Overview

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Contribution to the Electrochemical Sensors and Biosensors field by the Electrochemical and Analytical Chemistry research areas at UAM-I

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Abstract. An overview of the electrochemical sensors and biosensors developed at Universidad Autónoma Metropolitana Unidad Iztapalapa, is summarized by describing the principal contributions in the field performed by the Electrochemistry and Analytical Chemistry research areas of the Chemistry Department. Here, we divided the contributions into four main groups: fabrication and characterization of surfaces, electrochemical sensors and biosensors with environmental applications, electrochemical sensors and biosensors with applications in the food industry, and electrochemical sensors and biosensors with biomedical applications in the healthcare industry; the foundation of the Institution, the creation of the Electrochemistry and Analytical Chemistry research areas, and the first electrochemical sensor development at the institution are cited in the historical context of the scientific electrochemical sensors and biosensors timeline.
Keywords: Electrochemical sensors; electrochemical biosensors; environmental industry; food industry; biomedical industry.

Resumen. Una visión general de los sensores y biosensores electroquímicos desarrollados en la Universidad Autónoma Metropolitana Unidad Iztapalapa, se resume describiendo los principales aportes en el campo realizados por las áreas de investigación de Electroquímica y Química Analítica del Departamento de Química de esta institución. Hemos dividimos las contribuciones en cuatro grupos principales: fabricación y caracterización de superficies, sensores y biosensores electroquímicos con aplicaciones ambientales, sensores y biosensores electroquímicos con aplicaciones en la industria alimentaria, y sensores y biosensores electroquímicos con aplicaciones biomédicas en la industria para el cuidado de la salud; la creación de la Institución, las áreas de investigación de electroquímica y química analítica, así como el primer sensor electroquímico desarrollado en la Institución, se citan en el contexto histórico de la cronología científica de los sensores y biosensores electroquímicos en el mundo.
Palabras clave: Sensores electroquímicos; biosensores electroquímicos; industria del medio ambiente; industria alimentaria; industria biomédica.

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Abbreviations

ISE	Ion Selective Electrode
WE	Working Electrode
RE	Reference Electrode
d.c.	Direct current
a.c.	Alternating current
DME	Drop Mercury Electrode
ISFET	Ion-sensitive field-effect transistor
GOx	Glucose Oxidase
O_2	Oxygen
UAM-I	Universidad Autónoma Metropolitana Unidad Iztapalapa
UAM-A	Universidad Autónoma Metropolitana Unidad Azcapotzalco
UAM-X	Universidad Autónoma Metropolitana Unidad Xochimilco
CGM	Continuous Glucose Monitoring
CPE	Carbon Paste Electrode
AFM	Atomic Force Microscopy
PLA	Poly (lactic acid)
PPy	Poly pyrrole
GCE	Glassy Carbon Electrode
AgNPs	Silver nanoparticles
HDX	Hexadecane
MetDTF	O,O’-2(,2´-biphenylene)dithiophosphate methyl
NSAIDs	Nonsteroidal anti-inflammatory drugs
AChE	Acetylcholinesterase enzyme
ACh	Acetylthiocholine
LOD	Low detection limit
SPE	Screen-Printed Electrodes (SPE)
Ag/AgCl	Silver/silver chloride electrode
PVA-SBQ	Poly-vinyl alcohol with SBQ
AWP	Azide-unit pendant Water-soluble Photopolymer

PVA-AWP	Poly-Vinyl Alcohol with AWP
PVC	Poly-Vinyl Chloride
NaTPB	Sodium tetraphenyl borate
SDS	Sodium dodecyl sulfate
SPR	Surface plasmon resonance
FIA	Flow injection analysis
Hg	Mercury
Cd	Cadmium
Pb	Lead
CD	Cyclodextrin
DCF	Diclofenac
LSV	Linear Sweep Voltammetry
Ag/AgIbu	Silver and silver ibuprofonate
Ibu	Ibuprofen
MTZ	Metronidazole
G	Guanine
A	Adenine
2-Hp-β-CD	2-hidroxipropyl-β-cyclodextrin
USP	United States Pharmacopeia Convention of 2007 30
GA	Glutaraldehyde
CA	Caffeic Acid
Pt	Platinum
DS-	Dodecyl sulfate ion
MWCNT	Multi Walled Carbon Nano Tubes
AA	Ascorbic Acid
UA	Uric Acid
CNT	Carbon Nano Tubes
PEI	Polyethylenimine
CGM	Continues Glucose Monitoring
CTAB	Cetyltrimethylammonium bromide

PPO	Poly phenol oxidase
SWCNT	Single Wall Carbon Nano Tubes
TCNQ	Tetracyanoquinodimethane
PB	Prussian blue
Fe_3O_4	Ferrite
DNA	Deoxyribonucleic acid
8-OHdG	8-Hydroxy-2'-deoxyguanosine
Au	Gold
AuNPs	gold nanoparticles
APC	Anomatous Polyposis Coli
SPGE	Screen Printed Gold Electrode

Introduction

Electrochemical sensors and biosensors have emerged as analytical tools required for the detection and quantification of the compounds in the environment of study. Considering the IUPAC definition, a chemical sensor “is a device that transforms chemical information, ranging from the concentration of a specific sample component to total composition analysis into an analytically useful signal” [1]. A biosensor “is a device that uses specific biochemical reactions mediated by isolated enzymes, immunosystems, tissues, organelles or whole cells to detect chemical compounds usually by electrical, thermal or optical signal [2].

Sensors and biosensors are constituted of two essential units; the first is the receptor, where the chemical information is changed into a measured signal by the second unit, known as the transducer, which is the device that transforms the measured signal into useful analytical information [1], according to the transducer utilized for processing the signal, the sensors and biosensors can be classified as optical, electrical, mass sensitive, magnetic, thermometric or electrochemical; additionally, at each division, we can find subdivisions according to the type of signal, transducer material, size, or application field [1,3-7].

According to the kind of signal generated, the electrochemical sensors and biosensors are classified as potentiometric, amperometric, impedimetric, or conductometric, and also can be classified according to the application field. Fig. 1 shows the scheme of the cells employed for potentiometry (two sensors system) and voltammetry (three sensors system); it should be noted that voltammetric measurements can also be performed with a two electrodes system.

Potentiometric sensors measure the potential of an indicator electrode, also known as Working Electrode (WE). WE include the Ion Selective Electrode (ISE), metal/metal oxide, or redox electrode against a Reference Electrode (RE) [1]. The sensing method is based on the Nernst equation. In this scenario, the most known electrode is the pH electrode or glass electrode [8].

Voltammetric sensors measure the current in d.c. (direct current) or a.c. (alternating current) mode, the amperometric sensors are included in this group. WE of this type include inert, chemically active, and modified electrodes [1]. In the beginning, the methods that used these voltammetric sensors were the polarography with the drop mercury electrodes (DME); the boom of these sensors was from 1950 to 1960 when even the DME was used for heavy metals determination; currently, the voltammetric determinations are performed employing different materials and nanomaterials that modified the surface of the WE [8].

The *Ion-Sensitive Field-Effect Transistor (ISFET)* plays a crucial role in measuring the ion concentration in a solution. It does so by the interaction between the analyte and sensor coating providing a clear understanding of its function.

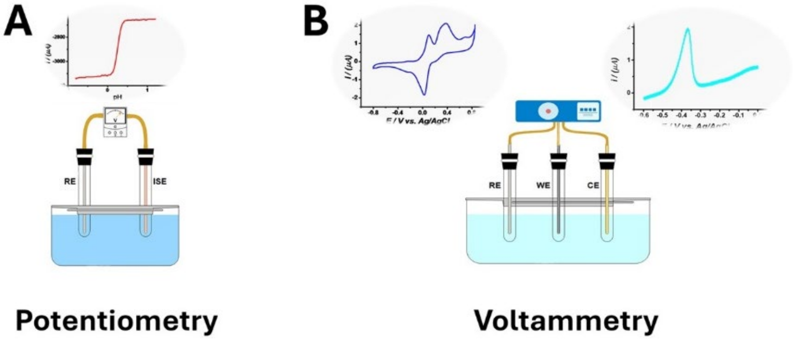

Fig. 1. Electrochemical cell for **(A)** potentiometric and **(B)** Voltammetric measurement.

In general, since the development of the first glass electrode, which is considered the first electrochemical sensor, and according to Karl-Heinz and Kurt, 2010 [8], was developed by Max Cremer in 1906, the sensors have found applications in several fields, including aerospace, computer science, agriculture, environment, and healthcare, the role of the system is to generate data that can be processed to obtain information about the surrounding of a sample under study.

Electrochemical biosensors can also be applied in environment monitoring (air, soil, and aquatic) to detection of pollutants, nutrients, and general composition in the food and pharmaceutical industries during fabrication. Both electrochemical sensors and biosensors have applications in the health field. The most known electrochemical biosensor is the biosensor for glucose monitoring. The first biosensor for glucose monitoring was developed in 1962; it consisted of the immobilization of the enzyme Glucose Oxidase (GOx) onto an Oxygen (O_2) Clark electrode [9,10]; this kind of biosensor has been the target of several research groups in academia and the biotech industry; whence, several of the academy's research performed have been transferred to biotech companies. From the beginning until now, to improve the quality of this kind of biosensors, different electrode materials, polymers, and immobilization methods have been applied to develop the current continuous blood glucose monitoring (CGM); the technology has been extended for other biomarkers in the health care, food, and environmental fields.

With electrochemical sensors and biosensors marketing stride in diverse industries, the impact on the global economy is undeniable. In 2022 the market for electrochemical sensors in the USA was USD 8019.9 million. Projections suggest that this figure will soar to USD 11290 million by 2028, underscoring the market´s rapid growth and potential.

The electrochemical sensors and biosensors research field keeps growing in the World; scientific research shows that more than 6680 research papers during 2023 and 2024 have been published in the “electrochemical sensors” and “electrochemical biosensors” according to Web of Science Research with a refined topic by article type [11]. The Chemistry Department at Universidad Autónoma Metropolitana Unidad Iztapalapa (UAM-I), specifically in the electrochemistry and analytical chemistry research areas, has contributed and continues their contribution to the topic as is describes in this overview.

Fig. 2 shows the timeline of the development of electrochemical sensors and biosensors, UAM-I foundation and its contribution to the field.

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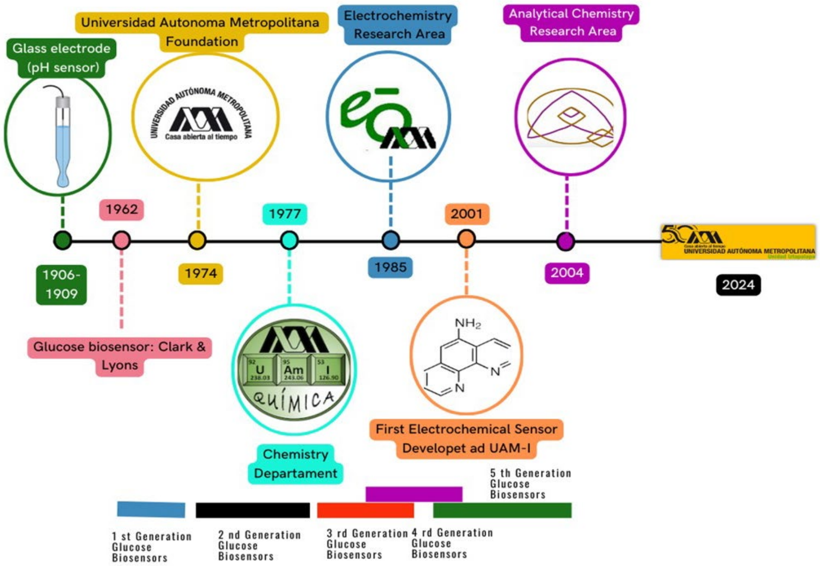

Fig. 2. Timeline of electrochemical sensors and biosensors, UAM-I foundation and its contribution to the field.

By 1909, the first glass electrode and glucose electro-oxidation in a lead anode was reported in the Atlas Power Company; although by 1937, glucose could be oxidized and reduced in basic media onto lead electrodes, it is during 1962 when GOx was immobilized into an O_2 electrode by Clark and Lyons, the response of this electrochemical sensor depended on the GOx enzyme, with the development of this biosensor the first glucose biosensor generation began, it was characterized by the GOx immobilization onto an electrode surface [9,10,12,13]. By the ´70s, the second generation of glucose biosensors with the employment of redox mediators and GOx began [12,13], during the ´70s, in Mexico, the Universidad Autónoma Metropolitana was founded with three campuses, Iztapalapa (UAM-I), Azcapotzalco (UAM-A) and Xochimilco (UAM-X), by 1974 year of creation of the UAM-I [14-16], the glucose biosensors were already commercialized in the optical version (reactive strips used for glucose detection in urine) [12,13]. In 1977, the Chemistry Department at UAM-I was created [15,16], while the optical immunosensors commenced [13] in the sensors field.

The ´80s were the heyday of the third generation of glucose electrochemical sensors; however, the second generation of glucose electrochemical biosensors strips were commercialized along with the first glucometers, which were available for medical doctors and health care spaces. In 1985, the Electrochemistry research area at UAM-I was created [16]; the objective of the area is and has been to study the processes associated with the charge transfer, which is the working principle of the electrochemical sensors and biosensors. At the end of the ´80s and the beginning of ´90s the fourth generation of glucose sensors was born, and other analytes became the target in the sensors and biosensors field, the Guclowatch went in and out of the market [13]. By the beginning of the XXI Century, the boom of the electrochemical sensors started with, the Continuous Glucose Monitoring (CGM) along with the incorporation of nano-materials, new polymers, and the beginning of the wearable devices that can be adapted to invasive and minimal invasive electrochemical sensors and biosensors, alongside the fifth glucose sensors generation brought the electrochemical sensors to the market as a real alternative to the conventional analytical protocols used for analytes detection and quantification [10,12,13].

In the last twenty-four years, the Electrochemistry area at UAM-I started its contribution to the electrochemical sensor field; the venture began in 2001 with the study of a conductive polymer, the (5-amino-1,10-phenantroline), which shows acid-base properties when was electropolymerized onto a carbon paste electrode (CPE). Therefore, the polymer was applied for pH measurements [17]. Three years later, in 2004 the Analytical Chemistry research area at the Chemistry Department at UAM-I was created [15]. During the twenty-first century, the two research areas (electrochemistry and analytical chemistry) have contributed to the electrochemical sensors and biosensors field. Here the overview of these contributions has been divided into four main applications: a) Fabrication and characterization surface, b) Electrochemical sensors and biosensors with environmental applications, c) electrochemical sensors and biosensors with applications in the food industry and d) electrochemical sensors and biosensors with biomedical applications in the healthcare field.

Fabrication and characterization of surfaces

When assembling an electrochemical platform for sensors or biosensors, all the steps are crucial. However, the first layer of the sensing platforms is critical; on those substrates the receptors and bioreceptors are located; thus, having a well-structured substrate is important for the optimal performance of the electrochemical device. The research carried out at UAM-I has provided relevant information in this field.

Specific material surface characteristics are required for particular biosensors development; in some cases, cells need to grow with specific traits; in this context, in 2003, the study of the morphology of poly(methyl methacrylate) (PMMA) as a model of lithography microfabrication obtained by ion beam method, the morphology studied by Atomic Force Microscopy (AFM), showed that depending on the generated geometry, different kind of cells could grow on the surface [18]. Gómez-Pachón et al. [19] 2013 studied poly(lactic acid) (PLA) fabricated by electrospun technique; in that case, nanofibers were obtained. In the two cases mentioned above, the morphologies were obtained by AFM, STM, and Scanning Tunneling Microscopy (STM); the results showed depending on the monomer and synthesis method; polymers can show different morphology with different properties, the synthesis method could be extended to other polymers for *in-vivo* or *in-vitro* sensors applications monitoring [20].

The *in-vivo* biosensing implies analytes determinations in the presence of different metabolites and also microorganisms; in those cases, additional information about the environment of the sensing sample can facilitate the studies. Batina et al., 2004 [21], studied the enamel by AFM, their results provided information about different morphologies of the enamel which depended of the presence of microorganisms; the results of the research can be helpful to the development of wearable platforms where sensors are mated to mouthguards or tattoos directly placed on the mouth [22,23].

Electrodeposit is a common step to modify the electrode surface to prepare the sensing layer, in this context the study of the variation of the electrodeposition as a function of the *in-situ* pH solution, Alvarez-Romero et al., 2005 [24], studied the electropolymerization effect of poly-pyrrole (PPy) electropolymerization onto Glassy Carbon Electrode (GCE) performed at different pH of the supporting electrolyte, the pH was monitored by an Ion Selective Field Effect Transistors (ISFET), Paramo-García et al., 2012 [25], also studied the pH effect on the electropolymerization of poly-pyrrole (PPy) onto a GCE , in addition of pH, the support electrolyte effect was studied by AFM. Besides electrochemical deposition, other methods have been performed for metal deposition, professor Batina´s research group fabricated silver nanoparticles (AgNPs), in vacuum by pulsed laser ablation the studies revealed that according to the laser used, different morphologies and sizes can be obtained, in their studies, the nanoparticles were characterized by TEM and AFM, it was found that the chemical environment is relevant for the materials deposition; thus, according of the required characteristics of a sensing platform for receptor interactions, different methods can be applied for materials deposition [26,27]. The self-assemble films of organic compounds such as cholesterol over a gold electrode studied by STM, from a technological point of view, provided information for a better understanding of metallic surface modifications, which could favor the metallic surface-receptors interaction needed at the design of sensors and biosensors with medical applications [28]. The electrochemical sensors studies begun responding to ideal media, nowadays, is required that sensors provide information from real systems; in this way, the cell growth and the recording of their electrical activity under different experimental conditions performed by Acosta-García et al., 2018 [29], showed that no significant differences were observed when the cell grew onto a gold or petri dish surface; however, the membrane capacitance was lower for the cells grown in a petri dish substrate, the results from this research take relevance at the development of implantable devices that nowadays require minimize the biofouling effect produced by the cells in the environment [30].

In the biosensors field, the recognition agent is vital; among the recognition agent, we can find the enzymes, and one of the most used enzymes is the GOx, typically obtained from the *Aspergillus Niger,* which is a mold that is characterized due to its capability to modify the metabolism of microbes, due to its applicableness Velasco-Alvarez et al., 2001 [31], exposed *Aspergillus Niger* to different electric current to observe the effect on the mold grow and the degradation of hexadecane (HDX) in an electrochemical cell with a ruthenium oxide electrode, the results showed that by applying an electrical current was possible to manipulate the metabolite of microorganism, which can be taken as advantage for enzymes production under certain conditions to favor specific characteristics.

As can be seen, the research performed at UAM-I has found relevant results in the fabrication. The surface characteristics obtained with different techniques, and conditions could help in the development of biosensors with favorable characteristics, according to the research performed.

Electrochemical sensors and biosensors with environmental applications

Electrochemical sensors are typically used to monitor soil, air, and aqueous media pollutants in the environmental field. The electrochemical sensors and biosensors developed at UAM-I have been applied to determine in aqueous media a wide variety of pollutants such as pesticides, heavy metals, phenolic compounds, and nonsteroidal anti-inflammatory drugs (NSAID)s. The contributions in this regard are described below.

Pesticides detection is relevant for humankind; these compounds possess mutagenic, teratogenic, and cancerogenic properties; pesticides are also known as nerve agents due to their mechanism of action; these compounds inhibit irreversibly the cholinesterase blocking the impulse from the nervous system, causing paralysis, and death of mammals. Most biosensors developed for pesticide detection are based on the inhibition of the Acetylcholinesterase enzyme (AChE) and the response to the neurotransmitter acetylthiocholine (ACh). Valdés-Ramírez et al., 2008, 2009 [32-34], designed and evaluated biosensors based on the AChE inhibition; in their studies AChE from drosophila melanogaster wild and genetically modified were used, the modified enzymes improved the low detection limit (LOD) as well as the specificity of the pesticides (carbamates or organophosphates). The enzymes were immobilized onto screen-printed electrodes (SPE), the working electrode (WE) was made from a mixture of carbon ink and Cobalt (II) phthalocyanine, which works as a redox mediator lowering the detection potential from 700 mV to 200 vs Ag/AgCl$_{\text{(pseudoreference electrode)}}$, the electrochemical detection was carried out by amperometric measurements of the current before and after biosensors inhibition; the AChE´s was entrapped into a polymeric membrane of poly-vinyl alcohol with SBQ units (PVA-SBQ) or poly-vinyl alcohol with azide-unit pendant water-soluble photopolymer (AWP) (PVA-AWP), the studied systems were employed for carbamates and organophosphates compounds in batch and flow injection system, those were applied for pesticides detection in river water, apple skin and in a mixture of two different pesticides along an artificial neural network to identify the pesticide present in the samples. Juárez-Gómez et al., 2020 [35] developed an ISE for pesticides detection; the sensing electrode is based on an Ag/AgCl as an internal electrode submerged into a reference ACh solution, the solution container was sealed by a selective membrane based on poly-vinyl-chloride (PVC), sodium tetraphenylborate (NaTPB), measures the ACh in solution after 60 minutes of incubation of the sensor in a propuxor pesticide and AChE.

In the agriculture field, besides the use of pesticides that help the good growth of grains, fruits, and vegetables, fertilizers are essential; in this regard, Álvarez-Romero et al., 2007 [36] developed a nitrite-selective composite electrochemical sensor, which was based on nitrite doped PPy, the electrochemical sensor is employed as recognition agent when is used in a 1:1 ratio with graphite, the sensor was constructed in a tubular shape to be incorporated to a flow injection analysis (FIA) system, the sensor response was sub-Nernstian, the mentioned electrochemical sensor can find applications in agriculture, pharmaceutical and food industries. When studying a real sample, a variety of chemical compounds are found; among those, surfactants employed in several industries can be found in soil and water samples. Rodríguez-Bravo et al., 2011 [37] developed an electrochemical sensor for the detection of sodium dodecyl sulfate (SDS), a chemical commonly used in the cleaning industry; the electrochemical sensor is based on the electropolymerization of PPy doped with SDS onto a carbon paste electrode, the developed sensor possesses a potentiometric response, and its surface was characterized by surface plasmon resonance (SPR).

Other pollutants considered toxic for humans and aquatic life organisms are heavy metals; mercury (Hg), cadmium (Cd), and lead (Pb) have been found in the aquatic and soil environments; in this regard at UAM-I, electrochemical sensors based on modified electrodes have been developed for detection and quantification of metals; the developed electrochemical sensors are based on the modification of CPE with polymers able to form inclusion compounds, the studied and applied polymers are based on α, β, or γ cyclodextrin (CD) or poly-coumaric acid, both were electropolymerized in acid media. Cyclodextrin forms a conductive polymer, while poly-coumaric acid can be a conductive film when it is electropolymerized in alcohol-acid solution media or non-conductive when it is electropolymerized in neutral aqueous media. In the research mentioned earlier, the detection of heavy metals was carried out by anodic stripping voltammetry, and the modified electrodes were employed in the quantification of ions of Cd^{2+}, Hg^{2+}, and Pb^{2+} in real samples [38-40]. Another electrochemical sensor developed for Hg is the one described, by Juárez-Gómez et al., 2016 [41], where a sensing platform based on an ISE for Hg is described the development consisted in a polymeric membrane containing O,O'-(2,2´-biphenylene)dithiophosphate methyl (MetDTF) as ionophore and PVC and NaTPB as a plasticizer, cocktail in different rations was drop cast onto a solid graphite/Araldit HT composite, the solid ISE was employed for potentiometric measurements of Hg^{2+} in aqueous solution, the sensor with better analytical parameter showed a Nernstian response, one of the main characteristics of this electrode, is the possibility of its use in acidic pH from 0 to 6. Ochoa-Pérez et al., 2024 [42], developed an ISE to detect Cr (VI); the sensor was based on β-cyclodextrin and carbon quantum dots, the selectivity of the sensor is based on the combination of both, which modified a GCE, the developed sensor can be used in acidic media without the interference of hydronium ions or other heavy metal ions, in comparison with other Cr(VI) ISE, this do not involve a plasticizer or ionophore thus its cost is cheaper.

Humankind has synthesized some drugs to relieve pain and reduce fever; among those, we can find non-steroidal anti-inflammatory drugs (NSAIDs); as a part of quality control is essential to establish methods that help to control the sample composition, and due to the massive use of these drugs, nowadays, it is possible to find the presence of NSAIDs in aquatic environments where its presence needs to be monitored; therefore, electrochemical sensors have been developed to detect and quantify these compounds. For the assembly of the electrochemical sensing platform, the chemistry of the target molecule, including the pKa´s, must be considered to establish the conditions for the redox processes of the molecule. In the case of two of the designed electrochemical sensors at UAM-I, [43,44], the detection and quantification of diclofenac (DCF) is based on the oxidation of the drug, the studies were performed by Linear Sweep Voltammetry (LSV) and amperometry, considering the pKa of the molecule to establish the redox mechanism. Rivera-Hernández et al., 2017 [45] developed an ISE based on silver and silver ibuprofonate (Ag/AgIbu); the sensor was prepared with a silver wire covered with silver ibuprofonate which is an insoluble salt; the electrochemical sensors responses in a Nernstian fashion; therefore, the Ibuprofen in a solution can be detected a quantify with the potentiometric system.

The electrochemical detection and quantification of antibiotics can also be performed utilizing other electrochemical sensors as the developed by González-Solís et al., 2023 [46]; the research introduced a methodology to quantify metronidazole (MTZ) employing CPE electrochemically modified with 2-hidroxipropyl-β-cyclodextrin (2-Hp-β-CD) in acidic media, the electrochemical MTZ was performed by CV under different concentrations of MTZ, the found results are in good agreement with the detection limits established by the United States Pharmacopeia Convention of 2007 30 (USP). Unfortunately, NSAIDs are not the only drugs that can be found in the environment; acetaminophen is another drug that can be found in water and soil. Valencia-Valencia et al., 2023 [47] developed an electrochemical sensor for acetaminophen detection; the sensor is based on the interactions of acetaminophen with guanine (G) and adenine (A); the sensor is fabricated into an SPE utilizing a DPV technique, it was found that G and A do not interact with acetaminophen, therefore it can be quantified in samples where these two nitrogen bases are present as it is found in real samples.

Another kind of electrode with environmental applications is ISFET. In some environmental and several industrial conditions, one of the analytical parameters that are required to measure and monitor is the pH, which commonly can be performed by employing a glass electrode; however, when there is not an aqueous solution, a variation of sensors is needed, this is the case of the ISFET developed by Alvarez-Romero et al., 2003 [48], in this research, the developed ISFET presented the duality of being used in aqueous and glacial acetic media, the electrochemical sensor was employed for pH measurements in the range from 4 to 10.4; the measurements of the cell potential consisted in the cell standard potential, the acetic acid autoprotolysis constant, and the dissociation constant of the base under analysis. The ISFET was utilized for the determination of the equilibrium constant of the protonated pyridine and diethylamine, which are solvents highly employed in the fabrication of paints, rubber, and pharmaceuticals, whence the relevance of electrochemical sensors of this type. This kind of electrochemical sensor can be applied in the electroplating processes as described in [24], where the pH of a plating bath was monitored with an ISFET sensor employing a FIA system; according to the results, the developed sensor and method show good agreement compared to obtained results with a commercial pH sensor.

Further, the mentioned applications of electrochemical sensors and biosensors in the environmental field for the care of the ecosystems is needed to know the interaction between the microorganisms and their environment; therefore, the interaction between *Leptospirillum sp,* an iron-oxidizing bacterium, and pyrite was studied by Saavedra et al. 2021 [49|, the studies provided information about the pyrite chemistry modification by the presence of the bacteria; the pyrite surface interaction-bacteria was studied by EIS. The electrochemical sensor provided information about the attachment or non-attachment of the bacteria to the mineral surface; an attachment of the bacteria can produce a biofilm covering the mineral; the research was performed with pyrite electrodes under acidic conditions at low frequencies.

The results from the developed electrochemical sensors and biosensors have shown that these kinds of devices can be used for pollutant detection as well as at quality control in the fabrication line for different compounds used in agriculture, pharmaceuticals, mining, and aquatic environments.

Electrochemical sensors and biosensors applied in the food industry

Besides the described electrochemical sensors and biosensors that can be applied for pesticide detection [32-35], which are widely used in agriculture to maintain healthy crops, at UAM-I, other biosensors with applications in the food industry are those developed mainly for quantification of specific compounds such as antioxidants, glucose, or pH in different real samples including tea infusions, wine coffee, medicinal plants and commercial beverages [17,50-54]. The human diet includes the consumption of legumes, cereals, and fruits; in these natural products, a considerable number of antioxidants can be found.

Antioxidants play a crucial role in neutralizing the free radicals generated by cellular metabolism [50-53]; in this regard, electrochemical biosensors have been developed to detect and quantify the antioxidant capacity of some natural products.

The developed biosensors are based on immobilizing enzymes such as Laccase or Tyrosinase. The enzymes were immobilized onto an SPE utilizing PVA-AWP or glutaraldehyde (GA); the sensors were calibrated for caffeic acid (CA), o-, m- and p-diphenol, or catechol; the biosensors were employed to determine the quantity of phenolic compounds and antioxidant capacity of tea infusions [49] and medicinal plants [52]. Juárez-Gómez et al., 2020 [53] utilized the enzyme Xanthine oxidase (XA) along a CPE; the electrode was used to determine the antioxidant capacity of beverages such as coffee, wine, and green tea infusions; the developed method presents potential applications for *in situ* determinations. Another antioxidant of interest in the food industry is resveratrol, which can be found in grapes and wines. At wine production the determination of resveratrol in the pipeline becomes important; thus, a bio-composite made of graphite powder, epoxy resin, and Lacasse was developed by Castro et al., 2009 [55]. In the food industry as well as in the biomedical field, the glucose monitoring in the production line is important for the quality control processes. The electrochemical biosensor that is described by Álvarez-Romero et al., 2004 [51] is focused on the development of a solid electrochemical biosensor based on a composite epoxy resin/graphite/GOx from *Aspergillus Niger;* the biosensor was fabricated in a tubular configuration and coupled to an FIA system with a tubular CE (epoxy resin/graphite), and a commercial Ag/AgCl RE, the results showed that the biosensor sensitivity and linearity depends on the ionic strength of the system, the biosensor presented two linear ranges (low concentrations 0.51 mM to 9.6 mM) and (high concentrations 10 mM to 1 M) when compared the last with the linear range from other biosensors which typically gets saturated at low glucose concentrations (5-10 mM); therefore, the developed glucose biosensor has potential applications for glucose quantification at beverage as well as intravenous glucose solutions. In the same regard, a glucose biosensor based on GOx/p-coumaric acid electropolymerization onto a platinum (Pt) electrode was designed by Valdés-Ramírez and Galicia, 2023 [54]; the characteristic of this biosensor is to employ a thin layer membrane to immobilized the GOx, the advantage of a thin polymeric membrane helps to the fast glucose and hydrogen peroxide diffusion traduced as a fast electrochemical response, the biosensor was utilized to quantify glucose in beverage samples; both developed glucose biosensors showed results with good agreement with the glucose concentration reported in the nutritional table of the test samples.

Other sensors with applications in the food industry are the electrochemical sensors for pH measurements, such as the first electrochemical sensor developed at UAM-I [17] and the ISFET sensors [24,48] for pH determination in nonaqueous solution; Álvarez-Romero et al., 2004 [56], developed an electrochemical sensor for dodecyl sulfate ion (DS-) detection, the sensor was fabricated in a tubular geometry and adapted to a FIA system; the relevance of this research is that DS- is an ion used in several industries including food, pharmaceutical, cleaning, and hygiene, in all those cases the DS- can be monitoring in the pipeline employing the tubular electrochemical sensor developed in the mentioned research.

As can be seen, the developed electrochemical sensors and biosensors possess potential applications in the food industry.

Electrochemical sensors and biosensors with biomedical applications in the healthcare field

The last but not least important application field of the electrochemical sensors and biosensors developed at UAM-I research groups is the biomedical field, specifically the health sector. As was mentioned before, the first glucose biosensor was developed in 1962.

Due to its importance as a biomarker for diabetes, glucose is the most studied analyte in the electrochemical sensors and biosensors development in both the academy and the biotech industry. At UAM-I, the glucose biosensors have been developed utilizing different materials and configurations, the developed biosensors were based on the immobilization of GOx onto various substrates such as GCE, CPE, Multi-Walled Carbon Nano Tubes (MWCNT), Pt, all the developed biosensors were applied in the quantification of glucose in real samples. In the research performed by Álvarez-Romero et al., 2004 [51], the enzyme GOx was incorporated as a part of a composite made of graphite and epoxy resin; the biosensor was designed in a tubular configuration which was coupled to an FIA system used for glucose determination in commercial solutions containing glucose such as oral solutions with electrolytes, peritoneal with electrolytes, and intravenous solution with electrolytes, in all cases the results were in a good agreement with reported in the composition labels. In the research performed by Lozano et al., 2010 [57], the glucose biosensor was based on GOx immobilized with Nafion® film onto carbon nanotube paste modified with an electropolymerized film of poly(Fe(III)-5-amino-phenanthroline, which works a redox mediator due to the presence of iron ions in the layer, the biosensor was utilized for glucose determination in human blood serum, no signal due to ascorbic acid (AA) or Uric acid (UA) was detected; therefore, the biosensor shows good selectivity against some of the common electroactive compounds present in real samples. The development of new biosensors includes the use of new materials as transductors, as well as new redox mediators or membranes; in the work described by Gutiérrez et al., 2011 [58], the GOx was absorbed into a layer of carbon nanotubes (CNT) dispersed in Nafion® and polyethylenimine (PEI) onto a GCE, the developed glucose biosensor showed selective response to glucose towards AA and UA. The glucose biosensor described by Ortiz et al., 2019 [59], was focused on the nanoarchitecture of MWCNT functionalized with concanavalin A and GOx; the biosensors were applied to determine glucose in blood human serum samples showing good correlation with glucose concentration compared to typical laboratory methods.

Nowadays, the biosensors in the market (sensor strips) can determine punctual glucose concentration in a small droop blood sample; the strips are coupled to tiny devices that provide a numerical value related to glucose concentration in the blood; those devices have been commercialized Worldwide helping the physicians and diabetic patients to control its glucose; however, due to the demand for new technologies in the medical care, have made possible the development and marketing of wearable devices such as the glucowatch, optical tattoo-sensors and the Continues Glucose Monitoring (CGM), the latest are implantable devices able of providing glucose concentration information every 5 minutes in real-time and *in-vivo.* Biotech companies such as DEXCOM, Medtronic, and Libre Freestyle have in the market CGMs that can last 14 days and that do not require glucose calibration; the success of these devices has been the fabrication method and the used materials that prevent the leak, the biofouling and long membrane conditioning, in this regard the researches continue exploring new materials; accordingly, Valdés-Ramírez and Galicia 2021 and 2023 [54,60], designed biosensing platforms based on ferulic acid and p-coumaric acid, which were electropolymerized to entrap GOx into a thin film that showed fast responses to glucose, anti-biofouling effect when tested in proteins solution, the glucose biosensors were used to determine glucose concentration in commercial beverage providing results with good agreement with the glucose concentration reported by the maker companies, due to their linear ranges both sensors showed potential applications for glucose samples and other biologic fluids.

Biomarkers such as UA, AA, cholesterol, dopamine, and some genes were employed to develop other electrochemical biosensors at UAM-I [61-74]. Dopamine is a neurotransmitter involved in body movements, memory processes, and pleasure; it has been a target molecule of several research. The electrochemical sensors related to dopamine are based on its selective oxidation, employing different electrode materials as well as modified electrodes. Alarcón-Angeles et al., 2008 [69], utilized MWCNT modified with β-CD; these electrochemical sensors allowed the electrochemical determination of dopamine through its oxidation, which was carried out in the presence of UA and AA under physiological conditions; the results were confirmed and supported by theoretical studies in the research performed by Palomar-Pardavé et al., 2014 [71], in this case an SPE electropolymerized with β-CD was used for dopamine determination in the presence of AA, compared to the research developed at [69], the determination was carried at acidic pH, and the AA signal displayed more cathodic potential while the dopamine peak moves to more anodic potential compared to the potentials at [69] where the determination was performed at neutral pH. Corona-Avendaño et al., 2010 [70], described the use of cetyltrimethylammonium bromide (CTAB) for the determination of dopamine in the presence of UA and AA; the measurements were carried out by differential pulse voltammetry (DPV). Another modified electrode for dopamine quantification is the one described by Colín-Orozco et al., 2012 [68]; they used a CPE modified with micelles of SDS, the measurements were performed by DPV, and the determinations were carried out in the presence of UA and AA; in the case of CTAB, the determination showed an oxidation peak at 700 mV, while the use of SDS reduces the oxidation of dopamine to 400 mV.

Another material that has been employed for dopamine electrochemical detection is graphene; this material was used to produce a graphene paste electrode, and the dopamine was oxidized directly onto its surface; compared with the previous electrochemical sensors for dopamine developed at UAM-I, the graphene electrode allows the oxidation at 100 mV, and the reduction of the oxidation potential means the application of less energy; the graphene sensor also employed for catechol detection, in that case, it was observed a reduction signal at -100 mV; the platform was also used for polyphenol oxidase (PPO) for determination of ethanol in alcohol beverages [72]. As MWCNT, single-wall carbon nanotubes (SWCNT) have also been employed in the design of an electrochemical platform for dopamine sensing. Gutiérrez et al., 2015 [73] developed an electrochemical sensor based on SWCNT modified and polylysine; the sensor allows the oxidation of dopamine at 200 mV. The above dopamine sensors show how the use of different electrode materials, allows the modification of the oxidation potentials for dopamine and other analytes; the electrode material used for a determination will depend on the specific conditions of the samples under study.

Cholesterol is another biomarker target in the development of biosensors, the development of a cholesterol biosensor based on cholesterol oxidase onto an SPE modified with different redox mediators including tetracyanoquinodimethane (TCNQ), Prussian blue (PB), and ferrite (Fe_3O_4); the analytical response of these electrochemical responses are based on the reduction of the H_2O_2 generated by the enzymatic reaction, the biosensors exhibit satisfactory analytical parameters and could be used for lipids determination at blood samples concentration as was reported by Hernández -Cruz et al., 2013 [74].

The Deoxyribonucleic acid (DNA) is a molecule that carries genetic information; its oxidative damages can be monitored through 8-Hydroxy-2'-deoxyguanosine (8-OHdG); therefore, an electrochemical sensor based on CNT dispersed on PEI onto a GCE was used for the 8-OHdG detection by DPV [75]. As the DNA molecule, genes can also be divided into small chains with a specific hybridization site when a specific part of a chain is immobilized onto an electrode, mainly a gold (Au) electrode or gold nanoparticles (AuNPs). Later, the electrode is incubated with the complementary chain, and the electrochemical response for an electrochemical probe provides a diminution of an electrochemical signal compared to the no complementary chain; this kind of biosensors can supply information about the presence of microorganisms or mutations in the DNA. Professor Batina´s research group, has applied this principle at the development of some genosensor, at [75] the detection of the p3 mutation, andk12p.1 mutation was described [77]. Also, a genosensor based on the mutation of the gene adenomatous polyposis coli (APC) is described in [78]. BAT-26 mutation, which helps in the early detection of cancer, was described by Garcia-Melo et al., 2024 [79]; in all the cases, a screen-printed gold electrode (SPGE) was used, and the electrochemical response was obtained by CV or DPV with a $Fe(CN)_6^{3-/4-}$ as an electrochemical probe. The size of the electrochemical sensors, easy manipulation, and fast response time made them promising tools to be applied for early diagnosis kits, as mentioned in [79].

The above electrochemical sensors and biosensors are systems focused on modifications of the WE; however, there is also needed to make research for the easy fabrication of the RE to mate them with tubular geometry for FIA systems or adapt them to implantable devices. In this regard, the development of Ag/AgCl reference electrodes based on composites was performed by Valdés-Ramírez et al., 2005, 2011 at [80,81]; in the first case, the Ag and AgCl were incorporated as a part of the composite. Thus, the RE could be fabricated at the desired geometry and size, while for the second case, Ag and AgCl were electrodeposited on the composite surface, which allows an easy surface regeneration; both reference electrodes were used for potentiometric and amperometric systems showing similar results to the obtained with commercial double-junction Ag/AgCl reference electrodes.

It is worth highlight that several researchers have been made the product of collaborations with other National and International institutions. These collaborations have been instrumental in the development of the electrochemical sensors and biosensors developed at UAM-I. The sensors have been applied to determine analytes with practical applications in the food, agriculture, pharmaceutical, and healthcare industries and have the potential to revolutionize these industries. The developed sensors have shown considerable analytical parameters that can be used as an alternative to the classical analytical methods in the determination of the analytes. Among the advantages of the electrochemical sensors, is possible to mention their fast and reproducible responses, most of the developed sensing systems do not need a sample preparation, thus electrochemical systems can be applied for *in-vivo* real-time measurements. These collaborations have been part of the projects of graduate students and research projects supported by CONACYT in the past and currently CONAHCYT.

Since 2001, the Electrochemistry and Analytical Chemistry research areas have been developing electrochemical sensors and biosensors, and the results have been published in more than 60 scientific articles published in national and international journals. The significant number of publications underscores the extensive research and development efforts in the field of electrochemical sensors and biosensors and the impact of our work in advancing this field. Fig. 3, shows the number of publications in the electrochemical sensors and biosensors field from the Electrochemistry and Analytical research areas of the Chemistry Department at Universidad Autónoma Metropolitana Unidad Iztapalapa.

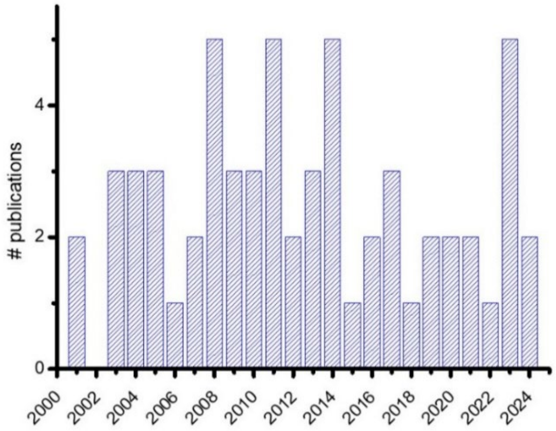

Fig. 3. Number of publications in the electrochemical sensors and biosensors field from the Electrochemistry and Analytical Chemistry research areas at Chemistry Department UAM-I.

The future of the research on the electrochemical sensors at UAM-I, as in the World, is the development of sensing and establishing collaborations with electrical engineers to miniaturize the whole sensing device. Provide society with accessible technology that is easy to use to help them monitor their biomarkers in the control and/or prevention of diseases, and or its environment to not overexpose themselves to pollutants generated by industries and anthropogenic activities.

Conclusions

Electrochemical sensors and biosensors emerged as a need for accessible devices that are easy to use, have fast responses, and provide accurate results as alternatives to the classical instruments used for analytical determinations of analytes of relevance in the food, environmental, and healthcare fields. The developed

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electrochemical sensors and biosensors have been designed in diverse geometries, shapes, and sizes; therefore, they are devices that can be adapted to different sensing systems, such as batch or flow systems. The developed sensors and biosensors can be single-use or multiple-use and provide punctual or continuous information. The transducers can be mated to different substrates so they can be fabricated employing diverse materials according to specific requirements. Currently, electrochemical sensors and biosensors, besides in-situ and in-vitro measurements, have been adapted to in-vivo systems. In this regard, wearable devices have been successfully developed for continuous monitoring. The electrochemical sensors and biosensors developed at UAM-I have successfully been applied for several analytes determination as nerve agents (pesticides), heavy metals, phenols, antioxidants, or biomarkers such as uric acid, dopamine, fragments of DNA chain, or glucose, all of them chemical compounds of relevance in the food, environment, and healthcare fields, these compounds are produced in different industries as textile, pharmaceutical, cleaning, agriculture, polymers, paints, aerospace, construction or by the anthropogenic activities. From 2001 till now, the UAM-I in the chemistry department and punctually at Electrochemistry and Analytical Chemistry areas have contributed with state-of-the-art electrochemical sensors and biosensors to determine analytes that are needed to monitor and control pollutants in the environment, particular chemical compounds in the production line in several industries, or biomarkers that provide information about the health of a patient, helping the physicians to provide a faster diagnosis and or controlling medications, all the electrochemical sensors and biosensors offer solutions to the local and worldwide community.

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