Nonelectrochemical Techniques in corrosion inhibition studies: Analytical techniques

Humphrey Sam Samuel[,] Ugo Nweke-Maraizu, Gani Johnson, and Emmanuel Edet Etim Received: 14 April 2023/Accepted 20 July 2023/Published 24 July 2023

Abstract: Corrosion is a widespread issue that impacts many different businesses and infrastructures, causing major financial losses and safety risks. Chemical substances known as corrosion inhibitors can be added to gases or liquids to slow down the rate of corrosion of metals in contact with an aggressive medium. The corrosion inhibition efficiency of a given inhibitor can be influenced by several factors including, temperature, concentration of inhibitor, the type of aggressive medium, etc. In this study, nonelectrochemical methods of analyzing the corrosion of metals have been reviewed. All the methods are observed to have a common principles of monitoring the changes in the structure, surface and other observable properties of the metal, in the absence and presence of the inhibitor. An inhibitor is accepted in this work, only if it can inhibit corrosion, when presence in a minute concentration. In the light of examining the chemical make-up and molecular structure of corrosion inhibitors, spectroscopic methods such as NMR and GCMS are upheld. FTIR and Raman spectroscopy are useful in analyzing the functional groups in the inhibitor or the corrosion products, without and in the presence of the inhibitor. XPS offers insight into the elemental composition and oxidation states of the metal surface and inhibitor film. It is the recommendation of the authors that a comprehensive approach analyzing to corrosion inhibition requires the adoption of different methods that can provide information concerning the inhibition.

Keywords: analytical techniques, spectroscopy, corrosion inhibition.

Humphrey Sam Samuel

Department of Chemical Sciences, Federal University Wukari, Taraba State, Nigeria Email; humphreysedeke@gmail.com

Ugo Nweke-Maraizu,

Department of Chemistry, Rivers State University, Nkpolu-Oroworukwo, Port Harcourt Email; nwekemaraizuugo@gmail.com

inan, <u>nwekemaraizuugo(*a*,g</u>ina

Gani Johnson

Department of Chemical Sciences, Federal University Wukari, Taraba State Email; johnsongani5@gmail.com

*Emmanuel Edet Etim

Department of Chemical Sciences, Federal University Wukari, Taraba State Email; <u>emmaetim@gmail.com</u> Orcid id: 0000-0001-8304-9771

1.0 Introduction

Corrosion is an electrochemical process that have been known as a major factor responsible for material degradation. Since most industries are faced with challenges arising from corrosion, the benefits of corrosion inhibitors cannot be over-emphasized. Some establish role of corrosion inhibitors are

- (i) Protection of metals against degradation
- (ii) Maintenance of the aesthetic value of the metal
- (iii) Prevention of industries from collapse
- (iv) Reduction of running costs that could have arisen from the replacement of damaged facilities

The classification of corrosion inhibitors can be based on several backgrounds including mode of action, source, chemical constitution, etc. Based on electrochemical action, corrosion inhibitors can be categorized as anodic, cathodic, or mixed inhibitors depending on how they work. Cathodic inhibitors act by lowering the oxygen content at the cathode surface, anodic inhibitors function by creating a protective oxide film on the anode surface. Mixed inhibitors function by fusing the two processes. (Devaraj and Srinivasan, 2017). Corrosion inhibitors can be categorized as organic or inorganic, depending on their chemical constitution. General, most active organic inhibitors have one or more of the following properties. viz, heteroatoms. heteroatoms, aromatic system, suitable pi-bond, functional groups, conjugated systems, bulky molecular structure, etc. On the other hand, inhibitors, containing at least one metal are classified as inorganic inhibitors; examples are chromates, molybdates, and phosphates make up inorganic inhibitors. Corrosion inhibitors can also be a classified as

mixed or migrating inhibitors based on how they are applied to concrete structures. While migrating inhibitors are sprayed to the surface of the concrete after it has set, admixed inhibitors are added directly to the concrete mix during batching (Ma *et al.*, 2022; Onen *et al.*, 2017; Etim *et al.*, 2022). Fig. 1 is a flow chart showing the major classification of corrosion inhibitors. In the chart, adsorption inhibitor is assigned to organic inhibitors. However, in practice, an adsorption inhibitor is any inhibitor that is characterized by any of the following features,

- (i) Increase in inhibition efficiency with increase in concentration of the inhibitor
- (ii) Fitness of one or more adsorption isotherms
- (iii) Define inhibition behaviour when temperature of the inhibition system is altered

The activation energy and the free energy of the adsorption should follow certain range

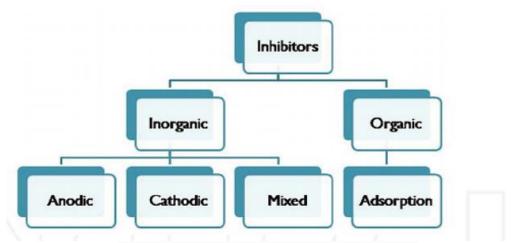


Fig. 1. Classifications of corrosion inhibitors

By elucidating the underlying causes of corrosion and assessing the effectiveness of corrosion inhibitors, analytical approaches are essential in investigations on corrosion inhibition (Ma L, *et al.*, 2020). Researchers can use these methods to characterize the corrosion process, evaluate the efficacy of inhibitors, and

create cutting-edge corrosion prevention measures. X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and infrared spectroscopy (IR) are used to analyze the molecular structure and chemical composition of corrosion inhibitors. The knowledge of the functional groups present in the inhibitor



molecules that is supplied by IR and Raman spectroscopy facilitates the finding of significant chemical interactions between inhibitors and metal surfaces (Fayomi *et al.*, 2018).

Combining experimental and computational methodologies creates a multidisciplinary framework for the study of corrosion inhibition. It offers possibilities for the design and optimization of cutting-edge, eco-friendly corrosion inhibitors for a range of applications. The purpose of this article is to provide illuminating information regarding the effectiveness and performance of the various analytical techniques applied in corrosion inhibition studies. These analytical methods can be combined and used to help researchers fully comprehend the causes driving corrosion. They can evaluate the effectiveness of corrosion inhibitors and direct the creation of successful corrosion prevention techniques. Atomic force microscopy (AFM), Raman spectroscopy, differential scanning calorimetry chromatography-mass (DSC), gas spectrometry (GC-MS), and liquid chromatography-mass spectrometry (LC-MS) are some of the analytical techniques covered in this article. (Devab and El-Etre, 2019).

2.0 Analytical techniques

2.1 Atomic Force Microscopy (AFM)

Corrosion inhibition investigations frequently employ the potent surface analysis method known as atomic force microscopy (AFM). Researchers can examine the morphological properties of corroded surfaces and the production of inhibitor films using AFM since high-resolution gives imaging it and topographical data of the metal surface (Chen H et al., 2020). This method contributes to the comprehension and creation of successful corrosion prevention methods by providing insightful information on the interactions between the metal surface, the corrosive environment, and the corrosion inhibitor. In investigations on corrosion inhibition, the



ability of AFM to photograph surfaces at the nanoscale level is one of its main advantages. A pointed tip is used in atomic force microscopy (AFM) to scan across a sample's surface while exerting constant force on the sample. A three-dimensional topographical map of the surface is created using the data from the cantilever's deflection, which bears the tip. AFM can be used to investigate how well inhibitors function to stop or lessen corrosion damage in the context of corrosion inhibition. By contrasting the topographical characteristics of corroded surfaces with and without inhibitors, researchers may evaluate the ability of inhibitors to prevent corrosion and slough off corrosion damage. AFM can be used in conjunction with other imaging modes to gather more detailed data. Phase imaging in AFM, for instance, may distinguish between materials with distinct mechanical properties. In order to characterize the compositions and distributions of the inhibitor coating and the metal surface, it can be helpful to discriminate between them. Also, the mechanical and adhesive characteristics of the inhibitor coating can be obtained using AFM-based force spectroscopy techniques, allowing for a deeper comprehension of its stability and protective capacity (Chen H, et al., 2020). AFM is also useful for in situ corrosion studies, which enable real-time observation of the corrosion process as well as the development and dissolution of the inhibitor coating. For a better understanding of the inhibitor's function and the detection of probable failure modes, in situ, AFM experiments offer dynamic information regarding the behavior of the inhibitor under corrosive conditions (Hanbing Chen et al., 2020).

2. 2. Raman spectroscopy

The powerful analytical technique of Raman spectroscopy has been widely applied in studies on corrosion inhibition. The molecular structure, chemical composition, and interactions of corrosion inhibitors with the metal surface, provides essential information. Due to its many advantages, such as nondestructive analysis, little sample preparation, and the ability to offer both qualitative and quantitative data, Raman spectroscopy is a flexible technique for the analysis of corrosion processes and the evaluation of corrosion inhibitors (Yasakau, 2021). The sample is illuminated by a laser beam during Raman spectroscopy, and the reflected light is captured and analyzed. The vibrational modes in the resulting Raman spectrum are used to describe the chemicals that are present in the sample. The Raman shifts, or the energy differences between the incident and dispersed light can be used to discriminate between different chemical species and functional groups. One of primary applications of Raman the spectroscopy in studies of corrosion inhibition is the discovery and Characterization of corrosion inhibitors. Comparing the reference and inhibitor-derived Raman spectra can help prove the existence of certain inhibitors. By analyzing the Raman shifts and intensity patterns, scientists can determine the inhibitor's chemical constituent and functional groups (wang et al., 2022).

The distribution and adsorption patterns of corrosion inhibitors on metal surfaces can be studied using Raman spectroscopy. Through the examination of changes in the surface of the metal, Raman spectra as a function of inhibitor concentration or exposure duration can aid researchers to assess the formation of inhibitor adsorption layers. The powerful analytical technique of Raman spectroscopy has been widely applied in studies on corrosion inhibition. The molecular structure, chemical composition, and interactions of corrosion inhibitors with the metal surface, it provides essential information. Due to its many advantages, such as non-destructive analysis, little sample preparation, and the ability to offer both qualitative and quantitative data, Raman spectroscopy is a flexible technique for the analysis of corrosion processes and the evaluation of corrosion inhibitors (Wang,



2022). Researchers can determine modifications in the electrical and structural properties of the metal produced by inhibitor adsorption by examining changes in the Raman spectra of the metal surface in the presence of inhibitors. This aids in the comprehension of passive film generation and corrosion process inhibition. The design and optimization of corrosion inhibitors can also benefit from the information that Raman spectroscopy can provide regarding the type and strength of chemical bonds that exist between the inhibitor and the metal surface (Nandhini, and Rajendran, 2017). It is occasionally possible to increase the analysis of corrosion inhibition mechanisms combining by Raman spectroscopy with other methods. For example, Raman microscopy makes it possible to map the distribution of inhibitor molecules on the metal surface with extreme spatial precision. Also, time-resolved Raman spectroscopy can be used to look into the dynamic mechanisms that occur during corrosion inhibition. By tracking changes in Raman spectra over time, researchers may monitor the emergence of corrosion processes, inhibitor deterioration, and the production of corrosion products. (Ma L. et al., 2020).

2.3. Differential scanning calorimetry (DSC)

Differential scanning calorimetry (DSC) is a frequently used analytical technique to assess the thermal behavior and stability of corrosion inhibitors. The Understanding of the pattern of changes in temperature, phase transitions, and interactions between corrosion inhibitors and the metal surface is possible with the use of DSC. This technique helps in the development and optimization of effective corrosion prevention techniques by analyzing the thermal stability, mechanisms of corrosion inhibitor degradation, and compatibility under various conditions (Abdulrahman, F., & Abass, 2018). DSC measures heat movement into or out of a sample as a function of temperature. The sample is heated or cooled to the same

temperature as a reference material, and any temperature difference between the two is recorded. The temperature differential, which symbolizes the heat flow associated with endothermic or exothermic processes, is displayed as a function of temperature to generate a DSC thermogram. Determining the thermal stability and breakdown temperatures of corrosion inhibitors is the main use of DSC. Researchers can identify endothermic or exothermic peaks in the DSC thermogram that correlate to phase transitions, melting points, breakdown, or other thermal processes by submitting the inhibitor to a controlled temperature program. Such peaks can reveal details on the inhibitor's thermal behavior and stability, the pathways through which it degrades, and the determination of the inhibitor's ideal operating temperatures. (Abdulrahman and Abass, 2018).

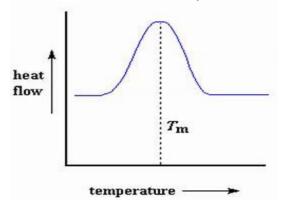


Fig. 2: Differential scanning calorimetry plot. Source: Differential Scanning Calorimetry (pslc.ws)

DSC can also be used to check whether corrosion inhibitors are compatible with other materials or system components. Researchers can evaluate any potential interactions or incompatibilities by conducting DSC experiments with mixes of the inhibitor and various metals or coatings. To guarantee that the corrosion inhibitor does not adversely affect the performance or stability of other components within the system, compatibility tests are crucial. DSC is helpful for researching how corrosion inhibitors and metal surfaces



interact. Researchers can track any changes in the thermal behavior of metal samples coated with the inhibitor using DSC studies in comparison to uncoated samples. Shifts in melting or decomposition temperatures may occur in the presence of an inhibitor layer and signify interactions (Yu, X., & Song, W. 2018). The evaluation of the inhibitor's efficacy is aided by this knowledge, which aids in understanding the behavior of adsorption and the creation of protective coatings. Additionally, DSC can be used in conjunction with other techniques to get more information. For example, DSC and thermogravimetric analysis (TGA) make it possible to evaluate heat flow and mass changes simultaneously. By illuminating the thermal stability, disintegration kinetics, and weight loss of corrosion inhibitors, this combination provides a comprehensive insight of their behavior at various temperatures. In addition to analyzing the thermal characteristics of corrosion inhibitors, DSC can be employed in studies on accelerated aging. Researchers can imitate long-term exposure circumstances by exposing inhibitors to high temperatures for a set amount of time, and then evaluate any modifications to the inhibitor's function or stability (Wang et al., 2022).

2. 4. Gas chromatography-mass spectroscopy (GC-MS)

Gas chromatography-mass spectrometry (GC-MS) is a typical analytical technique, mostly useful in analyzing organic corrosion inhibitors especially in qualitative and quantitative identification of chemical constituents. fragmentation pattern, etc. For detection and identification purposes, GC-MS uses mass spectrometry and gas chromatography. This enables researchers to study complex mixtures of organic compounds present in corrosion processes. GC-MS is heavily used in the identification, characterization, and evaluation of corrosion inhibitors and the byproducts of their breakdown. This helps in the development of useful corrosion prevention methods. For

GC-MS analysis, a sample is first placed into a gas chromatograph.

Based on their chemistry and boiling points, the components can be separated. After that, the isolated chemicals are added to a mass spectrometer. The chemicals are ionized by the mass spectrometer, which then calculates the ratios of mass to charge. Their molecular architectures are revealed by this. It is possible to identify certain chemicals present in the sample by comparing the generated mass spectra to spectral libraries (Yasakau, 2020). The identification and quantification of corrosion inhibitors is one of the main uses of GC-MS in corrosion inhibition investigations. The exact inhibitors utilized can be located and their presence verified by examining the sample's mass spectra and chromatographic peaks. The gas chromatography separation's retention durations can be used to learn more about the compound's volatility and elution behavior. This makes it easier to distinguish it from other chemicals in the mixture and identify it. The molecular structure is determined by the mass spectra produced by the mass spectrometer. GC-MS enables the analysis of other organic species in the corrosion system, such as organic pollutants or reaction byproducts, in addition to the identification and quantification of corrosion inhibitors. This thorough examination aids in comprehending the general chemistry and make-up of the corrosion environment as well as how it affects corrosion inhibition (I.A.W. Ma et al., 2022). However, researchers can measure corrosion inhibitors and the byproducts of their destruction using GC-MS. Researchers can calculate the concentration of inhibitors present in the sample bv incorporating relevant internal standards or calibration curves. The effectiveness of the inhibitor, variations in concentration over time. and the influence of environmental factors on their degradation are all revealed bv quantitative analysis. Additionally, GC-MS analysis can be used in conjunction with other



methods to improve our comprehension of corrosion inhibition mechanisms. As an example, the simultaneous measurement of mass loss and identification of volatile degradation products is made possible by linking GC-MS with thermal analysis methods like thermogravimetric analysis (TGA). This combination offers insightful information on the temperature stability, decomposition kinetics, and volatile species emitted during corrosion inhibitor degradation (Zhang & Dong, 2019).

2.5 Liquid chromatography-mass spectroscopy (LC-MS)

In investigations on corrosion inhibition, liquid chromatography-mass spectrometry (LC-MS) is a very adaptable and popular analytical approach. combines liquid It the chromatographic separation powers with mass spectrometry detection and identification abilities, making it possible to analyze a variety of organic substances, including corrosion inhibitors and the breakdown products of those compounds. The examination of corrosion inhibitors' transformation and degradation processes, as well as their identification, quantification, and characterization, are all made possible by this technique (Mohan, et al., 2018). Liquid chromatography is first used to separate the sample for LC-MS analysis based on its chemical composition and interactions with the stationary phase. Once inside the mass spectrometer, the separated chemicals are ionized and subjected to mass-to-charge ratio analyses. Specific substances found in the sample can be identified by comparing the generated mass spectra to spectral libraries. The identification and confirmation of corrosion inhibitors is one of the main uses of LC-MS in corrosion inhibition investigations. Researchers can pinpoint the precise inhibitors utilized and validate their presence in the sample by examining the chromatographic peaks and mass spectra. The liquid chromatography retention durations give information about how the molecule interacts

with the stationary phase, assisting in its identification. The molecular structure and fragmentation pattern of the inhibitor is determined by the mass spectra produced by the mass spectrometer, further validating its identification (Ismail, and Ahmed, 2019).

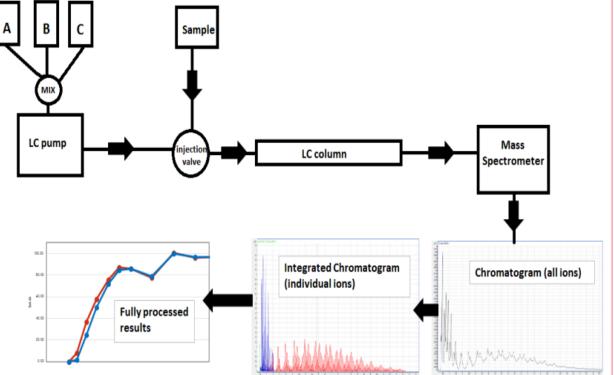
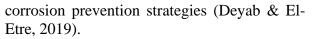


Fig. 3:. Schematic representation of the LCMS workflow. A, B and C on the top left part of the figure represent the mobile phase which can be mixed from 3 different solvents to create a gradient. (Source:Chromatographic Separation of Surfactants in Chemical EOR (researchgate.net)

The Understanding of corrosion inhibition processes can be improved by combining LC-MS with other techniques in addition to qualitative and quantitative investigation. For instance, combining LC-MS with HRMS improves the accuracy and precision of identifying and characterizing corrosion inhibitors and the products of their breakdown. To improve the separation and analysis of particular compounds or complicated mixtures, LC-MS can be combined with additional separation techniques like gas chromatography or solid-phase extraction. Researchers can gain thorough insights into the chemistry and behavior of corrosion inhibitors by combining LC-MS with other analytical techniques, the development of successful helping



3.0 Case Study

Several successful inhibitors have been analyzed using the above-listed methods. In Table 1, examples of corrosion inhibitors that have been investigated by various researchers are presented. It is seen from the results that most investigations employ several methods to drawn inferences.

The results presented in Table 1 above is a reflection of the techniques that dominate the analytical aspect of corrosion studies, especially electrochemical methods, weight loss, SEM, FTIR and GCMS (mostly for plant samples. The computational or quantum chemical approach is relatively new.



Corrosion inhibitor	Class	Analytical method	Reference
substituted 1,3,4-	Organic	Potentiodynamic	Raviprabha and Bhat
oxadiazole		polarization, electrochemical	(2023)
		impedance spectroscopy, SEM	
FD and ACP	Organic	Weight loss, FTIR,	Gopalakrishnan et al.
		electrochemical, hydrogen evolution	(2023)
Schiff base	Inorganic	Potentiodynamic	Betti et al., (2023)
		polarization, electrochemical	
		impedance spectroscopy, SEM	
Carica papya leaf	Organic	Weight loss, GCMS, FTIR,	Eddy et al., (2022)
extract	extract	SEM,	•
imidazo[1,2-a]	Organic	EIS, PDP, SEM, DFT, MD	Daoudi et al. (2023)
pyridine derivatives			
Aromatic oxaldehye	Organic	Weight loss, FTIR, Computational, SEM	Eddy and Ita (2011)
Ossimium	Organic	Weight loss, GCMS, FTIR,	Eddy et al. (2010)
gratissimium leaf	extract	Gasometric,	
extract			
Erythromycin	Drug	Weight loss, hydrogen evolution, FTIR	Ogoko et al. (2010)

Table 1: List of some studied corrosion inhibitors and analytical methods employed

4.0 Conclusion

The above review reveals that several analytical chemistry techniques exist for corrosion monitoring and can technically be viewed as methods that provide imaging information on the surface morphology (such as AFM, SE< TEM, XPSetc), methods that provide chemical composition (such as GCMS, XRD, etc), methods that provide information on functional grounds or vibration of bonds (such as FTIR, Raman, etc), methods that provide information on inhibition efficiency, which include, weight loss, EIS, PDP, gasometric and thermometric. Revelations from the information presented in this review, it can be stated that analytical chemistry provides the foundation for investigating the functionality of corrosion inhibitors and the

latter cannot be assessed without the former. We hereby recommend that an adequate review of the theory of analytical chemistry is necessary and should be considered as the first step before undertaking any corrosion inhibition research.

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Declarations

The authors declare that they have no conflict of interest.

Data availability

All data used in this study will be readily available to the public.

Consent for publication

Not Applicable

Availability of data and materials

The publisher has the right to make the data Public.

Competing interests

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Authors' contributions

HSS, UNM and UNM were involved in literature review, writing and drafting, revision and editing while EEE was involved in conceptualization, writing and drafting, revision and editing.

