Perspective

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The Long and Winding Road to Build a Credible Mexican Electrochemistry

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Abstract. The recognition as emeritus researcher granted by the Mexican Council of Science and Technology a year ago (2020) appeared as a reminder of the beginning of my career of the challenges, friendships and experiences lived at the extrema during these years up to now. Though I am grateful for the opportunity granted by the Journal of the Mexican Chemical Society to write this article, summarizing all the experiences and teachings in a single perspective article would be a titanic task. Nevertheless, I am committed to highlighting some key moments from my journey so far. It is my hope that this manuscript will inspire and motivate the next generation of electrochemistry researchers and commemorate the unforgettable experiences shared with my students, colleagues, and friends.
Keywords: Electrochemistry; research team; academic career.

Resumen. El reconocimiento como investigador nacional emérito que me fue otorgado por el Consejo Nacional de Ciencia y Tecnología hace un año (2022) ha detonado el reencuentro con las experiencias vividas al inicio de mi carrera que comprenden retos, amistades y múltiples experiencias, vividas con gran intensidad desde esos días (1984) hasta el día de hoy. Agradezco la oportunidad que me ha concedido la Sociedad Química de México de escribir este artículo, resumir todas las experiencias vividas y aprendizajes adquiridos en este artículo de perspectiva ha sido una labor titánica. De esta manera se describen los momentos claves que fueron apareciendo en mi carrera científica y que forjaron mis conocimientos y habilidades. Deseo que este escrito inspire y motive a la siguiente generación de investigadores electroquímicos y que así mismo, sea un reconocimiento a las experiencias inolvidables compartidas con mis estudiantes, colegas y amigos.
Palabras clave: Electroquímica; grupo de investigación; carrera académica.

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Discovering the path of research

In my period of secondary school years in Mexico City, my curiosity about understanding the processes of nature began, particularly those associated with the plants in our garden. There, I designed "experiments" to unravel, for example, the shape of the ovaries of the "lilies of Mary"; from this, I realized that it was necessary to know the fundamentals of the processes, so I began to devour the books on the subject that existed at home. Later during my high school years, I discovered the pleasure of being in an open-shelf libraries (few existed at that time, the library of the Congress of the Union becoming my favorite). In this way, I immersed myself in the vast universe of knowledge, that I nourished with reading every day from the moment I finished my school hours at Prepa 9 until they kicked me out at closing time. In this way and with the guidance of my Chemistry and Biology teachers, my curiosity for photosynthesis blossomed. Instead of completing a monograph to conclude the course, I proposed the Project: "The influence of wavelength on oxygen production in algae", which I pursued at ESIQIE under the mentorship of a professor who happened to be the brother of a friend, Elsa.

This magical – but clear, incipient – experience of planning, experimenting, and understanding that I gained in this project, I faced a great dilemma to selecting my major for higher studies. Evaluating the curricula of the available colleges that would bring me closer to investigating natural processes, and with the interest of photosynthesis, I enrolled in the Bachelor of Pharmaceutical Chemistry Biologist (QFB) program at the FES Cuautitlán UNAM. However, it seemed to me that this degree lacked the necessary rigor, and I decided to switch to a Chemistry major. This decision caused me many conflicts with my father, leading to an increased demand for work in his business, since he had dreamt of me pursuing law or medicine; the anger was such that any achievement through my studies was always totally disregarded. Despite this, the pleasure of knowledge and the diversity of chemistry models that allow people to look natural process have been the energy that fills the professional and human being called Nacho González with fullness. This attitude in my work as assistant professor of Analytical Chemistry, at the FES Cuautitlán, allowed me to be invited to pursue a PhD in Analytical Chemistry at the University of Paris VI, France. It was during my doctoral research that I discovered the wonderful world of electrochemistry; my fascination was such that I decided to work body and soul, for a dream: to establish the "Mexican School of Electrochemistry". When one works tirelessly toward a dream, your soul intrudes, and the phrases that have accompanied my research work have come from there, when I have had to face a particular challenge.

Identifying and initiating the dream that has been the engine of my life

In the eighties, after returning to Mexico after completing my doctoral studies and before the very incipient formation of research areas, I faced several challenges to integrate and participate in the development of this tradition. There were several answers to these challenges, among which I may cite some: Stop doing research assuming that Mexico lacked adequate infrastructure, I could have continued the collaboration with the research group I have worked with to complete the doctorate; or finally, I could have proposed projects associated with tradition and economic interest of Mexico. Despite the competitive disadvantages of the third option, which however over time would provide me with more satisfaction, I opted for this one recklessly (at that time I did not realize this).

The final objective of my research was clear: consolidate the development and application of electrochemistry in Mexico, and to transcend in the economic and technological fields of particular interest to our country. Throughout my career and due to the lack of specialists in the different branches of electrochemistry, I adopted a very peculiar strategy by developing a specific line of research in three or four doctoral projects, so that these future doctors could be the ones who continued and consolidated that line of research. Subsequently, I initiated another line of research developed by another three, four, or even five doctoral students who would inherit this knowledge, and so on. This strategy fostered the diversity of fields in which I have ventured, which undoubtedly impacted the level of my specialization, but expanded the diversity of the lines cultivated in electrochemistry (fundamental and applied) in Mexico by different groups that have been nucleated from the doctors thus trained.

Throughout my career, my students and close collaborators have been able to witness my personality and discern the genuine intentions behind of some of the phrases they hear at congresses and other academic events such as thesis evaluations. It is undeniable that some of these phrases and my way of expressing myself have managed to instill a certain halo of bewilderment and even fear in unwary students and young researchers. These phrases are not just occurrences but include in many ways the true intention of my work as a researcher, as well as my personal and professional achievements.

The challenges to solve that forged the phrases that identify with my being and my work

From some of the phrases that I always mention I will make a recap of some of the research topics and my vision of electrochemistry that I hope I succeeded in sharing with my students and collaborators.

We can only innovate when we understand the operational principle of a process

In the visit of a deputy director of Pemex to UAM-I in the nineties, he raised the problem of maintaining the distillation towers and catalytic reactors of the refineries, since they presented much greater corrosion damage than predicted by online evaluations (such as the corrosimeter and the weight loss measurements of metal coupons, which were commonly tests to evaluate corrosion). According to what was known in the sector, it was reported that with the evaluation using various electrochemical techniques, it would be possible to have a measurement of corrosion rates closer to the observed damage. Therefore, he commissioned us with this project. This represented a great challenge, since I had no experience in corrosion, nor in projects linking with Pemex (Mexican Oil Company). A multidisciplinary team was set up (with a logistics management, Teresita Oropeza, and an operation management, Margarita Miranda), which collected information and experimental strategies to face the challenge, especially in the use of polarization curves, electrochemical noise, electrochemical impedance spectroscopy, wheel testing, among others.

The corrosion rates evaluated with these techniques were different from each other, higher than those found from weight loss experiments, and different from those reported in the literature for corrosion of carbon steel in sour environments. These results indicated that the films formed in the corrosion process had properties very different from those assumed for corrosion in aqueous media, and especially from that in the "sour media" reported in the literature. On the other hand, the corresponding NACE standard proposed a composition of the sour medium very different from that formed in the dome of catalytic reactors in Mexico. So, if we wanted to evaluate with certainty the corrosion rate of carbon steel in the "sour media" characteristic of the catalytic towers at the PEMEX refineries, *we had to understand the principle of operation of the process, and then innovate.*

For this project, we found that some residual seawater remained trapped in the crude oil, which was further released during distillation and in the catalytic towers, thus forming condensed sour media where ammonium sulfide and cyanide ions were present. This phenomenon was of special interest for PEMEX, since the very specific characteristics of Mexican crude oil, the nature of the process, and the operation conditions made it impossible to formulate strategies to stop corrosion in pipelines and containers of catalytic plants, nor use other strategies employed in other countries. For this specific case, two important operation principles were unknown: firstly, the urgent need to understand the very nature of the specific films produced during corrosion in sour media (including sulfides, ammonia, and cyanides) and their effect on film properties such as conductivity and the diffusion of species. Secondly, once this knowledge was acquired, it was essential to propose new corrosion inhibitors capable of stopping the critical processes.

For the first time, the characterization of corrosion products in carbon steel was provided by a combination of spectroscopic and electrochemical techniques. It should be noted that during the period of this research, the tradition in corrosion science was limited to a few characterizations without a real understanding of the product formation mechanisms, and therefore, the utilization of XPS and electrochemical microscopies was somewhat disruptive [1]

Fig. 1**(I)** depicts one of the main results, the formation of films composed of different Fe compounds with oxygen and sulfur, showing a very clear Fe gradient as a function of the film depth the different zones indicate regions where the concentration of species drastically changes, SEM analysis of transversal zones of affected materials (Fig. 1**(II)**) indicates that effectively Fe_xS_y films are being formed. However not only in the surface the concentration of sulfides is higher, but the entire composition of species is changing; Fig. 1**(III)** and 1IV depict Fe2p and O1s XPS spectra as function of the sputtering time, using this information, the components at each dept were determined. Moreover, the fact that an Fe gradient was formed, indicated that not only film formation was driven by "chemical diffusion" of iron species through the corrosion film, but there was also a diffusion of H° produced during the reduction of hydrogen sulfides at the film /electrolyte interface; it turned out that both processes had a profound effect on the nature of the film. The slower the diffusion of H°, the higher its pressure buildup, which results in blistering and steel fracturing effects.

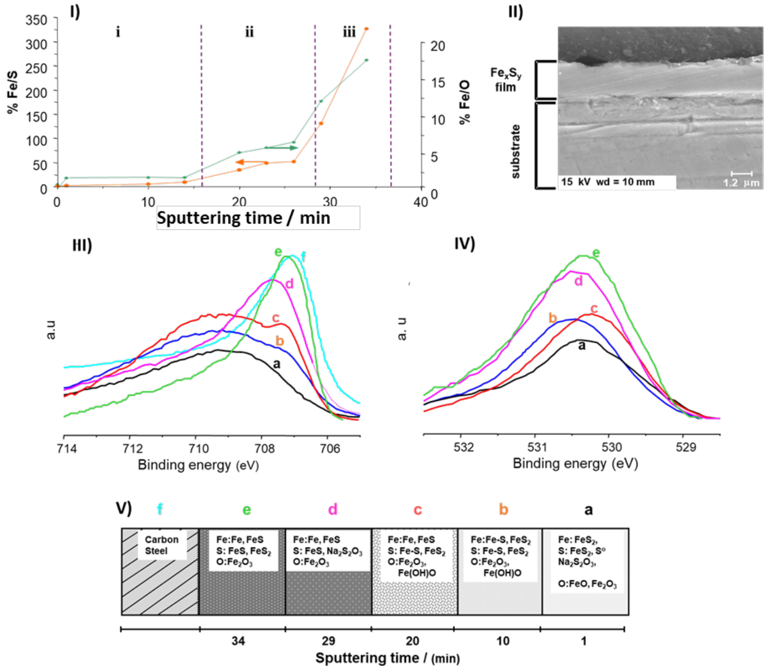

Fig. 1. Combination of spectroscopic and imaging methods for the analysis of corrosion films formed on carbon steel in sour media. **(I)** XPS analysis of the ratio Fe/S and Fe/O as function of the sputtering time, **(II)** SEM of the transversal cut of a exposed sample, **(III)** Fe 2p XPS spectra of exposed samples as function of sputtering time, **(IV)** O 1s XPS spectra of exposed samples as function of the sputtering time and **(V)** XPS detected compounds and the labeling of dept used for XPS analysis in III and IV. Figure adapted from (1).

The diffusion of species through corrosion films was thought to be the key point to avoid corrosion; however, still more important is the development of strategies capable of determining diffusion changes induced by corrosion inhibitors. In this regard, electrochemical impedance spectroscopy (EIS) was used as a fundamental method to determine the effect of diffusion. In addition to traditional EIS examination, where only charge transfer resistances are determined, our approach consisted of fitting spectra to equivalent electric circuits; nonetheless, the method was implemented still one step further. The specific components of the electrical circuit, once fitted to the time constant spectrum (RC electric circuit) are plotted individually. In this way, their positions in the frequency axis of the Bode diagram indirectly indicate the natural rate of each process, in this case, the diffusion of species [3]. Therefore, effective corrosion inhibitors capable of stop H° diffusion are the only ones capable of stopping steel fracturing. Once the structure of the material was determined, as well as the implications on the mechanism of corrosion and the dominant factors, we were able to innovate on the development of corrosion inhibitors, therefore causing a great impact on one of the main industrial problems in Mexico in the eighties. [4]

Someone already came up with the idea before, what happens is that it is no longer fashionable, just do an exhaustive review find it and improve it

The ever-increasing stringency of environmental regulations is compelling academia to implement new processes that are more efficient and environmentally friendly. One of such processes is the leaching of mining concentrates and minerals as well as the treatment of the concomitant residual waters. In the mineral leaching process there occurs simultaneously an oxidation process (typically of the mineral sulfides), and the reduction of the oxidant (i.e., dioxygen or Fe^{3+}). The semiconductive and especially non-conductive properties of minerals have represented a challenge for their electrochemical study (mainly for oxidation characterization). In the decades of the 70s and 80s several alternatives emerged to face this challenge, among which are the use of carbon paste electrodes with both conductive and non-conductive binders. The use of pure minerals and limited strategies to relate the electrochemical behavior with the reactivity of minerals promoted the loss of interest in this type of studies, despising their versatility for the design of hydrometallurgical processes. We took this information back ten years later for the study of concentrates and mineral residues; novel electrochemical and spectroscopic strategies were designed to establish the mechanisms of dissolution of these complex matrices [5]. At the beginning it was difficult to place our articles, since it was a "topic that was no longer fashionable", but our perseverance - and especially the evidence on the applicability of what has been developed - have placed us in a preponderant place in the subject worldwide. The innovative part in which we participated was that of implementation of carbon paste electrodes to study the processing of natural minerals, flotation concentrates, and minerals residues. Moreover, chemical perturbation programs and advanced characterization methods were employed to analyze the species formed during the oxidation of mineral sulfides [5].

The dissolution mechanism of chalcopyrite was studied at carbon paste electrodes with a combination of electrochemical and spectroscopic techniques. These studies aimed at developing new methods to extract copper from $CuFeS_2$ (the most refractory mineral in hydrometallurgical processes) and finding the conditions to improve copper dissolution without the formation of passivation layers. Our novel protocol included the formation of species at different anodic potentials, followed by rinsing and testing again in a renewed solution which was subsequently subject to electrochemical methods to characterize the species in solution (see Fig. 2(**I**)) [6].

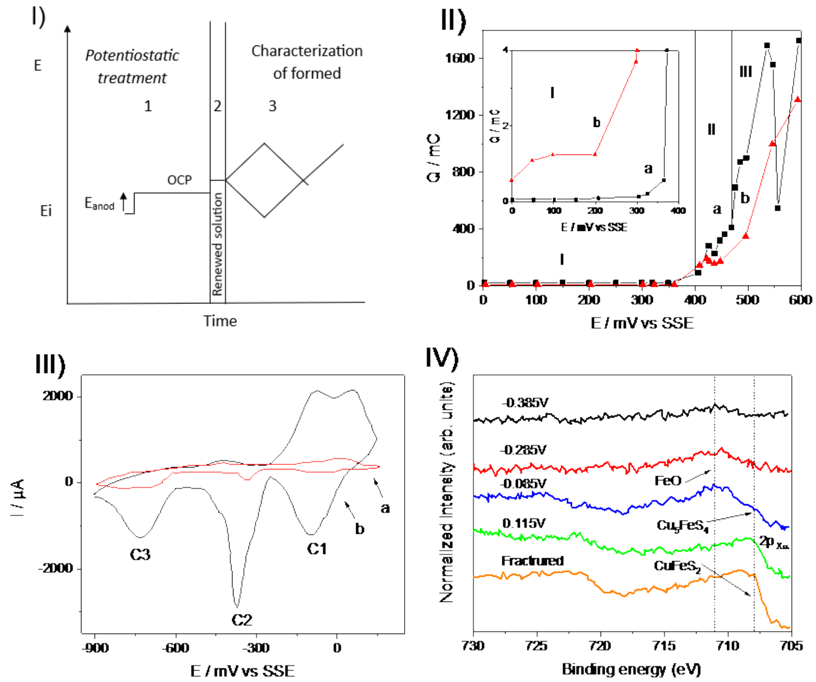

Fig. 2. Electrochemical and spectroscopical characterization of chalcopyrite dissolution products using carbon paste electrodes. **(I)** protocol used for the study of the formation of species, **(II)** Comparison of charge during oxidation of electrodes with pure chalcopyrite (trace a in black) and chalcopyrite concentrates (trace b in red),

(III) Cyclic voltammetry of electrodes of pure chalcopiryte **(a)** and chalcopyrite concentrates **(b)**, previously modified at 550 mV in 1.7 M H_2SO_4, **(IV)** XPS analysis of the compounds previously formed by anodic treatment. At different potentials indicated in the figure. Figures adapted from D. Nava et al [6,7].

To understand the complex phenomena occurring during leaching, a comparison of carbon paste electrodes with pure chalcopyrite (trace **(a)** in Fig. 2**(II)**, and Fig. 2**(III)**) were compared against those of chalcopyrite concentrates (trace **(b)** in Fig. 2**(II)**, and Fig. 2**(III)**) [7]. Fig. 2**(II)** indicates the charge associated to the oxidation process on both electrodes at every potential. The appearance of three distinctive regions is evident (see reference [6] for the electrochemical reactions associated to each region). Here, pure chalcopyrite presents a higher current in zones II and III, while the currents associated with chalcopyrite concentrates in zone I start at lower potentials. Therefore, the concentrate produces an active layer that further leads to the formation of several redox pairs able to effectively lixiviate copper (Fig. 2**(III)** trace **(a)**). Chalcopyrite concentrates contain higher amounts of other minerals, for instance galena, pyrite, among others. Galena creates a galvanic interaction among the phases present in the concentrates that effectively form this active layer in comparison to pure chalcopyrite, which forms passive layers [7]. Further characterization (Fig. 2**(IV)**) indicated that the phases formed at every potential were different indeed, enabling us to detect and differentiate between active and passive layer components [6,7].

The electrochemical response of pyritic samples and mining wastes, in conjunction with evolution of the chemical quality of the leach solution, constitutes a novel methodology to establish and understand the factors involved during Acid Rock Drainage generation (ARD), the main environmental concern of the mining industry [8].

The most important step in mining corresponds to the process of dissolving the ore, so all efforts were directed to this stage. The result of this research, in addition to the articles and training of PhD students, has been the registration of national and international patents in which the process of leaching and recovery of silver is established (the ten registered national patents, some of which have already been applied, support my commitment to technological development). In particular, I take great pride in the development (in collaboration with Prof. Gretchen Lapidus) and successful implementation of a cyanide-free process for the recovery of Ag, a contribution of utmost importance since our process is friendly to the environment [9]. This development has been scaled to the pilot plant level at the company Servicios Administrativos Peñoles in Torreón, Mexico.

The study and control of hydrometallurgical processes have consolidated a collaboration with the mining industry during the last twenty years, in which other innovations have been carried out to existing processes. The experience in this type of linkage with the industry has allowed the development electroremediation of processes for the treatment of waters contaminated with metals and recalcitrant organic compounds. Here, the novel strategy that has been established for the design and construction of electrochemical reactors has been paramount for the development of robust processes (see below).

Electroplating (i.e., the build up of metallic electrochemical coatings of surfaces) has been widely and empirically applied in Mexico and motivated an investigation of the early stages of the process leading to the development of robust macroelectrolysis processes. The use of predominance/existence zone diagrams of the chemical species of an electrodeposit bath (i.e., chemical speciation) [10] allowed the design of chemical compositions to tune the solvation sphere of the ions to be deposited and thus control the type and texture of the deposit [11]. On the other hand, experimental strategies (e.g., substrates, perturbations, characterization) and innovative models were developed to study the formation, growth rate, and dimensionality of the early nuclei of deposited metals [12]. These disruptive contributions with respect to electrodeposits have made it possible to design metal preparation processes with different textures and properties, such as: thin and compact films in metal refining [13], solar energy absorbing films [14], and even the formation of metal powders for the continuous recovery of metal ions from different industrial effluents. Likewise, it has been possible to design, form, and characterize metal oxides with specific properties to be used as electro- and photocatalysts [15] for the advanced oxidation of organic matter.

This is truly peeking into the intimate life of the electrons

Oxide-reduction chemical reactions are the basis of processes that occur in nature: respiration, photosynthesis, and corrosion, to name a few. In these processes there is a charge transfer that is detected with

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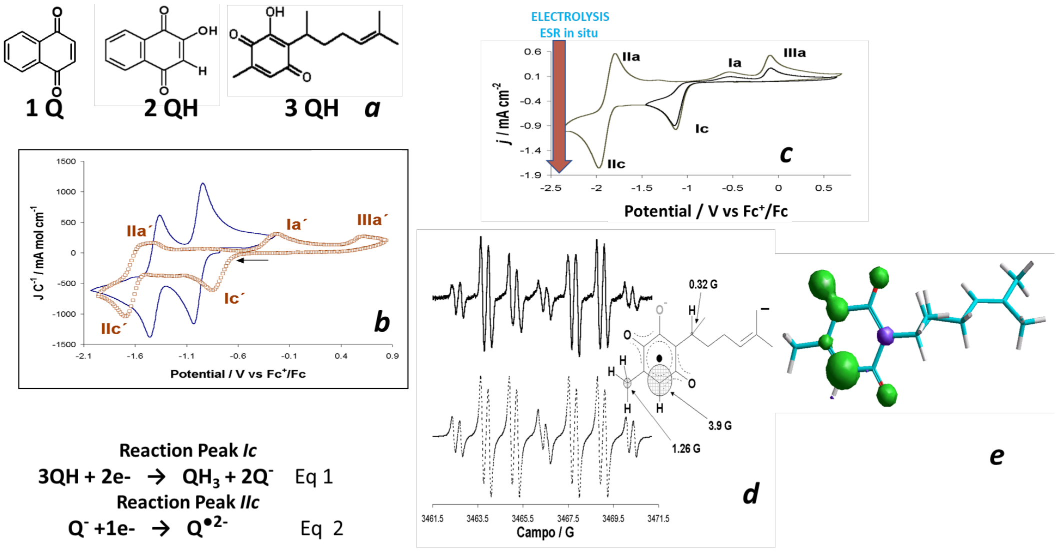
the change of the oxidation number of the oxidant and the reducer, but "the electrons" involved do not manifest since these reactions are usually carried out in media that are not electronic conductors. On the other hand, when an electrical potential difference is established in an electronic conductor, the current intensity measures how fast the electrons move, without them being "seen" either. In this line, the electrochemical study of natural products based on quinones led us to implement an *in situ* characterization, whereby a cation radical is formed through an electrochemical reaction and this is detected in the same electrochemical cell by electron spin spectroscopy ("the electron remains in the radical"). The analysis of the ESR spectrum allows to establish what are the interactions of this with the hydrogen atoms of the molecules, opening all the possibilities to establish the structure of the molecules in the solvent where they are located. After so much searching this is "*truly peeking into the intimate life of the electrons*" and that is why I am very excited.

Electrochemistry in general - but also organic electrochemistry in particular - deals with experiments with limited information, where mostly the current, potential and time are our only variables. With this limited information (which is still very useful) we usually calculate kinetic and mechanistic information; however, the feeling that the elusive electron is non existing under certain conditions forces us to use spectroscopic tools to get deeper insights on the nature of the electron and its interactions.

Quinones, for example, having a hydroxy function in their structure are important in biological systems due to their capacity to generate free radicals via redox reactions, which ultimately lead to the generation of reactive oxygen species [16]. The overall mechanism of radical formations and their reactivities are important for the design of synthetic and natural drugs and other environmental applications. In our case, the real nature of the reduction products was debatable since some authors proposed that the reaction proceeded by a monoelectronic reduction leading to a radical anion, while others proposed an electrochemical-chemical (EC) mechanism. To determine the real nature of these electrochemical processes we proposed the use of spectroscopic methods to directly “observe” the electrons as follows.

Three quinoid compounds were used to demonstrate the formation of radical anions during reduction (Fig. 3**(a)**) [17]. Those compounds are very different: compound 1 does not have a hydroxy function, compound 2 has hydroxylated components and so does compound 3. A voltammetric study (Fig. 3**(b)**) indicated that compound 2 undergoes two redox processes: the first one (*IC*) associated to an irreversible reduction (i.e, Eq. 1, Fig. 3**(b)**); on the other hand, if process (*IIC*) were partially reversible, it should be associated to the reduction of the anion formed during the first reduction process for hydroxyquinone deprotonation which had not been demonstrated at that point. To demonstrate the formation of a dianion radical by reaction (2), the electrochemical cell was coupled with ESR equipment and tested at the peak potentials (Fig. 3**(c)**); any signal detected should be associated to radicals formed during the electrolysis at the specific potential [17]. The results were amazing! For instance, the spectrum of perezonate species produced at more negative potential confirmed the presence of radicals (Fig. 3**(d)**); moreover, the hyperfine interactions can be simulated by the interactions between proton groups in the 5, 7, and 14 positions with the unpaired electron and therefore the structure of radical anion o perezonate was described [18] (Fig. 3**(e)**).



Reaction Peak *Ic*

$3QH + 2e^- \rightarrow QH_3 + 2Q^-$ Eq 1

Reaction Peak *IIc*

$Q^- + 1e^- \rightarrow Q^{\bullet 2-}$ Eq 2

Fig. 3. Electrochemical characterization coupled to ESR experiments leading to the exciting word of electrons within molecules, **(a)** Compounds used to determine the formation of radicals, **(b)** Voltammetric response for quinones 1Q (continuous line) and 2QH (dashed line) and electrochemical reactions associated to peak reduction, (**c**) Voltammetric response for 3QH, perezone, **(d)** ESR in situ spectra obtained during electrolysis of perezone (compound 3QH) at potential marked in, **(e)**. The spin density structure evaluated from the analysis of the experimental data is shown in the figure. Figures adapted for C. Frontana et al [17,18].

The results obtained demonstrated for the first time the formation of radical dianions during the reduction of natural hydroxyquinones, opening new routes for the identification of cytotoxic effects of this kind of compounds [18]. Moreover, the stoichiometry and the estimation of the thermodynamic association constants of hydrogen bonding between these molecules and nitro compounds became possible [19].

The use of spectroscopic tools coupled with electrochemical techniques opened the route for further studies like the structural properties of polymers [20], reaction mechanisms in coordination compounds and [21], among others.

Size does matter: Design and scale up electrochemical reactors

The design and characterization of electrochemical cells currently moves from empirical design to the application of models of hydraulics, mass transfer, and their impact on current distribution and potential. Our group has implemented strategies to design and scale, in an efficient and robust way, electrochemical reactors (from the laboratory to industrial reactors of large volume of operation) [22] for various applications ranging from the recovery of gold and silver dusts [13], and the leaching of concentrates less polluting than using cyanide, to the treatment of effluents from the electroplating [23] and textile industries [24], effluents with high fat and oil content, and currently, with arsenic [25], among others. The efficiency and selectivity of these reactors allows them to be coupled with biological reactors in a novel way [26]. This has consolidated the development of Electrochemical Engineering in Mexico for different applications, and we have made this way of studying electrochemical reactors fashionable in the world.

Electrochemistry: a tool or a science?

“Electrochemistry (a key basic science) has evolved huge industrial applications such as the chloro-alkali process, aluminum winning, needs for sustainable energy and climate change (batteries, supercapacitors,

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fuel cells, electrolyzers for oxygen and hydrogen production), chemical sensors. It spreads over many areas such as chemistry, physics, material science as well as environmental engineering [27]. Because of this diversity of technologies and the competition to offer novel devices, "electrochemistry research" is only related to "electrochemical characterization" in a routine manner and complementary to other characterization and evaluation strategies. To achieve the development of disruptive systems that respond to the needs arising for the fulfillment of the sustainable development goals (SDG) of the United Nations, it is mandatory to review and investigate the basic principles of electrochemistry in this new universe: for example, the study of the electronic/ionic conductive interface, and the transfer of charge in water in salts electrolytes (WISE and in eutetic solvents: as well as the use of the tools of this century, such as Artificial Intelligence and Machine Learning. To get a glimpse into the state of the art in this new research of electrochemistry as a science, I suggest consulting the Special Issue on Emerging Opinions, "New generation electrochemists make a big impact" Edited by Prof. Richard G. Compton and Hasuck Kim published in Current Opinion in Electrochemistry (2022) [27]. Reading this special issue will certainly motivate research into the elemental principles of electrochemical science in the new era.

Lastly, it is important to note that these research topics are not exhaustive since I have surely not mentioned in this writing several of the "scientific adventures" with my collaborators, students, and friends to whom I am deeply indebted. These forty years of living from, with, and for electrochemistry have allowed me to form my vision of electrochemistry. Now it is necessary to reflect on where to walk in this fantastic discipline.

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