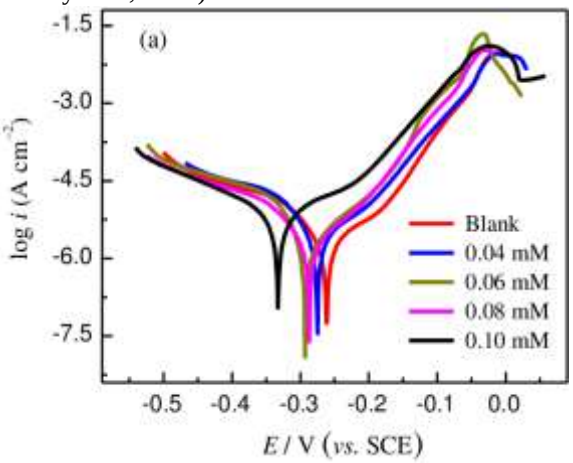

Fig 1: Potentiodynamic polarization curves for copper in 3% NaCl in the presence of different concentrations of DSA. Source: https://www.researchgate.net/figure/a-Potentiodynamic-polarization-curves-for-copper-in-3-NaCl-in-the-presence-of_fig7_271536961

Electrochemical impedance spectroscopy (EIS): The investigation of the electrochemical behavior of metals in corrosive settings is carried out with the use of the potent technology known as electrochemical impedance spectroscopy (EIS). Applying an AC potential signal to the metal surface and observing the current response over a range of frequencies are the key steps in this process. The collected impedance information is then evaluated to ascertain a number of variables, including corrosion rate, double-layer capacitance, and charge transfer resistance. EIS sheds

light on corrosion processes and corrosion inhibitor performance, particularly in terms of their capacity to alter the electrochemical behavior of the metal surface (Wang, 2005). Galvanostatic and potentiostatic methods, cyclic voltammetry, linear polarization resistance, and electrochemical noise analysis are other electrochemical techniques. In CV, a potential sweep is applied to the metal surface, and the current response is then measured. To calculate various metrics like polarization resistance and Tafel slopes, the collected data is then evaluated. ENA entails tracking changes in current or potential over time in order to gain knowledge about the rate of corrosion and the efficiency of the inhibitors (Berradja and Abdenacer 2019).

Weight loss measurements: Measurements of weight loss provide various benefits for corrosion studies. They don't need expensive or specialized equipment, and they are rather straightforward. However, there are some restrictions on weight loss measurements. They give averaged rates of corrosion over the entire exposed surface, which could leave out localized or pitting corrosion. Additionally, the specimens must be sacrificed for weight loss measurements, making it a destructive procedure (Etim *et al;* 2022). This method is well-known and simple for determining corrosion rates and gauging the efficiency of corrosion inhibitors. This method entails exposing metal samples to corrosive conditions for a predetermined amount of time while monitoring the weight loss that results. The metal's rate of corrosion is then determined using the weight loss. As a result, it is frequently used with other techniques, including electrochemical techniques, to gain a deeper understanding of the corrosion behavior of metals (Berradja and Abdenacer 2019).

The following steps are commonly included in the weight loss measurement process:

i. Making Specimens: From the substance of interest, metal specimens are made, typically in the shape of coupons or discs. To get rid of any impurities or oxide coatings that can obstruct the corrosion process, the specimens are thoroughly cleaned.

ii. Immersion or Exposure to Corrosive Environment: The specimens are subjected to the relevant corrosive environment for a predetermined amount of time. Depending on the application or study, the corrosive environment may be a solution, gas, or atmospheric conditions (Onen *et al;* 2000).

iii. Removal and Cleaning: Following the exposure period, the specimens are carefully taken out of the corrosive environment, rinsed with an appropriate solvent to get rid of any residue or products of corrosion that may have adhered to them, and then dried.

iv. *Evaluating Weight Loss*: The specimens' weight loss is calculated by dividing their starting weight by their final weight following exposure. According to Belarbi *et al;* (2016), weight loss serves as a proxy for the degree of corrosion that took place throughout the exposure period. (Etim *et al;* 2017).

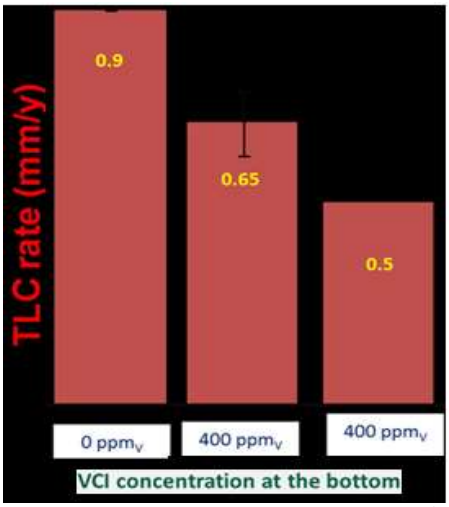

Fig 2. Corrosion rate by weight loss measurement of the uninhibited and inhibited TLC specimens (WCR = 0.6 mL/m ^{2} /s). (Belarbi *et al;* 2016).

Corrosion rate calculations: Calculations of corrosion rates offer useful quantitative information for determining the extent of corrosion, contrasting the efficacy of various corrosion inhibitors, and estimating the effectiveness of corrosion control techniques in practical settings. These calculations support the identification of corrosion mechanisms, the creation of corrosion mitigation techniques, and the choice of appropriate materials and inhibitors (Onen et al; 2004). In studies on corrosion inhibition, corrosion rate calculations are essential because they enable a quantitative assessment of the corrosion process and the efficiency of corrosion inhibitors. Corrosion rates are often represented in terms of mass loss per unit area per unit time, or corrosion current density, which is measured in amperes per square meter (A/m^2). Based on experimental data gathered using procedures like weight loss measurements, electrochemical measurements, or corrosion coupon analysis, a variety of approaches can be used to compute corrosion rates. Several popular techniques for calculating corrosion rate include:

i. Weight Loss Method: As was previously said, the corrosion rate may be estimated by multiplying the metal specimen's weight loss by the sum of the exposed surface area and exposure duration. This method provides an average corrosion

rate over the entire exposed surface (Ushie *et al;* 2017).

ii. The Tafel Extrapolation Method makes use of potentiodynamic polarization measurements' polarization data. The corrosion current density (icorr) can be calculated by extrapolating the Tafel slopes of the anodic and cathodic branches of the polarization curve. Faraday's law, which links the corrosion current density to the metal dissolution rate (Baboian and Treseder 2002) is then used to compute the corrosion rate.

iii. The linear relationship between the corrosion current and the applied voltage in the passive area of the polarization curve is the foundation of the linear polarization resistance (LPR) approach. The slope of the linear part of the polarization curve can be used to calculate the polarization resistance (Rp).

iv. Mass Loss and Time Method: With this approach, weight loss measurements are used to track the metal specimen's mass loss over time. The corrosion rate can be calculated from the slope of the linear regression line by visualizing the mass loss as a function of time (Shinggu *et al;* 2023).

It is significant to remember that the proper corrosion rate calculation approach is determined by the experimental data at hand and the particular corrosion situation being examined. The constraints and presumptions related to each calculating method must also be taken into account (Hindawi, 2014).

Surface Analysis Techniques: Surface analysis techniques, such as Fourier-transform infrared spectroscopy (FTIR), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD), are invaluable tools in corrosion inhibition studies because they provide detailed information about the morphological, chemical, and compositional changes occurring on the metal surface both before and after exposure to corrosive environments and corrosion inhibitors. The molecular make-up, chemical bonds, elemental distribution, and crystallographic characteristics of the metal surface, corrosion byproducts, and corrosion inhibitors are all covered in detail. These methods help us better understand how corrosion works, how effective corrosion inhibitors are, and how to build corrosion control methods. Scanning electron microscopy (SEM) and atomic force microscopy (AFM) are two extensively utilized surface analysis methods in corrosion inhibition research and X-ray photoelectron spectroscopy (XPS) others include Fourier-transform infrared spectroscopy (FTIR), Energy-dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD) (Hindawi, 2014).

Scanning electron microscopy (SEM): A strong technique called scanning electron microscopy (SEM) makes it possible to characterize the surface morphology and microstructure of metal materials and to image them with high resolution. A focused electron beam used in SEM scans the sample surface, creating signals that provide information about the surface topography, elemental makeup, and microstructural traits (Zhang *et al;* 2005). SEM can be used to assess the impact of corrosion inhibitors on the metal surface by comparing the morphology and microstructure of the metal surface before and after exposure to the inhibitor. You can use this technique to check for corrosion products on the metal surface as well.

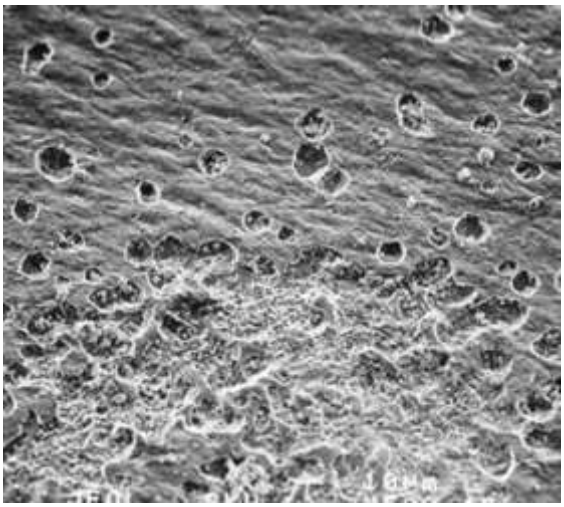

Fig 3: According to Zhang *et al.* (2005), a major corrosion problem developed at the aluminum current collector's edge in cell M9.

The following are the main steps in a SEM analysis:

i. Sample Preparation: To create a flat and smooth surface, metal samples are cut, ground, and polished. The samples may also go through additional processes, such as etching to disclose microstructural features or removing corrosion products, according on the particular requirements (Osigbemhe *et al;* 2022a; 2022b).

ii. Imaging: The ready samples are placed inside the SEM chamber, where an electron beam is directed at them. As the beam moves across the surface, secondary electrons (SE) and backscattered electrons (BSE) are produced as a result of the interaction between the electrons and the sample. In order to build an image of the sample surface, these signals are identified. Information regarding surface topography is provided through SE imaging. It is possible to construct an image of the sample surface using these signals, which are then detected. In contrast to BSE imaging, which offers insights into atomic number and compositional changes, SE imaging provides

information regarding surface topography (Osigbemhe *et al;* 2022c).

iii. Elemental analysis can be carried out using SEM in conjunction with energy-dispersive X-ray spectroscopy (EDS) or wavelength-dispersive X-ray spectroscopy (WDS). EDS/WDS detectors pick up the X-rays that the sample emits when it comes into contact with the electron beam. It is possible to perform both qualitative and quantitative elemental analysis since the discovered X-rays are indicative of the elements that are present in the sample. As a result, deposits, corrosion products, and the distribution of components on the metal surface is identified (Andrew *et al;* 2018).

2. X-ray photoelectron spectroscopy (XPS)

In investigations on corrosion inhibition, XPS is yet another potent tool. By examining the energy distribution of the photoelectrons released from the sample surface when it is subjected to X-rays, XPS can reveal information on the chemical makeup of the metal surface. By contrasting the chemical composition of the metal surface before and after exposure to the inhibitor, XPS can be used to detect the existence of corrosion products on the metal surface and evaluate the impacts of corrosion inhibitors on the metal surface (Dwivedi *et al;* 2017).

The following are the main steps in XPS analysis:

i. Creating the Sample: To provide a clean surface free of contaminants, metal samples are prepared by cleaning and degassing. To reduce air and moisture contamination, the sample is often placed in an ultra-high vacuum (UHV) chamber (Etim *et al;* 2022).

ii. X-ray Excitation: When a specific energy level of X-rays is focused onto the sample surface, photoemission from the atoms in the surface's topmost atomic layers results According to Samuel et al; (2023), the kinetic energy and intensity of the photoelectrons are measured.

iii. The released photoelectrons are subjected to spectral analysis to establish their binding energies. (Etim *et al;* 2017).

XPS analysis can reveal information on the surface chemistry, oxidation states, chemical composition of the metal, and corrosion products. Determine the existence of corrosion inhibitors, passivation layers, surface coatings, and corrosion inhibitor interactions in particular. XPS data can be used to analyze the chemical composition of the metal surface both before and after exposure to corrosive conditions and inhibitory treatments.

3. Fourier-Transform Infrared Spectroscopy (FTIR)

The molecular makeup and chemical interactions of both organic and inorganic compounds can be examined using a technique called Fourier-transform infrared spectroscopy (FTIR). The functional groups on the metal surface and corrosion products are located using the effective technique of Fourier-transform infrared spectroscopy (FTIR). FTIR is used to calculate the sample's infrared radiation absorption. The absorption spectrum reveals the functional groups present on the sample surface and the corrosion products (Deepak, 2017).

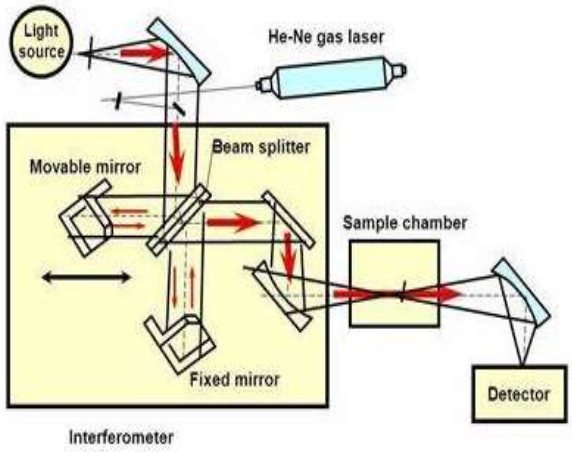

Fig 4.Block diagram of an FTIR spectrometer

The primary steps in FTIR analysis are as follows:

i. Sample Preparation: Several methods, such as deposition onto a suitable substrate, direct examination of the metal surface, and characterization of the extracted corrosion products, are employed to prepare metal samples or corrosion products for FTIR analysis. The materials are often made as thin films or powders to allow for infrared (IR) transmission or reflection measurements (Khan *et al;* 2018).

ii. Acquiring the infrared spectrum: Various infrared radiation wavelengths are subjected to the ready samples. The sample causes the IR radiation to emit molecular vibrations that are specific to the substance's functional groups. By determining how much light is reflected or transmitted as a function of angle (Etim *et al;* 2017).

iii. Spectral analysis: The IR spectra that have been gathered are evaluated to identify the distinctive absorption bands that are associated with specific chemical species and functional groups. As a result, it is possible to identify and analyze the organic compounds, corrosion inhibitors, and corrosion products that are present on the metal surface (Etim *et al;* 2017).

The chemical properties of the compounds present on the metal surface that are revealed by FTIR analysis include corrosion inhibitors and how they interact with the metal. It can be used to determine whether functional groups that are important to the

mechanisms of adsorption, passivation, or inhibition are present. FTIR is particularly useful for studying organic inhibitors, as well as the creation and longevity of inhibitor coatings, according to Baker *et al;* (2014).

Energy-Dispersive X-ray Spectroscopy (EDS): In investigations on corrosion inhibition, energy-dispersive X-ray spectroscopy (EDS) is another effective method. By examining the energy distribution of X-rays released from the sample surface when exposed to an electron beam, EDS can reveal details on the elemental composition of the metal surface and corrosion products (Baker *et al;* 2014).

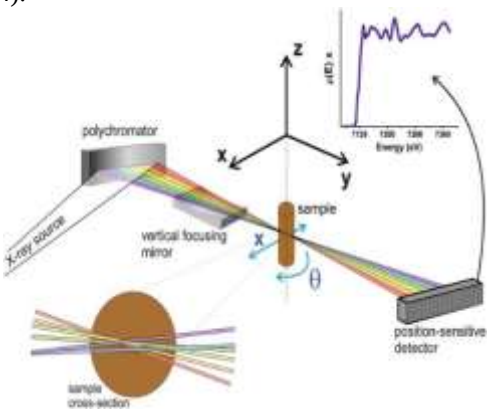

Fig 5. Energy-dispersive X-ray absorption spectroscopy tomography experimental setup (Sanchez *et al;* 2017)

Following are the primary steps in an EDS analysis:

i. Sample Preparation: Metal samples are appropriately cleaned and polished to prepare them for SEM-EDS examination. For imaging and analysis, the samples are loaded into the SEM chamber.

ii. X-ray Excitation and Detection: The SEM emits a concentrated electron beam that interacts with the sample surface to excite and detect the presence of X-rays, which are distinctive of the elements in the sample. The energy of the released X-rays is discovered and measured using EDS detectors.

iii. Elemental Analysis: The elements that are present on the metal surface are determined using the energy data from the observed X-rays. The X-ray intensity gives semi-quantitative or quantitative information about the distribution and composition of the elements.

iv. The elemental makeup of the metal surface, the presence of corrosion products, and the distribution of the elements on the surface are all revealed by EDS analysis. It aids in determining the existence of deposited species, the distribution of corrosion inhibitors, and the elemental changes brought on by corrosion processes. EDS can also be used to create elemental maps, which display the spatial distribution of elements on the metal surface.

X-ray Diffraction (XRD): X-ray diffraction (XRD) is a powerful technique for determining the phase and crystallographic structure of materials, including corrosion products and surface layers. The formation of protective layers, the composition of corrosion products, and the impact of corrosion inhibitors on the crystallographic properties of the metal surface are all investigated using XRD in studies on corrosion inhibition. One example of how corrosion inhibitors alter the crystallographic properties of the metal surface is the use of organic inhibitors to prevent corrosion on iron or steel.

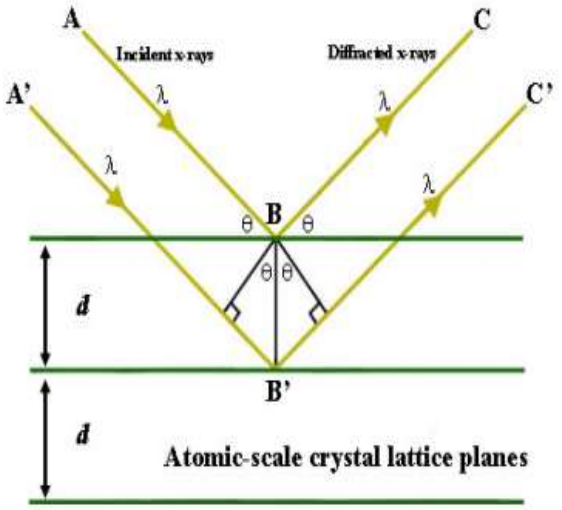

Fig 6: diagram of diffracted X-rays Source: https://serc.carleton.edu/msu_nanotech/methods/BraggsLaw. html

The primary steps in XRD analysis are as follows:

i. Sample Preparation: Before being used in an XRD analysis, metal samples or corrosion products are ground or pulverized into fine powders. According to Raji *et al;* (2012), the powders are normally put on a sample holder and correctly aligned.

ii. X-ray Diffraction: The sample is exposed to X-ray radiation, which interacts with the crystal lattice to scatter the X-rays. A diffraction pattern is created when the scattered X-rays are subjected to constructive and destructive interference. The material's crystal planes and lattice spacing are represented by peaks in the diffraction pattern.

iii. Data Analysis: To ascertain the crystal structure, lattice parameters, and identify the phases present in the sample, specialist software is used to evaluate the produced diffraction pattern (Etim *et al;* 2020a).

The crystalline structure and phase composition of corrosion products, surface films, and deposited species are vitally understood through XRD investigation. It aids in comprehending how corrosion

is inhibited, how protective layers form, and how structural changes to the metal surface result from corrosion processes (Rani *et al;* 2012).

Conclusion: Due to their invaluable insights into corrosion processes, the efficiency of corrosion inhibitors, and the creation of corrosion control schemes, experimental methodologies are crucial in studies examining corrosion inhibition. In order to quantify corrosion rates, polarization behavior, and impedance factors, corrosion measurement techniques are crucial. These techniques include potentiodynamic polarization, electrochemical impedance spectroscopy (EIS), and galvanostatic and potentiostatic procedures. Potentiodynamic polarization, which is dependent on the applied potential, is used to measure the current density. These experimental methods can be used in conjunction to help researchers develop a thorough grasp of corrosion inhibition. The gathered information and insights can then be utilized to create corrosion prevention plans, choose the best corrosion inhibitors, and enhance the performance of materials in varied corrosive situations.

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